A STUDY OF THE MANUFACTURE OF SULPHURIC ACID BY THE TOWER METHOD

Thesis for the Degree of M. S.

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Sulphuric acid

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By

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PART ONE

A. INTRODUCTION.

Sulphuric acid is one of the well known heavy chemicals which is produced in heavy chemical industrial plants. It is by far the most widely used compound in various sorts of chemical industries. Its use in the chemical field is comparatively the same as steel is in the automotive industries. The daily output capacity of sulphuric measures up to steel based on hundreds of tons per day.

In the past few years a great deal of study and effort have been spent toward bettering the operative conditions of sulphuric acid plants, notably, the reduction of chamber space. European countries have made more progress in this direction than the United States, consequently, more of such newer plants are found in Europe for the daily production of sulphuric acid. England is the ranking country in latest designs and more especially her "Mills-Packard Process."

It is desirous to make sulphuric acid by a tower method, yet employing the chamber process reactions. Also, not only to reduce chamber capacity in converting sulphur to sulphuric acid, but to eliminate them completely. To accomplish this point in mind it would reduce the cost of construction enormously since the lead chambers are expensive and bulky to build. Not only that, but ground space would be cut down considerably which is of vital importance. A more rapid cycle would save time in comparison with the chamber process when the sulphur enters the plant and leaves as sulphuric acid. Thus, a study of sulphuric acid by the tower method was thought to answer the point in mind.

B. HISTORY OF MAKING SULPHURIC ACID.

Small quantities of free dilute sulphuric acid were found in some lakes and streams, notably, Rio Vinagre River in South America, and Lake Mount Indian in Java. It has been said that sulphuric acid was either formed by spontaneous oxidation of natural sulphides, or by the action of water containing cholrides on certain silicates and sulphates together under the influence of internal terrestrial heat. If the drainage water from copper mines should run over pyrites beds in the presence of moist air a sufficient amount of acid will be produced which would have a highly corrosive action on equipment and machinery.

Sulphuric acid has been known for centuries as an artificial chemical compound obtained by distillation of sulphates, especially alum and ferrous sulphate. In this way a strong and powerful corrosive acid distillate was produced.

Some have shown when saltpeter and brimestone were burned conjointly in small amounts in moist air, or in vessels containing water to absorb the produced fumes, sulphur trioxide, an acid liquid was formed which upon evaporation of water left the concentrated sulphuric acid.

Few understood the identity of sulphuric acid and its importance. Not until about 1740 when Ward set up at Richmond, England a small plant for the production of sulphuric acid. He called it "per campanan" made by the bell, the so called shape of the glass vessel. In this vessel eight parts of sulphur and one part of saltpeter were burned together. The lower part containing water was kept warm by the use of a heated sand bath. Charges were fired inside of the bell at regular intervals so that the fumes produced by one charge could be condensed before another one was made, ultimately, the watery acid obtained became strong enough for ordinary uses. After evaporation in glass retorts, the process grew commercially to a full extent, and comparatively profitable in cost production with that of distilling sulphates. Thus, the manufacture of non-fuming sulphuric acid from sulphur developed into one of the largest chemical industries at that time.

After certain developments of the coal tar color industries, they had created a considerable demand for fuming acid which obseleted Ward's process.

1. Production of Fuming Sulphuric Acid.

A large number of various processes in the manufacture of fuming sulphuric acid had been patented from time to time and consequently put into actual operation for commercial purposes. These maybe conveniently grouped into three divisions. (1) Those depending upon the action of heat on some metallic sulphate whereby decomposition took place to evolve sulphur trioxide. Further decomposition of sulphur trioxide resulted into sulphur dioxide and oxygen which was not desirable, so temperatures had to be under careful control. (2) Those in which the essential action was the surface action offerred by solid materials in bringing the two gases, sulphur dioxide, and oxygen, in contact to form sulphur trioxide. (3) And those based on other reactions which have been held secretly and reserved. The first two principles can be combined inasmuch as the sulphur dioxide and oxygen could be united again after original decomposition in the distillation of metallic sulphates. Such a plan of combination was processed and patented by Neal in 1876.

a. Distillation Method of Metallic Sulphates.

The substance largely employed at any rate was sulphate of iron, formerly in form of "copperas" or "green vitroil" (Ferrus Sulphate). It was carefully dried to deprive most of its water of crystallization and partially peroxidize it during the process. Secondly, the ferric sulphate as a crude mass was prepared by long continued weathering of certain salty minerals containing iron sulphide disseminated throughout. Lixivation of the mass dissolved out the mixed ferrous, ferric, and aluminic sulphates which were later evaporated to remove the water. Finally the evaporated sulphates were gently roasted to dehydrate the mass and completely peroxide the iron present.

On heating such materials, a series of clay retorts were mounted together in a large gallory furnace. Sulphur trioxide was expelled at sufficiently high temperatures. It was condensed in suitable receivers previously charged with small amounts of water or weaker solutions of sulphuric acid from a primary run. In the same manner several batches were worked off into the same set of receivers. The remaining residue of impure ferric oxide, more or less brightly tinted, settled in the retort was so far from being valueless that it constituted quite an important by-product for the use of various shades of pigment.

Heretofore few people had only seen a few pounds of acid made. The described process produced 98 per cent sulphuric acid and two per cent of water per ton of commercial acid.

Other numerous methods turned out few tons of fuming sulphuric acid per day. One of these processes was patented by R. Schubert who worked with diminished pressures thus effecting the distillation method whereby the sulphates were decomposed at lower temperatures. He also passed the evolved vapors through heated platinized asbestos to cause the reunion of sulphur dioxide and oxygen formed by breaking up the evolved sulphur trioxide under the influence of heat. Sanstadt in 1875 bettered Schubert's method in substituting magnesium sulphate in place of ferric sulphate, because it gave off sulphur trioxide at lower temperatures.

Scheurer-Kestner found that by heating a mixture of two parts of calcium sulphate and one part of ferric oxide to a bright red heat, a great deal of sulphuric anhydride was evolved and comparatively very little was lost as sulphur dioxide and oxygen. It was said other sulphates of diabasic metals could have been substituted for calcium sulphate.

S. Pitt had patented a process whereby sulphuric anhydride was obtained from acid residues of petroleum refining. This consisted of converting the residue into ferric sulphate by addition of red ocher and heating in air to 300° - 350° centrigrade. In this manner one-third of sulphur went to sulphuric acid while the remainder went to ferric sulphate. J. D. Stark completed the same method in which the residue, ferric sulphate, was fed into a retort by means of an endless screw. Heat was applied to drive off sulphur trioxide and all the sulphur dioxide formed was again reunited in passing it through platinized asbestos tubes. The residual ferric oxide was utilized over again with the acid residue of petroleum refineries. In all, this process was a continuous one.

b. Surface Action Process.

It has long been known, as a laboratory experiment, when a mixture of sulphur dioxide and oxygen was passed through a tube containing heated asbestos, that immediately sulphur trioxide was formed. For many years such a plan had been used in vitroil-making. Phillips patented a similar process in 1831. He burned brimstone in excess of air so that the gaseous mixture of sulphur dioxide and excess air could be passed and brought in contact with platinized asbestos. But, such a method involving expensive platinum could not compete commercially with the nitre process. From this conclusion the nitre process became a popular one. Subsequently, the same reasons were applied to other cases where attemps were made in producing vitroil by catalytic agents due to high costs. Yet, in 1875 Winkler pointed out similar reactions could compare with the distillation methods. In the same year Squire and Messel patented a process in Britain based upon a principle where platinized pumice stone acted as a catalytic agent as well as affording surface action. Their idea met difficulty in applying the plan industrially, due to a rapid corrosive action turning out upon the plant where sulphuric acid was decomposed by heat.

Numerous improvements from time to time have been made. Winkler patented the use of a variety of catalytic agents of high activity, such substances as asbestos, slag wool, infusorial earth, and even organic bodies like cellulose, cotton-wood impregnated with platinum black by soaking in platinum chloride solution and reducing with sodium formate. Surface acting materials of this kind were sufficiently active to enable sulphuric acid to be easily made from burner gases containing as little as four per cent of sulphur dioxide.

Rath in 1882 took out a patent for the use of ordinary burner-gases (Sulphur or Pyrites burnt in air). By passing these gases through purifiers, the dust and aqueous vapors were retained behind. Finally the clean and dried gases were forced through vertical iron tubes containing surface acting materials between a temperature of a low and full red heat.

c. Miscellaneous Processes.

Reactions other than the ones described have been capable of giving rise to fuming acid, and have accordingly been patented. These have not been used to any full extent. Among those was Gruber's nitrosyl sulphuric acid method. This plan depended upon the formation of nitrosyl sulphuric acid (Chamber Crystals) which were prepared by the well known reaction of ordinary burner gases and oxides of nitrogen in the presence of steam. The action of these in another vessel with hot sulphur dioxide mixed with air or oxygen eliminated the oxides of nitrogen. Shown as follows:

1. $2SO_2(OH)(NO_2) + SO_2 + Air \longrightarrow 2NO + H_2SO_4 + 2SO_3$

2. Manufacture of Non-Fuming Acid.

Since Ward's time, changes in the nature of sulphuric acid plants were in the increase of dimensions rather than involving material variation in most general processes. About 1746 Roebuck substituted lead vessels or chambers in place of the glass bells. Later steam was used and generated in a separate boiler. A regulated continuous air supply was maintained instead of intermittent amounts which had been controlled by valves. A separate sulphur burner was also introduced as a source of sulphur dioxide rather than from pyrites or other metallic sulphides. The addition of the Gay-Lussac and Glover towers was proposed and used. These towers introduced an economical move whereby nitrous fumes were recovered instead of passing away with the spent air. The heat given off from the sulphur burners was employed for the purpose of concentrating dilute acids obtained from the chambers, also for vaporizing water and a saving in additional fuel to generate steam for the chambers. Minor variations and adjustments in the mode of supplying nitrous fumes had been made. The routine of processes were carried out scientifically under more precision and skilled supervision. Thus larger yields came into reality and a saving in cost of production resulted in larger profits.

a. The Lead Chamber Process.

The essential characteristic of the chamber process is the action of the oxides of nitrogen in the presence of air, namely, for conveying oxygen to the sulphur dioxide. The spent oxides of nitrogen are recovered in the Gay-Lussac tower (to be fully explained later) to be used over and over again to react with more sulphur dioxide until oxidation is practically completed.

Either suplhur or pyrites can be burned for source of sulphur dioxide. When using brimstone, about 95 per cent of it is sulphur along with about five per cent of ash which contains sand, calcium sulphate, pyrites, and a little bituminous matter along with silica, arsenic, zinc, lead, antimony, selenium, mercury, thallium and other elements.

The start of the operation begins with the burning of either brimstone or pyrites in their suitable burners. At any rate, sulphur dioxide is generated and represented by the following equations:

1. From Brimstone.

$$S + O_2 \longrightarrow SO_2 + \text{heat}$$

2. From Pyrites.

$$4\ FeS_2 + 11\ O_2 \longrightarrow 8\ SO_2 + 2\ Fe_2O_3 + \text{heat}$$

The sulphur dioxide leaves its respective burner and passes through a flue attached to the burner and the other end is connected to the bottom of the Glover tower. Nitre pots filled with saltpeter and little sulphuric acid lie inside on the bottom of the flue line. These are heated from the under side of the flue line by the heat of the sulphur or pyrites burners. Thus the sulphur dioxide passes over these nitre pots and air as well which carry along the nitrogen dioxide generated as shown by the following equations:

3. $NaNO_3 + H_2SO_4 + \text{heat} \longrightarrow NaHSO_4 + HNO_3$
4. $NaHSO_4 + NaNO_3 + \text{heat} \longrightarrow Na_2SO_4 + HNO_3$
5. $2\ HNO_3 + \text{heat} \longrightarrow 2\ NO_2 + H_2O + \frac{1}{2}\ O_2$

The gases, sulphur dioxide, nitrogen dioxide and air after passing over the pots and entering the Glover tower through the flue line, enter what is known as the oxidization zone of the Glover tower.

In this zone the suplhur dioxide is oxidized to sulphur trioxide by the action of the nitrogen dioxide and the surface action of the packing of the Glover tower as follows:

6. $SO_2 + NO_2 \longrightarrow SO_3 + NO + \text{heat}$

Next in line the gases sulphur trioxide, nitric oxide, and air pass up the remaining packed area (to be explained later) and consequently, enter the top of the first lead chamber, there being 3 to 12 chambers which lie between the Glover and Gay-Lussac towers. In the first lead chamber the gases are showered with steam from above by use of a sprayer affair or an atomizer. In this manner the 53° Be' acid is formed:

7. $SO_3 + H_2O \longrightarrow H_2SO_4 + \text{heat}$

The same takes place in the succeeding chambers and more sulphuric acid is formed from sulphur trioxide which escaped from being completely showered in the first chamber. These chambers are so arranged and baffled in order that it will take a longer time for sulphur trioxide to pass through the entire lead chambers to insure all of the absorption of sulphur trioxide as sulphuric acid. The chambers play another role from the one just detailed. Theoretically, all of sulphur dioxide was supposed to be oxidized in the Glover tower, but actually this does not take place. Thus some of the sulphur dioxide does pass into the chambers, and since there is also more nitrogen dioxide due to rapid oxidation of nitric oxide to nitrogen dioxide with excess air present, more sulphur trioxide is formed in the chambers and accordingly showered to form sulphuric acid or commonly called "Chamber Acid." In conclusion the chambers finish the work the Glover tower fails to complete. This "Chamber Acid" is drawn off at the bottom of the chambers as 53 Be' acid.

Insofar as the nitrogen dioxide has contributed its function and being the key to the entire set up, it would leave as waste gas with the spent air which contributed the oxygen in oxidizing the nitric oxide to nitrogen dioxide. For this reason the Gay-Lussac tower aids in the recovery of the oxides of nitrogen which are costly to lose.

The Gay-Lussac tower of earlier days was constructed of sheet-lead metal supported by wooden scaffolding in the same fashion as were the lead chambers. Both square and circular types were built; although, the latter ones had double advantage of offering more cubic space for a given area of sheet-lead, and an equal distribution of absorbing acid in all parts of the tower.

Hard-burnt coke was somewhat employed for packing of these towers, and various other materials such as fragments of glass and porcelain. Other substances capable of resisting acid reactions were utilized. Coke seems to fulfill the call when it comes to regular contour of packing and surfacing, only others answered better against acid resistance. Coke also had a tendency to reduce the higher oxides of nitrogen to the nitric oxide form which was not desirable from the stand point of complete recovery of nitrogen oxides. In doing so the coke soon wore down to a crumbled state which packed the bottom tightly, consequently, a blockage of entering gases resulted. Such a case would interfere with the operation of the tower. Yet coke was satisfactorily employed providing the tower temperatures were kept low enough to prevent reduction of higher oxides of nitrogen. In the end the use of coke did not over balance the cost of cooling facilities. To remedy this situation perforated stoneware plates were used extensively in columns and they answered well in washing the gases, abosrbing, and surfacing too.

Now, as the nitrogen dioxide, nitrogen and spent air enter at the bottom of the packed tower from the bottom of the last chamber, 60°Be' sulphuric acid is sprayed over the entire packing. Since the gases can only pass upward through the uniform packing, the trickling down 60°Be' acid absorbs the nitrogen dioxide while the nitrogen and air escape out at the top of the Gay-Lussac tower.

Sixty degree Be' sulphuric acid is used because at this strength the nitrogen dioxide gas is best dissolved or absorbed as nitrosyl sulphuric acid:

$$8. \quad 2\ NO_2 + H_2SO4 \longrightarrow SO_5NH + HNO_3$$

This equation representing the manner in which the oxides of nitrogen are recovered is basic from the standpoint of the Gay-Lussac tower operation.

To return the nitrosyl sulphuric acid and nitric acid to the Glover tower it follows the route now to be discussed. First, the acid will be called Gay-Lussac acid owing its name to the tower name. The Gay-Lussac acid is drawn off at the bottom of the said tower where it gathers after trickling down the packing. After being drawn off into a suitable reservoir, it is pumped from here to a tank which lies on the top of the Glover tower.

Owing to the higher temperatures of the gases of the Glover tower, it was far more strongly built than the Gay-Lussac tower. The Glover towers were also sheet-lead lined as well as the Gay-Lussac towers, but thicker plates were used to a stoutness of 20 to 25 pounds per square foot in the upper part and double this amount in the bottom portion. These towers were packed with fire brick in order to withstand heat, and acid reactions as well. Such towers were also circularly constructed for the same reasons supplementing the Gay-Lussac type.

The function of the Glover tower toward the chamber process will now be explained. The Gay-Lussac acid which had been pumped into the tank resting on top of the Glover tower drains down through a pipe and finally comes out in a spray over the entire packing. Some sort of a sprayer resistive to acid is employed to distribute the Gay-Lussac acid. There are three zones in this tower, named in order from the top to bottom; the top zone,or oxidation zone,for sulphur dioxide to sulphur trioxide at the expense of nitrogen dioxide, the middle zone or denitration zone where the oxides of nitrogen of nitrosly sulphuric acid are released, the bottom zone, or concentration area.

As the Gay-Lussac acid trickles down through the top zone, it eventually works down into the middle zone or denitration zone. Since the trickling acid is of 60°Be', it is diluted by the up coming steam from the concentration zone. The final dilution becomes 58°Be' at which concentration the oxides of nitrogen are released as follows:

9. $SO_5NH + HNO_3 \longrightarrow 2NO_2 + H_2SO_4$

10. $2SO_5NH + H_2O \longrightarrow 2H_2SO_4 + NO + NO_2$

Both of these reactions will take place. The ninth happens in the manner illustrated since nitrosyl sulphuric is unstable in concentrations at or below 58°Be'. The tenth will break up if steam hits the molecule of nitrosyl sulphuric acid. Only, it is believed that the first predominates. As soon as the oxides of nitrogen are released they are immediately carried up into the above oxidation zone by the up coming air from the sulphur burners. At the same instant sulphur dioxide is likewise being carried along with the air. Thus the nitrogen dioxide and sulphur dioxide come in contact in the oxidation zone producing more sulphur trioxide (Equation No.6) and passing into first chamber on through out the whole cycle. In the same journey the oxides of nitrogen go along with the sulphur trioxide as previously explained.

Now, the 58°Be' acid keeps on trickling through until it reaches the concentration zone, the one situated at the very bottom of the Glover tower. As the hot gases from the sulphur burner come into the Glover tower, they make their first passage through the bottom zone, or the concentration zone. The hot gases drive off the water as steam from the 58°Be' acid by the time it trickles and gathers into a lead pan receiver. From this lead pan the acid is drained into some suitable reservoir and from it pumped into a tank resting upon the top of the Gay-Lussac tower. The acid drained off from the Glover tower is called Glover acid, owing its name to the tower name.

Note that in describing and explaining the three zones each one was named after its type of operation. Thus concludes the operation of the chamber process except that a little discussion on construction and comparison of each unit will now be explained.

The lead chambers are built of sheet-lead about 5 to 7 pounds per square foot. Some sort of scaffolding is necessary to support the chambers. These chambers, generally eight in all, are named after the material made of. Each was 20 to 30 feet wide, 16 to 25 feet high, and 250 to 300 feet long with a total capacity of 100,000 to 150,000 cubic feet.

About 14 cubic feet of chamber space is required to burn efficiently one pound of sulphur per 24 hour day. Ten to 15 pounds of pressure is used to force in the steam into the chambers to insure constant spraying. Generally automatic regulators take care of this work.

The comparison of towers employed are as follows: The Gay-Lussac tower is from 8 to 10 feet on the inside diameter and 30 to 50 feet high with a capacity of 1 to 2 per cent of the chamber space. The Glover tower is 8 to 14 feet in diameter and 20 to 30 feet high with a total capacity of 550 to 600 cubic feet. The Glover tower as previously mentioned is somewhat stronger built. Both are sheet-lead lined and properly scaffolded to support the lining. The picture of the chamber process is with the Glover tower at one end of the chambers and the Gay-Lussac tower at the other end. In such manner that the chambers lie between the two upright standing towers. The Glover tower contains three zones where as the Gay-Lussac contains only one.

The concentration zone is loosely and coarsely packed with materials larger in cross-section than the packings of the upper zones. The denitration zone is packed a little tighter with finer packings. The oxidation zone is packed more closely with finest packing in cross-section than either lower zones. The Gay-Lussac tower is packed of uniform size throughout the entire height.

In conclusion the "chamber process" is theoretically a perfect cycle, but due to some loss which is based on oxides of nitrogen escaping out of the Gay-Lussac tower, the plant is 92 per cent efficient. Very little amounts of sulphuric acid are lost.

Recent years have led to the development of sulphuric acid plants to obtain more suitable methods than the widely employed chamber process, but most searchers could only revise or make additional changes to the chamber method. Thus Mills-Packard method was successful in reducing the chamber space. They stumbled on their idea one day while the acid plant was in operation when a heavy rain fell and the plant seemed to operate more efficiently than ever before. Mills and Packard observed keenly that the chamber temperature dropped as compared to normal days. They realized that it could only be due to the cooling of chambers at the expense of heavy rain. Lead is used to dissipate all the heat from the inside gases, but not as desired by Mills-Packard from that time on.

The above discovery gave them a lead to alter and make additional changes in their chambers. Consequently, the design of water-cooled chambers followed and were used extensively in Great Britain and some other countries, but not as yet in the United States. Cooling was accomplished by running water in lead troughs which encircled the outside of chambers. Chamber design is essentially truncated cones with lead curtains supported by steel frames. As mentioned above, the water-cooling-troughs encircle the truncated cone. A great number of these lead troughs increased with the height of chambers in order to accommodate sufficient cooling in removal of heat evolved from reaction of gases within the chambers.

The advantages claimed for such chambers are as follows:

1. The chamber space required per unit weight of sulphur burned is reduced from one-third to one-half of the usual space per 24 hours.
2. Material saving in cost of construction per unit capacity is reduced from 30 per cent to 40 per cent.
3. A substantial saving in ground space per unit of production capacity.
4. Longer life of lead chambers.
5. Usually no buildings are necessary for housing the chambers.
6. Niter consumption per unit of sulphur made into acid is no higher than that of ordinary types of chambers.
7. Feasibility of combing the chambers with rectangular chambers of existing plants.

Actual operating results show for these chambers only 3.5 to 4.5 cubic feet of chamber space as compared with the best ordinary type which is 8 to 10 cubic feet with non-cooling facilities. From 3 to 4 per cent of niter consumption is all that is necessary to complete operation based on sulphur burned.

Great care must be exercised in building the Mills-Packard chamber. If they are too high, chance of wind storms will overthrow them unless supported extra heavily.

The following temperatures were recorded for the Mills-Packard chambers, 47 feet high[1]

Chamber Number	Temperature °C
1	77 - 84
2	62 - 72
3	60 - 70
4	50 - 60
5	30 - 40
6	20 - 30

Eldon L. Larison of Anaconda, Montana, who was their consulting engineer in 1920, worked on acid-proof masonary for making sulphuric acid. Anaconda Copper Company was confronted with the principle waste gas, sulphur dioxide, from copper sulphide smelters. This waste was detrimental to nearby civilization as well as to green growth. Such a problem had to be dealt in the face of the common law which protected the neighborhood from any disagreeable or irritable gas. Thus Mr. Larison was asked how such a handicap could be properly harnessed.

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1. From Chemical and Metallurgical Engineering, Volume 24, No. 18; Page 786, 1921 by Andrew M. Fairlie.

His only answer was to utilize it in the manufacture of sulphuric acid since sulphur dioxide could be gotten for nothing inasmuch as the company was only interested in copper refining.

The reactions, in making sulphuric acid, generate considerable amount of heat which ordinarily is dissipated by the lead walls of the plant. The radiation capacity of lead chambers is a fixed relation, and a rather definite limit of production is set beyond which the chambers fail to function properly. It has long been known and recognized that the reaction velocity in the chamber process could be increased by vigorous mixing of gases, causing them to impinge upon the surface wetted with acid. Thus Larison constructed an acid-proof masonry plant. The heat of reaction was removed by a continuous flow or circulation of sulphuric acid over the acid-resisting tower packing. Yet the concentration was so regulated to avoid the absorbtion of nitrogen dioxide.

The first all masonry acid-proof tower was erected in 1911 at Nitrolee, South Carolina. It was built of stone and acid-proof cement imported from Germany. Later the nitric acid producers took up the construction of all masonry towers along with the phospheric acid plants. By 1914 American made chemical bricks and acid-proof cement were used.

In the year of 1914, the first all masonry acid-proof plant for sulphuric acid was set up in Macon, Georgia. Since that time all masonry equipment became popular for Glover and Gay-Lussac towers and acid concentrators, in all parts of United States.

Ordinary packing for towers became obselete because of cracking upon intense heat, thus, came in the demand for checker-work chemical brick. Graded sizes of lump quartz or combination of brick and quartz were made.

Water atomizers displaced hot steam for the sake of cooling conveniences and facilities.

In 1914 brimestone was resumed in place of pyrites which had superseded the original use of brimstone. Due to the world war restrictions on importation for Spanish pyrites was the initial move toward brimstone. Many plants were constructed for the purpose of supplying sulphuric acid for high explosives. Such being the situation brimstone industries once more began to flourish in the South. The demand for sulphuric acid kept the chamber plants more active than in the previous years.

Opl of Kruschau, Austria, patented a nitration method in the United States to produce sulphuric acid by the least use of lead chambers. Kaltenbach developed two other nitration methods which had been put to commerical use. One was called the "Pipe Process" and the other was called the "Packed Cell Process."

b. The Pipe Process.

After considerable study of thermal balances between heat entering and leaving - (1) The Glover Tower; (2) The first two chambers out of a total of four; (3) And the last two chambers - Kaltenbach came to the conclusion that the chamber temperatures were **an** influencing factor. He also concluded that unsatisfactory contact between the reacting gases and liquids, the inefficiency of lead surfaces for dissipation of reaction heat, and bulky expensive construction were the defects in the chamber process.

To overcome these observed faults, the pipe process was devised and patented by Kaltenbach. Its main characteristics are according to the designer as follows:

1. The immediate dissipation of heat involved in the nearby vicinity of its generation;
2. The possibility of controlling simultaneously both reaction temperatures and acid concentration to obtain favorable conditions;
3. Intimate contact between the reacting gases and liquids.

The operation of this system will not be explained since it is about the same as the "cell Packed Process" which will soon follow.

The advantages of the pipe process may be listed in order:

1. Great ease of controlling reaction temperatures by means of circulating water in the jackets and of cooled acids in the tubes;
2. Effective utilization of the tubes surface for the interchange of heat;
3. Reduction of lead and other construction materials;
4. Ease of isolating one or more tubes for cleaning or repairing without stopping the operation of the plant;
5. Possibility of limiting number of tubes used in the production;
6. Complete independence of atmospheric conditions;
7. Employment of heat of reaction for concentrating the weaker acids to commercial standards;

8. Finally, elimination of water atomizers.

c. The Packed Cell Process.

This method was fully developed by Larison. His research consisted as follows: First, to construct an apparatus in such a manner that the gas mixture from the Glover tower would impinge vigorously on the packing surfaces which are wetted by sulphuric acid of a low specific gravity. The concentration of sulphuric acid would have to be just low enough so that it could not absorb oxides of nitrogen. Second, to control the temperature of the reacting gases by a direct circulation contact with quantities of cooler weak acid. Third, having provided for the removal of reaction heat by means other than radiation through the metallic walls, to dispense completely with the lead chambers, and finally adopt acid-proof masonry towers internally packed to new design.

Details of the Packed Cell plant:

A column of acid resisting brick was introduced for two reasons. One that it was most effective in causing vigorous mixing of gases. The other provided a large amount of wet surface for cooling, impingement, and condensation of acid. This unit is incased in a masonry structure, and the reacting gases are driven through the brick column by use of fans. Particular care must be employed in laying the bricks as packing so that the cross sectional area will not offer unreasonable resistance to the passage of a predetermined gas volume. Due to structural considerations these packed columns are built in series of cells or small towers.

It has been possible to design and construct an acid plant without the aid of the lead chambers. Such a plant consisted of the Glover tower, a set of packed cells, and a Gay-Lussac tower. This type of set up will make acid at a rate corresponding to one cubic foot of gross packed cell volume per pound of sulphur burned per 24 hours. When operating with a seven to 8 per cent of sulphur dioxide mixture, about 10 minutes were required for converting sulphur dioxide to sulphuric acid. Such a plant was built at Anaconda, Montana producing 25 tons of 50° to 60° Be' acid per day.

The Anaconda plant utilizes sulphur dioxide gas from roasting copper concentrates in a wedge furnace. Sulphur dioxide is drawn, cooled, and blown in by a blower which forces it into the Glover tower of standard size and dimensions. Two 4 x 4 x 40 foot coolers receive and cool the out flowing acid. This division of the plant is just as would be provided for any chamber plant of equal capacity.

From the top of the Glover tower the gases pass through a pair of flues to enter the packed cells. There are five cell packs in one block. These cells are packed with acid resisting bricks of standard shape. The results show no difference in efficiency whether the gases travelled down or up. Each cell is provided with its own acid distribution system. The newly made acid is drawn at the bottom of each cell and passed into a series of coolers. The cooled acid is pumped around by centrifugal pumps. The gravity of the acid must be 48° - 50° Be' because at this concentration, it will not take up sulphur dioxide or oxides of nitrogen. This is accomplished by running water into the distributor tanks as required. A very precise control of the gas temperatures can be maintained by varying the flow of acids in the cells.

The average daily niter consumption during the same run of period was 670 pounds of sodium nitrate or 6 - 7 per cent based on the sulphur in made acid. As the operation became more accustomed to plant practice, the niter consumption decreased to 4 - 5 per cent.

PART TWO

USE OF A GLASS TOWER FOR LABORATORY RESEARCH

A. LABORATORY EXPERIMENTAL STUDY AND RESEARCH.

1. Discussion.

About three weeks during the summer was spent in the library searching and studying any available literature that pertained to the manufacture of sulphuric acid. Eight or 10 most widely commercialized processes used in the early seventeenth century to the present day were carefully studied in details as to the theory involved in each method. Most of the sulphuric acid plants seem to have originated as developements or modifications of the chamber process which still holds the lead in output of commercial sulphuric acid.

Having read considerable literature, most men had devoted their time in studying and investigating the function of the chambers. Many improvements have been centered around the chambers where very little work was done on the towers and other vital attachments. In this problem it is desirous to depense with the chambers completely and give thought to development of the towers and their minor supplementary units. Nevertheless, the same chemical reactions will be confined to this tower method as those used in the chamber process. In order to accomplish this movement it will be necessary to work with the gas-liquid phase more so than in the ordinary methods. The chambers consist of nothing but large empty space since they are based primarily on the gas phase. The towers consist of packing which is practically hinged on the gas-liquid phase. This being true, it seems possible to do away with the spacious chambers.

Since the lead chambers not only provided excess volume for gases to react, they also removed the heat of chemical reaction by radiation through the lead walls. This point must be dealt with in the tower method, because it is necessary to remove the heat of reaction. It can be solved by working with materials which will radiate heat to the outside and be able to resist acid reaction. Some means of cooling facilities will have to be designed. A water-jacketed tower appears to aid the problem for laboratory work.

2. Construction Of The Glass Tower.

The first part of Fall term was spent in building a glass water-jacketed tower. Glass answered well to acid-resistance and a chance to observe what will take place within the tower. It is also a good conductor of heat so that water could carry the heat away after it once passed to the outside of the tower walls.

The entire set up in reality contains two towers in one. The top half (2) (Refer to Blue Print No.1) known as the Gay-Lussac while the lower half is called the Glover tower (3). Both of these towers are connected with a suitable rubber stopper through which small necessary glass tubes (D)&(E) pass so that the air and oxides of nitrogen can enter into the Gay-Lussac tower (2) from tower (3), and the Gay-Lussac acid can drain into the lower half, or Glover tower (3). (For better understanding refer to accompanying Blue Print No.1. All blue prints are enclosed in the pocket of the back cover of this thesis. It is advised to take out blue print No.1 and refer to it as the construction of the glass tower is explained.) Acid-proof cement was used to cover the rubber stoppers. Rubber will not resist corrosion to oxides of nitrogen nor sulphuric acid to any appreciable extent.

Inasmuch as the connection had to be air tight, final assurance could be depended upon the acid-proof cement. The acid-proof cement was mixed with sodium silicate to a plastic state and applied around the rubber stopper and ends of the glass tower. Over night the cement dried and became as hard as rock.

Both the Gay-Lussac (2) and Glover (3) towers as one rest upon a wide mouth bottle (K) which is joined to the bottom of the glass tower (3) with a rubber stopper and acid-proof cement as described above. Four-quarter inch glass tubes are inserted through this rubber stopper (10) so that gases can enter into the wide mouth bottle which will be called the reaction chamber (7). One glass tube admits nitrogen dioxide from the generator (L). One glass tube lets in the sulphur dioxide from its source (T). Another serves the means of passing air into the reaction chamber. The last tube has its one end reach to the bottom of the wide mouth bottle in order to draw out the newly made acid with a vacuum. This reaction chamber has a capacity of seven and one-half liters. (One must not confuse himself when chamber is mentioned here because its purpose is entirely different from the terminology in the lead chamber process.) Its use in this set up serves as a support for the glass tower, catches and collects the acids that trickle down from the above tower, and aids as an entrance for the gases in passing into the Glover tower.

a. Glover Tower (3).

This Glover tower is 48 inches high and two inches on the inside diameter. The walls of the tower are one-eighth of an inch thick.

It is packed with fine glass wool which is cut into two inch strips. Particular care was exercised in packing the tower so that it offered least resistance to passage of gases and likewise to the counterflow of trickling acids. A 250 cc. separatory funnel (I) rests on top of the Glover tower with its stem passing through the rubber stopper. This container holds dilute sulphuric acid which is employed during operation for the absorbtion of new sulphur trioxide which is made in the Glover tower. A small perforated lead pan (F) lies on top of the wool packing. It serves the purpose of equally distributing the dilute sulphuric acid which drips into it from the 250 cc. separatory funnel (I). The Glover tower is designed with two zones. The oxidation zone (H) takes up the bottom half of the tower. Here the sulphur dioxide is oxidized to sulphur trioxide by nitrogen dioxide. The upper half known as the absorption zone (HF) takes on the sulphur trioxide made in the lower part (H). A centigrade thermometer was placed in the packing and protrudes into the oxidation zone (H). The idea in mind was, mainly, to study oxidation reaction temperatures as a means of controlling chemical reactions. If the readings were low they indicated lack of oxidation since it is known that such a reaction gives off enormous quantities of heat. For this reason the lower half was water-jacketed (A) to remove the internal heat. The tower itself is fastened with a condenser clamp (11) to a ring stand well supported onto a table to prevent the set up from tipping over because air passing through it is more than apt to turn over the whole tower.

b. Gay-Lussac Tower (2).

This tower being similar in construction to the Glover tower (3) rests upon the latter. A piece of glass tubing two inches in diameter and 42 inches long constitutes the entire make up (2). The bottom end is enclosed by a lead cup (G) sealed with acid-proof cement. Two half inch glass tubes (D)&(E) passing up from the Glover extend up through the bottom of the Gay-Lussac tower, or the mentioned lead cup. (Refer to blue print No. 1 again). The short tube (D) permits the oxides of nitrogen and air to enter the Gay-Lussac tower from the Glover tower. The other longer one (E) carries the Gay-Lussac down two-thirds of Glover at the point (H) and is released. The lead cup (G) mentioned serves as a collecting pan for the trickling acid in its tower. The bottom part of this tower is packed with beads (12) so that the acid could freely drip into the bottom tower; otherwise if glass wool (C) were packed all the way down, it would hold the acid from draining because of the fine close packing. From here up, glass wool filled the remainder of the tower. A thermometer was placed in about the center of tower (2) so that temperatures could be observed. Another perforated lead cup (B) rests upon the packing (C) of this tower to act as a distributor for the 60°Be' acid which is in the above 250 cc. separatory funnel (A). The top of the tower is capped with a rubber stopper through which passes the stem of the 250 cc. separatory funnel (A), the half-inch flue line (W), and a thermometer. The thermometer was inserted to obtain the temperature of the spent air for final calculations.

This stopper is not covered with acid-proof cement as other joints are since nothing but air attaches it. It merely aids as a closed top to the set up and holds the above described attachments in their respective positions. This tower like the Glover tower is fastened with a condenser clamp (13) to the same ring stand.

c. Supplementary Attachments.

These are numerous and it is necessary to mention each one's function and purpose.

A six liter erlenmeyer flask constitutes the entire body of the generator (L). It has its top closed with rubber stopper and acid-proof cement. Three one-eighth inch glass tube lines pass into it through the rubber stopper. One is the stem of glass funnel (M) which was used to pour through nitric and sulphuric acid. Since this generator has its top permanently closed and air tight sealed, a vacuum line (14) was necessary to draw off the spent acids. The third line (C) is the flue line for the passage of nitrogen dioxide gas from the generator (L) into the reaction chamber (K) previously described. Some of the air is passed into the generator through the vacuum line so it can bubble through the mixed acid and carry along with it the nitrogen dioxide gas into the reaction (K). The whole producer is heated with a bunsen burner (N) to release the oxide of nitrogen. (Refer to how each was run on page 34).

The newly made acid contained some oxides of nitrogen which had to be removed. Thus a six liter erlenmeyer flask (S) provided as a means of this removal. It is capped with a rubber stopper through which three one-eighth inch glass tubes pass. One was used to draw off the newly made acid with a vacuum from the reaction chamber (K). The other served as a flue line for the oxides of nitrogen which were driven off by heat (AA) and carried by air into the reaction chamber (K) through the third glass tube.

A ten liter battery jar (6) was used to cool the reaction chamber (K) which contained the newly made and spent acids. Of course, this particular cooling chamber in a commercial plant of this design would not be necessary. In this case it was desirous so that a complete heat-balance could be calculated to account for all the heat of reaction. The inlet cooling water (X) came in at the bottom through a glass tube which extended vertically beneath the surface and almost touching the bottom of the battery jar (6). The outlet cooling water was drawn off with a vacuum into a five gallon bottle (7).

In order to measure the consumed volumes of sulphur dioxide and air, one twenty-light dry gas meter (V) and one thirty-light dry gas meter were obtained from Consumers Power Company in Lansing. The smaller one was used for sulphur dioxide while the larger one was used for the air. Two manometers (T)&(P) had to be made; one for each meter. Particular care was necessary not to exert more than two pounds pressure per square inch upon these meters. Any amount over two pounds would give inaccurate readings and probably ruin the meters. For this reason the air passed through the manometers first and then into the dry gas meters. Note that the temperature of sulphur dioxide (V) and air (R) were accounted for by respectively passing each into its small bottle and out again. Each bottle was sealed tightly with a rubber stopper. Inlet and outlet glass tubes as well as a thermometer were inserted through the rubber stopper.

B. PROCEDURE FOR EACH RUN.

First, 60°Be' sulphuric acid was poured into the separatory funnel (A) which rests on top of the Gay-Lussac tower (2). (For better understanding refer to the accompanying blue print No. 1. All blue prints are enclosed in the pocket of the back cover of this thesis. It is advised to take out the corresponding blue print and refer to it as each detail is described and explained). By opening the stopcock (A), the acid runs into the perforated lead cup (B) situated on top of the packing (C) of tower (2). The cup distributes the acid in a spray which trickles down over (C). Thus the saturation of the surface material of the tower was maintained while the test was made. The purpose of this is to make it possible for the oxides of nitrogen passing from (H) through (D) to be recovered by the 60°Be' acid. These oxides of nitrogen react with the said acid to form nitrosyl sulphuric acid as follows:

1. $2NO_2 + H_2SO_4 \longrightarrow SO_5NH + HNO_3$

The nitrosyl sulphuric acid mixed with the 60°Be' acid, and both are collected in (G) as they pass into (E) which carries them into (H) where dilution took place with the dilute acid. It then runs from (I) through another perforated lead cup (F) distributing it over the packing (J). When the dilute acid and the 60°Be' acid containing the nitrosyl sulphuric acid meet the following reaction takes place:

2. $2SO_5NH + H_2O \longrightarrow 2H_2SO_4 + NO_2 + NO$

Nitrosyl sulphuric is stable in 60°Be' sulphuric acid and as soon as water gets to it, dilution takes place and nitrosyl sulphuric acid breaks up as shown by reaction 2. Consequently, the oxides of nitrogen are released in

(H), and the diluted 60°Be' acid passes down into (K).

The original source of oxides of nitrogen were introduced from (L) called the nitrogen dioxide generator. Five hundred cc. of HNO_3 (1.39 Sp.Gr.) were poured into (L) through the funnel (M), also, 500 cc. of concentrated sulphuric acid (66°Be') were added to the same container through (M). Having both acids in (L) as mixed acids, these are heated by the bunsen burner (N). Upon heating, the oxides of nitrogen are liberated from the solution and pass through the flue line (L) into the reaction chamber (K).

Particular care was exercised in heating (L). If too much heat is applied to the mixed acids, the nitric acid will distill over, and this is not desirable; so by using a lower flame (N) a greater portion will remain in (L). Concentrated sulphuric acid was used because it has more affinity for water than do the oxides of nitrogen. Thus the sulphuric acid could take on its maximum water of hydration. Concentrated nitric acid was employed because it contained little water, thereby, lessening the possibility of having too much water present to form nitric acid as the oxides of nitrogen were once evolved.

The air was introduced through the manometer (P), then over into the air meter (Q) from where it passed into the temperature recording unit (R). From (R) it was transmitted into (S) and finally into the reaction chamber (K). As the oxides of nitrogen were generated in (L) and evolved over (O) into (K), the air carried them up in the Glover tower (G). This was continued until the reaction chamber (K) and the Glover tower were filled. Since the oxides of nitrogen (NO_2) are of reddish brown color, it was easily observed when the reaction chamber and Glover tower were filled with them.

At this point the SO_2 was admitted into the set-up. From the SO_2 cylinder it was passed through the manometer (T), then over into the SO_2 meter (U), next into the temperature recording unit (V) and finally into the reaction chamber (K). The same air that carries the NO_2 from (K) into the tower (3) simultaneously carries the SO_2.

The SO_2 and NO_2 both react in the tower (3) at (H) called the oxidation zone with the following reaction taking place -----

3. $SO_2 + NO_2 \longrightarrow SO_3 + NO +$ Heat.

SO_2 is oxidized to SO_3 by NO_2 at the same time evolving heat. This heat is known as positive heat of reaction. From and between the points (H) and (F) of the Glover tower (3) the SO_3 is carried up by the air and absorbed by counter flow of dilute sulphuric acid from (F). Thus the concentration of this weak acid is built up corresponding to the amount of SO_3 formed. Now, the built-up acid trickles below (H) and into (K). From (K) the newly made acid and 60° Be' acid which came down from (A) were drawn out by means of a vacuum. This was done to draw out the product where it could be heated in (S) to drive off the oxides of nitrogen which happen to be absorbed in solution. As soon as all the product is drawn into (S) the vacuum is released and air comes in through the same line (vacuum line) carrying back the NO_2 gases into (K) while (S) is being heated.

Going back to reaction (3) the NO gases were carried into the Gay-Lussac tower (2) by the air through (D). The NO is oxidized to NO_2 on its way up by the oxygen present in the air in the following manner ---

4. $NO + \frac{1}{2}O \longrightarrow NO$ - heat.

This reaction differs from 3 insofar as evolution of heat is concerned. Instead of giving off positive heat it takes on heat from the immediate vicinity. Such a reaction is known as endothermic while reaction 3 is exothermic. As the NO_2 enters the Gay-Lussac tower (2) through the flue line (D) with the air carrying it along, it is absorbed by the 60° Be' Acid coming down from (A). Reaction No. 1 takes place within this area (C). The air keeps on passing and leaves the tower (2) through the vent line (W). It is known as spent air consisting mostly of nitrogen since its oxygen was taken up by reaction 4. Thus the cycle of this process is completed. It will go on as explained during time the run is continued.

It is necessary to explain the different points of the set-up while the test was being made. The manometers (T) and (P) were made up to insure proper pressures on the meters (U) and (G). If the pressure exerted upon these meters was greater than two pounds per square inch, they would not register properly the respective volumes of SO_2 and air.

The meters were employed for four reasons. First, to be able to account for the amount of SO_2 and air introduced so that calculations can be made for the material balance. Second, the amount of acid made from the SO_2 and third, the heat balance, and finally as a means for controlling the process. The air should theoretically be passed in at a ratio of 3.6 to 1 by volume. At this degree enough oxygen is available to indirectly convert SO_2 to SO_3; although,it is best to exceed the ratio to insure a sufficient amount of oxygen.

The temperature recording units (R) for air and (V) for SO_2 were employed so that data could be obtained for heat balance calculations of the process.

Since this problem involves making sulphuric acid by a tower method as pointed out in the introduction, it is known that heat of reaction will have to be removed in order that the process will carry on efficiently; also that the heat of reaction will be given off in the oxidation zone (H) of tower (3). For this reason it was water-jacketed, indicated in the blue print as (4). As the heat is liberated from the oxidation zone, it is conducted to the outside of the tower where it is picked up by the cooling water circulating in (4). The reaction chamber is kept cool in the battery jar (6). Water passes in from (x) and is divided so that half of it goes into (4) and the other half into (6). From (6) the spent water is drawn off by a vacuum into (7) where the amount can be accounted for. Likewise, from (4) it was drawn into a suitable container and also recorded.

In order to study reaction temperatures of the Glover and Gay-Lussac towers while the run was in progress, thermometers were inserted in tower (3) and (2). In (3) the thermometer was placed in the oxidation zone while in (2) in the center of it. Thus if there was a rise in temperature of tower (3), it indicated that oxidation of SO_2 to SO_3 was taking place. The other in tower (2) was used to study Gay-Lussac tower temperatures from the standpoint of controlling the heat of that area. In practice such towers should be cold in order to have good recovery of oxides of nitrogen.

While the run was in operation, data was recorded every fifteen minutes. Such an interval was chosen because it took that long to record the temperatures of inlet air (R), SO_2 at (V), and water at (X), and of outlet air at (W), outlet water of the reaction chamber at (U), and of the water-jacket at (2). Other data was also taken such as volume of air at (Q) and of SO_2 at (V); pressure of air at (P), of SO_2 at (V); pressure of air at (P), of SO_2 at (T), and the weight of outlet cooling. water from reaction chamber at (7) was received in a pail and weighed as such. Of course another bucket was replaced while the one containing the water of the previous interval was being weighed. The containers (A) and (I) had to be refilled when they became emptied. This was done during each interval, and amounts of each filling were measured out in cc's. Temperatures of these input acids were also tabulated.

Such procedure was followed out for each run. Some of these runs were from 2 to 3 hours long while others were shorter due to some difficulties arising at which points the tests had to be concluded.

1. Laboratory Runs.

The following symbols were used to denote various recorded data.

T_S - Inlet temperature of SO_2 degrees C.

T_{A_1} - Inlet temperature of air " ".

T_A - Outlet " " spent air degrees C.

T_0 - Inlet " " cooling water degrees C.

T_1 - Outlet " " " " of the Glover tower degrees C.

T_2 - Outlet " " " " " " reaction chamber degrees C.

T_3 - Temperature of Glover tower.

T_4 - " " Gay-Lussac tower.

P_2 - Pressure of inlet air in pounds per square inch.

P_1 - " " " SO_2 in pounds per square inch.

V_2 - Volume of inlet air in cubic feet.

V_1 - " " " SO_2 in " ".

W_1 - Weight of cooling water in pounds from the Glover tower.

W_2 - " " " " " " " " reaction chamber.

The following pages include laboratory runs and discussion of each.

Run No. 1

Time in Min.	T_S	T_{A_1}	T_A	T_0	T_1	T_2	T_3	T_4	P_2	P_1	V_1	V_2	W_1	W_2
0	27.°	27.0°	27.0	19.°0	19.0	19.0	27.0	27.0	0	0	0	0	0	0
15	21.°	21.0	25.0	19	25.0	28.5	32.0	31.0	1.06	1.06	1.0	2.0	7.58	5.16
30	22.	22.0	25.5	19	25.5	28.5	32.0	45.0	1.96	1.96	4.0	6.5	7.60	4.84
45	22.	22.0	25.0	19	25.0	29.0	32.0	45.0	1.62	1.56	7.5	12.0	7.12	5.60
60	22.	22.0	24.5	19	24.5	28.5	33.5	45.0	2.16	2.00	9.0	14.0	7.13	5.40
75	22.	22.0	24.0	19	24.0	27.5	35.0	45.0	2.16	2.00	10.0	16.0	5.10	4.00
90	22.	22.0	24.0	19	24.5	27.5	34.0	46.0	2.66	2.37	11.5	21.0	4.80	4.80
105	22.	22.0	23.5	19	23.0	26.5	32.0	45.0	2.50	2.12	13.0	26.0	4.10	2.57
Average	22.	22.0	24.5	19.	24.5	28.0	32.9	42.8	2.06	1.89	13.0	26.0	43.61	32.57

Temp. 23.°C

Input

	H_2So_4		HNO_3
SP. Gr.	1.69	1.08	1.39
Grams.	1268.0	1028.0	146.0
Vol. cc.	750.0	950.0	105.0

Temp. 27.°C

Output

H_2SO_4	
Specific Gravity	1.38
Grams.	2528.0
Vol. cc.	1831.0

DISCUSSION OF LABORATORY RUNS

Run No. 1

Observation of the results on this run showed that it did not turn out very satisfactorily. The ratio of air to SO_2 theoretically should have been 3.6 to 1. Data showed a 2 to 1 ratio. By this indication most of the NO was lost due to lack of oxidation to NO_2.

All the average temperatures show that there couldn't have been much oxidation between SO_2 with NO_2, because the thermometer readings were low. If this reaction had gone to the full extent and with the 13 cubic feet of SO_2, the thermometer readings would have been higher.

The temperatures of the Gay-Lussac tower were higher than at any other point. For a satisfactory run the Gay-Lussac tower temperatures should be low. Something must have taken place in the Gay-Lussac tower to raise the temperature.

Run No. 2

Time in Min.	T_S	T_{A_1}	T_A	T_0	T_1	T_2	T_3	T_4	P_1	P_2	V_1	V_2	W_1	W_2
0	22.0	22.0	22.0	18.0°	18.0	18.0	23.0	23.0	0	0	0	0	0	0
15	23.0	23.0	25.5	18	23.0	24.0	28.0	28.0	0.16	0.25	2.0	4.5	7.23	6.2
30	21.5	22.5	25.0	18	20.0	24.0	29.0	28.5	0.56	0.81	2.7	6.3	7.46	5.6
45	22.0	22.0	24.0	18	20.0	22.0	29.0	30.0	1.31	1.62	3.0	8.3	6.58	5.65
60	23.5	22.0	24.5	18	20.5	21.5	26.5	34.0	1.75	2.00	3.5	10.0	6.35	5.10
75	24.0	23.5	26.0	18	21.0	21.5	27.0	*35.0	1.87	2.16	3.7	11.8	6.30	5.00
90	25.0	23.7	26.5	18	21.5	22.0	30.0	38.0	1.87	2.12	3.9	13.5	6.12	4.47
105	26.0	24.8	27.3	18	21.5	21.5	30.0	37.5	1.62	1.87	4.1	15.0	6.00	4.24
120	26.0	25.8	28.3	18	21.5	22.0	29.0	37.0	1.86	2.00	4.3	17.4	5.82	4.16
135	26.0	26.0	28.5	18	23.0	23.0	30.3	35.0	1.86	2.00	4.5	20.0	5.02	4.28
150	25.0	26.0	28.5	18	24.0	23.0	31.5	36.0	1.86	2.00	4.7	23.5	4.50	3.92
165	24.0	25.0	27.5	18	25.0	23.0	31.0	37.0	1.86	2.00	4.8	27.0	4.00	3.56
180	24.0	24.5	27.0	18	24.5	23.0	31.5	37.0	1.86	2.00	4.9	29.0	3.82	2.16
195	24.0	24.5	27.0	18	24.0	23.0	31.5	39.0	1.86	2.00	5.0	31.0	2.00	2.00
210	24.0	24.5	26.0	18	24.5	23.5	31.5	38.0	1.86	2.00	5.1	33.5	2.00	1.75
Average	24.0	24.0	26.6	18.0	22.4°	22.7	29.7	32.0	1.54	1.77	5.1	33.5	73.2	57.84

*Dilution of 60° acid with 10.8° acid.

	Input			Output
	H_2SO_4		HNO_3	H_2SO_4
Sp. Gr.	1.70	1.08	1.39	1.39
Wt. Grams	850.0	1080	152	2250
Vol. cc.	500.0	1000.0	108.2	1620
Temp.°C	23	23	23	26

Run No. 2

This run ran for three and one-half hours whereas Run No. 1 was one and three-quarters hours long. The object was to cut down on the flow of SO_2 and lengthen the time to cut down on the velocity of SO_2 in passing through the Glover tower. According to the temperature, again the readings were about the same as those of the previous run.

The efficiency of converting SO_2 to H_2SO_4 increased from 15.85% of the first run to 61.8% for this one. The increase may be due to the excess amount of air used in comparison with theoretical amounts. Another reason for such increase in efficiency arises from the fact that Gay-Lussac tower temperatures were lower. This run showed a decrease of 10° C. over the first one in the Gay-Lussac tower. Still these temperatures were higher than those of Glover tower.

At the end of 75 minutes the Gay-Lussac temperature began to increase which may be due to the water content of the air or dilution with 12% acid.

Run No. 3

Time in Min.	T_S	T_{A_1}	T_A	P_1	P_2	V_1	V_2
0	22.0	22.0	22	0	0	0	0
30	19.0	19	36	1.00	1.12	0.3	3.0
60	18.0	19	38	0.62	0.75	0.6	6.0
90	18.0	18	40	1.28	1.38	1.4	10.0
120	18.0	18	46	1.87	2.00	1.9	14.0
150	18.0	19	46	1.87	2.00	2.4	15.0
180	18.0	19	46	2.00	2.12	3.2	17.0
Average	18	18.7	42	1.44	1.56	3.2	17.0

	Input			Output
	H_2SO_4		HNO_3	H_2SO_4
Sp. Gr.	1.71	1.08	1.39	1.29
Wt. Grams	762.0	1080	160.0	1892.0
Vol. cc.	445.0	1000	115.0	1468.0
Temp. °C	22.0	22.0	22.0	27.0

Run No. 4

Time Min.	T_S	T_{A_1}	T_A	T_0	T_1	T_2	T_3	T_4	P_1	P_2	V_1	V_2	W_1	W_2
0	23.0	22.0	23	22	22	22	23	23	0	0	0	0	0	0
10	21.0	20.0	22	22	22.5	22	24	22	1.25	1.5	0.25	2.0	3.10	2.71
20	19.0	19	22	21.5	23	22	25	22	0.75	0.87	0.50	3.0	3.12	2.79
30	18.0	18	21	21	23	22	24	21	1.25	1.5	0.70	4.33	3.71	2.80
40	18	18	20	20	22	22	23	20	1.75	1.87	0.90	6.00	3.41	2.79
50	19	19.5	21	20	22	21¾	26.5	21	1.75	1.87	1.2	7.30	2.90	2.91
60	19	20	21	20.5	21	21	25	21	1.87	2.00	1.3	8.30	2.50	2.71
70	18.5	19	21	20	21	21	21	21	1.87	2.00	1.7	9.70	6.00	4.0
80	18.5	18	20	20	21	21	21	20	1.56	1.81	1.8	10.2	6.72	4.1
90	18.5	18	21	20.75	21	21	22	21	1.75	1.87	1.9	12.0	5.98	4.14
100	18.5	19	30	20.25	22	22	23	30	1.00	1.12	2.4	14.0	5.62	4.70
110	19	19	30	20.25	22	22	23	30	1.00	1.12	2.7	16.7	5.70	3.92
120	19	19.5	30	19	22	22	23	30	1.00	1.12	2.9	16.7	4.60	3.47
Average	18.8	18.9	23.2	20.7	21.6	21.8	23.3	23.2	1.4	1.56	2.9	16.7	53.33	42.04

	Input			Output
	H_2SO_4		HNO_3	H_2SO_4
Sp. Gr.	1.71	1.08	1.39	1.30
Wt. Grams	710.0	1080.0	148.0	1900.0
Vol. cc.	415.0	1000.0	106.2	1460.0
Temp. °C	22	22	22	25

Run No. 3

The use of cooling water was omitted in this run to see how important a function it really served. Note, that all the temperatures increased considerably; not due to more oxidation in the Glover tower because the efficiency was lowered. The water does remove considerable heat which needs to be taken out. A drop of efficiency of 60% to 15% proved it.

Run No. 4

All the temperatures did not rise more than a degree or two through out over the initial temperatures before the start of run. Still there was a 40% efficient conversion of SO_2 to H_2SO_4 acid. This information brings to light that other factors influence conversion of SO_2 to H_2SO_4 besides the showing of oxidation which in turn is indicated by the thermometers.

Run No. 5

Min. Time	T_S	T_{A_1}	T_A	T_0	T_1	T_2	T_3	T_4	P_1	P_2	V_1	V_2	W_1	W_2
0	20.°	20.0	20.0	20.0	20.0	20.0	20	20	0	0	0	0	0	0
10	18	18	20	20	20.5	21.0	19	23	0.25	0.5	0.2	2.0	2.16	1.92
20	18	18	21	20	20.5	21	19	23	0.75	0.93	0.3	3.3	2.18	1.87
30	17	18	20	20	20.5	21	20	24	0.50	0.68	0.5	4.0	2.17	1.96
40	17	18	20	20.5	21.5	21	20.15	26	0.56	0.75	0.9	5.1	2.18	1.82
50	17	18	20	21.0	22.0	21	20.0	29	0.87	1.00	1.2	6.2	2.16	1.70
60	16.5	19	21	21.0	23.0	21	21	29	1.87	1.00	1.5	7.5	2.19	1.82
70	17	19	21	21.0	22.5	21	21	30	0.25	0.56	1.8	9.5	2.18	1.9
80	17	19	21	21.0	22.0	22	22	31	0.45	0.62	2.2	10.8	2.20	1.96
90	18	19	21	21.0	23.0	22	22	30	0.62	0.83	3.0	13.0	2.10	2.0
100	18	18.5	21	21.0	24.0	22.5	23	32	1.25	1.50	3.8	15.4	2.16	1.86
110	17	18.5	22	21.0	24.0	22.5	23.5	31	1.06	1.18	4.6	17.6	2.17	1.72
120	17	19	21	21.0	24.0	23.5	23.0	32	1.12	1.25	5.2	19.5	2.19	1.9
Average	17.3	18.5	20.9	20.9	22.2	21.5	21.1	28.3	0.81	0.90	5.2	19.5	26.04	22.43

	Input			Output
	H_2SO_4		HNO_3	H_2SO_4
Sp. Gr.	1.71	1.08	1.39	1.40
Wt. Grams	707.0	540.0	161	1316.0
Vol. cc.	413.0	500.0	115.8	940.0
Temp. C	20	20	20	23

Run No. 6

Time Min.	T_S	T_{A_1}	T_A	T_0	T_1	T_2	T_3	T_4	P_1	V_2	V_1	P_2	W_1	W_2
0	19	20	19	22.5	22.5	22.5	23	20	0	0	0	0	0	0
10	18.7	19	21.5	22.	22.0	22	23	21	0.81	1.3	0	1.06	4.88	2.71
20	19.0	19.7	22.0	21.3	22.5	22	23	21	0.87	3.5	0.27	1.06	4.15	2.56
30	19.0	19.7	21.7	21	22.2	21.7	23	21	0.87	4.8	0.5	1.06	5.15	3.43
40	18.8	19.	21.5	20.5	21.5	20.5	23	21	0.2	6.0	0.6	0.37	4.69	3.11
50	19.0	19.5	21.	20.5	21.0	21.	22	21	0.2	7.3	0.8	0.37	4.04	2.39
60	19.0	18	21.	20.5	21.0	20.5	20	21	0.2	9.0	1.0	0.40	4.13	2.60
70	18.5	18.2	22	21	21.0	21	21	21	0.2	10.9	1.2	0.35	3.93	2.07
80	18.0	19	21.5	21.7	22.0	21.7	24	21	0.2	12.3	1.5	0.37	4.44	1.93
90	18.0	18.7	22	22	22	22	25	21	0.4	13.5	2.0	0.7	3.88	1.48
100	18.5	19.2	22	22.5	22.5	22.5	24	22	0.4	15.4	2.5	0.7	4.03	1.48
110	18.5	19	22	22.5	22.5	22.5	23	21.	0.2	16.9	3.0	0.4	3.53	1.31
120	18.5	19	21.5	23	23	23	24	22	0.2	18.5	3.3	0.4	3.53	3.31
130	18.5	19	21	23	23	23	24	22	0.2	20.	4.0	0.4	3.15	3.12
140	18.5	19	21	23.5	23.5	23.5	25	21	0.2	21.5	4.2	0.4	3.15	2.21
150	18.5	19.3	21	24	24	24	24	21	0.25	23.2	4.3	0.5	3.26	1.91
160	18.5	19.5	21	24	24	24	25	21	0.31	24.8	4.5	0.62	3.26	2.10
170	18.5	19	21	23.5	24	23.5	25	21	0.31	27.0	4.6	0.62	2.22	1.66
180	18.5	19	21	24	24	24	25	21	0.31	28.3	4.8	0.62	3.46	3.46
Average	18.5	19	21.6	22.2	22.4	22.3	23.4	21.	0.365	28.3	4.8	0.58	67.88	42.83

	Input			Output
	H_2SO_4		HNO_3	H_2SO_4
Sp. Gr.	1.69	1.08	1.39	1.45
Wt. Grams	1690	1080	170.	3300
Vol. cc.	1000	1000	122.	2090
Temp. °C	22.5	22.5	22.5	24

Run No. 7

Time Min.	T_S	T_A	T_A	T_0	T_1	T_2	T_3	T_4	P_1	P_2	V_1	V_2	W_1	W_2
0	19.5	20	20	18.5	18.5	20	19.5	20	0	0	0	0	0	0
15	19.5	19.5	20.5	19.0	21	20	21.	21	0.61	0.75	0.2	2.0	5.36	3.6
30	19.5	19.5	20	19.0	21	20	21	21	0.9	1.1	0.45	3.0	3.96	2.45
45	19.5	20.0	20.5	20	21.5	20	22	21	0.9	1.1	0.6	5.5	3.87	2.96
60	19.5	21	21	20	21.5	20.5	24	21	1.3	1.45	1.4	6.0	3.03	4.48
75	21.5	21	20	20	21	21	25	21	1.75	1.87	2.0	8.4	3.09	4.53
90	21.0	20	20.5	21	21	21	24.5	22	1.87	2.00	2.3	10.0	2.42	3.03
105	22.0	20.5	21.5	21	21.5	21.5	27	33	1.87	2.00	2.5	11.0	3.09	2.96
120	21.5	21.5	24.0	22	23	22.5	27	24	1.87	2.00	3.0	13.0	3.78	2.27
135	20	19.5	23.	20.5	22	22.5	25	22	1.87	2.00	3.5	15.3	2.81	3.39
150	20	19.0	22.	19.5	22	21.0	23	22	1.87	2.00	4.0	17.0	3.12	5.45
Average	20.4	20.2	21.3	20.2	21.55	21.0	23.95	21.8	1.48	1.62	4.0	17.0	29.02	35.12

	Input			Output
	H_2SO_4		HNO_3	H_2SO_4
Sp. Gr.	1.69	1.08	1.39	1.42
Wt. Grams	1540	1080	156	2840
Vol. cc	920	1000	112	1990
Temp. °C	20	20	20	24

Run No. 8

Time Min.	T_S	T_{A_1}	T_A	T_0	T_1	T_2	T_3	T_4	P_1	P_2	V_1	V_2	W_1	W_2
0	19.5	19.	21	19	19	19	20	21	0	0	0	0	0	0
15	17.5	17.5	20.5	19	20	19.5	23	21	0.2	0.3	0.7	1.7	5.78	5.63
30	17	17	19	20	21.5	20	23	20	0.15	0.27	0.8	4.0	3.84	2.75
45	16	16	18	19.5	23	19.5	23	19	0.2	0.3	1.2	6.0	3.33	2.42
60	17	17.0	17	20	23	20	23	19	0.2	0.3	1.8	8.8	6.24	2.75
75	19.5	19.5	18	20	23.5	20	23	19.5	0.2	0.3	2.0	11.2	6.06	2.84
90	21	21	20	20.5	23.5	20.5	23	20.5	0.2	0.3	2.2	13.6	5.96	2.66
105	21	21	21	21	23.5	21	23	20.5	0.2	0.3	2.5	16.0	6.06	3.87
120	19	19	21	20.5	24.	21	23	20.5	0.2	0.3	2.8	18.3	5.12	3.19
135	20	20	21.	20.5	23.	21	22.5	21.0	0.2	0.3	2.9	20.5	5.54	3.19
150	20	20	21	20.5	21.5	21	22.5	21.0	0.2	0.3	2.9	22.7	4.78	2.6
Average	18.8	18.8	19.65	20.15	22.65	20.35	23.	20.2	0.2	0.3	2.9	22.7	52.71	31.9

	Input			Output
	H_2SO_4		HNO_3	H_2SO_4
Sp. Gr.	1.71	1.08	1.39	1.44
Wt. Grams	1805.	1080	165	3150
Vol. cc.	1055	1000	118.7	2095
Temp. °C	20	20	20	21

Runs No. 5 to 8.

These four runs did not give any new information than had the previous runs. Only one thing left to consider and that is the oxides of nitrogen. Much of SO_2 was not converted to H_2SO_4 and this reaction being dependent upon the influence of oxides of nitrogen left the problem open to the key of the process.

These eight runs were made by generating the NO_2 gas and admitting it as such. It was thought to try and mix the HNO_3 with the 60° acid in the Gay-Lussac tower. This was done. HNO_3 and H_2SO_4 were mixed and used in the Gay-Lussac tower in the usual manner as the 60° H_2SO_4 was heretofore.

Runs 6, 7 and 8 were made under additional height of Gay-Lussac tower which showed constant efficiency but not a satisfactory one.

Up to this point glass beads were used as packing material. These were removed and replaced with glass wool.

Run No. 9

Time Min.	T_S	T_{A_1}	T_A	T_0	T_1	T_2	T_3	T_4	P_1	P_2	V_1	V_2	W_1	W_2
0	21.5	21.5	21.5	19.5	19.5	19.5	21.5	21.5	0	0	0	0	0	0
15	22.0	21.	22.	19.5	23.	21.7	22	21.5	0.2	0.25	0.3	2.0	5.18	7.18
30	22.5	20	22	19.5	22	22	24	21.5	0.35	0.4	0.7	4.6	4.86	14.83
45	21.0	19.5	22	19.5	26	23	31	25	0.2	0.3	1.5	7.2	2.83	6.5
60	20.0	19.5	32	16.5	34	24	37	26	0.3	0.4	3.25	14.2	1.7	12.0
75	19.5	20	29	16.5	31	23	38	18	0.4	0.45	4.9	17.4	1.35	6.96
90	19.5	20	25	15.5	19	21	35	19	-	-	6.8	21.6	2.52	7.46
Average	20.7	20.	25.3	17.8	25.9	22.3	31.2	21.8	0.29	0.36	6.8	21.6	18.44	54.93

	Input			Output
	H_2SO_4		HNO_3	H_2SO_4
Sp. Gr.	1.63	1.08	1.39	1.40
Wt. Grams	1392	1175	378	3440
Vol. cc.	854	1087	272	2460
Temp. °C	21.5	21.5	21.5	23

Run No. 10

Time Min.	T_S	T_{A_1}	T_A	T_0	T_1	T_2	T_3	T_4	P_1	P_2	V_1	V_2	W_1	W_2
0	23	23	24	20.5	20.5	20.5	24	24	0	0	0	0	0	0
15	22	21	25	21	22.5	22	30	28	0.2	0.3	0.5	1.5	2.90	6.16
30	22	21	25	19	29.0	22	37	27	0.25	0.35	1.0	2.5	1.7	2.78
45	21	20	26	19	30	22.5	75	36	0.4	0.50	2.0	3.0	6.66	9.21
60	21.5	20	36	19.5	25	22.	47	27	0.25	0.40	3.0	4.2	12.9	10.45
75	21.5	19.5	28	19.5	28	23	43	33	0.4	0.62	4.0	6.25	8.5	9.66
90	20.	18	24	19	31	23	46	39	0.4	0.62	5.1	9.3	6.5	9.82
105	19.5	17.5	24	18.5	37	25	50	38	0.7	1.00	6.2	11.2	4.22	13.3
120	18.5	16.5	28	18.5	37	25	58	51	0.7	1.00	7.0	14.1	3.75	13.6
135	19.	17.	27	18.5	30	23	48	51	0.4	0.75	8.1	17.5	7.42	19.9
150	18.5	18.	26	18.5	30	21	37	32	0.2	0.35	9.27	19.2	1.09	17.78
Average	20.35	18.85	27.9	19.1	29.0	22.85	47.1	36.2	0.39	0.589	9.27	19.2	55.64	112.66

	Input			Output
	H_2SO_4		HNO_3	H_2SO_4
Sp. Gr.	1.63	1.08	1.39	1.42
Wt. Grams	2300	2100	304	5315
Vol. cc.	1410	1944	219	3740
Temp. °C	24°	24°	24°	26°

Run No. 11

Time Min.	T_S	T_{A_1}	T_A	T_0	T_1	T_2	T_3	T_4	P_1	P_2	V_1	V_2	W_1	W_2
0	24	24	24	19	19	19	20	24	0	0	0	0	0	0
15	23	23	33	19	20	20	22	85	0.25	0.37	0.6	1.7	11.34	5.18
30	21	21	25	19	24	20	46	56	0.35	0.50	1.2	4.1	5.50	7.31
45	20	19	22	19	24	20	47	48	0.35	0.50	1.8	6.5	6.43	6.87
60	19.5	17	31.5	18	24	20	47	77	0.38	0.60	2.2	9.0	6.97	7.25
75	19.	17	25.	16	24	20	55	86	0.38	0.60	2.9	11.2	6.81	9.69
90	18	16.5	22	15	23	19	53	83	0.38	0.60	4.0	13.8	7.81	12.56
105	17	16.5	35	16	24	18	55	80	0.35	0.50	5.0	15.5	7.62	12.78
120	16	15.	57	16	20	18	30	89	0.38	0.60	6.0	17.6	7.19	11.37
135	16	15.	42	17	19	18	31	70	0.38	0.60	6.5	19.9	7.25	11.13
150	16	15	23	18	20	18	55	56	0.38	0.60	7.0	21.5	6.50	10.
165	16	15	21	18	21	18	55	60	0.38	0.60	7.2	22.0	6.25	9.75
Average	18.3	17.2	30.5	17.3	22.1	19.0	45.	71.7	0.348	0.55	7.2	22.0	79.67	103.89

	Input			Output
	H_2SO_4		HNO_3	H_2SO_4
Sp. Gr.	1.8	1.08	1.39	1.55
Wt. Grams	1888.	1055	254	3466
Vol. cc.	1021	968	183	2230
Temp. °C	22	22	22	24

Run No. 12

Time Min.	T_S	T_{A_1}	T_A	T_0	T_1	T_2	T_3	T_4	P_1	P_2	V_1	V_2	W_1	W_2
0	20	20	23	23	23	23	21	23	0	0	0	0	0	0
15	20	19	23	23	24	23	37	32	0.17	0.37	0.6	2.5	9.22	8.62
30	20	18	23	20	25	23	52	37	0.25	0.37	1.15	4.4	8.63	10.38
45	20	18	23	18.5	23	22.5	57	54	0.27	0.37	1.70	6.0	9.06	12.83
60	20	19	24.5	18.5	22	21	51	68	0.30	0.40	2.50	7.9	9.75	8.63
75	20	19	*55	18.5	21	21	43	70	0.30	0.4	3.10	9.5	9.83	9.72
90	20	19	35	18	21	20	40	70	0.25	0.37	3.70	10.6	9.10	7.75
105	20	19	29	17	21	20	45	68	0.22	0.37	4.50	12.1	8.22	7.75
120	20	19	31	15	19.5	19.5	40	68	0.30	0.4	4.90	13.8	7.37	8.34
135	20	18	31	15	18.0	19.	35	70	0.30	0.4	6.0	16.0	8.43	10.31
150	20	18	38	15	18.0	19	40	85	0.30	0.4	7.0	18.0	8.10	5.31
Average	20	18.6	31.25	17.85	21.25	20.8	44.0	63.2	0.266	0.385	7.0	18.0	87.71	89.64

	Input			Output
	H_2SO_4		HNO_3	H_2SO_4
Sp. Gr.	1.8	1.08	1.39	1.60
Wt. Grams	1129	800	148	2670
Vol. cc.	627.5	740	106.6	1669
Temp. °C	22	22	22	22

* Gay-Lussac acid turned violet color.

Run No. 13

Time Min.	T_S	T_{A_1}	T_A	T_0	T_1	T_2	T_3	T_4	P_1	P_2	V_1	V_2	W_1	W_2
0	24	21	21.0	19	19	19	21	21	0	0	0	0	0	0
15	24	21	24	19	20	21	30	23	0.3	0.65	0.5	3.0	8.09	6.63
30	24	19	24	19	21.5	21	32	23	0.3	0.6	1.0	6.0	5.50	7.00
45	23	17	26	19	21.0	21	36	24.5	0.3	0.6	1.65	9.2	5.67	7.25
60	22	17	27	18	24.5	21	42	25	0.3	0.6	2.5	12.5	5.80	7.00
75	21	17	25	17	25	23.5	42	27	0.25	0.5	3.6	14.7	4.00	5.87
90	20	16	23	16	23	22	52	35	0.35	0.4	4.6	16.9	5.28	5.50
105	20	15	24	15	26.5	25	60	63	0.40	0.5	5.8	19.1	3.00	8.66
120	19	14.5	28	14	22	22	58	69	0.35	0.6	6.7	21.4	10.00	10.28
135	19	15.	37	16	21.	21	63	67	0.35	0.5	7.9	23.7	5.25	6.75
150	19	14.5	28	16	22	21.	45	40	0.35	0.5	9.0	25.9	6.75	8.9
Average	21.1	16.5	25.5	16.9	22.65	21.8	46	39.6	0.325	0.535	9.0	25.9	59.34	73.8

	Input			Output
	H_2SO_4		HNO_3	H_2SO_4
Sp. Gr.	1.83	1.08	1.39	1.65
Wt. Grams	1360	1100	156	3300
Vol. cc.	742.5	1020	112	2000
Temp. °C	21	21	21	25

Run No. 9.

One prominent factor stood out and that was oxidation between SO_2 and NO_2. The difference in temperature between outlet and inlet cooling water was more marked than in the previous runs. The conversion of SO_2 to H_2SO_4 was 88.9%. This was either due to the wool packing or else the oxides of nitrogen were ample enough by mixing HNO_3 with H_2SO_4. Although in the end this is not desirous, since the HNO_3 acid must be removed from the final product.

Runs No. 10, 11, 12 and 13.

These were all about the same as 9. Since there is good conversion, it is now desirous to build up the concentrations of future runs. This can only be accomplished by increasing the flow of air and SO_2, or decreasing the downward flow of used acids to absorb the newly made SO_3 gas. Having received enough information on tower methods, it was thought to reproduce the same results on a larger scale or called pilot plant research.

2. Data and Calculated Results.

Page (59) contains averaged data for each separate run made in the laboratory.

Page (60) contains averaged input and output data and computed results.

a. Average Data for Each Run Made in Laboratory.

No. of Run	Time of Run in Hrs.	Temperature Degrees Centigrade								Pressure lbs. per sq. inch		Volume in Cubic Feet		Weight of Outlet Cooling Water in lbs.	
		In-let SO_2	In-let Air	Out-let Air	Inlet Cooling Water	Outlet Cooling Water		Towers							
						Glover Tower	Reaction Chamber	Glover	Gay-Lussac	Air	SO_2	Air	SO_2	Reaction Chamber	Glover Tower
1	1.75	22.0	22.0	24.5	19.0	24.5	28.0	32.9	42.8	2.06	1.87	26.0	13.00	32.57	43.61
2	3.50	24.0	24.0	26.6	18.0	22.4	22.7	29.7	32.0	1.77	1.54	33.5	5.10	57.84	73.20
3[1]	3.00	18.0	18.7	42.0	-	-	-	-	-	1.56	1.44	17.0	3.20	-	-
4	2.00	18.8	18.9	23.2	20.70	21.8	21.60	26.1	23.2	1.56	1.40	16.7	2.90	42.04	53.33
5	2.00	17.3	18.5	26.9	20.90	22.2	21.50	21.1	28.3	0.90	0.81	19.5	5.20	22.43	26.04
6[2]	3.00	18.5	19.0	22.6	22.2	22.4	22.30	21.8	21.0	0.58	0.365	28.3	4.80	42.83	67.88
7	2.50	20.4	20.2	21.3	20.2	21.55	21.00	23.95	21.8	1.627	1.481	17.0	4.00	35.12	29.02
8	2.50	18.8	18.8	19.7	20.15	22.65	20.35	20.7	20.2	0.30	0.20	22.7	2.90	52.71	31.90
9[3]	1.50	20.7	20.0	25.3	17.8	25.9	22.3	31.2	21.8	0.36	0.29	21.6	6.80	54.93	18.44
10	2.50	20.3	18.85	27.90	19.1	29.0	22.85	47.1	36.2	0.589	0.39	19.2	9.27	112.66	55.64
11	2.75	21.1	16.5	25.5	16.9	22.65	21.8	46.0	39.6	0.535	0.325	22.0	7.20	103.89	79.67
12	2.50	20.0	18.6	31.25	17.85	21.25	20.8	44.0	63.2	0.385	0.266	18.0	7.00	89.64	87.71
13	2.50	21.1	16.5	25.5	16.9	22.65	21.8	46.0	39.6	0.535	0.325	25.9	9.00	73.80	59.34

1 - This run was made without cooling the tower.

2 - Addition of Gay-Lussac tower to equal height of Glover tower.

b. Input and Output Data for H_2SO_4 and Results.

No. of Run	Time of Run in Hrs.	H_2SO_4 Employed in Recovery of Oxides of Nitrogen			H_2SO_4 (Dilute) Employed in Absorption zone for new SO_3			Average Sp.Gr. of Input Acids	Gross Product				Grams of 100% H_2SO_4			Tower Eff. in % for SO_2 to H_2SO_4
		Volume cc.	Weight Grams	Sp. Gr.	Volume cc.	Weight Grams	Sp. Gr.		Sp. Gr.	Be'	Volume cc.	Weight Grams	Input	Output		
														Actual	Theor.	
1	1.75	750.0	1268.0	1.69	950.0	1028.0	1.08	1.34	1.38	40.3	1831.0	2528.0	1010.0	240.0	1525.0	15.85
2	3.50	500.0	850.0	1.70	1000.0	1080.0	1.08	1.29	1.39	41.0	1620.0	2250.0	745.0	370.0	598.0	61.80
3	3.00	445.0	762.0	1.71	1000.0	1080.0	1.08	1.27	1.29	33.0	1468.0	1892.0	670.0	60.0	376.0	15.95
4	2.00	415.0	710.0	1.71	1000.0	1080.0	1.08	1.26	1.30	33.7	1460.0	1900.0	624.0	136.0	339.0	40.00
5	2.00	413.0	707.0	1.71	500.0	540.0	1.08	1.36	1.40	41.4	940.0	1316.0	576.0	149.0	597.0	24.90
6	3.00	1000.0	1690.0	1.69	1000.0	1080.0	1.08	1.38	1.45	45.0	2090.0	3300.0	1360.0	325.0	564.0	57.60
7	2.50	920.0	1540.0	1.69	1000.0	1080.0	1.08	1.37	1.42	42.8	1999.0	2840.0	1250.0	243.0	469.0	51.80
8	2.50	1055.0	1805.0	1.71	1000.0	1080.0	1.08	1.41	1.44	44.3	2095.0	3150.0	1483.0	184.0	339.0	54.25
9	1.50	854.0	1392.0	1.63	1087.0	1173.0	1.08	1.32	1.40	41.4	2460.0	3440.0	1850.0	709.0	799.0	88.9
10	2.50	1410.0	2298.0	1.63	1944.0	2100.0	1.08	1.31	1.42	42.8	3740.0	3740.0	1902.0	1003.0	1090.0	92.0
11	2.75	1021.0	1839.0	1.80	968.0	1045.0	1.08	1.47	1.55	51.5	2230.0	3466.0	1785.0	480.0	846.0	56.7
12	2.50	627.5	1128.0	1.80	740.0	800.0	1.08	1.41	1.60	54.2	1669.0	2670.0	1086.0	754.0	823.0	89.9
13	2.50	742.5	1360.0	1.84	1020.0	1100.0	1.08	1.40	1.65	57.2	2000.0	3300.0	1404.0	1018.0	1022.0	96.5

Input and Output Data Continued from Previous Page.

No. of Run	Ratio of Input Air in Cu. Ft. to SO_3 in Cu. Ft.		Ratio of Newly Made 100% H_2SO_4 to 100% Used in Grams		Cu. Ft. of Tower Space per lb of S per 24 hrs.	B.T.U. [redacted] in Cooling Water
	Theoretical	Actual	Theoretical	Actual		
1	3.68	2.00	1.51	0.37	0.07	2260
2	3.68	6.50	0.81	0.50	0.09	5440
3	3.68	5.30	0.56	0.10	0.45	-
4	3.68	5.75	0.54	0.22	0.15	3035
5	3.68	3.75	1.04	0.26	0.13	1325
6	3.68	6.00	0.42	0.24	0.09	671
7	3.68	4.25	0.38	0.20	0.10	2640
8	3.68	7.83	0.23	0.12	0.13	1508
9	3.68	3.18	0.42	0.38	0.02	3041
10	3.68	2.07	0.57	0.53	0.02	7030
11	3.68	3.05	0.47	0.27	0.05	7520
12	3.68	2.57	0.75	0.69	0.30	6660
13	3.68	2.88	0.73	0.735	0.023	5570

c. Heat Balance - (Based at 0°C and 760 mm. Press.).

The heat balance was calculated for the run showing best results. This happened to be run No. 13.

Summary Heat Balance of Glover Tower.

Input		
Not due to any chem. reactions.	Calories	%
Heat content of saturated air	1,032	0.2
Heat content of sulphur dioxide	51	-
Heat content of nitric acid	1,900	0.3
Heat content of sulphuric acid 60° Be'	12,580	2.0
Heat content of sulphuric acid 10.8° Be'	20,800	3.3
Total	36,363	
Heat due to reactions.		
Heat of formation of sulphuric acid	540,000	86.5
Heat of dilution 60° acid to 41° Be'	48,200	7.7
Sum Total	624,563	100.0
Output		
Heat content carried by air to Gay-Lussac tower	50,100	8.0
Heat content carried by final product	4,125	.7
Heat of centrating acid 10.8° Be' to 41° Be	5,100	.8
Heat of centrating acid 41° Be' to 57.2° Be	82,000	13.0
Heat taken out by cooling water	352,000	56.5
Heat lost by radiation	131,338	21.0
Sum Total	624,563	100.0

Summary Heat Balance of Gay-Lussac Tower.

	Calories	%
Input		
Heat content carried by air from Glover to Gay-Lussac tower	50,000	82.8
Heat content of 60° H_2SO_4	9,330	15.5
Heat content of HNO_3 acid	1,050	1.7
Total	60,380	100.0
Output		
Heat content carried into Glover tower by 60° acid.	11,130	18.4
Heat content carried by HNO_3 acid into Glover tower	1,682	2.9
Negative heat of reaction by oxides of nitrogen	9,650	16.0
Heat content of air in leaving Gay-Lussac tower	6,360	10.5
Heat lost by radiation	31,558	52.2
Total	60,380	100.0

Summary Heat Balance of Whole Tower.

Input		
Glover tower	624,563 cal.	91.4%
Gay-Lussac tower	60,380 cal.	8.6%
Total	684,943 cal.	100 %

Output

Glover tower	624,563 cal.	91.4%
Gay-Lussac tower	60,380 cal.	8.6%
Total	684,943 cal.	100.0%

d. Material Balance.

The material-balance was calculated for the same run as for heat-balance.

Summary Material Balance.

Input

Saturated air	931.0	grams.	22.0%
Sulphur dioxide	680.0	"	16.1%
Nitric acid	156.0	"	3.7%
60° Be' H_2SO_4	1360.0	"	32.2%
10.8° Be' H_2SO_4	1100.0	"	26.0%
Total	4,227.0	"	100.0%

Output

Spent Air	635.2	grams.	15.0%
Water in Gay-Lussac	136.7	"	3.2%
60° Be' H_2SO_4	1360.0	"	32.2%
10.8° Be' H_2SO_4	1100.0	"	26.0%
Grams. of SO_3	832.0	"	19.7%
HNO_3 Acid	148.9	"	3.5%
Loss of Oxides of N_2	14.2	"	0.4%
Total	4,227.0	grams.	100.0%

3. Sample Calculations for Run No. 13.

a. Results of laboratory data.

(1) Average Sp. Gr. of input acids.

742.5 cc. (Sp. Gr. 1.83) = 1365 grams.

1020 cc. (Sp. Gr. 1.08) = 1100 grams.

1762.5 " 2465 grams.

Dividing,

2465 ÷ 1762.5 = ...1.40 Sp. Gr.

(2) Input, 100% H_2SO_4 in grams.

1.83 Sp. Gr. = 94% H_2SO_4

1.08 Sp. Gr. = 12% H_2SO_4

Then,

1365 x 0.94 = 1272 grams 100% H_2SO_4.

1100 x 0.12 = 132 grams 100% H_2SO_4.

Total.......................................1404 grams.

(3) Gross output, 100% H_2SO_4 in grams.

1.65 Sp. Gr. = 73.4% H_2SO_4.

Weight of output = 3300 grams.

Multiplying,

3300 X 0.734 = ...2422 grams.

(4) Net output, 100% H_2SO_4 in grams.

Subtracting (2) from (3) or,

2422 - 1404 = ...1018 grams.

(5) Tower efficiency in % of converting SO_2 to H_2SO_4.

9 cu. ft. of SO_2 consumed.

1 cu. ft. of SO_2 weighs 0.169 lbs.

at 29" Hg.

1 lt. = 454 grams.

Therefore,

$9 \times 0.169 \times 454 = 680$ grams of SO_2.

$SO_2 + \frac{1}{2}O_2 + H_2O \rightarrow H_2SO_4$.

Mol. Wt. of $H_2SO_4 = 98$.

Mol. Wt. of $SO_2 = 64$.

Then,

$680 \times 98 \div 64 = 1022$ grams of H_2SO_4 (theoretical).

Actual conversion from (4) = 1018 grams.

Thus,

$1018 \div 1022 \times 100\% =$ 96.5%

(6) Tower spaced used/one pound sulphur/burned 24 hours.

Capacity of tower calculated as follows.

Height of Glover tower 48 inches and 2" in diam.

Height of Gay-Lussac tower 42" and 2" in diam.

$48 \times 1^2\ 3.1416 = 151$ cu. in. capacity of Glover tower.

$42 \times 1^2\ 3.1416 = 132$ cu. in. capacity of Gay-Lussac tower.

Total capacity, $151 + 132 = 287$ cu. in.

$287 \div 1728 = 0.166$ cu. ft.

680 grams of SO_2 contains $(680 \div 454)(32 \div 64) = 0.75$ lbs. of sulphur.

0.75 lbs of sulphur used in 2.5 hours.

Dividing,

0.166 ÷ 0.75 = 0.221 cu. ft. per 2.5 hours.

Then,

0.221 x 2.5 ÷ 24 = 0.023 cu. ft. or tower space required per pound of sulphur burned per 24 hours..............................0.023 cu. ft.

b. Heat-Balance, based 0° C. and 29" Hg.

GLOVER TOWER

INPUT

Before start of chemical reactions.

1. Heat content of saturated air.

Inlet temperature 16.5° C. = 61.8° F.

From accompanying chart.

Lotent heat (B.T.U./lb. H_2O) for 61.8 F. .....1057 B.T.U.

Humid heat (B.T.U./ °F/ lb. dry air) for 61.8° F. 0.243

Humidity (lbs. H_2O/lb. dry air) for 61.8° F. 0.012

Then,

1057 x 0.012 = ...12.70 B. T. U.

0.243 (61.8 - 32) = ... 7.25 B. T. U.

Total B. T. U./lb. dry air at 16.8° C........19.95 B. T. U.

1 B. T. U. = 252 cal.

1 cu. ft. Sat. air weighs at 29" Hg. 0.079 lbs.

25.9 cu. ft. of air used.

Now,

252 x 0.079 x 25.9 x 19.95 = ...10.32 cal.

(2) Heat content of sulphur dioxide.

Inlet temperature 21.1° C. = 70° F.

From accompanying chart.

B. T. U. / lb. mol. SO_2 / °F for 70° F = 9.1

Density of SO_2 , 29" Hg. lbs. per cu. ft. 0.169

9 cu. ft. of SO_2 utilized.

Then,

9.0 x 0.169 = 1.421 lbs. of SO_2.

Pound mol. of SO_2 = 64 lbs.

Or,

9.1 x 1.421 ÷ 64 = ...0.202 B. T. U.

252 x 0.202 = ... 51.0 cal.

(3) Heat content of HNO_3 (78.18%).

Inlet temperature 21° C.

From accompanying table.

Heat capacity in cal. / gram of solu.

per °C. for 78.18% HNO_3 = 0.58

Weight of HNO_3 = 156 grams.

Then,

0.58 (21-0) 156 = ... 1,900 cal.

(4) Heat content of H_2SO_4 (12%).

Input temperature 21° C.

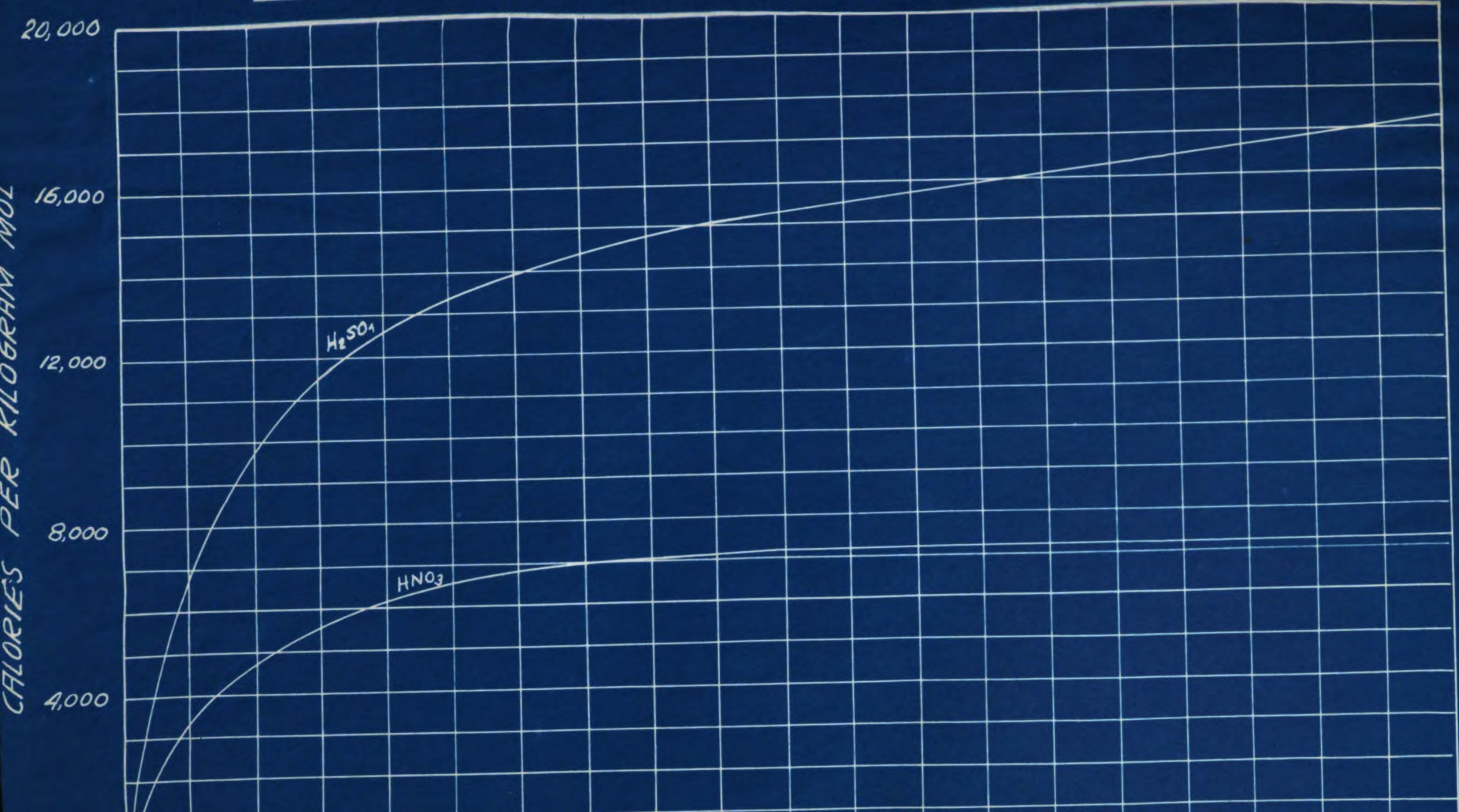
Heat capacity, cal. / gram / °C for 12% acid = 0.9

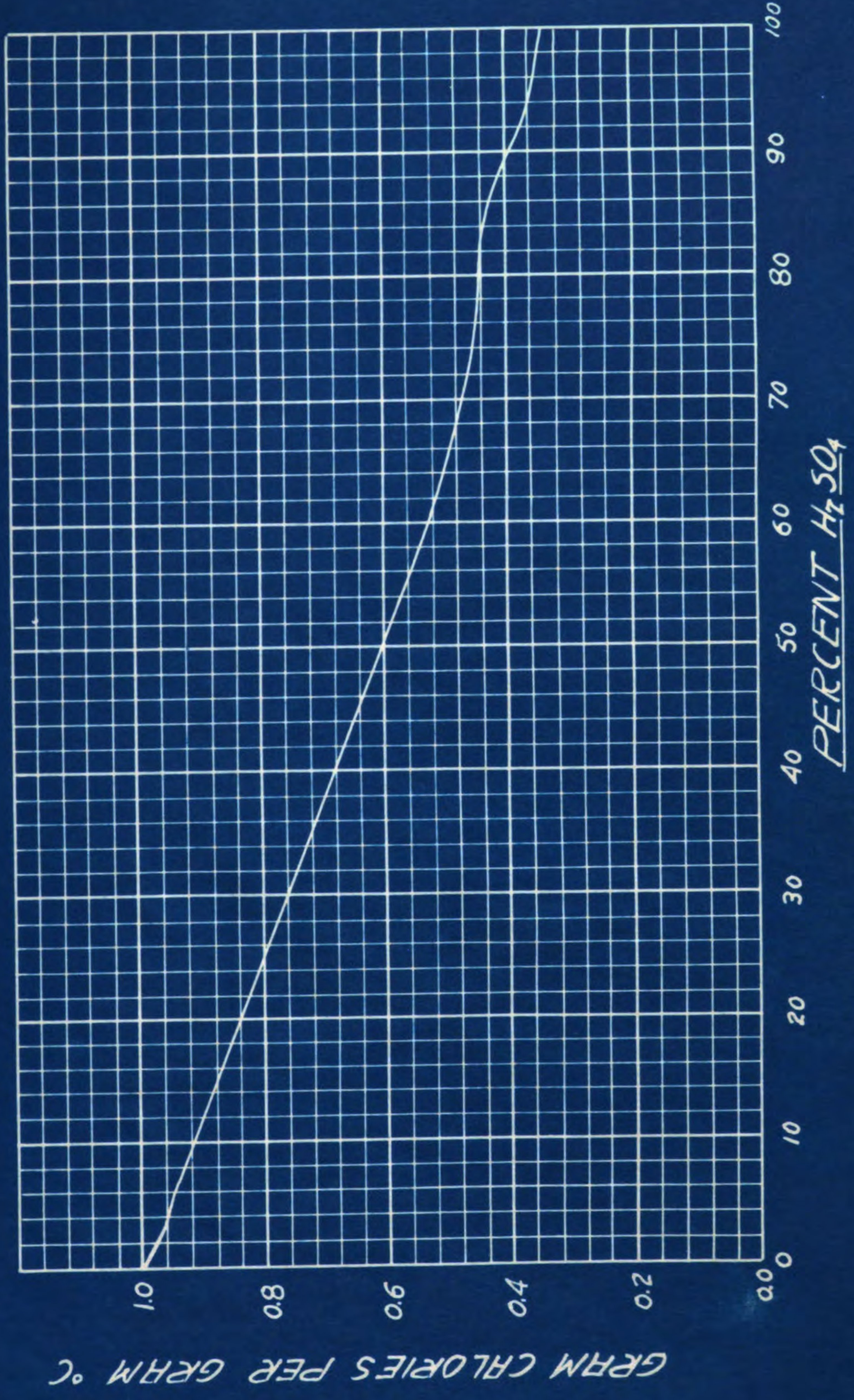
Weight of H_2SO_4 acid = 1100 grams

Then,

0.9 (21-0) 1100 = ...20,800 cal.

INTEGRAL HEATS of SOLUTION of ACIDS IN WATER AT 18°C





C = TOTAL HEAT CAPACITY, CALORIES PER GRAM PER °C c heat capacity in calories per gram of solution per °centigrade.

Percentage HNO_3	c
0	1.00
1.0	.985
2.5	.965
5.0	.935
10.0	.888
15.0	.848
20.0	.807
25.0	.770
30.0	.737
40.0	.669
45.0	.662
50.0	.655
60.0	.634
70.0	.610
80.0	.581
90.0	.553
98.0	.475
100.0	.460

(5) Heat content of H_2SO_4 (60°).

Input temperature 21° C.

Heat capacity, cal. / gram / °C. for 60° acid = 0.44

Weight of 60° acid = 1360 grams.

Then,

0.44 (21-0) 1360 = ...12,580 cal.

Total heat entered before start of run = 36,363 cal.

Heat due to Chemical Reaction.

(6) Heat of formation of H_2SO_4. H_2SO_4 is formed from SO_2 gas, liquid water and oxygen. Actually the conversion takes place in two steps with intermediate formations. However, the net effect is the same as though the following reaction preceeded --

$SO_2\ (g) + \frac{1}{2}O_2\ (g) + H_2O\ (l) \rightarrow H_2SO_4 + Q$

Where Q = Total heat of formation in calories.

$SO_2\ (g) = S + O_2 - 69{,}400$ cal.

$O_2\ (g) = 0$ (zero).

$H_2O = H_2 + \frac{1}{2}O_2 - 68{,}310$ cal.

$H_2SO_4 = H_2 + S + 2\ O_2 - 189{,}750$ cal.

Then,

$SO_2 - 69{,}400 + 0 + H_2O - 68{,}310 = H_2SO_4 - 189{,}750 + Q$

Q = 52040 cal. / gram mol. of H_2SO_4.

1018 grams of H_2SO_4 were made.

Mol. of H_2SO_4 = 98 grams.

Q X wt. of acid (100%) ÷ 98

52040 x 1018 ÷ 98 = ...540,000 cal.

(7) Heat of dilution of 60° acid.

Heat of dilution accompanying a change in concentration may be calculated by subtracting the heat of solution at the initial concentration from the final concentration.

Mols. of H_2O / mol. of H_2SO_4 in 60° acid.

60° acid = 78% H_2SO_4 & 22% H_2O.

$22 \div 18 = 1.23$ mols. of H_2O.

$78 \div 98 = 0.796$ mols. of H_2SO_4.

$1.23 \div 0.796 = 1.53$ mols. of H_2O / mol. of H_2SO_4.

From accompanying chart.

Heat of solution for 1.53 mols. of water / mol. of H_2SO_4 = 8,500 cal. per gram mol.

Weight of 60° acid = 1360 grams.

Then,

$1360 \times 8{,}500 \div 98 = 91{,}800$ cal. heat of initial concentration.

1.4 Sp. Gr. of final concentration = 51. % H_2SO_4 and 49% H_2O.

$49 \div 18 = 2.22$ mols. of H_2O.

$51 \div 98 = 0.52$ mols. of H_2SO_4.

$2.22 \div 0.52 = 4.28$ mols. of H_2O per mol. H_2SO_4.

From accompanying chart.

Heat of solution for 4.28 mols. of H_2O per mol. H_2SO_4 = 13,000 calories per gram mol.

Final heat of solution =

1060.8 x 13,000 ÷ 98 = ...140,000 cal.

Subtracting, or total heat solution,

140,000 - 91,800 = ...48,200 cal.

Sum total of input heat in Glover tower 624,563.cal.

OUTPUT

(8) Heat content carried out by spent air to Gay-Lussac tower

at 46° C. or 114.8° F.

Latent heat (B.T.U. / lb. H_2O) for 114.8° F. = 1028 B.T.U.

Humidity (lbs. H O / lb. dry air) for 114.8° F. = 0.065.

Then,

1028 x 0.065 = ...61.8 B.T.U.

Humid heat (B.T.U. / F. / lb. dry air), 0.35 for 114.8° F.

Or,

0.35 (114.8 - 32) = ...29.0 B.T.U.

Total B.T.U. / lb. air ....96.8 B.T.U.

Weight of outlet air, 2.05 lbs.

96.8 x 2.05 = 198.8 B.T.U.

198.8 x 252 = ...50,100 cal.

(9) Heat content carried down by final product.

Weight of final product = 3300 grams.

1.65 Sp. Gr. of final product.

Temperature 25° C. of final product.

Heat capacity cal. / gram / °C. for 1.65 Sp. Gr. acid

........0.5

Then,

0.5 (25 - 0) 3300 = ...4,125 cal.

(10) Heat of centrating acid (12% or 10.8° Be' to 41° Be').

Initial concentration = 10.8° Be'.

10.8° Be' acid = 12% H_2SO_4 & 88% H_2O.

88 ÷ 18 = 4.88 mols. of H_2O.

12 ÷ 98 = 0.122 mols. of H_2SO_4.

4.88 ÷ 0.122 = 40 mols. of H_2O / mol H_2SO_4 / mol. gram H_2SO_4

From accompanying chart.

Heat of solution for 12% acid = 16,800 cal. per gram mol.

Then,

0.12 x 1100 x 16,800 ÷ 98 = 22,600 cal.

Final concentration = 41° Be'

From (page 71) 41° Be' acid contains 4.28 mols. of water per mol. H_2SO_4 and heat of solution = 13,000 cal. / gram mol. 1100 grams of 12% were used.

Then,

1100 x 0.12 x 13,000 ÷ 98 = ...17,500 cal.

Subtracting,

22,600 - 17,500 = ... 5,100 cal.

(11) Heat of concentration 1.4 Sp. Gr. acid which is average Sp. Gr. for input acid and was built up to 1.65 Sp. Gr. by the newly made 100% H_2SO_4.

Weight of input acid = 2460 grams.

Weight of output acid = 3300 grams.

Initial heat of solution for 1.4 Sp. Gr. acid =

13,000 cal. / gram mol.

Then,

13,000 x 2460 x .51 ÷ 98 = ... 166,500 cal.

Final heat of solution for 1.65 Sp. Gr. acid.

1.65 Sp. Gr. = 73.5% H_2SO_4 & 26.5% H_2O.

26.5 ÷ 18 = 1.47 mols. H_2O.

73.5 ÷ 98 = 0.75 mols. H_2SO_4.

1.47 ÷ 0.75 = 1.96 mols. of H_2O. per mol. of acid.

Heat of solution from chart for 1.96 mols. of H_2O / mol. of H_2SO_4 = 10,000 cal. / gram mol.

Then,

3300 x 0.735 x 10,000 ÷ 98 = 248,500 cal.

Subtracting,

248,500 - 166,500 cal. = ... 82,000 cal.

(12) Heat removed by cooling water.

Wt. of cooling water from oxidation zone = 59.34 lbs., temperature 73° F.

Weight of cooling water from reaction chamber = 73.8 lbs., temperature 71.2° F.

Inlet temperature of water = 61.80 F.

Then,

B.T.U. from Glover tower.

(73.0 - 61.8) 59.34 = ... 644

B.T.U. from reaction chamber.

(71.2 - 61.8) 73.8 = ... 694

Total B.T.U......................... 1338

Total calories = 1,338 x 252 = ...352,000 cal.

(13) Heat lost by radiation ....131,338 cal.

Total heat output from Glover tower ......524,563 cal.

GAY-LUSSAC TOWER

INPUT

(14) Heat content in air from Glover tower, same as output to Gay-Lussac (page 73). .... 51,000 cal.

(15) Heat content of 60° acid.

Heat capacity, cal. / gram / °C. for 78% acid = 0.44

Temperature of acid = 21° C.

Weight of acid = 1360 grams.

Then,

0.44 (21 - 0) 1360 = ... 9,330 cal.

(16) Heat content of HNO_3 acid.

Heat capacity in cal. / gram of solution / °C. for

78.18% = 0.58

Temperature of HNO_3 = 21° C.

Weight of HNO_3 = 156 grams.

Then,

0.58 (21 - 0) 156 = ... 1,050 cal.

Total input of heat for Gay-Lussac tower .. 60,380 cal.

OUTPUT

(17) Heat content carried down by 60° acid into Glover tower.

Weight of acid = 1360 grams.

Input temperature 21° C.

Temperature leaving tower 39.6° C.

Heat capacity $= 0.44$

Then,

$0.44\ (39.6 - 21)\ 1360 =$ .... 11,130 cal.

(18) Heat content carried by HNO_3 (78.18%) acid into Glover tower.

Heat capacity $= 0.58$

Weight of acid $= 156$ grams.

Temperature out $= 39.6°$ C.

Temperature out $= 21.0°$ C.

Then,

$0.58\ (39.6 - 21)\ 156 =$ .... 1,682 cal.

(19) Heat absorbed by $NO \rightarrow NO_2$

$NO_2 + SO_2 \rightarrow SO_3 + NO$ - heat

$SO_3 + H_2O \longrightarrow H_2SO_4$

1018 grams of H_2SO_4 were made.

$80 \times 1018 \div 98 = 830$ grams of SO_3.

To make 830 grams of SO_3 it required x grams of NO_2 .

Or,

$x = 830 \times 46 \div 80 = 477$ grams of NO_2.

Heat of formation of $NO_2 = -930$

Now,

$930 \times 477 \div 46 =$ .... 9,650 cal.

(20) Heat taken out by spent air.

205 lbs. dry air used.

Temperature 25.5° C. $=$ 77.9° F.

Humid heat (B.T.U. / °F. / lb. dry air) $= 0.268$

Then,

0.268 (77.9 - 32) 2.05 = 25.25 B.T.U.

25.25 x 252 = ...6,360 cal.

(21) Lost heat due to radiation = ...31,558 cal.

Total output heat from Gay-Lussac tower........60,380 cal.

c. Material - Balance.

INPUT

(1) Saturated air.

One cu. fut. at 29" Hg weighs 0.079 lbs.

25.9 cu. ft. of air consumed.

Then,

25.9 x 0.079 = 2.05 lbs. of air.

2.05 x 454 = ... 931.0 grams.

(2) Sulphur dioxide.

One cu. ft. of SO_2 at 29" Hg. weighs 0.169 lbs.

9 cu. ft. of SO_2 consumed.

Or,

9 x 0.169 = 1.521 lbs. of SO_2.

1.521 x 454 = ... 680.0 grams.

(3) Weight of HNO_3 = ... 156.0 grams.

(4) Weight of 60° H_2SO_4 =.......................... 1,360.0 grams.

(5) Weight of 12% H_2SO_4 =.......................... 1,100.0 grams.

Total input.................................. 4,227.0 grams.

OUTPUT

(6) Spent air.

Inlet air - water content.

Pounds of H_2O / lb. saturated air for 16.5° C. = 0.012

25.9 cu. ft. of air consumed.

Then,

25.9 x 0.079 x .012 x 454 = 136.7 grams of H_2O

Weight of inlet air = 931 grams.

Subtracting,

931 - 136.7 = 794.3 grams.

Spend air - oxygen content used.

1018 grams of H_2SO_4 made.

Mol. weight of $\frac{1}{2}$ O_2 = 16

Or,

1018 x 16 ÷ 98 = 166.4 grams of oxygen consumed.

Oxygen 20% by volume in air..

794.0 x 0.2 = 158 grams of oxygen used in converting SO_2 to SO_3 from air.

166.4 grams were actually used.

So,

166.4 - 158 = 8.4 grams of oxygen was deficient.

Finally,

794.0 - 158 = or air spend .... 635.0 grams.

(7)	Water held back by Gay-Lussac acid	136.7 grams.
(8)	Weight of 60° acid leaving tower	1360.0 grams.
(9)	Weight of 12% acid leaving tower	1100.0 grams.
(10)	Weight of acid made minus water or remainder is SO_3, 1018 x 80 ÷ 98 =	832.0 grams.
(11)	Weight of HNO_3 acid recovered	141.8 grams.
(12)	Loss of HNO_3 as NO_2	14.8 grams.
	Total output	4,227.0 grams.

C. CONCLUSIONS.

From observation of data and results many factors are open for discussion. With little experimental work on sulphuric acid thus far it appears that there is a lot to consider about sulphuric acid. Reactions taking place in the tower method are affected by outside factors such as temperature, pressure, and velocities of reaction which are influenced mainly by type of packing and finally tower space.

Possible reactions taking place may be divided into three groups as follows -- those which take place in a homogeneous gas phase.

1. $2\ NO + O_2 \rightarrow 2\ NO_2$ - heat
2. $2\ NO + \frac{1}{2}\ O_2 \rightarrow N_2O_3$ - heat
3. $N_2O_3 + \frac{1}{2}\ O_2 \rightarrow N_2O_4$ - heat

It is said reaction No. 2 takes place with great speed while No. 3 completes No. 2 and comparatively slowly. High temperature will affect No. 2 in breaking up N_2O_3 to NO and NO_2. There is a second series taking place in a heterogeneous gas-liquid interface phase and repre-

sented by the following equations --

1. $SO_2 + H_2O \longrightarrow H_2SO_3$

2. $H_2SO_3 + NO_2 \longrightarrow (H_2SO_4)\ NO$, known as violet acid.

3. $2(H_2SO_4)\ NO + \frac{1}{2}\ O_2(NO_2) \longrightarrow 2\ SO_5NH + H_2O(NO)$

4. $2\ SO_2NH + SO_2 + 2H_2O \longrightarrow 2(H_2SO_4)\ NO + H_2SO_4$

Reaction No. 1 takes place very readily, but is unstable, yet with ample supply of NO_2 it will produce as shown under No. 2. This reaction can be detected by its violet color and was observed in both towers. Violet acid can be produced with concentrated H_2SO_4 and (NO) under pressure. The latter may have taken place in the tower, because with a sufficient supply of air the violet color disappears. In either case, heat is evolved. Insofar as it does take place in the Glover tower, it is desirous since in the end H_2SO_4 will be made; but, not in the Gay-Lussac tower where the temperatures should be low. In the Gay-Lussac tower the NO and NO_2 finally are recovered by the H_2SO_4 to form nitrosyl sulphuric acid which is the customary way of bringing back the oxides of nitrogen into the Glover tower. As mentioned before, high temperatures somewhat affect this reaction. Other reactions which occur in the liquid phase may be listed as follows --

1. $(H_2SO_4)\ NO \longrightarrow H_2SO_4 + NO$

2. $H_2O + 2\ SO_5NH \longrightarrow 2H_2SO_4 + NO + NO_2$

3. $SO_5NH + HNO_3 \longrightarrow H_2SO_4 + 2NO_2$

These reactions were evidenced in the Glover tower and the reaction chamber. By closely observing the liquid trickling down the packing it seemed clear and colorless when all at once in the oxidation zone NO_2 gas was noticed by its reddish-brown color. At this instant reaction No. 2 took place due to the H_2O contained in the dilute acid which met the down coming nitrosyl sulphuric acid from the Gay-Lussac tower. The same took place in the reaction chamber where there must have been some HNO_3 and reaction No. 3 resulted. The HNO_3 came from two sources, from intermediate reactions from above and from the nitrogen dioxide generator which distilled some of it over.

Thus, by harnessing the work with the gas-liquid and liquid phase, it may be possible to eliminate the chambers the points to develope in this problem.

In conclusion, some runs were promising while others were not. Better results were obtained with the glass wool packing rather than with the glass beads. In study with a similar construction of tower in pilot plant, the glass wool packing will be employed.

PART THREE

STUDY WITH AN ACID-PROOF TILE TOWER FOR PILOT PLANT RESEARCH

A. PILOT PLANT OPERATIONS.

1. Discussion:

After considerable information had been obtained on preparation and study of sulphuric acid on an experimental scale in laboratory, it was desirous to duplicate the same in the pilot plant. (Pilot plant research is nothing more than reproducing the same as of experimental, only on a considerable larger scale.)

A new complete tower had been rebuilt out of the one in pilot plant where others had devoted time and study in search of similar results. The rebuilt tower, and from now on, will be referred to as the "tile tower" mainly to its construction of glazed acid-resisting tile. The tile tower was designed to differ from the old tower in some respects because of information gotten from experimental study in laboratory. Still, this tile tower is quite analogous in principle to both the old tower and the glass tower.

When the glass tower was built and set up, the top half which heretofore had been referred to as the Gay-Lussac tower, was not provided with cooling facilities. The object was that the chemical reactions taking place in that region were of negative heat of reaction and cooling was not necessary. (By negative heat of reaction it is meant - heat is taken on due to chemical reaction. Heat will be absorbed from close-by surroundings.) The lower half or the Glover tower had to be water cooled due to the chemical tower will be water cooled from source of inlet gases at the bottom to the outlet waste air at the top.

If the Gay-Lussac tower were also cooled, the heat that would ordinarily remain in it would break up the nitrosyl sulphuric acid into oxides of nitrogen and sulphuric acid, or merely reverse the reaction which is not desirous. Due to such decomposition, a loss of nitrogen dioxide and nitric oxide would result and consequently a drop in efficiency. Since the oxides of nitrogen are the key to the entire process, they need to be considered and delt with. In conclusion, the proposed plan can only be tested out for further comments to be made, if any.

B. CONSTRUCTION OF ACID-PROOF TILE TOWER.

The old tower was torn down to the cement base (FF). (Refer to Blue Print No. 2). A lead cup (N) six inches in diameter and eight inches deep was directly inserted, or imbedded in the center of the concrete base (FF). This foundation is thirty inches square and thirty-six inches high from the ground floor. A half-inch drain in the bottom of the lead cup was made of lead pipe to allow the newly made and spent acids to drain through it. An acid-resistive sprigot (C) was attached with acid-proof cement at the outer end of the lead drain pipe which has its inner end burned to the bottom of the lead cup (N). Thus a permanent connection was sound against gas or acid leaks.

A lead grate was burned upon the open end of the lead cup (N) reactions being of positive heat of reaction giving off heat. In drawing up the conclusions from experimental results, it was clearly seen that the Gay-Lussac tower was much more hotter than the Glover tower. The only source of heat that could have possibly entered into the Gay-Lussac tower was primarily due to the air carrying the heat up from the lower half of the glass tower. It is believed with such a tower method in the manufacture of sulphuric acid that the entire tower should be equiped with means of cooling. From the study of the chamber process, the most widely one used in commercial work, the gasses leaving the Glover tower are very hot and by passing through the lead chambers practically all of the heat is removed before entering the Gay-Lussac tower. (Refer to history of Chamber Process on manufacture of H_2SO_4). The heat is removed from the hot gases by being dissipated in the lead walls of the chamber. Since in the tower method, the chambers are completely left out. Then it stands to reason that time involved for the hot gases to leave the Glover tower and enter the Gay-Lussac tower is much more shorter than the hot gases leaving the Glover tower enroute through the chambers and into the Gay-Lussac tower of the "Chamber Process." At first upon the construction of the glass tower, it was thought the heat of reaction would be completely removed by cooling the Glover tower. Theoretically it should have, but actual it did not. For this reason the entire whose walls are one-inch thick, namely, to support the glass wool packing and finally to permit acids to trickle down from the above tower (3) into the lead cup (N). From here they were drawn off as described above.

a. Glover tower (3).

After the lead grate had been sealed tightly upon the lead cup so that gases couldn't escape through its burned joint, a six inch glazed acid-proof tile (MM) was set up right upon the lead grate (LL). These acid-proof tiles which will constitute the tower are two feet in length. The bottom of the first tile was cemented to the lead cup (N) with acid-proof cement which took about two days to dry out thoroughly. Thus an air tight joint was made. This sealed connection made up the bottom of the Glover tower (3). Care in setting the tiles was necessary, in which they were vertically lined with a plumb line, so that the concurrent flow of acids could wet the inside circular wall of the tower and the packing.

The second glazed acid-proof tile (MM) (numbering from the bottom up) was placed upon the top of the first, and sealed securely to insure leakage of gases and acids. A problem resulted in trying to use suitable materials with which to seal the joints between the tile. At first the whole bell of the tile was packed with acid-proof cement. Sodium silicate mixed with powder acid-proof cement to a plastic state was applied into the bell joints. The sodium silicate being in nature of a viscous liquid hardens when exposed to the atmosphere. Thus, trouble had been encountered whereby the plastic cement only dried on the outside surface or the one exposed to the air. This being the situation, the cement beneath the dried crust was as soft as when it was packed into the bell joints. In conclusion, such a connection with an unstable center was not desirable since it did not offer positive assurance against leaks and finally a very poor binder for the safety of keeping the tiles from tipping over. The tiles must be permanently set and vertically lined.

After a little time had been spent on study of applicable materials for joint purposes, it was thought that a small layer of acid-proof cement would do, just small enough to dry completely through its thickness. Still this idea left some doubt. Next plaster of paris had been mentioned by Mr. Reed, namely it being a salt ($CaSO_4$) of sulphuric acid would not be attached by the acid itself. Plaster of paris was mixed with a little water to the form of a mud and introduced as such into the bells of the tiles. So with a small layer of acid-proof cement and a considerably larger layer of plaster of paris, the uncertainty had been relieved. If the first layer should permit the sulphuric acid or gases to escape, then the second would prevent further leakage. Plaster of paris fulfilled one phase as an acid resistant and also as an air-tight connection since it expands upon solidification due to water of hydration.

Up to this point the joints were sound in respect from the inside to the outside of the inner tower. Since this tower was to be water-jacketed (4) for cooling purposes, the water would slightly dissolve the outer layer which is exposed to it. To remedy this cause a very small coat of asphalt was painted over the plaster of paris to ward off the water. Insofar as the bell joint was considered now, it was quite air-tight in regard to water entering in, or gases and acids escaping out. With this point in view it is not strong enough to hold the tiles from tilting over, because the compressibility of larger layer is too high. By packing in a layer of concrete above, or on top of the asphalt coat and again painting another coat of asphalt on it, the joint was sound in every respect and fulfilled all requirements.

Three tiles were worked up in this manner and constituted what has heretofore been referred to as the Glover tower (3). The inside of this tower was packed with fine glass wool (F) in such a mode that the least amount of resistance would be offered when the acids trickled down, or gases traveled upward, and lastly, producted sufficient surfacing area.

Having progressed to this degree, the set-up contained the base (H) and the Glover tower (3). Before adding the top half known as the Gay-Lussac tower (2), two sections of sewer tile (MN) twelve inches in diameter, and two feet long were set around the Glover tower and made up the bottom of the water-jacket(4). The first sewer tile was set in the base (N). Both it and the first tile of the Glover tower were imbedded four inches deep into the concrete base (Refer to Blue Print No. 2). In this position they were securely held and both columns being firmly fortified by the base, thus insuring the danger of tipping over. The second sewer pipe was placed in its location on the first tile (Again numbering from the bottom up). The sealing of these bell joints was an easy task in comparison with those of the inner tower. Rope oakum, asphalt, and a considerably larger layer of concrete, respectively affirmed the connection from water seepage or tilting of sewer tiles as the water-jacket was carried upward in its construction.

Having set up the two sewer tiles of the water-jacket, the top of the Glover tower was completed. A perforated lead pan (E) six inches in diameter lies on top of the glass wool packing (F). It serves the object of despensing equally the dilute acid over the packing. The dilue acid (J) makes its way into the perforated distributor (E) through the dilute acid line (K). Two to three inches above the rim of the perforated pan a one-inch lead plate (D) and nine inches in diameter rests in the shoulder of the bell of the third tile of the Glover tower (3). Both the plate and distributor were connected by the Gay-Lussac acid line (M) which extended flush from the top of lead plate (D) down four feet into (G) of the Glover tower (3), with the packing (F) centered around it. The entire unit was supported by the bell shoulder. This same lead disc was air-tight sealed with acid-proof cement. Its purpose served as; first, the bottom of the Gay-Lussac tower (2); secondly, the top of the Glover tower (3); and lastly, collected the Gay-Lussac acid (A) which in turn drained into the mentioned small pipe line (M) and in the end emptied into the oxidation zone (G) of the Glover tower (3). In conclusion a vent line (I), half an inch in diameter and three inches above the surface of the lead plate (D) was burned in so that only the spent air could pass through it from the Glover tower (3) to enter into the Gay-Lussac tower (2) without any acid draining in over it, because it is desirous to have the acid collect on the lead plate (D) and drain down its proper line (M). Thus the two towers were securely connected.

b. Gay-Lussac tower (2).

Next in order the dilute acid line (K) made of half-inch lead pipe was installed. It extended downward from the top of Gay-Lussac tower (2) to about two inches below the plate (D) in the bottom of the Gay-Lussac tower. Note, that it is a U-tube affair (F) whose design was necessary against upward pressures in order to allow the dilute acid (J) to fill the perforated pan (E) on top of the packing (F) of the Glover tower (3). (Refer to Blue Print No. 2). Consequently a free flowing stream of dilute acid was constantly maintained.

Continuing in further construction, the fourth acid-proof tile (OO) was placed in its designated position on top of the lead plate (D) and it was sealed in the bell shoulder of the third tile up. Similarily, fifth and sixth tiles were set in their respective places, and correspondingly the joints were filled or packed with various layers heretofore described in the construction of the Glover tower (3). More glass wool (C) was used to pack these three tiles which formed the Gay-Lussac tower (2). Same care was exercised for uniform packing as in the lower half (3) of the set-up for similar reasons.

Another lead perforated pan (B) serving the purpose as a distributor rested on top of the latter packing (C) and was also held by the shoulder of bell of the sixth tile. $\underline{6}0^{\circ}$ acid (A) runs into this pan (B) which evenly sprayed it over the packing (C). Consequently, the acid trickled down through the packing (C) of the Gay-Lussac tower (2). The tip of the dilute acid line (K) and air vent (EE) passed through this cup (B) and both were held in place by being burned in place with an acetylene torch. The air vent permitted the air to leave the set-up. This completed all the work on the inner tower. The remaining three sections of the water-jacket (4) were inserted and cemented together. An entry (BB) for inlet water was put in the bottom while a similar one (CC) at the very top carried out the spent cooling water. This concluded all work in construction of the entire tower.

C. Minor Attachments.

(1) Dilute acid reservoir (J)

A fifteen liter soft glass bottle fulfilled the need as a container for dilute acid to flow into the dilute acid line (K) at a uniform rate while the pilot plant run was in operation. It was graduated into liters whereby the difference of levels of acid before and after gave the amount used in each test. A siphon made of quarter-inch glass tubing conveyed the dilute acid into its respective line (K). It is held firmly in the mouth of the bottle (J) with a suitable rubber stopper. Also, a four centimeter funnel was inserted through the stopper for adding more weak acid into the reservior (J) when it became empty. A small size peep glass tube was forced through the same stopper to allow the air to escape when more acid was poured into the container.

(2) Reservoir for 60° Be' acid (A)

This container is identically the same as the one (J), only, its 60° acid flows into the perforated lead pan (B) lying on the top of the Gay-Lussac tower (2). The acid is thus showered over the packing (C). In the end it was received in the bottom (D) of the Gay-Lussac tower from where it emptied into a half-inch lead pipe (M) which is four feet long. In this manner the Gay-Lussac acid containing the absorbed oxides of nitrogen returned them back into the oxidation zone (G) which made up the bottom half of the Glover tower (3). The top half was called the absorption zone which was showered with dilute acid. Both siphons are equiped with valves for regulation of flow of respective acid.

(3) Sulphur dioxide meter (X)

An ordinary house gas meter was loaned to the chemical engineering department by the Consumers' Power Company of Lansing. It was borrowed for measuring the volume of SO_2 in order to be able to obtain accurate data for calculation of results. SO_2 was emitted from its cylinder in which it was contained in the liquid state and expanded upon entrance into the atmosphere as a gas. By passing it through a manometer (W), then into the gas meter (X) and finally into the tower (3) through the entrance (7) at the bottom; this method supplied the constant flow of SO_2 during working conditions. The manometer (W) acts as a safety device over which a limited pressure on the gas meter (X) will cause inaccurate registering of cu. ft. of gas. It also fulfilled the task of indicating packing resistance to inflow of gases. A thermometer (Y) was inserted between the gas meter (X) and tower (3) to obtain the inlet temperature for computing the heat balance.

(4) Air meter (T)

Another larger common gas meter was borrowed from the Consumers' Power Company of Lansing for recording the volume of air passed per unit interval. It too was equiped with a manometer (S) for safety and a thermometer (V) to obtain inlet temperature of air. In this case a meter was necessary to control the ratio of air to SO_2 which should actually be 4 - 1 by volume at atmospheric conditions of pressure. The recorded volume of air offered information in regard to calculating the heat and material balances. Quarter-inch rubber tubing was used for bringing the SO_2 and air into the tower from their respective sources.

(5) Nitrogen dioxide generator (5)

Some convenient means of introducing the nitrogen dioxide had to be made in such a way that a constant introduction could be maintained. A six liter erlenmeyer flask (5) was employed and properly installed (refer to Blue Print No. 2). An air line passed into this flask through its open top. In like manner the funnel (Q) was inserted for admitting nitric and sulphuric acids into the generator (5). Lastly, a fume line (V) connected the mouth of the flask (5) with the entrance (7) of the tower (3). Acid-proof cement was packed into the opening mouth of the generator (5) to form a sealed cap to prevent escape of nitrous fumes except into the tower(3). A bunsen burner (R) supplied the heat to the mixed acid to drive off the oxides of nitrogen. After the exhaustion of oxides of nitrogen, the spent acid consisting practically of sulphuric acid and water was drawn out by a vacuum through the same line which admitted the air into the set-up while in operation. Then by refilling with a new mixture of acids, the generator was ready for use again.

Another identical erlenmeyer flask (5) fulfilled the requirement in driving off the oxides of nitrogen which came down with the newly made acid. So by draining the gross acid and pouring them into the container through the funnel (Z), the oxides of nitrogen were expelled by heat of a bunsen burner. This unit is located to the right of the tower entrance (7).

The tower entrance (7) is of importance to consider. A way of introducing SO_2, air, and nitrous fumes was necessary insofar as the operation was concerned. An air-tight entrance had to be made to admit the gases named and yet prevent passage of them other than into the tower (3). For this point in mind a 600 cc. beaker (7) was used. The flue lines (V) and (AA) of the 6 liter flasks pass over the tip of the beaker (7) and protrude about one inch below the acid-proof cement cap which closes the top of the 600 cc. beaker (7). Through each of these flue lines the air comes in and brings along the oxides of nitrogen from the generator (5) and the newly made acid. Then one end of a larger glass tube also protrudes one inch below the surface of cap on the beaker (7) and the other end leads into the tower. The latter end is sealed into the lead cup (N) in center at the concrete base (FF). Thus there is a passage from the beaker (7) into the tower (3). A quarter-inch glass tube was inserted through the same cement cap for admittance of sulphur dioxide. A similar size glass tube was placed through the cap so that it reached the bottom of beaker (7) where any condensate could be drawn off by a vacuum.

In conclusion of constructing the tower and its minor units, such a set-up was used in the study of preparation of sulphuric acid by a tower method in pilot plant.

B. HOW EACH RUN WAS MADE.

The pilot plant sulphuric acid tower is about 15 feet high. (Refer to Blue Print No. 2 which is enclosed in the pocket of the back cover of this thesis. It is advised to take out the said plan and refer to it in how each run was made.)

60° acid was held in a graduate reservoir (A) from where it was siphoned over into the perforated lead pan (B). This lead pan distributes the 60° acid over the packing (C) contained in the Gay-Lussac tower (2). Finally the acid trickling down the packing absorbs the (NO_2) passing up from (G) through (H) and (I). The following reaction results as absorption of NO_2 by the 60° acid takes place --

$$1.\quad 2NO_2 + H_2SO_4 \longrightarrow SO_5NH + HNO_3$$

Nitrosyl sulphuric acid (SO_5NH), nitric acid (HNO_3) and the 60° acid (H_2SO_4) are collected in (D). From (D) the mixed acids drained into the Gay-Lussac acid line (M) of the Glover tower (3) down to (G). At (G) the mixed acid was diluted by the down-coming dilute acid from its reservoir (J) siphoned over into the dilute acid (K) which carried it over the trap (L). From here it was brought into the perforated lead pan (E), distributing it over the packing (F) of the tower (3) until both the dilute acid and 60° acid from (M) met at (G). At (G) the following reaction took place --

$$2.\quad 2SO_5NH + H_2O \longrightarrow NO_2 + NO + 2H_2SO_4$$

Nitrogen dioxide (NO_2) and nitric oxide (NO) remained at (G) while the diluted sulphuric acid (H_2SO_4) trickled into lead cup (N). From (N) it is drawn out through acid-proof sprigot (O) into a large bottle (P).

The original source of NO_2 was generated in the six liter erlenmeyer flask (5). 1500 cc. of nitric acid (1.39 Sp. Gr.) were poured through the funnel (Q) into (5). The same was followed by 1000 cc. of concentrated sulphuric acid (1.84 Sp. Gr.). Then the stop-cock of funnel (Q) was closed. By heating (5) with the bunsen burner (R) NO_2 was liberated from the solution of the mixed acids. The air was passed into the manometer (5), the air meter (T), the temperature recording unit (U) and into the generator (5). The air passing up through the heated mixed acids thus carried along the liberated NO_2 over the flue line (V) into the tower entrance (7) from where both passed up into the tower (3) through the packing. Heating of the container (5) and flow of air were continued until sufficient amount of nitrogen dioxides were generated.

When the above was completed, SO_2 was introduced from its source through the manometer (W), SO_2 meter (X), the temperature recording unit (Y), and the tower entrance (7) from where it passed into the tower (3). At (6) the SO_2 and NO_2 reacted to form SO_3 as follows --

3. $SO_2 + NO_2 \longrightarrow SO_3 + NO$

Sulphur trioxide (SO_3) is absorbed above the point (G) by the down trickling dilute acid from (E) and the following reaction resulted --

4. $SO_3 + H_2O \longrightarrow H_2SO_4 + \text{heat}$

In this manner the dilute acid was concentrated by taking on SO_3. Now, the built up acid trickled down into the receiving lead cup (N) from where it was drawn off through the sprigot (O) into (P). Some oxides of nitrogen were carried along into (P). Thus the gross output was emptied into (5) through the funnel (Z). Applying heat to (5) with a bunsen burner the oxides were liberated. The air passed into (5) and bubbled up through the gross acid in such a manner that it carried along the oxides of nitrogen over the flue line (AA) into the tower entrance (7) and finally in the tower (3) at (G) where more SO_2 met it to form SO_3.

Now going back to reaction 3, the NO is carried up into tower (2) by the air through (H) and (I). On its way up the air oxidized the NO to NO_2 --

5. $NO + \frac{1}{2}O_2 \longrightarrow NO_2$ - heat.

Again the NO_2 is absorbed by the trickling acid in (C) of (2) and reaction No. 1 was reproduced. The air deprived of its oxygen, to NO, passed up through (EE) and made its escape into the atmosphere. Thus the cycle of process of gases, air and counter-flow of acids is outlined and explained.

Points of interest were watched and observed toward the function of the tower method for making sulphuric acid.

Data was recovered every fifteen minutes for 2 to 3 hours. Since the tower was fifteen feet high and in order to record the outlet temperature of air and water, a ladder was used to climb up.

The following data was taken on tabulated data paper -- the temperatures of inlet air at (U), of inlet SO_2 at (Y), of inlet water at (BB); and outlet of air at (EE) and outlet water at (CC). The difference of levels of reservoirs (A) and (J) gave the amount of input acids in liters. Volumes of air at (T) and of SO_2 at (X) were written down along with corresponding pressures at (S) and (W). Some means of accounting for the weight of water had to be solved.

This was done by passing the outlet water of water-jacket (4) at the top at (CC). From (CC) it was led into a tank 48 inches in diameter and 3 feet high. So by measuring the difference of level of the water for each interval gave the volume of water passed. From its volume the weight was calculated.

With the use of meters the ratio of air to SO_2 was controlled. The ratio being actually 4 - 1. Difference of inlet and outlet temperatures were watched. An increase of such showed reactions of SO_2 to H_2SO_4 were taking place.

Such care and procedure was exercised in making all runs. Results were calculated which pertained to the possibility of developing a tower method for commercial purposes.

1. Laboratory runs of Pilot Plant.

The following pages contain data taken on each run. Curves were also plotted to study each run. After this, discussion of each trial is made.

Run No. 1

Pilot Plant Data Taken During Operation of Tile Tower

Time in Min.	Temperature °Centigrade					Inch. of Cooling Water	Liters of Input Acids		Vol. in Cu. Ft.		Press. lbs. per Sq. In.		Gross Output Product		
	Inlet SO_2	Inlet Air	Outlet Air	Cooling H_2O			Dilute	60° Be	SO_2	Air	So_2	Air	Temp. °C	Sp.Gr.	Vol. in cc.
				Inlet	Outlet										
0	-	-	-	16	16	0.5	0	0	-	-	-	-	-	-	-
15	-	-	-	16	16	3.5	0.3	0.3	-	-	-	-	-	-	-
30	-	-	-	16	16	6.5	0.6	0.7	-	-	-	-	-	-	-
45	-	-	-	16	18	9.5	0.9	1.4	-	-	-	-	-	-	-
60	18.0	18	26	16	17	12.5	1.2	1.5	-	-	-	-	-	-	-
75	17.5	11.5	28	16	17.5	15.25	1.6	2.1	7.0	19.5	0.75	5.4	31	1.54	825.0
90	17.0	11.0	30	16	17.5	18.25	1.9	2.7	11.5	37.7	0.80	6.2	35	1.53	565.0
105	17.0	11.0	35	16	18.2	21.00	2.3	3.0	16.5	58.0	1.10	6.0	32	1.60	315.0
120	18.0	11.5	50	16	18.0	23.75	2.6	3.2	21.4	77.5	1.10	6.0	26	1.60	160.0
135	18.0	11.5	37	16	17.0	26.50	3.1	3.5	25.5	97.0	0.60	6.0	28	1.60	170.0
150	18.0	11.5	36	16	17.0	29.00	3.5	3.8	26.0	117.0	0.40	6.0	32	1.52	180.0
165	18.0	11.5	34	16	18.0	31.50	4.2	4.2	30.0	139.0	1.10	6.0	34	1.52	1600.0
180	18.0	11.5	32	16	18.0	34.00	4.95	4.5	35.0	162.0	1.10	6.0	32	1.60	1150.0
195	18.0	-	-	16	18.5	36.00	-	-	-	-	-	-	31	1.62	1010.0
210	18.0	-	-	16	18.5	38.00	-	-	-	-	-	-	30	1.65	1000.0
225	18.0	-	-	16	19.0	41.75	-	-	-	-	-	-	29	1.65	1210.0
240	18.0	-	-	16	19.0	43.50	-	-	-	-	-	-	29	1.65	*2300.0
Aver.	18.0	11.3	31.0	16	18.0	43.00	4.95	4.5	35.0	162.0	0.9	6.0	31.2	1.58	10485.0

* Volume drained next day due to being held back by the packing.

Run No. 1

Calculated 100% H_2SO_4 for 15 Min. Intervals

Time in Min.	Grams 100% H_2SO_4 for Each 15 Min. Interval			
	Gross Output	Input	Actually Made	Theoretical
15	807	326	481	820
30	545	415	130	526
45	302	481	-	585
60	177	140	37	574
75	188	617.9	-	480
90	199	593	-	58.5
105	1510	345	964.1	468.0
120	985	237	748	585.0
135	950	365	584.5	
150	1150	345.9	804.1	
165	1470	494	976	
180	*2790	415	*2375	
Totals	11,073	4,981.2	6,091.7	4,096.5

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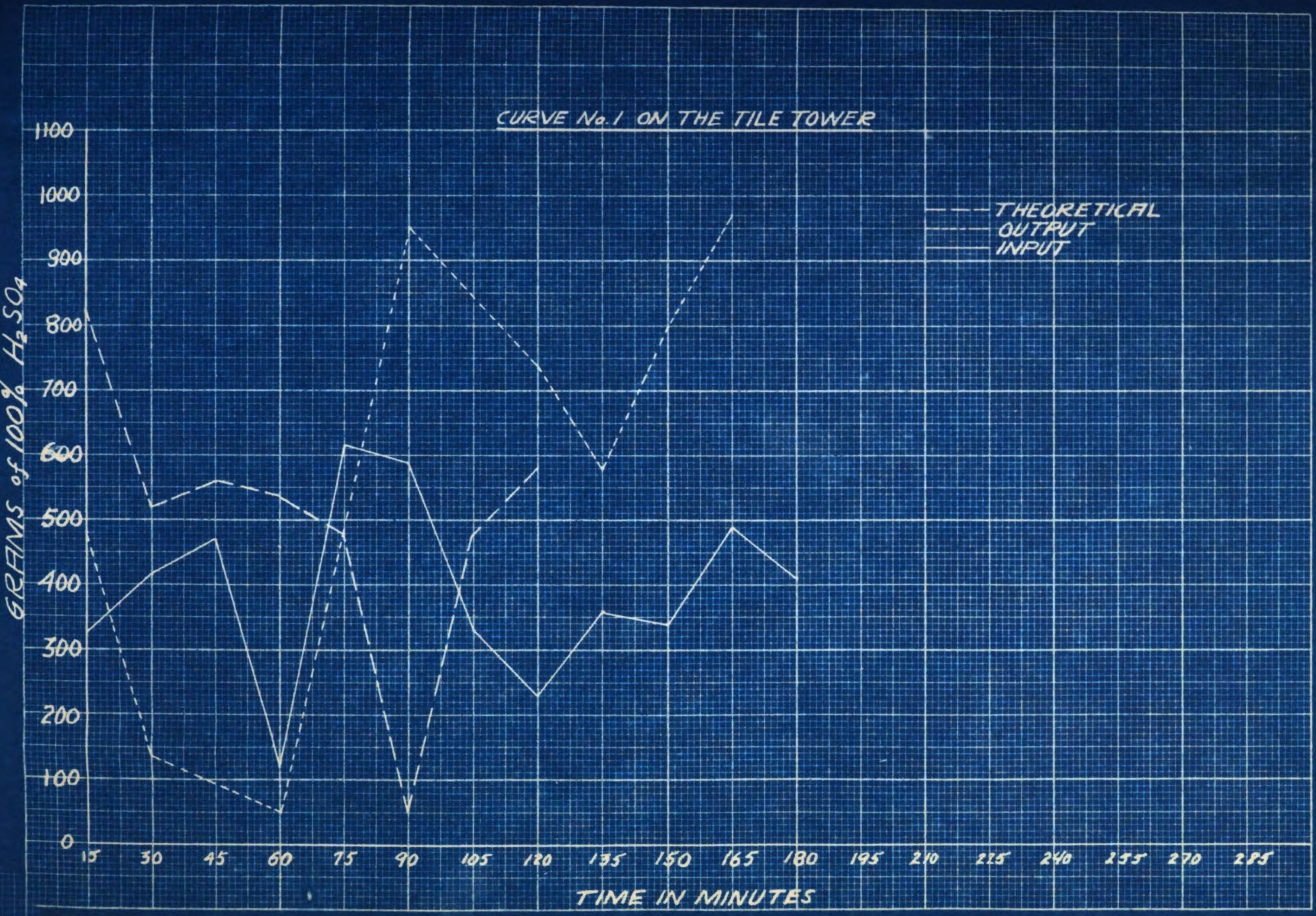
* This indicates amounts remaining in tower and drawn off next day. The wool packing retained these amounts due to the large surface offered. Thus the residual acid could not be drawn off at the 15 minute interval.



CURVE No. 1 ON THE TILE TOWER

Discussion of Run No. 1 made in pilot plant.

First of all, calculations for 100% sulphuric acid were computed for each 15 minute interval for gross output, input, and theoretical amounts. The dilute and 60°Be' acids were combined as one representing total input. Then the difference between gross output and input gave the amount of newly made sulphuric acid. The theoretical was determined from the volume of sulphur dioxide consumed. These results were computed for 15 minute intervals so they could be plotted and studdied from the standpoint of curves.

So by plotting the time as the abscissa and grams of 100% H_2SO_4 as the ordinate (refer to previous page), three curves were drawn out, one representing the input acid, another the output while the last the theoretical.

Interesting information can be seen by closely observing the plotted results. Theoretical and actual amounts of acid follow the same path only separated uniformly apart. Since the curves follow the same path it shows that as much acid was made as the SO_2 was admitted. These curves also bring out the point that the sulphur dioxide was not passed in at a uniform rate. If this were carried out, the curves would have been horizontal with the abscissa. It appears that the input acids have some affect too, only its best not to rely on it as yet.

The difference of temperature of inlet and outlet air indicated good signs. The same was true of inlet and outlet temperatures of cooling water. In conclussion the run turned out satisfactory and thus far, the same can be reproduced as was of laboratory tests in laboratory research.

Run No. 2

Pilot Plant Data Taken During Operation of Tile Tower

Time of Min.	Temperature °Centigrade					Vol. Cu. Ft.		Press. lbs. per Sq. In.		Liters of Input H_2SO_4		Diam. Tank 48" Height of Outlet	Gross Output Product		
	Inlet SO_2	Inlet Air	Outlet Air	Cooling Water Inlet	Cooling Water Outlet	SO_2	Air	SO_2	Air	Dilute 1.05 S.G.	60° HNO_3 1.39	Cooling H_2O	Temp.	Sp.Gr.	Volume cc
0	18	18	25	16	16	-	-	-	-	2.4	6.75	0.5	-	-	-
15	18	18	25	16	16	-	-	-	-	2.9	7.40	2.75	-	-	-
30	18	18	25	16	17	-	-	-	-	3.3	8.00	5.00	-	-	-
45	18	18	25	16	17	-	-	-	-	3.7	8.60	7.25	-	-	-
60	18	18	25	16	18	-	-	-	-	4.1	9.20	9.50	-	-	-
75	17	12	27	16	18	5.35	24	0.30	7.0	5.0	9.70	12.00	32	1.58	1050.0
90	17	11	35	16	18	9.65	58	1.00	6.75	6.4	10.40	14.50	33	1.40	850.0
105	17	11	36	16	18	16.6	90	1.40	6.75	7.2	11.00	17.00	33	1.30	725.0
120	17	11	36	16	19	24.1	130	1.40	7.00	7.9	11.40	19.00	30	1.20	900.0
135	-	-	-	16	19	-	-	-	-			21.00	29	1.14	1260.0
150	-	-	-	16	19.5	-	-	-	-			22.00	29	1.30	1470.0
165	-	-	-	16	19	-	-	-	-			24.50	28	1.50	925.0
180	-	-	-	16	19	-	-	-	-			25.75	27	1.60	*1600.0
Total or Average	17.0	11.0	33.2	16	18	24.1	130	1.02	6.87	5.5	4.65	22.25	30.1	1.39	9580.0

* This volume drained over night.

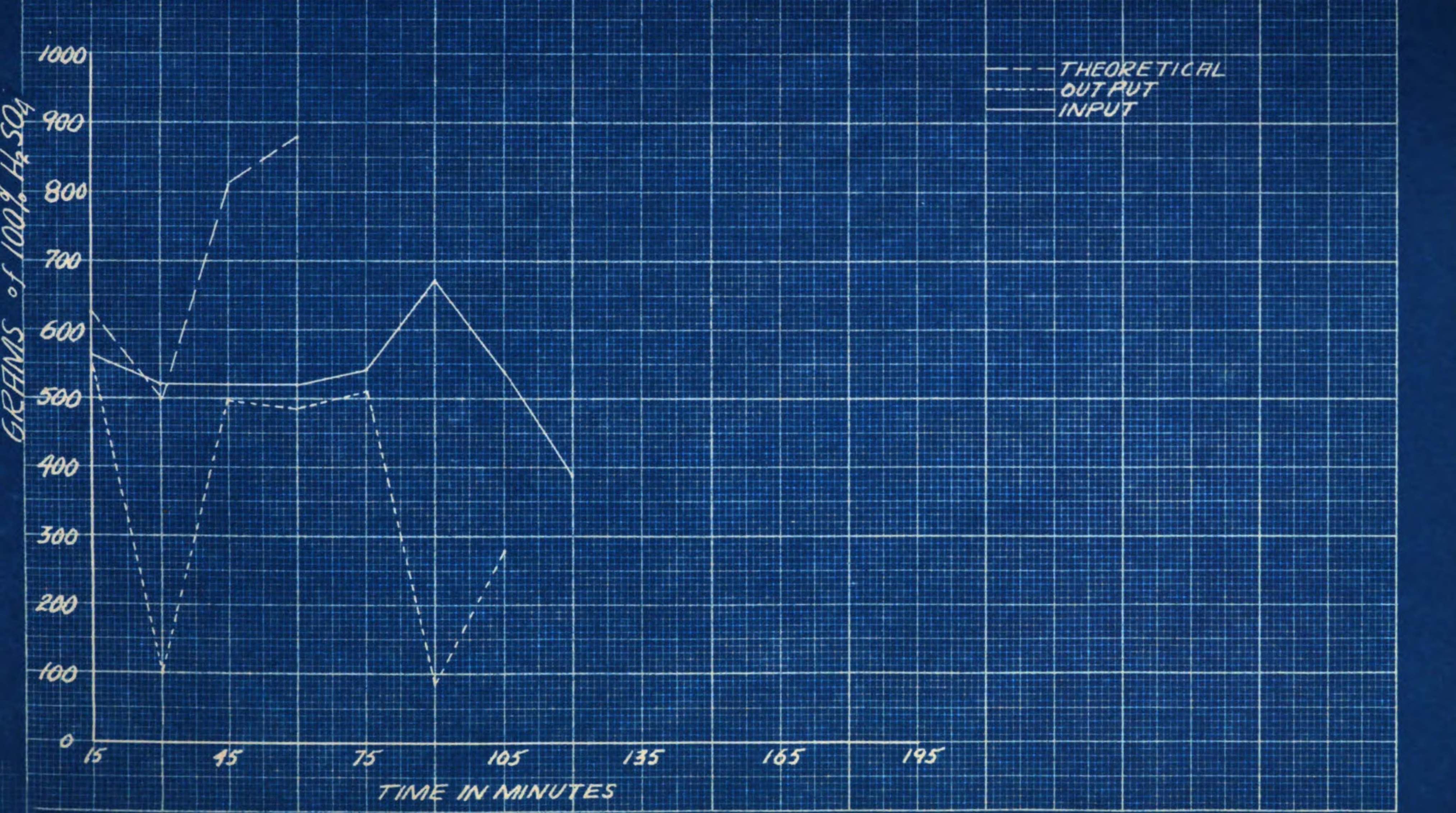
Run No. 2

Calculated 100% H_2SO_4 for 15 Min. Intervals

Time in Min.	Grams 100% H_2SO_4 During Each 15 Minutes			
	Gross Output	Input	Actually Made	Theoretical
15	1120	565.4	554.6	626
30	614	518.5	95.5	500
45	377	518.5	-	813
60	313	518.5	-	877
75	288	557.8	-	-
90	765	679.2	85.8	-
105	837	549.7	287.3	-
120	*2217.4	391.6	*1510.0	-
Totals	6532.4	3999.2	2533.2	2816

* Indicates amount remaining in tower and drawn off the following day. The glass wool packing retained these amounts due to the large surface offered. Thus the residual acid could not be drawn off at the last 15 minute interval.

CURVE No. 2 ON THE TILE TOWER



Discussion of Run No. 2 made in pilot plant.

Right off hand this run was to short. It had to be stopped due to a leak breaking out in the generator of oxides of nitrogen. Still the data was saved to determine more results and study them with use of curves. Again the same analogous results are reproduced in that the theoretical curve and actual followed similar paths.

Particular care had been used to control regular input volume of sulphur dioxide, but with least success. Yet favorable conditions were represented by temperature readings. The efficiency of the tower was 90.5% which is very good.

Another run was necessary to procure additional facts.

Run No. 3

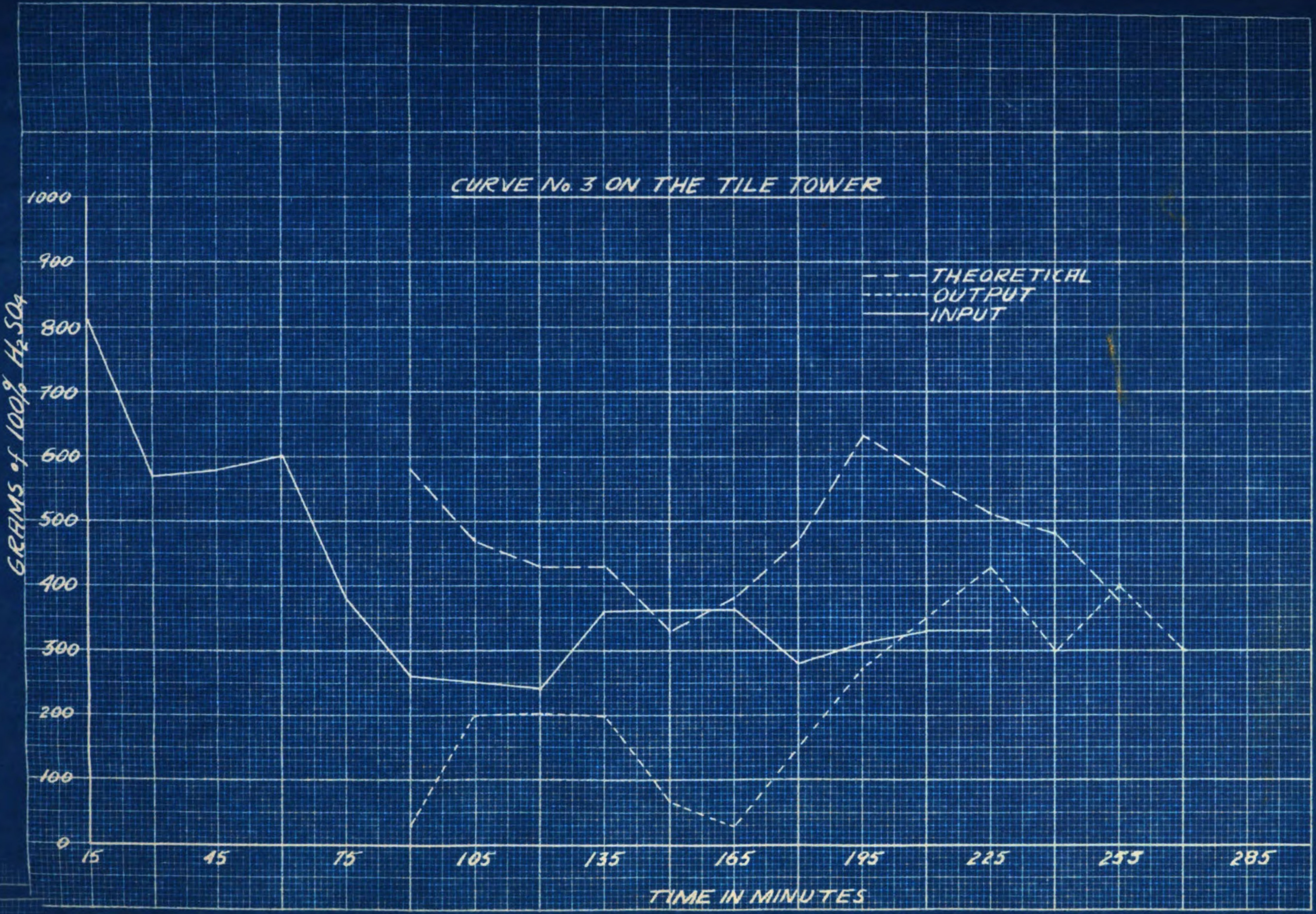
Pilot Plant Data Taken During Operation of Tile Tower

Time in Min.	Temperature °Centigrade					Vol. Cu. Ft.		Press. lbs. per Sq. In.		Liters of Input Acid		Inches of Cooling Water	Gross Output Product		
	Inlet SO_2	Inlet Air	Outlet Air	Cooling Water Inlet	Cooling Water Outlet	SO_2	Air	SO_2	Air	1.05	1.72-1.70*		Temp.	Sp.Gr.	Vol. in cc.
0	-	-	-	16	16	-	-	-	-	7.9	12.34	0.5	-	-	-
15	-	-	-	16	16	-	-	-	-	7.9	13.34	2.62	-	-	-
30	-	-	-	16	16	-	-	-	-	7.9	14.04	3.37	-	-	-
45	-	-	-	16	17	-	-	-	-	7.9	14.74	4.75	-	-	-
60	-	-	-	16	17	-	-	-	-	8.4	15.44	5.70	-	-	-
75	17	12	25	16	17	2.67	16.0	0.25	2.75	9.15	16.14	6.45	30	1.55	775
90	16	12	25	16	17.5	4.80	30.0	.30	2.75	9.45	16.34	7.50	31	1.52	500
105	15	12	24	16	17.5	6.80	43.0	.30	2.75	9.65	500 cc.*	8.50	29	1.51	340
120	15	12	24	16	18.25	8.80	57.0	.35	3.20	9.85	250 "	9.50	28	1.55	275
135	15	12	31	16	18.25	10.50	71.0	.37	3.45	10.25	250 "	10.50	25	1.56	285
150	16	12	32	16	18.25	12.30	86.0	.40	2.50	10.65	250 "	11.50	28	1.58	375
165	18	12	31	16	19	14.50	104.0	.50	2.50	11.00	200 "	12.50	31	1.55	350
180	18	13	28	16	19	17.30	116.0	.50	3.00	11.25	215 "	13.50	34	1.50	725
195	18	14	28	16	20	19.90	130.0	.40	3.00	11.55	235 "	14.50	40	1.44	680
210	19	15	29	16	20	22.20	148.0	.50	4.30	11.85	24 "	15.50	39	1.36	565
225	18	15	29	16	20	24.40	169.0	.55	3.50	12.00	-	16.50	40	1.40	350
240	18	15	29	16	21	25.20	194.0	.55	3.50	-	-	16.50	38	1.42	720
255	19	15	33	16	21	-	212.0	.30	3.50	-	-	17.50	36	1.43	290
270				16	21	-	-	-	-	-	-	18.50	31	1.45	1150
285				16	21	-	-	-	-	-	-	19.50	30	1.38	450
300				16	21	-	-	-	-	-	-	20.50	30	1.42	450
315				16	21	-	-	-	-	-	-	21.50	31	1.60	500
330				16	21	-	-	-	-	-	-	22/50	29	1.60	550
345				16	21	-	-	-	-	-	-	23.50	29	1.60	550
360				16	21	-	-	-	-	-	-	25.50	29	1.60	*500
Aver.	17	14	29.5	16	18.95	25.2	212.0	0.38	3.12	4.10	3060 2140	25.00	31.5	1.53	10105.0

* Remainder in tower and drained next day.

Calculated 100% H_2SO_4 for 15 Minute Intervals

Time in Min.	Grams of 100% H_2SO_4 for Each 15 Min. Interval			
	Gross Output	Input	Actually Made	Theoretical
15	775	809	-	-
30	471	565	-	-
45	309	565	-	-
60	275	605	-	-
75	292	383	-	-
90	375	266	25.8	313
105	318	178	197.2	251
120	568	681	204	235
135	430	364	203	235
150	408	360	65	175
165	201	364	37	213
180	580	286	269	251
195	236	310	-	338
210	770	336	439	310
225	336	331	302	272
240	498	-	404	262
255	558	-	302	200
270	615	-	384	-
285	558	-	255	-
Totals	11,543	7,486	2,057	2,960.0



CURVE No. 3 ON THE TILE TOWER

Discussion of Run No. 3 made in pilot plant.

This run wasn't as good as the previous one. Observing the representative curves on the preceeding page show no uniformity as desired and intended for. Other points in comparison were about the same. The input materials were controlled to ge regular at each interval, but the results show it to be different.

The efficiency was lower than either of the first two runs. It is believed that too much heed was taken in passing to small of quantities of input materials. It may be true of input acids which were insufficient to produce thorough wetting of the packing. This being true the gases coming in contact could only have reacted with the wetted portions and those contacting with the dry parts escaped unreacted.

Run No. 4

Pilot Plant Data Taken During Operation of Tile Tower

Time in Min.	Temperature °Centigrade					Inches of Cooling Water	Liters of Input Acids		Vol. in Cu. Ft.		Press. lbs. per Sq. In.		Gross Output Product		
	Inlet	Inlet	Outlet	Cooling Water											
	SO_2	Air	Air	Inlet	Outlet		Sp.Gr. 1.12	Sp.Gr. 1.72	SO_2	Air	SO_2	Air	Temp. °C	Sp.Gr.	Vol.cc.
0	-	-	-	-	-	-	5.4	-	-	-	-	-	-	-	-
15	-	-	-	-	-	-	5.6	-	-	-	-	-	-	-	-
30	-	-	-	-	-	-	5.8	-	-	-	-	-	-	-	-
45	-	-	-	-	-	-	6.05	0.1	-	-	-	-	-	-	-
60	20	21	25	16	17	1.00	6.35	0.6	0	0	0	0	-	-	-
75	20	18	30	16	17	2.75	6.60	1.0	5.95	26	1.5	6.75	31	1.5	260
90	19	18	30	16	18.5	5.50	6.95	1.5	11.90	51	1.6	7.75	38	1.4	170
105	18	16	35	16	20.0	8.25	7.20	2.2	18.20	78	1.4	9.30	34	1.42	195
120	17	15	32	15	19	10.25	7.50	2.8	25.20	110	1.4	9.25	64[1]	1.45	1275
135	17	15	35	15	19	12.50	7.80	3.4	32.10	135	1.5	9.25	50	1.32	1650
150	17	15	35	15	21	15.00	8.10	4.0	34.00	172	1.3	9.00	50	1.28	1415
165	17	14	45	14	21	17.25	8.40	4.6	38.00	203	1.4	8.80	45	1.15	2065
180	17	14	35	14	20	19.00	8.65	5.2	42.2	221	1.4	8.80	38	1.13	2290
195	-	-	-	14	18	22.00	-	-	-	-	-	-	35	1.15	4050
210	-	-	-	14	18	24.00	-	-	-	-	-	-	30	1.16	1950
225	-	-	-	14	18	-	-	-	-	-	-	-	27	1.10	1575
240	-	-	-	14	18	-	-	-	-	-	-	-	26	1.21	2865[2]
Aver.	17.8	16.2	34.6	14.8	19.8	23.00	3.25	5.1	42.2	221	1.43	8.40			

[1] Leak broke out of inner tile due to cracking, and cooling water seeped in.

[2] Volume drawn out the next day due to packing holding it.

Discussion of Run No. 4 made in pilot plant.

While the tower was in operation, the Glover tower broke out into a leak due to the cracking of the top tile (Refer to Blue Print No. 2). The only reason that can be given for its cracking was due to the heat of dilution plus the reaction heat. This happened since it was desirous to increase the efficiency by increasing the tower capacity by passing in more SO_2 and input acids. As a result, immediate quantities of heat were given off at the expense of chemical heat of reaction and heat of dilution. Thus the tile served its purpose to resistance of acids but not to excessive heat wear.

By referring to the data taken on run No. 4 made in pilot plant on the tile tower, one can tell by the comparison of input average specific gravity (1.35) with the output specific gravity (1.21) that dilution took place. Two reasons can be given for this lowering; one, that the average input specific gravity was higher than the output which even with small efficiency of the tower should have been somewhat increased, secondly, the output volume exceeded the input more than could have been accounted for. While the tower was under operation these two points were considered; and, after being convinced that the water was making its way into the inner tower from the outer jacket, the run was shut down.

This test was of value for it showed that tile constructed towers can not meet up with heat difficulties. Yet, such type of material will withstand resistance to acid. Since the given problem involves consideration of acid-resistance and heat, other more suitable material will have to be used. This means in order to proceed another tower needs to be constructed in place of the tile tower.

2. Data and Calculated results.

Average data for each run in pilot plant is contained on page 112.

Input and output materials and computed results for each separate run are on page 113.

Heat balance of Run No. 3 is on page 115.

Material balance of Run No. 3 is on page 116.

a. Average Data for Each Separate Run Made in Pilot Plant

No. of Run	Time in Hrs.	Temperature °Centigrade					Volume Cu. Ft.		Pressure lbs. per Sq. Inch		Gross Output			Amount of Cooling Water Passed	
		Inlet SO_2	Inlet Air	Outlet Air	Cooling Water		SO_2	Air	SO_2	Air	Temp. cc.	Sp. Gr.	Volume cc.	Height Measured in Inches	Calculated in Pounds
					Inlet	Outlet									
1	2.0	18	11.3	31.0	16	18	35	162	0.90	6.00	31.2	1.58	10485	43.00	2810
2	1.00	17	11.0	33.2	16	18	24.1	130	1.02	6.87	30.1	1.39	9580	25.25	1650
3	3.25	17	14.0	29.5	16	18.95	25.2	212	0.38	3.12	31.5	1.53	10105	16.32	1060
4	2.00	17.8	16.5	34.6	14.8	19.8	42.2	221	1.43	8.40	39.5	1.21	19650	23.00	1500

b. Input and Output Materials and Results Compiled for Each Pilot Plant Run

No. of Run	Time in Hrs.	Input H_2SO_4 Acid						Average Input Sp. Gr.	Gross Output				Grams of 100% H_2SO_4		
		60° Be^1 H_2SO_4			Dilute H_2SO_4										
		Vol. cc.	Wt. Gr.	Sp. Gr.	Vol. cc.	Wt. Gr.	Sp. Gr.		Sp. Gr.	Be^1	Volume in cc.	Wt. in Grams	Input	Output	Theoretical
1	2.00	4,500	7,740	1.72	4,950	5,550	1.12	1.36	1.58	53.4	10,485	16,550	4,981.2	6,091.7	4,096.5
2	1.00	3,559	6,110	1.72	5,500	5,775	1.05	1.31	1.39	41.5	9,580	13,300	3,999.2	2,533.2	2,816.0
3	3.25	3,060 2,140	5,260 3,640	1.72 1.70	4,100	4,300	1.05	1.42	1.53	50.3	10,105	15,470	7,486.0	2,057.0	2,960.0
4	2.00	5,100	8,769	1.72	3,250	3,600	1.12	1.35	1.21	25.2	19,650	23,800	5,085.0	1,975.0	4,930.0

Input and Output Materials and Results Continued from Previous Page

Tower Eff. in %	Ratios			Cu. Ft. of Tower Space per lb. of S per 24 hrs.	Heat Carried Away by Cooling Water in B.T.U.
	Output to Input Acids	Air to SO_2			
		Theoretical	Actual		
148.5	1.22	3.6	4.6	0.057	7,850
90.5	0.63	3.6	5.4	0.042	5,940
69.4	0.27	3.6	8.5	0.131	5,250
40.0	0.39	3.6	5.3	0.065	43,500

C. Heat Balance - calculated at 0° and 29" Hg.

Summary Heat Balance of Tile Tower.

INPUT

Before start of chemical reactions.

(1)	Heat content of Saturated air	311.00 B.T.U.	3.5%
(2)	Heat content of SO_2	0.59 B.T.U.	0.1%
(3)	Heat content of HNO_3 (78.18%)..........	33.60 B.T.U.	0.4%
(4)	Heat content of H_2SO_4 (1.05 Sp. Gr.)...	341.00 B.T.U.	3.8%
(5)	Heat content of Gay-Lussac acid.......	327.00 B.T.U.	3.6%
	Total heat before reaction............	1013.19 B.T.U.	-

Heat after chemical reaction.

(6)	Heat of formation of H_2SO_4..........	4,315.00 B.T.U.	47.7%
(7)	Heat of dilution	3,680.00 B.T.U.	40.9%
	Total.............................	9,008.19 B.T.U.	100.0%

OUTPUT

(1)	Heat content carried away by air....	875.00 B.T.U.	9.7%
(2)	Heat content carried away by acid...	970.00 B.T.U.	10.8%
(3)	Heat of concentration...............	1,130.00 B.T.U.	12.5%
(4)	Heat removed by cooling water.......	5,250.00 B.T.U.	58.3%
(5)	Heat absorbed by $NO \rightarrow NO_2$.........	77.40 B.T.U.	0.9%
(6)	Heat lost by radiation..............	705.79 B.T.U.	8.8%
	Total.............................	9,008.19 B.T.U.	100.0%

D. Material Balance.

INPUT

(1)	Saturated air	16.00 lbs.
(2)	Sulphur dioxide	4.25 lbs.
(3)	HNO_3 acid (1.39 Sp. Gr.)	1.53 lbs.
(4)	Total H_2SO_4	29.47 lbs.
	Total input	51.25 lbs.

OUTPUT

(1)	Spent air	15.08 lbs.
(2)	Weight of output product	33.99 lbs.
(3)	Weight of water held back from air	0.18 lbs.
(4)	Weight of HNO_3 acid recovered	1.28 lbs.
(5)	Acid left in the packing	0.47 lbs.
(6)	Loss of oxides of nitrogen	0.25 lbs.
	Total output	51.25 lbs.

3. Sample Calculations (Run No. 3).

a. Results of pilot plant data on the tile tower.

(1) Average Sp. Gr. of input acids.

Input acids-----

H_2SO_4 (Sp. Gr. 1.72), 3,060 cc.

H_2SO_4 (Sp. Gr. 1.70), 2,140 cc.

H_2SO_4 (Sp. Gr. 1.05), 4,100 cc.

Multiplying,

3,060 x 1.72 = 5,260 grams.

2,140 x 1.70 = 3,640 grams.

4,100 x 1.05 = 4,300 grams.

9,300 cc. 13,200 grams.

Dividing,

13,200 ÷ 9,300 = ...1.43 Sp. Gr.

(2) Input of 100%, H_2SO_4 in grams.

1.72 Sp. Gr., 80% H_2SO_4

1.70 Sp. Gr., 78.5% H_2SO_4

1.05 Sp. Gr., 7.7% H_2SO_4

Multiplying,

5,260 x 0.8 = 4,300 grams H_2SO_4

3,640 x 0.775 = 2,855 grams H_2SO_4

4,300 x 0.077 = 331 grams H_2SO_4

Total .... 7,486 grams.

(3) Gross Output of 100% H_2SO_4.

1.53 Sp. Gr., 63% H_2SO_4

Weight of gross output, 15,185 grams.

Multiplying,

15,185 x 0.63 = ... 9,539 grams.

(4) Net output of 100% H_2SO_4.

Subtracting (2) from (3)

9,539 - 7,486 = ... 2,057 grams.

(5) % efficiency of tower in converting sulphur dioxide into sulphuric acid.

25.2 cu. ft. of SO_2 consumed.

1.cu. ft. of SO_2 weighs 0.169 lbs.

One pound is equivalent to 454 grams.

Therefore,

25.2 x 0.169 x 454 = 1,934 grams of SO_2.

$SO_2 + \frac{1}{2}O_2 + H_2O \longrightarrow H_2SO_4$ + heat

Mol. weight of SO_2 = 64 grams.

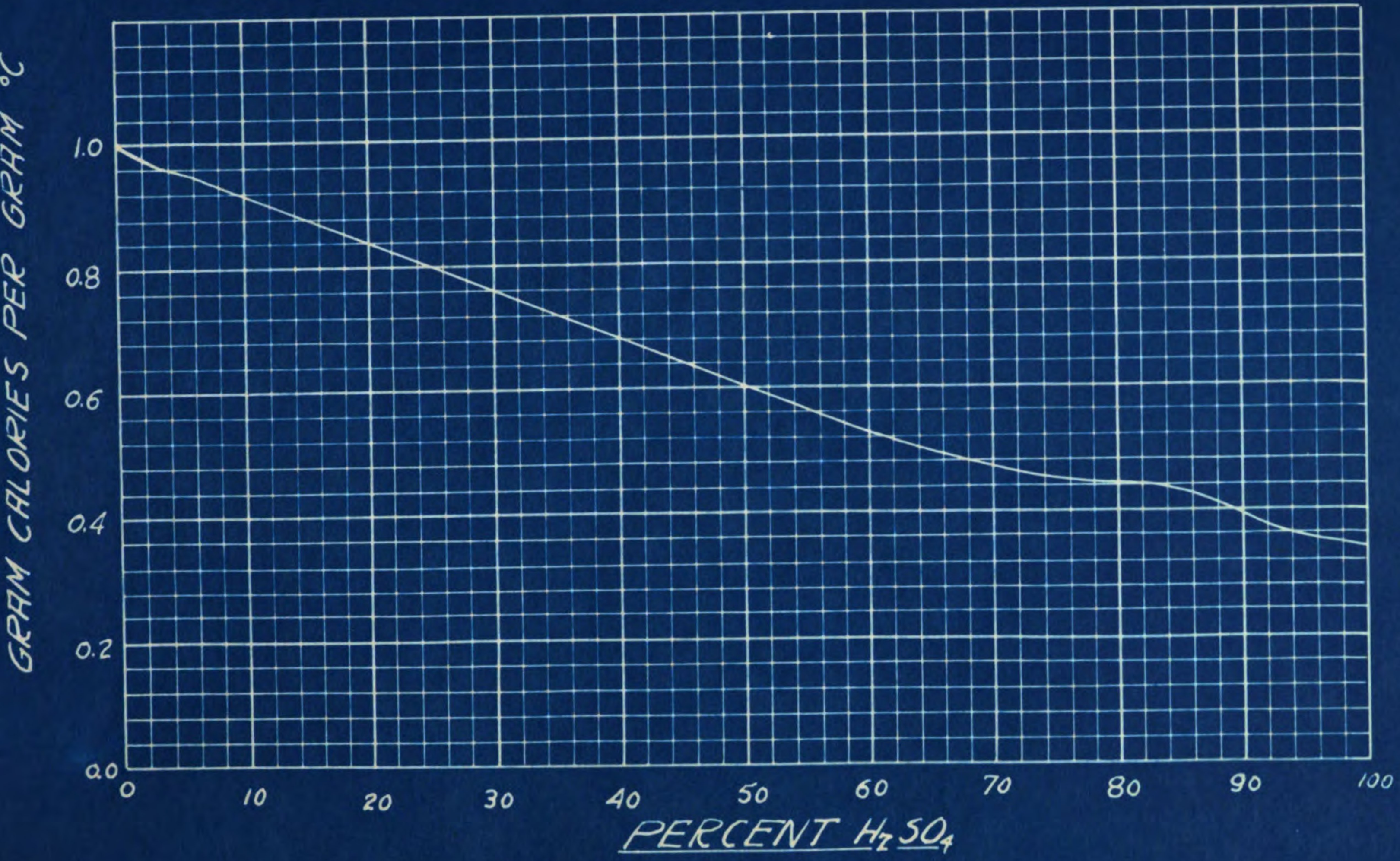
Then,

1,934 x 98 ÷ 64 = 2,960 grams of H_2SO_4 or theoretical amount.

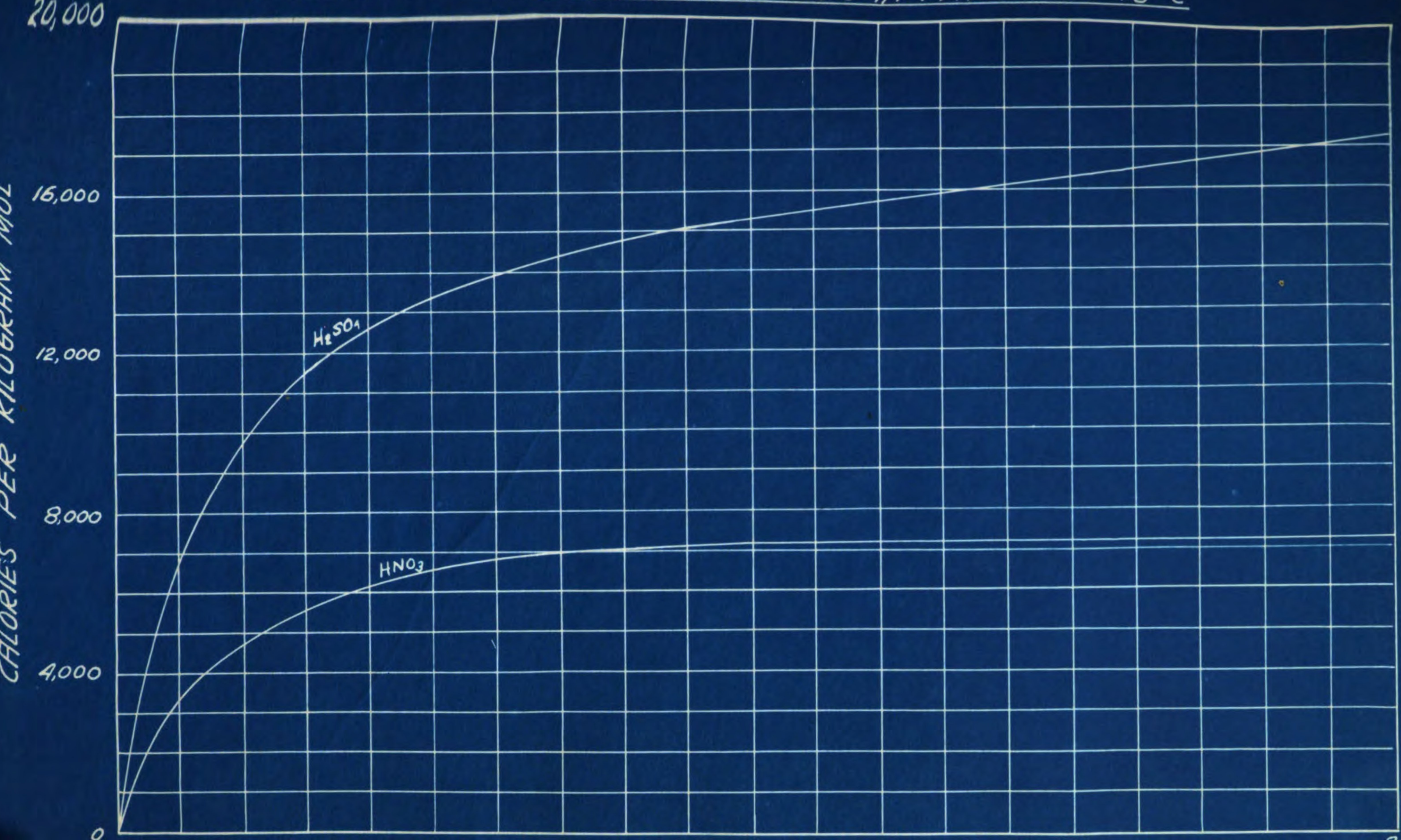
So,

2,057 ÷ 2,960 x 100 = ... 69.4%

C = TOTAL HEAT CAPACITY, CALORIES PER GRAM PER °C



INTEGRAL HEATS of SOLUTION of ACIDS IN WATER AT 18°C



(6) Tower space used / lb. of S. burned / 24 hours to acid.

Capacity of whole tower.

Volume of Gay-Lussac tower, 1.08 cu. ft.

Volume of Glover tower, 0.98 cu. ft.

Total.1.................... 2.06 cu. ft.

Weight of S in 1,934 grams of SO_2.

1,934 x 32 ÷ 64 = 967.0 grams of S.

967.0 ÷ 454 = 2.13 lbs. of S.

2.13 lbs of S was used in 3.25 hours / 2.06 cu. ft. volume of tower.

Dividing,

2.06 ÷ 2.13 = 0.967 cu. ft. / 3.25 hours.

Then,

0.967 x 3.25 ÷ 24 = ... 0.131 cu. ft.

b. Heat balance, based at 0° c and 29" Hg.

INPUT

Before start of chemical reactions.

(1). Heat content of sat. air.

Inlet temperature, 57.2° F.

From accompanying humidity chart.

Latent heat (B.T.U. / lb. H_2O) for

57.2°F. ....1.059 B.T.U.

Humid heat (B.T.U. / $^\circ$F. / lb. dry air) for

57.2° F. 0.247

Humidity (lbs. H_2O / lb. dry air)

for 57.2° F 0.011

Multiplying,

1,059 x 0.011= .... 11.6 B.T.U.

0.247 (57.2 - 32) = ... 6.2 B.T.U.

Total heat / lb. dry air............... 17.8 B.T.U.

1 cu. ft. of air weighs 0.079 lbs.

Cu. ft. of air consumed, 221.

Now,

221 x 0.079 x 17.8 = ... 311.0 B.T.U.

(2) Heat content of SO_2.

Inlet temperature 17° C. = 62.6° F.

From accompanying chart,

B.T.U. / lb. mol. / °F. for 62.6° F., 8.9

Density of SO_2 at 29" Hg. ,lbs. / cu. ft., 0.169

25.2 cu. ft. of SO_2 used.

Pound Mol. of SO_2 , 64 lbs.

Then,

25.2 x 0.169 x 8.9 ÷ 64 = ... 0.59 B.T.U.

(3) Heat content of HNO_3 (78.18%).

Input temperature 21° C.

From accompanying chart,

Heat capacity in cal. / gram of solu. / °C. for

78.18% HNO_3 .... 0.58

Volume of HNO_3, 1.39 Sp. Gr., 500 cc.

Multiplying,

500 x 1.39 = 695.00 grams of HNO_3.

0.58 (21 - 0) 695 = 8,470 cal.

1 B.T.U., 252 cal.

Dividing,

8,470 ÷ 252 = ...33.6 B.T.U.

(4) Heat content of H_2SO_4 (1.05 Sp. Gr.).

Input temperature, 21° C.

From accompanying chart.

Heat capacity, cal. / gram / °C for

1.05 Sp. Gr. = 7.7% H_2SO_4, 0.95

Weight of H_2SO_4 ,4,300 grams.

Then,

0.95 (21 - 0) 4,300 = 85,900 cal.

85,900 ÷ 252 = ...341. B.T.U.

(5) Heat content of Gay-Lussac acid.

Input temperature 21° C.

Heat capacity, cal. / gram / °C for

1.72 Sp. Gr., 80% H_2SO_4, 0.442

1.70 Sp. Gr., 78.5 % H_2SO_4, 0.440

Weight of 80% H_2SO_4, 5,260 grams.

Weight of 78.5% H_2SO_4, 3,640 grams.

Then,

0.442 (21 - 0) 5,260 = 48,800 cal.

0.44 (21 - 0) 3,640 = 35,600 cal.

Total .... 82,400 cal.

82,400 ÷ 252 = ... 327 B.T.U.

Total heat taken in before start of reactions........................ 1,013.19 B.T.U.

(6) Heat of formation of H_2SO_4.

Sulphuric acid is made from SO_2 gas, oxygen gas, and liquid water. Actually the conversion takes place in two steps with intermediate formations. However, the net effect is the same as though the following reaction proceeded in this manner —

$$SO_2 (g) + \tfrac{1}{2}O_2 (g) + H_2O (l) \rightarrow H_2SO_4 (l) + Q$$

Where Q = total heat of formation in cal.

SO_2 (g) = $S + O_2 - 69,400$ cal.

O_2 (g) = 0

H_2O (l) = $H_2 + \frac{1}{2}O_2 - 68,310$ cal.

Adding algebraically,

$$SO_2 - 69,400 + \tfrac{1}{2}O_2 + H_2O - 68,310 \rightarrow H_2SO_4 - 189,750 - Q$$

Q = 52,040 cal. / gram mol. of H_2SO_4.

2,057 grams were made.

Mol. of H_2SO_4, 98 grams.

Multiplying and dividing,

Q x weight of acid ÷ 98 = total heat of formation.

52,040 x 2,057 ÷ 98 = 1,088,000 cal.

1,088,000 ÷ 252 = ...4,315 B.T.U.

(7) Heat of dilution of 1.72 Sp. Gr. acid.

Heat of dilution of 1.70 Sp. Gr. acid.

Heat of dilution accompanying a change in concentration may be calculated by subtracting the heat of solution at the initial concentration from that of the final concentration.

No. of mols. of H_2O / mol. H_2SO_4 in,

1.72 Sp. Gr. acid, 80% H_2SO_4, 20% H_2O.

1.70 Sp. Gr. acid, 78.5% H_2SO_4, 21.5% H_2O

20 ÷ 18 = 1.110 mols. of H_2O.

21 ÷ 18 = 1.195 mols. of H_2O.

80 ÷ 98 = 0.816 mols. of H_2SO_4.

78.5 ÷ 98 = 0.801 mols. of H_2SO_4.

Then,

Mols. of H_2O / mol. of H_2SO_4,

1.110 ÷ 0.816, or 1.36

1.195 ÷ 0.801, or 1.49

From accompanying chart, heat of solution for 1.36 mols. of water / mol. H_2SO_4, 8,000 cal. and for 1.49, 8,300 cal.

Weight of 1.72 Sp. Gr. acid, 5,260 grams.

Weight of 1.70 Sp. Gr. acid, 3,640 grams.

Multiplying,

5,260 x 8000 ÷ 98 = 430,000 cal.

3,640 x 8300 ÷ 98 = 308,000 cal.

Total initial heat of solution ....738,000 cal.

No. of mols. of H_2O / mol. H_2SO_4 in 1.42 Sp. Gr. acid;

52.5% H_2SO_4, 47.5% H_2O, or final concentration.

$47.5 \div 18 = 2.640$ mols. of H_2O.

$52.5 \div 98 = 0.535$ mols. of H_2SO_4.

$2.64 \div 0.535 - 4.93$ mols. of H_2O / mol. of H_2SO_4.

Heat of solution for 4.93 mols. of H_2O / mol. of H_2SO_4,

13,300 cal.

Weight of 1.42 Sp. Gr. acid, 13,000 grams.

Then,

$13{,}000 \times 13{,}300 \div 98 = 1{,}765$ cal., or

final heat of solution.

Subtracting initial heat of solution from final

heat of solution,

$1{,}765{,}000 - 738{,}000 = 927{,}000$ cal.

Total heat of dilution is

$927{,}000 \div 252$ = ...3,680 B.T.U.

Total input heat of tile tower .... 9,008.19 B.T.U.

OUTPUT

(8) Heat content carried out by air into atmosphere.

212 cu. ft. of air consumed.

Outlet temperature 29.5° C. or 85.1° F.

Humidity (lbs. H_2O / lb. dry air) for 85.1° F., 0.036

Latent heat (B.T.U. / lb. H_2O) for 85.1° F., 1045 B.T.U.

Humid heat (B.T.U. / °F. / lb. dry air) for

85.1° F, 0.275

Multiplying,

1045 x 0.036 = ...37.6 B.T.U.

0.275 (85.1 - 32) = ...14.6 B.T.U.

Total B.T.U. / lb. dry air = ...52.2

1 lb. air weighs at 29" Hg. 0.079 lbs.

212 x 0.079 x 52.2 = ... 875 B.T.U.

(9) Heat content carried out by gross product.

Volume of gross product, 10,105 cc..

Sp. Gr. of gross product, 1.53

1.53 Sp. Gr. acid, 63% H_2SO_4.

Temperature of product, 31.5°C.

Heat capacity, cal. / gram./ °C. for 63% acid, 0.5

Weight of acid = 10,105 x 1.53 or 15,530 grams.

0.5 (31.5 - 0) 15,530 ÷ 252 = ... 970 B.T.U.

(10) Heat of concentration of 1.42 Sp. Gr. to 1.53 Sp. Gr. of final product.

1.42 Sp. Gr.; 52.5% H_2SO_4, 47.5% H_2O.

1.53 Sp. Gr.; 63% H_2SO_4, 37% H_2O.

47.5 ÷ 18 = 2.64 mols. of H_2O.

37.0 ÷ 18 = 2.05 mols. of H_2O.

52.5 ÷ 98 = 0.535 mols. of H_2SO_4.

63.0 ÷ 98 = 0.643 mols. of H_2SO_4.

Dividing,

$2.64 \div 0.535 = 4.94$ mols. of H_2O / mol. H_2SO_4.

$2.05 \div 0.643 = 3.19$ mols. of H_2O / mol. H_2SO_4.

From chart, heat of solution for

4.94 mols. of H_2O / mol. H_2SO_4, 13,300 cal..

3.19 mols. of H_2O / mol. H_2SO_4, <u>11,500 cal.</u>

Heat taken by concentration ...... 1,800 cal.

Weight of final product, 15,530 grams.

Then,

$(15,530 \div 98)\ (1800 \div 252) =$ ....1,130 B.T.U.

(11) Heat removed by cooling water.

Weight of cooling water, 1060 lbs.

Outlet temperature, 18.95° C., or 65.75° F.

Inlet temperature, 16.00° C., or 60.80° F.

Multiplying,

$(65.75 - 60.8)\ 1060 =$ ....5,250 B.T.U.

(12) Heat absorbed by $NO \rightarrow NO_2$.

$NO_2 + SO_2 \rightarrow SO_3 + NO$

$SO_3 + H_2O \rightarrow H_2SO_4$

Grams of acid made, 2,057.

Mol. weight of SO_3, 80 grams.

Now,

$(80 \div 98)\ 2057 = 1672$ grams of SO_3.

NO_2 required by SO_2 in grams.

Mol. Weight of NO_2, 46 grams,

Or,

(46 ÷ 80) x 1672 = 960 grams of NO_2.

Heat of formation of NO_2, (-930 cal.)

per gram mol.

Multiplying and dividing,

930 x 960 ÷ 46 = ...19,500 cal.

19,500 ÷ 252 = ... 77.4 B.T.U.

(13) Heat lost due to Radiation........................ 705.79 B.T.U.

Total heat output............................... 9,008.19 B.T.U.

C. Material Balance.

INPUT

(1) Saturated air.

1 cu. ft. of air at 29" Hg. weighs 0.079 lbs.

212 cu. ft. consumed.

Multiplying,

212 x 0.079 = ...16.00 lbs.

(2) Sulphur dioxide.

1 cu. ft. SO_2 at 29" Hg. weighs 0.169 lbs.

25.2 cu. ft. consumed.

Multiplying,

25.2 x 0.169 = ... 4.25 lbs.

(3) HNO_3 acid (1.39 Sp. Gr.)

500 cc. used.

Multiplying and dividing,

500 x 1.39 ÷ 454 = ... 1.53 lbs.

(4) H_2SO_4 (1.72 Sp. Gr.).
" (1.70 " ").
" (1.05 " ").

Amounts used.

3,060 cc. of 1.72 Sp. Gr.
2,140 cc. of 1.70 Sp. Gr.
4,100 cc. of 1.05 Sp. Gr.

Multiplying and dividing,

3,060 x 1.72 ÷ 454 = 11.60 lbs.
2,140 x 1.70 ÷ 454 = 8.02 lbs.
4,100 x 1.05 ÷ 454 = 9.85 lbs.

Total ....29.47 lbs.

Total input into tower.........................51.25 lbs.

OUTPUT

(5) Spent air.

Inlet air minus water content.

Pounds H_2O / lb. sat. air for 14°C. or 57.2°F, 0.011.

Weight of air passed, 16.0 lbs.

Weight of water content, 16 x 0.011 = ...0.18 lbs.

Subtracting,
16 - 0.18 = 15.82 lbs. of dry air.

Dry air minus oxygen content or actual air spent.

Weight of H_2SO_4 made, 2,057 grams.

Mol. Weight of $\frac{1}{2}O_2$, 16 grams,

Or,

(16 ÷ 98) 2,057 = 334 grams of oxygen used.

334 ÷ 454 = 0.74 lbs.

Subtracting finally,

15.82 - 0.74 = ...15.08 lbs.

(6) Weight of output acids.

1.72 Sp. Gr., 16.60 lbs.

1.70 " " , 8.02 lbs.

1.05 " " , 9.47 lbs.

Total ....29.47 lbs.

Newly made.

2,057 ÷ 454 = 4.52 lbs.

Total ....33.99 lbs.

(7) Water content retained.......................0.18 lbs.

(8) Weight of recovered HNO_3.....................1.28 lbs.

(9) Loss of HNO_3 acid............................0.25 lbs.

(10) Loss of acid in tower.......................0.47 lbs.

Total output................................51.25 lbs.

C. CONCLUSIONS

The study of preparation of sulphuric acid on a larger scale as compared with laboratory work proved to be quite successful. Similar results as of laboratory were equally reproduced. One thing failed to do was that in obtaining sufficient data and information which should have been similar for all tests, This may be due to some outside factors which need looking into in order to obtain constant results of like nature.

The tile structure failed in more vital respects than it proved suitable in others. As for sake of resisting acid wear, it was not affected in the least. When it was subjected to proper cooling, it was inadequate for removal of immediate heat of dilution and of formation. In comparing the temperature of inlet air with the outlet air, the difference was too great insofar as the Gay-Lussac temperatures were concerned. As it has been pointed out before, the sole key to the operation is the function of oxides of nitrogen. If the Gay-Lussac tower temperatures are high, the oxides will be driven off due to lack of formation of nitrosyl sulphuric acid which reacts best at lower temperatures. Thus, if there is a constant excessive loss of nitrous fumes as the case appeared to be in all runs, insufficient oxidation of sulphur dioxide results.

It was desirous to use just a little over the theoretical amount of HNO_3 to produce the required share of nitrous fumes.

By doing so, the efficiency dropped due to large losses of oxides of nitrogen. Sometimes the temperatures of the Gay-Lussac tower were momentarily high because the immediate heat was not removed fast enough by conduction through the acid-proof masonry tile, thus it remained in the tower. Then the up-passing air no doubt carried it to the Gay-Lussac tower where it prevented the formation of nitrosyl sulphuric acid which in turn could not be carried down into the oxidation zone of the Glover tower. Consequently, the oxides were held free in the Gay-Lussac tower or else carried out into the atmosphere. Now, as soon as the oxides of nitrogen are not carried to the oxidation zone, the sulphur dioxide makes its way into the Gay-Lussac tower and there the oxidation takes place still raising the temperature. Such a case was actually observed. When the supply of SO_2 was turned off momentarily the temperatures decreased. By that time more nitrosyl sulphuric acid was formed, and recovery of oxides of nitrogen resulted which made their way into the oxidation zone. The sulphur dioxide valve was opened again to admit more SO_2 into the system in order to continue the run. In conclusion the tile was a very poor conductor of heat.

Too many joints were created with the two-foot length tiles and changes for leaks were possible. This defect as far as the process is concerned had no influence on it, yet it left an uneasy feeling about the stability of the towers.

Inasmuch as withstanding high temperatures in the Glover tower, the tile cracked upon intense heat. Commercially such material could not be utilized. Of course, for the purpose of pilot plant study it was alright to use right off hand, but difficulty arose in the end. This being the case the data obtained could not be relied upon for designing a commercial plant. It would be best to work with tower material that could also be used in construction of an industrial plant.

The use of glass wool packing was excellent for the abundant surfacing in promotion of the gas-liquid phase. It also weathered the heat and acid wear in the most desirable fashion.

To obtain further information a new tower will have to be constructed of some type of acid resistive alloy.

PART FOUR

STUDY WITH A LEAD TOWER
FOR PILOT PLANT RESEARCH

A. PILOT PLANT OPERATIONS.

1. Discussion.

In reference to what has been set out to do as explained in the discussion of part III, continuance will be carried on from that point.

Since the tile tower could not stand under the influence of heat, a tower of more suitable material was made. Lead, right off hand, answered well insofar as to acid-resistance and conduction of heat for pilot plant research. Yet, for industrial use a more resistive alloy would be better because originally it was desirous to dispense with lead completely as well as the lead chambers.

The biggest point in progress toward development at that stage was to increase output per unit capacity. By that stage, it is meant when the tile tower cracked due to excessive heat. In the end the lead tower should stand up better against heat, but not nearly so well against acid wear. Still, it should weather through to obtain the concluding information as to prospects of developement of such a method for commercial use. Again it is briefly stated that maximum output per unit time will be tried for by making more runs. A faster rate of SO_2 , air, and NO_2 will be supplied per same unit volume per unit time to obtain the ultimate daily output with a satisfactory efficiency.

2. Construction of Lead tower.

There was not any change in this set-up in comparison with the tile tower described in part III (page 85). The only difference is that the inner tower is made of lead still containing two sections, the top half (3) or the Gay-Lussac tower, and lower as the Glover (15), front view, (Refer to Blue Print No. 3).

First, the previous pilot plant tower was torn down to the concrete base (G). The lead cup (DD) was removed to be reconstructed and inserted back in its place. A short half-inch lead pipe (4), sideview, is burned to outer side of (DD), and it extends into the cup (DD) at the bottom in such a manner that the acids can drain out of (DD) through (4) and over the sprigot (H), sideview, and into the receiver (I), front view. Next, the entrance line (K) from tower entrance (9), side view, was joined to the top of the cup (DD) so that the gases could enter tower (7) and (8), side view. The lead grate (L) was burned upon (DD) to form the bottom of tower (7). It also supported the inside packing and permitted gases to pass through the packing.

(a) Construction of Glover tower (7), (side view).

Two sheets of lead 3/16 inches thick were rolled cylindrically to form a six inch diameter tower and six feet long (7). The bottom of (7) was burned around the lead grate (L) and on top of the lead cup (DD). Thus an air tight joint (Z) prevented the escape of gases to the outside or leakage of water into

(7) from water-jacket (2). Half of this tower (7) was packed with glass wool (M). Then the Gay-Lussac acid line (N) had to be suspended into (7) and the glass wool packed around (N).

Four lead lugs (O) an inch square and inch and half long were burned quarterly apart inside of the circular tower (7) down four inches from the top of (7). A perforated lead pan (P) six inches in diameter and two inches deep rests upon the lugs (C). Then on top of the lead pan (P) a solid lead plate (Q) one inch thick and 6 inches in diameter was burned to the inside wall of tower (7). Through the center of (Q) and (P), passes the lead line (N) burned so that acids cannot leak along side of (N) at the points where (N) passes through (Q) and (P). Short vent lines half an inch in diameter and inch and half high pass through (P) and (Q) so that the oxides of nitrogen and air can enter into tower (8) from (7). This discussion constitutes the top of Glover tower (7) and the bottom of tower (8), or the Gay-Lussac.

(b) Gay-Lussac tower (8), (Side view in Blue Print No. 3).

The two lower sewer tiles (16) and (17) were inserted with the lower one (16) imbedded four inches below the top of the concrete base (G). Now, the remainder of the inner lead tower (8) was finished. Two more lead sheets 3/16 inches thick were rolled and make up tower (8), 6 inches in diameter and 50 inches high. This was lowered into the top of tower (7), and outside of (7) was burned against the outside of (8). (This is clearly shown by studying the cutout section (X) of the side view in Blue Print No. 3.)

The bottom of (8) was filled with coarse packing (R) about six inches deep, so that the Gay-Lussac acid could drain into (N) and at the same time permit air to pass through the vents of (Q) and (P). From (R) up, glass wool was packed around the dilute acid (S). At the top of (8) and resting on the glass wool packing (M), another lead perforated pan (B) was burned to the inside wall of (8). Through (B) passes the line (S) and the air vent (T) which permits the spent air to escape into the atmosphere.

The remaining three tiles (12), (13), and (14) of the water-jacket were set in and cemented with corresponding materials, rope oakum, asphalt, and portland cement mixed with a little sand. This completed all the work on the tower.

(c) Minor Units (Front View of Blue Print No. 3).

Acid reservoirs (1) were made of 15 liter bottles and graduated as such. Two siphons (20) and (21) were made of quarter-inch glass tubing equiped with glass stop-cocks. Rubber stoppers close the mouths of bottles (1), and through them pass the shorter legs of the siphons (20) and (21). Thus the siphons are firmly held in the shown position. Two funnels (22) and (23) also respectively pass through the same rubber stoppers of bottles (1). Required input acids were entered through these funnels. Likewise, two peep tubes were inserted through the rubber stoppers along the side of the funnel stems in the bottles (1) so that air could escape when the reservoirs were being filled with more acid of required strength.

The NO_2 generator (U) and NO_2 removed from newly made acid in (V) are made of 12 liter round bottom pyrex flasks. These are supported by the tripods (W) and (Y), and the C lamps (AA) and (BB). The mouths of each are filled in with a mixture of asbestos and portland cement (CC). Asbestos, portland cement and water were mixed to a plastic state and applied as such. These caps (CC) not only form a cap, but also hold in place the flue lines (EE) and (FF), funnels (GG) and (HH), and the air lines (II) and (LL). Bunsen burners (TT) and (SS) were used to heat (U) and (V) respectively.

The manometers (KK) and (MM) were made to obtain the pressures of the inlet air and SO_2. Gas meters (NN) and (OO) registered respectively the volume of SO_2 and air passed into the system. Also the control of ratio of air to SO_2 was maintained with (NN) and (OO). Theoretically the ratio should be 3.6 of air to 1.0 of SO_2 by volume. Actually this degree was exceeded so that a sufficient amount of air entered to provide ample supply of oxygen to the process. Temperature recording units (PP) and (RR) were devised for obtaining the inlet temperatures of SO_2 and air. This concluded the work on building the lead tower and its minor units.

B. HOW EACH RUN WAS MADE.

The runs were made on the lead tower identically the same as those on the tile tower. Similar data was also taken.

1. Pilot plant runs on the Lead Tower.

The following pages contain the tabulated data for each separate run. Grams of 100% input, output and theoretical amounts of acid were plotted as three curves. These amounts were calculated for each interval and plotted against the time intervals. The curves were studied and explained in discussion of each run.

Run No. 1 on Lead Tower

Data Taken on the Lead Tower in Pilot Plant Operation

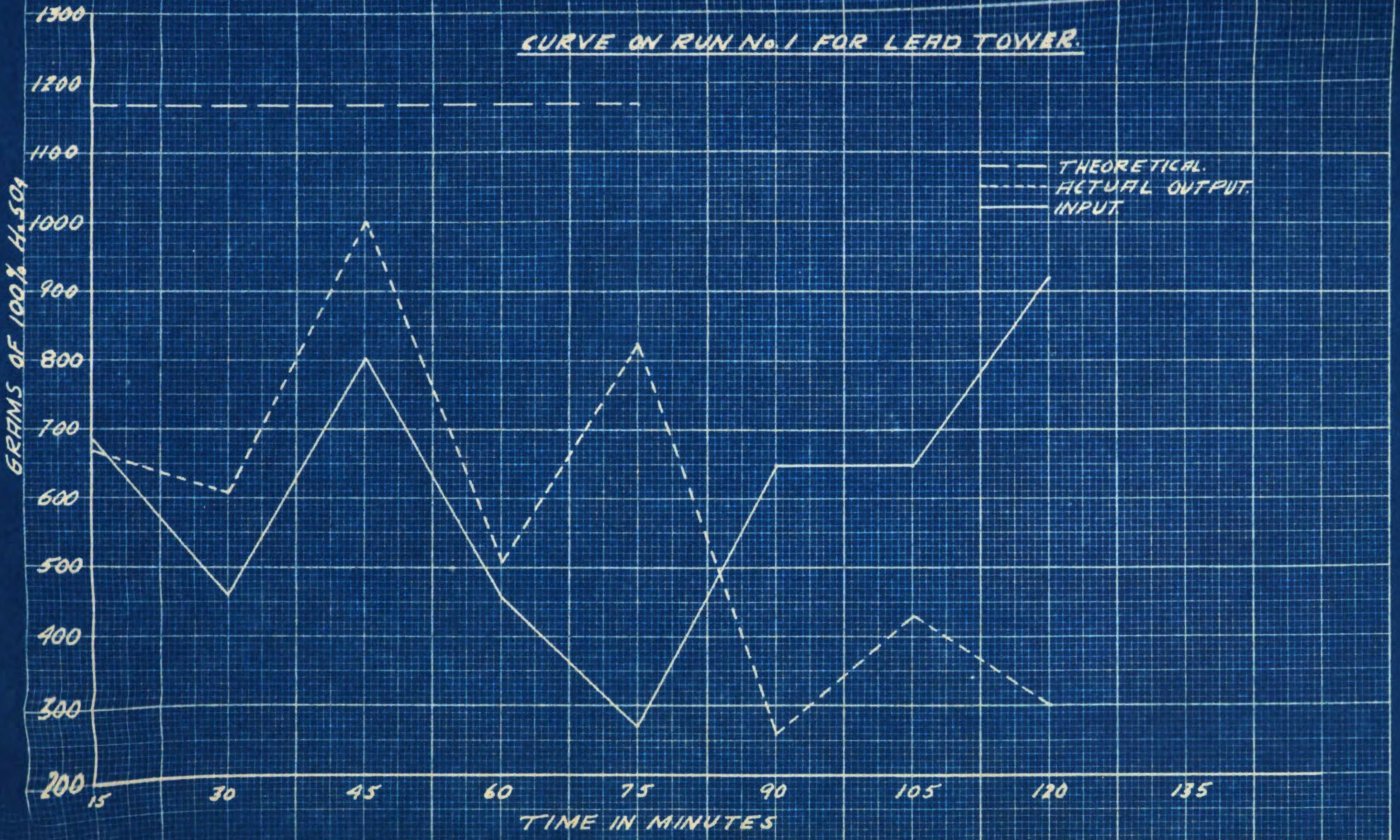
Time in Min.	Temperatures °Centigrade					Inches of Cooling H_2O	Input Acids		Vol. in Cu. Ft.		Press lbs. per Sq. In.		Gross Output Product		
	Inlet SO_2	Inlet Air	Outlet Air	Cooling Water Inlet	Cooling Water Outlet		Sp. Gr. 1.08	Sp. Gr. 1.70	SO_2	Air	SO_2	Air	Temp. °C	Sp. Gr.	Vol. in cc.
0	-	-	-	-	-	-	2.2	5.00	-	-	-	-	0	0	0
15	-	-	-	-	-	-	2.4	5.50	-	-	-	-	55	1.5	1475
30	-	-	-	-	-	-	2.6	5.85	-	-	-	-	55	1.4	1525
45	-	-	-	-	-	-	2.7	6.45	-	-	-	-	50	1.35	2995
60	-	-	-	-	-	-	2.9	6.80	-	-	-	-	50	1.55	1000
75	24	24	24	14.0	21	0,5	3.00	7.00	0	0	0	0	55	1.55	1100
90	23	20	30	13.5	18	4.0	4.00	7.40	10	30	3.0	9.00	55	1.55	910
105	23	19	40	13.0	18	7.5	5.00	7.80	20	62	3.0	9.25	50	1.60	970
120	22	18	70	13.0	17	11.0	6.00	8.40	30	98	3.0	10.00	50	1.60	1100
135	21	18	75	13.0	17	14.5	7.00	9.30	40	132	3.0	9.70	50	1.60	1080
Aver.	22.5	18.7	53.5	13.12	17.5	14.0	4.8	4.30	40.	132	3.0	9.23	51.5	1.48	11755

Input of 500 cc. 1.39 Sp. Gr. HNO_3 Temp. 24 °C.

Run No. 1 on Lead Tower

100 % H_2SO_4 Calculated for 15 Minute Intervals

Time in Min.	100% H_2SO_4 in Grams			
	Gross Output	Input	Net Output	Theoretical
15	1360	686.	674	1170
30	1068	460.	608	1170
45	1842	803.	1039	1170
60	1000	486.	514	1170
75	1100	276	824	-
90	910	650	260	-
105	1080	650	430	-
120	1220	920	300	-
135	1192	1310	-	-
Totals	10,770	6,461	4,309	4,680



CURVE ON RUN No. 1 FOR LEAD TOWER.

Discussion of Run No. 1 for the lead tower.

First of all the efficiency was very good which was 92%. When the test was made, it lacked sufficient amount of nitrogen dioxide because the odor of SO_2 leaving out of the air vent on top of Gay-Lussac tower indicated the loss. Observing the curves on the previous page show that the input acids have some influence on the operation of the tower. This indication presents that wetting of the surface is essential. SO_2 was carefully regulated to admit it at a constant rate and it did not influence the operation. Yet previous runs of the tile tower showed that it did. Without any doubt it should have in this run. This leads to discussion as to some authority that the SO_2 passes into solution with water to form sulphurous acid which reacts with the NO_2 as follows --

1. $SO_2 + H_2O \rightarrow H_2SO_3$
2. $2H_2SO_4 + NO_2 \rightarrow 2(H_2SO_4)NO$

The $(H_2SO_4)NO$ is called the violet acid. It in turn reacts with more NO_2 and oxygen as follows --

3. $2(H_2SO_4)NO + \frac{1}{2}O_2 + NO_2 \rightarrow 2SO_5NH + H_2O(NO)$
4. $2SO_5NH + SO_2 + 2H_2O \rightarrow 2(H_2SO_4)NO + H_2SO_4$
5. $(H_2SO_4)NO \rightarrow H_2SO_4 + NO$

These reactions are greatly influenced by pressure. The pressures were considerable higher than in the previous runs. The internal resistance of the packing may have increased the pressure because greater quantities of gases were passed per unit time. Also some of these reactions are of heterogeneous type on the gas-liquid phase. So it seems logical that this could have happened along with wetting of input acids upon the packing to produce a homogeneous liquid-phase. Insofar as the process is concerned there isn't any harm if the reactions pursue this course.

Temperature conditions are favorable except the Gay-Lussac tower temperature.

Run No. 2 on Lead Tower

Data Taken on the Lead Tower in Pilot Plant Operation

Time in Min.	Temperatures °Centigrade					Inches of Cooling H_2O	Input Acids		Vol. in Cu. Ft.		Press lbs. per Sq. In.		Gross Output Prod.		
	Inlet SO_2	Inlet Air	Outlet Air	Cooling Water			Sp.Gr. 1.08	Sp.Gr. 1.70	SO_2	Air	SO_2	Air	Temp. °C	Sp. Gr.	Volume in cc.
				Inlet	Outlet										
0							3.4	1.50					0	0	0
15							3.75	1.96					30	1.40	550
30							4.10	2.42					35	1.44	670
45							4.45	2.88					35	1.47	1260
60							4.60	3.34					35	1.44	1680
75	24	24	26	13	13	0.5	4.80	3.80	0	0	0	0	40	1.50	1350
90	19	14	28	13	16	4.0	5.40	4.30	12.0	40	5.0	13.1	50	1.45	1975
105	18	13	62	13	21	7.5	6.20	5.60	23.0	76	6.7	13.4	50	1.50	1225
120	17	13	35	13	21	11.0	7.30	7.60	35.0	114	7.2	13.2	50	1.58	1200
135	17	13	40	13	20	14.5	8.00	8.60	44.0	152	5.0	14.0	50	1.68	3135[1]
Aver.	18.7	13.12	41.2	13	19.5	14.0	4.60	7.10	44.0	152	5.97	13.4	41.6	1.54	13035

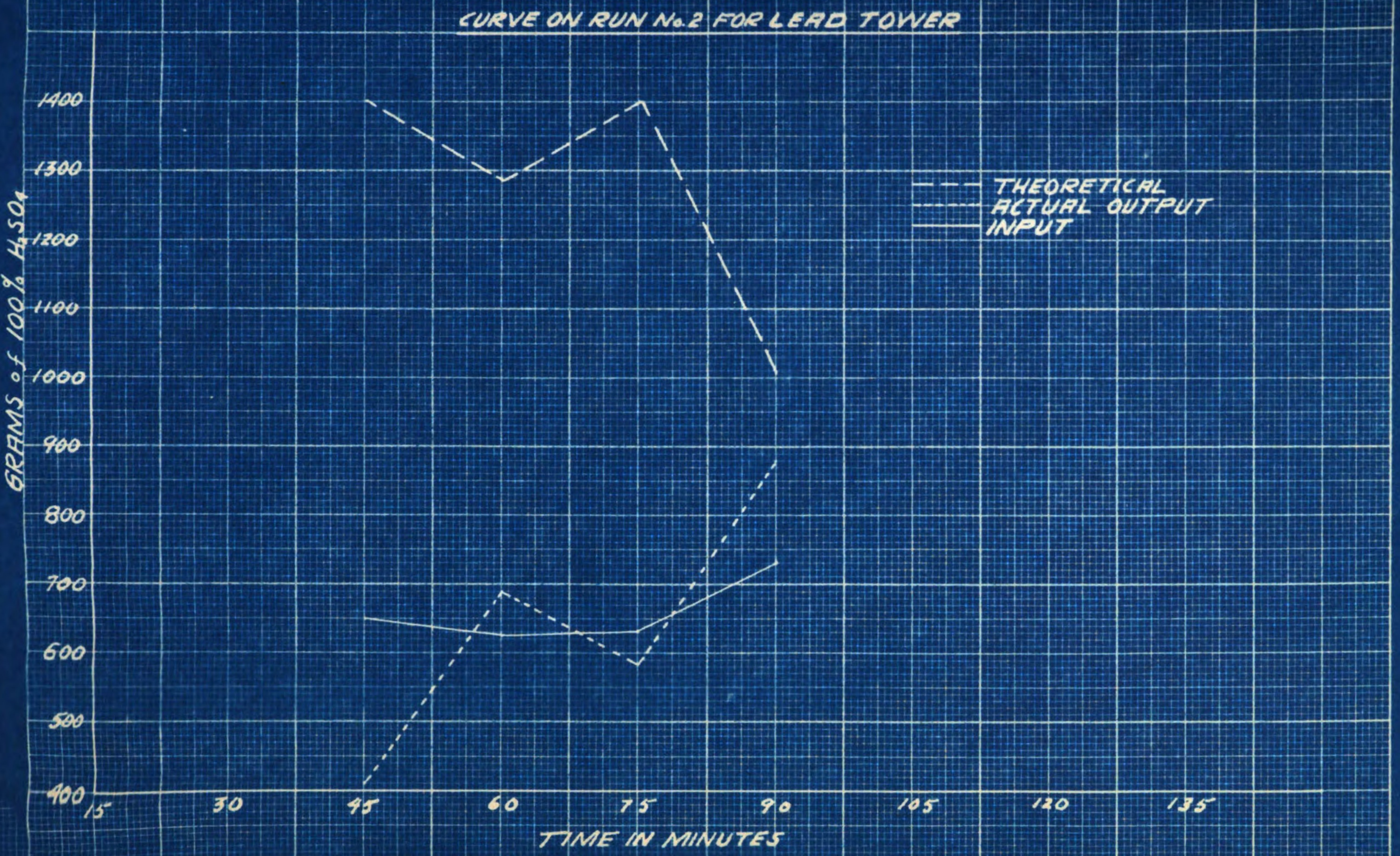
[1] Drawn off the following day.

Run No. 2 on Lead Tower

100 % H_2SO_4 Calculated for 15 Minute Intervals

Time in Min.	100% H_2SO_4 in Grams			
	Gross Output	Input	Net Output	Theoretical
15	387	652		
30	526	665		
45	1060	652	408	1400
60	1320	626	694	1285
75	1220	633	587	1400
90	1620	738	882	1050
105	1110	1029	81	
120	1280	1462		
135	4020[1]	1410	3610	
Total	12,543	7,867	4,676	5,135

[1] Drawn off the following day.



CURVE ON RUN No.2 FOR LEAD TOWER

Discussion of Run No. 2 for the lead tower.

The efficiency of this run was 91% or 1% less than in run No. 1. Yet this is satisfactory for it seems that the metallic tower produces uniform results for the two runs thus far made. Only a slight increase in capacity output was gained per hour which is somewhat encouraging from the standpoint of commercial output. More runs will be made toward the latter mentioned point.

The curves are not as expected to be since the entire results could not be plotted. From this consideration much cannot be relied upon them.

The run as a whole was promising in many respects. The outlet air temperatures were lower by 12° C. which is quite a drop for the size of tower employed. A bigger difference in cooling water temperatures was observed since an equal amount of water was passed in both cases. Part of the increase in difference may be due to use of more input acids whose heat of dilution was correspondingly greater in comparison with the first run. By closely comparing the amount of dilute acid passed in each case it seems that most of heat removed was due to heat of formation rather than heat of dilution.

In final conclusion the metallic tower to study with for reduction of cubic volume per pound sulphur burned in 24 hours is turning out better as matched with the tile tower.

Run No. 3 on Lead Tower

Data Taken on the Lead Tower in Pilot Plant Operation

Time in Min.	Temperature °Centigrade					Inches	Input Acids		Vol. in Cu. Ft.		Press Lbs. per Sq. In.		Gross Output Product		
	Inlet SO_2	Inlet Air	Outlet Air	Cooling Water Inlet	Cooling Water Outlet	Cooling H_2O	Sp. Gr. 1.08	Sp. Gr. 1.70	SO_2	Air	SO_2	Air	Temp. °C	Sp. Gr.	Volume in cc.
0							0.5	1.2							
15							0.8	1.9							
30							1.0	2.6							
45	22	22	25	13	13	0.5	1.5	3.4	0	0	1.6	8.1	30	1.68	260
60	18	14	50	13	16	2.5	2.0	4.0	6.25	25	1.6	8.2	35	1.65	700
75	17	14	50	13	18	4.5	2.5	4.5	12.50	51	1.7	8.0	40	1.55	1195
90	17	14	60	13	18	7.0	3.0	5.0	18.75	78	1.6	8.1	40	1.60	1080
105	17	14	40	13	19	9.5	3.5	5.5	25.00	103	1.7	8.0	45	1.62	1050
120	17	14	35	13	19	11.7	4.1	6.1	31.25	127	1.7	8.2	45	1.60	1050
135	17	14	30	13	18	13.7	4.8	6.7	37.50	152	1.5	8.2	45	1.60	1040
150	17	14	35	13	19	15.7	5.5	7.2	43.75	175	1.6	8.1	50	1.60	1275
165	17	14	35	13	19	17.7	5.8	7.7	50.0	201	1.6	8.1	50	1.61	1115
180				13	19	19.7							55	1.61	1025
195				13	18	22.0							55	1.60	1150
210				13	18	24.0							55	1.60	1150
225				13	17	26.0							55	1.60	1405[1]
Aver.	17	14	42.	13	18.3	25.5	5.3	6.5	50.0	201	1.6	8.1	49.1	1.61	13495

Input - 500 cc. of 1.39 Sp. Gr. HNO_3, Temp. 22° C.

[1] Drawn off the following day.

Run No. 3 on Lead Tower

100 % H_2SO_4 Calculated for 15 Minute Intervals

Time in Min.	100% H_2SO_4 in Grams			
	Gross Output	Input	Net Output	Theoretical
15				
30				
45	332			
60	850	963		
75	1210	950	260	
90	1200	1121	79	
105	1200	855	345	730
120	1180	725	455	730
135	1150	725	425	730
150	1410	725	685	730
165	1253	863	390	730
180	1160	881	279	730
195	1275	751	524	730
210	1275	699	576	730
225	1558[1]			
Total	14871	9258	5613	5840

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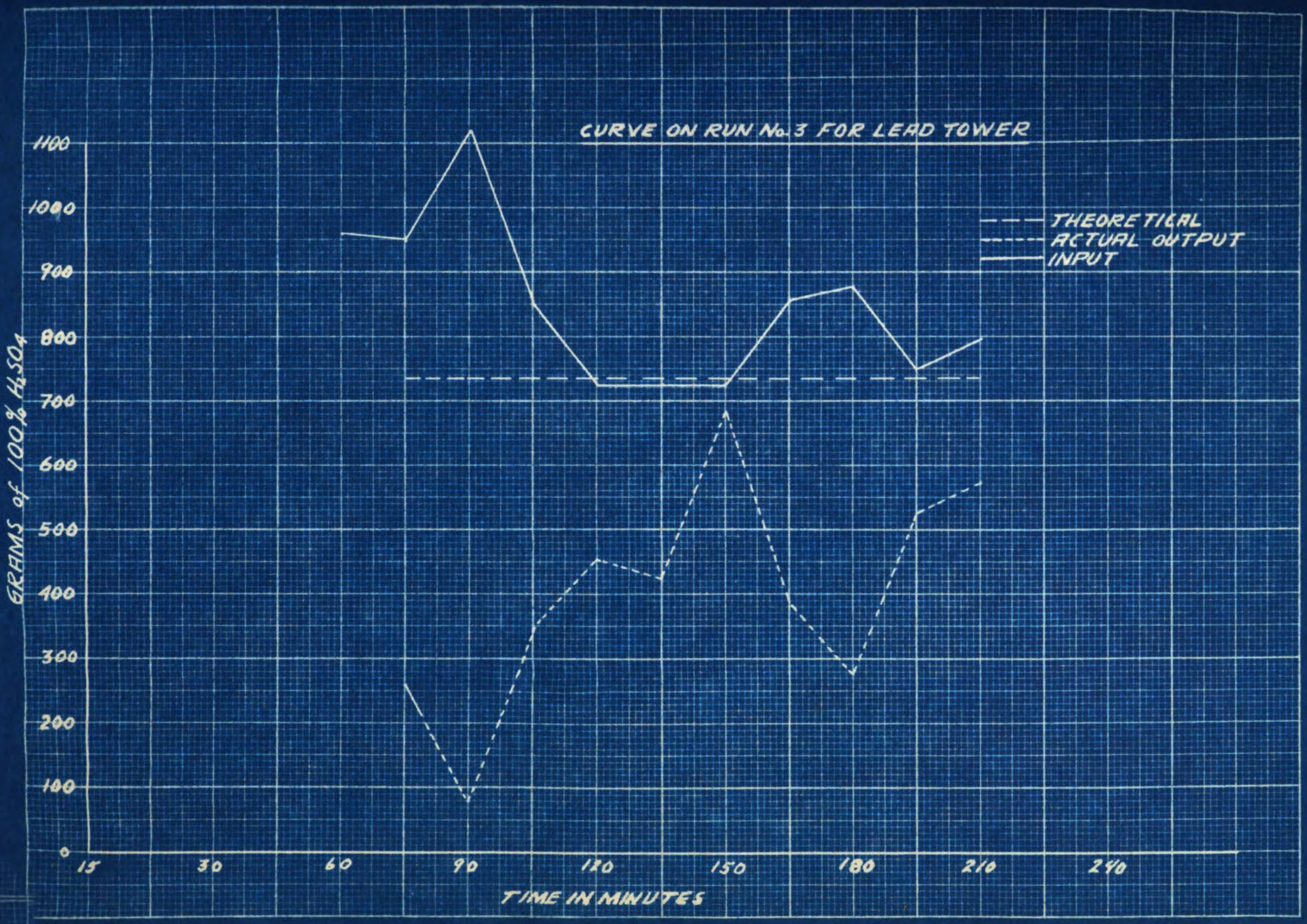
1. Drawn off the following day.



CURVE ON RUN No. 3 FOR LEAD TOWER

Discussion of Run No. 3 for the lead tower.

The efficiency increased over the prior two tests. The curves seem to represent some queer facts. The reasons based on other plottings heretofore are contrary insofar as these curves show it. There appears open for discussion that other factors influence the reactions besides the flow of SO_2 and input acids wetting the packing. Pressures do have some effect through vigorous impinging of gases upon the wetted surfaces. Yet, careful control was used to regulate the admittance of SO_2 and air whereby not only constant supply of gases were maintained but regular impingment of them upon the wet packing as well. Proper temperatures have aided the efficiency, and in observing the data in regard to them, they are satisfactory. The one thing left to consider and that is the packing. It may have held the acids in certain places due to close surfacing of the fine glass wool packing. If this turns out to be the case, then those parts of the tower will be idle.

The output capacity was lower in this run after it was hopeful to increase it. Another run will be made in order to see if it actually can be accomplished, and to increase the efficiency.

One thing worth noticing in this run is that the output specific gravity was practically constant which in commercial conditions is important.

Run No. 4 on Lead Tower

Data Taken on the Lead Tower in Pilot Plant Operation

Time in Min.	Temperature °Centigrade					Inches Cooling H_2O	Input Acids		Vol. in Cu. Ft.		Press Lbs. per Sq. In.		Gross Output Prod.		
	Inlet SO_2	Inlet Air	Outlet Air	Cooling Water Inlet	Cooling Water Outlet		Sp.Gr. 1.08	Sp.Gr. 1.70	SO_2	Air	SO_2	Air	Temp. °C.	Sp. Gr.	Volume in cc.
0							0.50	2.20							
15							1.25	2.70							
30							1.95	3.30							
45				13	13	1.0	2.70	3.8							
60	20	20	28	13	16	2.0	3.4	4.3	35.5	0	0	0	35	1.55	1100
75	14	12	55	13	22	3.25	4.2	4.8	47.0	50	5.0	16.0	35	1.45	875
90	14	12	70	13	24	4.50	5.5	5.3	60.0	102	5.5	17.2	45	1.35	935
105	14	10	60	13	26	5.75	6.0	5.7	72.0	151	5.0	16.8	50	1.40	1220
120	14	10	48	13	27	7.00	7.0	6.1	83.0	204	5.0	17.0	60	1.50	2000
135	14	10	48	13	29	8.25	8.0	6.6	92.0	260	5.5	17.4	60	1.50	1370
150	14	10	35	13	28	9.50		6.9	102.5	312	5.0	17.1	60	1.55	1050
165				13	23	10.75			109.0	358	4.0	17.0	60	1.55	1540
180				13	21	12.00							50	1.60	1300
195				13	18	13.25							45	1.60	1345
210				13	17	14.50							35	1.70	2600[1]
Aver.	14.0	10.6	53.0	13	23.7	13.50	7.5	4.7	73.5	358	5.0	17.0	48.6	1.66	14335

Input 500 cc. of 1.39 Sp. Gr. HNO_3.

[1] Drawn off the following day.

Run No. 4 on Lead Tower

100 % H_2SO_4 Calculated for 15 Minute Intervals

Time in Min.	100% H_2SO_4 in Grams			
	Gross Output	Input	Net Output	Theoretical
0				
15				
30				
45	1100			
60	705	767		
75	575	880		
90	880	773	107	
105	1810	760	1050	1340
120	1240	773	467	1520
135	1050	760	290	1410
150	1540	855	685	1285
165	1440	658	782	1050
180	1490	700	790	1227
195	3440[1]	396	3044	876
Total	17600	7322	8298	8908

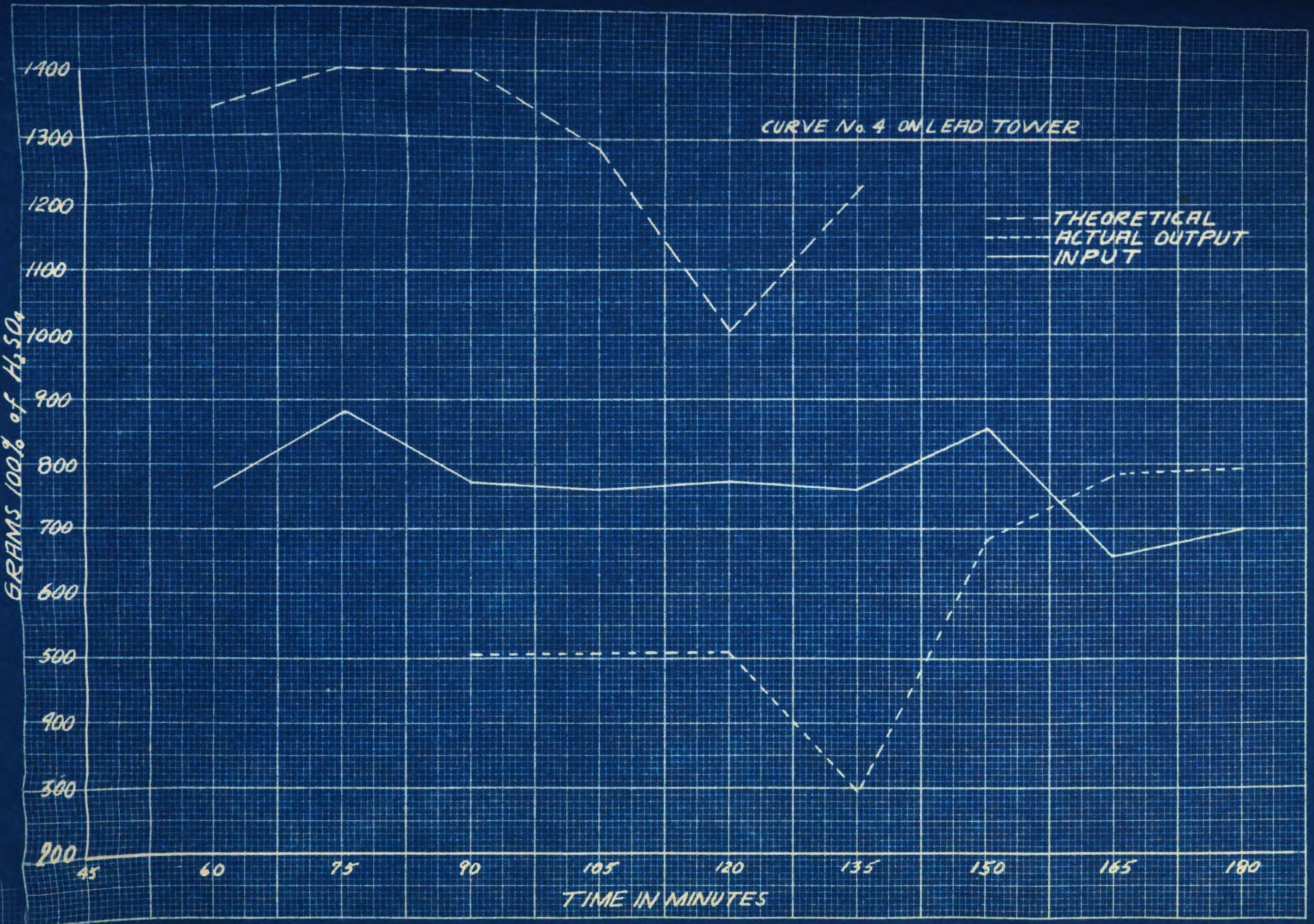
1. Drawn off the following day.

Discussion on Run No. 4 for the lead tower.

The efficiency was good in this test, yet it turned out very good in regard to the amount of input acids. A greater net output exceeded the input basing on 100% sulphuric acid in comparison of all trials. Heretofore, other runs employed more input acid than they showed for net output. Generally in previous runs a larger amount of 60° acid was passed in as compared to the amount of dilute acid where in this case the reversal took place which is more desirous.

Comparison of these four runs it appears that about 45 cubic feet of SO_2 consumed per hour gives the best efficiency. There was a vast difference in cooling water temperatures indicating a large removal of heat. The heat of reaction no doubt was larger than heat of dilution basing it on the amount of sulphuric acid made.

The curves indicate facts that are reliable. The SO_2 curve in this case influenced the newly made acid. In considering what some authorities have said about this fact, yet, it is believed they must have worked with conditions which predominated in Run No. 2 on the lead tower. The tower input acids still show their aid toward the reacting conditions. Temperatures were slightly higher which had some effect on the final efficiency. This defect could only be remedied by working with higher towers in which the water could have a better chance to remove all the available heat.


CURVE No. 4 ON LEAD TOWER
THEORETICAL
ACTUAL OUTPUT
INPUT
GRAMS 100% of H_2SO_4
1400
1300
1200
1100
1000
900
800
700
600
500
400
300
200
45
60
75
90
105
120
135
150
165
180
TIME IN MINUTES

2. Data and Calculated Results.

The following page contains the average data for each run made with the lead tower in pilot plant research.

The next following page has the input and output data and results calculated for each run.

a. Average Data for Each Separate Run Made in Pilot Plant

No. of Run	Time in Hrs.	Temperature °Centigrade					Volume in Cubic Feet		Press lbs. per Sq.In.		Gross Output			Amount Cooling Water Passed	
		Inlet SO_2	Inlet Air	Outlet Air	Cooling Water		SO_2	Air	SO_2	Air	Temp. °C	Sp.Gr.	Volume in cc.	Height Measured in Inches	Calculated in Pounds
					Inlet	Outlet									
1	1.0	22.5	18.7	53.5	13.12	17.5	40.0	132	3.0	9.23	51.5	1.48	11,755	14	910
2	1.0	18.7	13.12	41.2	13.0	19.5	44.0	152	5.97	13.4	41.6	1.54	13,035	14	910
3	2.0	17	14	42.0	13.0	18.3	50.0	201	1.6	8.1	49.1	1.61	13,495	25	1630
4	1.5	14	10.6	53.0	13.0	23.7	73.5	358	5.0	17.0	48.6	1.66	14.335	13.50	880

b. Input and Output Materials and Results Compiled for Each Pilot Plant Run

No. of Run	Time in Hrs.	Input H_2SO_4 Acid						Average Input Sp. Gr.	Gross Output				Grams of 100% H_2SO_4		
		60° Be[1] H_2SO_4			Dilute H_2SO_4										
		Vol.cc.	Wt.Gr.	Sp.Gr.	Vol.cc.	Wt.Gr.	Sp.Gr.		Sp.Gr.	Be[1]	Volume in cc.	Wt. in Grams	Input	Output	Theoretical
1	1.00	4,300	7,300	1.70	4,800	5,180	1.08	1.37	1.48	47	11,755	17,400	6,461	4,309	4,680
2	1.00	7,100	12,100	1.70	4,600	4,970	1.08	1.45	1.54	51	13,035	20,000	7,867	4,676	5,135
3	2.00	6,500	11,060	1.70	5,300	5,715	1.08	1.42	1.61	55	13,495	21,350	9,258	5,613	5,840
4	1.50	4,700	8,000	1.70	7,500	8,100	1.08	1.32	1.66	58	14,335	23,800	7,322	8,298	8,908

Input and Output Materials and Results Continued from Previous Page

Tower Eff. in %	Ratios			Cubic Feet of Tower per lb. of S per 24 Inches	Heat Carried Away by Cooling Water in B.T.U.
	Output to Input Acids	Air to SO_2			
		Theoretical	Actual		
92.0	0.66	3.6	3.3	0.21	7,350
90.0	0.60	3.6	3.5	0.19	10,670
95.0	0.61	3.6	4.0	0.17	15,500
93.4	1.13	3.6	4.8	0.17	17,000

c. Heat Balance - calculated at 0° C and 29" Hg.

Summary Heat Balance of Lead Tower.

INPUT

Before start of chemical reactions.

(1)	Heat content of sat. air	401.0 B.T.U.	1.40%
(2)	Heat content of SO_2	1.9 B.T.U.	0.01%
(3)	Heat content of HNO_3	33.6 B.T.U.	0.12%
(4)	Heat content of Gay-Lussac acid	294.0 B.T.U.	1.04%
(5)	Heat content of dilute acid...........	610.0 B.T.U.	2.16%
	Total before reaction	1,340.5 B.T.U.	

Heat after chemical reactions.

(6)	Heat of formation of H_2SO_4.........	17,480.0 B.T.U.	62.0%
(7)	Heat of dilution ,..................	9,370.0 B.T.U.	33.27%
	Total	28,190.5 B.T.U.	100.00%

OUTPUT

(1)	Heat carried out by air	3,960.0 B.T.U.	14.57%
(2)	Heat carried down by gross acid ...	1,930.0 B.T.U.	6.84%
(3)	Heat of concentration	4,830.0 B.T.U.	16.63%
(4)	Heat removed by H_2O	17,000.0 B.T.U.	60.20%
(5)	Heat absorbed by $NO \rightarrow NO_2$	312.0 B.T.U.	1.15%
(6)	Heat lost by radiation	158.5 B.T.U.	0.61%
	Total	28,190.5 B.T.U.	100.00%

d. Material Balance.

INPUT

(1)	Saturated air	28.4 lbs.	36.5%
(2)	SO_2	12.4 lbs.	16.0%
(3)	HNO_3 acid	1.5 lbs.	1.9%
(4)	Gay-Lussac acid	17.6 lbs.	22.3%
(5)	Dilute acid	17.8 lbs.	23.3%
	Total	77.6 lbs.	100.0%

OUTPUT

(1)	Spent air	25.4 lbs.	32.8%
(2)	Recovered HNO_3	1.1 lbs.	1.4%
(3)	Loss of HNO_3 [illegible]	0.4 lbs.	0.5%
(4)	Gay-Lussac acid	17.6 lbs.	22.3%
(5)	Dilute acid	17.8 lbs.	23.3%
(6)	Lbs. of SO_3	14.9 lbs.	19.2%
(7)	Loss	0.4 lbs.	0.5%
	Total	77.6 lbs.	100.0%

3. Sample Calculations (Run No. 4 on the Lead Tower)

Same set of calculations apply here as those on Run No. 3 made in pilot plant with the tile tower (Page 116)

C. CONCLUSIONS.

In the study of preparation of sulphuric acid with tower methods, it is evident that a metal tower is more suitable in acid manufacturing than tile towers are. It is very true that the latter's life would be longer under working conditions insofar as chemical reactions attack the materials enclosing them. Yet the heat will crack the tile tower whereas the metal one will withstand it nicely. Metal also answers well in conduction of heat which in tower method is of a vital importance. During the last few years certain alloys are capable of warding off acid reaction to a considerable extent. With such metal improvements, it is possible to employ metal towers with a fair degree of assurance.

Comparing the results of both towers in the pilot plant, the lead tower excelled the former in the most important points. One was that a higher and more uniform efficiency was obtained in each successive test. In the last analysis this is highly desirable from the standpoint of engineering research. Not only did the efficiency prove better than the tile tower, but also of the glass tower in laboratory study. It appears that a higher one can yet be procured in industrial conditions, of course using a metal tower of suitable acid resistance.

More comparisons with other factors influencing efficiency appeared more promising upon the use of a metal unit. Notably, the temperatures which can be controlled more easily by proper heat removal of chemical formation. Higher concentrated acids can be also produced. This phase as seen from the heat balance shows that much heat is taken up upon concentration, just as much so as evaporating to concentrate weaker solutions. To produce a higher concentration means passage of larger columes of SO_2 and air, consequently, immediate heat of formation is given off and a tile tower fails in this respect. Pressure affects efficiency insofar as it affects the chemical reactions. At various pressure different intermediate reactions take place of which some are beneficial while others are not. Pressures in turn were influenced by temperatures conditions which can only be controlled more readily in a metal tower by cooling and flow of input gases and air.

The type of packing used was the same in all cases, both laboratory study and pilot plant. It produced ample surface for the gas-liquid phase to work remarkably, only a great deal of resistance was offered by it toward the counterflow of gases. It seemed at times that too much pressure was exerted against the counter flow of acids too, thus relieving uniform wetting throughout the entire tower. This could be remedied by looser packing since in observing the results on"space used per pound of sulphur converted to sulphuric acid per twenty-four hours" could be increased slightly and yet have a lower volume in comparison with actual plants in operation all over the world. Outside of this there is a good possibility of putting such a process to commercial use. And even more-so to make it a continuous process which was not possible under the conditions in pilot plant equipment.

PART FIVE

DESIGN OF COMMERCIAL PLANT CAPACITY
50 TONS OF 53° Be' H_2SO_4 PER DAY

A. DISCUSSION

A plant capacity of 50 tons of 55°Be' acid per 24 hour day will be designed from the results obtained in pilot plant. The information procured in the research work appears to indicate that a tower method for making sulphuric acid could be employed commercially against other plants.

Different types of materials were considered for plant construction. Those which can withstand acid resistance to a fairly good degree, and can readily conduct heat were compared as to cost and length of life, or with least depreciation. After looking over certain alloys which the producers listed, their physical properties were studied, especially those pertaining to density, thermal coefficient of expansion, resistance to sulphuric and nitric acid at various temperatures, and specific heat conductivity.

Physical properties of nichrome alloy showed favorable possibilities to working conditions of tower methods for making sulphuric acid. Its analysis is composed of 12 per cent of copper, 60 per cent of nickel, 15 per cent of chromium and the balance is iron. The melting point of it is $1350^\circ C$ and the tower temperatures will not be over $200^\circ C$, so there is not any danger of heat of reaction melting this alloy. The thermal coefficient of expansion is 0.00017($0-100^\circ C$). Its specific heat of conductivity is 0.136 which is very good in comparison with the other listed alloys. The cost was listed at 76 cents per pound in small lots. This price quotation could be reduced when buying it in larger quantities for construction of a commercial plant. Its resistance to acid corrosion is recommended very favorable to H_2SO_4 and HNO_3.

Duriron can also be used. Its thermal coefficient of expansion per degree Fahrenheit is 0.0000156 which is considerably less than nichrome.

Its melting point is 2300 degrees Fahrenheit. This physical characteristic offers assurance against tower heat. Specific heat conductivity is 0.11 which is a little less than nichrome and still higher in comparison with others. Its price was listed at 67 cents a pound in small equipment lots weighing 150 pounds, yet if such material were bought in ton amounts, the price would be much lower. Density of duriron is 440 pounds per cubic foot. Resistance to corrosion of sulphuric acid (67 per cent) was tested and a depth of 0.00003 inches per year was penetrated. Resistance to corrosion of nitric acid (25 per cent) was 0.00002 inches deep per year. In final comparison duriron out-weighs nichrome in all respects except that it is easier to machine the latter because the first is extremely hard. Its analysis is 14 per cent silicon and the balance is iron.

If a tower were constructed of either nichrome or duriron, the cost would be very high. Thus lead was also considered which is much cheaper and costs about $4.10 per hundred pounds. In comparing its physical properties whose thermal coefficient of expansion is 0.00029 per degree C, this factor can readily match those of the two alloys mentioned above. The melting point of lead is between 325 and 327 degrees C which is considerably lower than duriron or nichrome and consequently less advantageous to tower heat. Its specific heat of conductivity is only 0.0302(0-100°C) which is a vital point to consider since heat must be quickly removed if a commercial tower is to function properly. As to acid corrosion, lead would not compare with nichrome or duriron. It could be used, but precise care must be exercised whereas not as much with the other two. By reducing this uneasiness less risk would be involved in running a commercial plant.

A commercial plant will be designed according to relative capacity of producing 50 tons of 53° acid per 24 hours.

B. PLANT DESIGN.

1. Size of Sulphur Burner.

a. Dimensions of Cylinder (A).

Refer to blue print No. 4 for each unit design and description. A Glen Falls type of burner will be used. Such a device requires 24 cubic feet per 24 hours to burn one ton of sulphur. Not quite 11 tons of sulphur are necessary to make 50 tons of 53° acid. Since the efficiency is about 95 per cent, 11 tons of sulphur will just about make 50 tons of 53° acid.

Multiplying:

24 X 11 = 264 cu.ft. are necessary to burn 11 tons of sulphur per 24 hours. Twenty feet will be the length of the cylinder for the Glen Falls sulphur burner.

Dividing:

264 ÷ 20 = 13.2 Sq. Ft., cross sectional area of the cylinder. 13 ÷ 3.1416 = 4.2 ft, the radius squared.

The square root of 4.2 = 2^+ feet, radius of cylinder.

The dimensions of the (A) will be 4 feet in diameter and 20 feet long.

b. Combustion Chamber (B).

Thirty cubic feet of chamber volume are necessary to burn one ton of sublimed sulphur from (A) per twenty four hours.

Multiplying:

30 X 11 = 330 cu. ft. to completely burn the sulphur from (A).

The length of the combustion chamber will be 10 feet.

330 ÷ 10 = 33 sq. ft., cross sectional area of combustion chamber.

Final dimensions will be 5 feet wide, 6.5 feet high and 10 feet long.

A steel cylinder will take care of sulphur as a material structure. The combustion chamber will have to be made out of fire brick whose radiation factor is small to prevent loss of heat. It is desirous to use all the heat available for units (C) and (M). (Refer to blue print no. 4).

2. Design of Unit (C) to Remove NO_2 from 60° Acid Which is Discharged from (D) into (C). Eleven Tons of Sulphur Burned will Produce as follows:

$$S + 1\tfrac{1}{2}O_2 - H_2O \rightarrow H_2SO_4$$

Mol. Wt. of S, 34 grams.

Mol. Wt. of H_2SO_4, 98 grams.

Then,

98 ÷ 32 X 11 = 33.65 Tons of 100 per cent H_2SO_4.

Sixty degree acid contains 78 per cent H_2SO_4 and 22 per cent water.

33.65 ÷ 0.78 = 43 Tons of 60° acid per 24 hours.

43 ÷ 24 = 1.8 Tons of 60° acid per hour.

The size of (C) will be based on holding 1.8 tons of 60° acid while being heated to drive off the oxides of nitrogen. One cubic foot of 60° acid at 20° C weighs 100 pounds.

1.8 X 2000 ÷ 100 = 36 cu. ft., or volume to hold 1.8 tons of acid per hour. Allowing for expansion of 60° acid from 20° to 80°C, also for space above the liquid surface of 60° acid in (C) because oxides of nitrogen will have to be carried back in to (E) with air bubbling through the 60° acid, so by doubling 36 cu. ft. will be a sufficient volume to handle the situation.

36 X 2 = 72 cu. ft., vol. of (C).

Eight feet will be the length of (C).

72 ÷ 8 = 9 sq. ft., or cross sectional area of (C).

Two feet wide, 4.5 feet high and 8 feet long will be the dimensions of (C).

3. Design of Acid Tower (E).

From the results obtained on the lead tower in pilot plant 15.6 pounds of 60° acid were produced in one hour per 2.25 cu. ft. of tower space. Weight of acid produced per day would be 375 pounds per 24 hours of 2.25 cu. ft. of tower capacity.

Volume required to manufacture 43 tons of 60° acid,

375 2.25 (43 X 2000) : X

X 2.25 X 86000 ÷ 375 = 520 cu. ft.

Tower (E) will be a cell packed design containing smaller towers (F) enclosed in one large unit (E) with water circulating between the walls of towers (F). The dimensions of towers (F) will be 2 feet in diameter and 40 feet high. Each tower (F) will contain the bottom portion as the Glover tower, 15 feet high, and the top part, the Gay-Lussac tower 25 feet high. Owing to change of packing from glass wool to coarser packing about the size of glass beads, the Gay-Lussac tower will not be considered only the Glover tower whose height is 15 feet.

Number of towers (F) that will occupy 520 cu. ft. of volume.

1^2 X 3.1416 X 15 = 47 cu. ft., Vol. of (F).

520 ÷ 47 = 11.05 towers (F), 11 towers will be enough to make 43 tons of 60° acid.

a. Cooling Space Between Towers (F) in (E).

Amount of heat to be removed per minute.

(1) Heat due to formation of converted SO_2 to H_2SO_4.

120,450 cal. per mol. of H_2SO_4.

120, 450 X 0.004 = 481.8 B.T.U. per mol. H_2SO_4.

481.8 X 453 ÷ 98 = 2230 B.T.U. per lb. H_2SO_4.

60 lbs. of 60° acid will be made per minute.

60° acid contains 78 per cent H_2SO_4.

60 X 0.78 = 44.8 lbs. of 100% H_2SO_4 per minute.

44.8 X 2230 = 110,556 B.T.U. given off per minute when 44.8 lbs. of H_2SO_4 will be made from SO_2.

(2) Heat of dilution.

Heat of dilution can be determined as the difference of heat of solution at the initial concentration from the final concentration.

60 lbs. of 60° acid and 60 lbs. of water are required to make 60 lbs. of 60° acid per minute.

So by calculating the average Sp. Gr. of input Gay-Lussac acid and the water, it can be considered as the initial concentration.

60 X 453 = 27200 grams of 60° acid.

60 X 453 = 27200 grams of water.

27200 ÷ 1.6 = 17000 cc. of 60° acid.

27200 ÷ 1.0 = 27200 cc. of water.

Adding:

27200 ÷ 27200 = 54400 grams of acid and water.

27200 ÷ 17000 = 44200 cc. of acid and water.

Dividing:

54400 ÷ 17000 = 1.2 Sp. Gr. of input acid and water.

1.6 sp. gr., initial concentration.

1.2 sp. gr., final " .

1.6 sp. gr., 78 % H_2SO_4 - 22% H_2O.

1.2 sp. gr., 28 % H_2SO_4 - 72% H_2O.

22 ÷ 18 = 1.220 mols. of H_2O.

78 ÷ 98 = 0.796 mols. of H_2SO_4.

72 ÷ 18 = 4.000 mols. of H_2O.

28 ÷ 98 = 0.359 mols. of H_2SO_4.

1.220 ÷ 0.796 = 1.53 mols. of H_2O per mol. of H_2SO_4.

4000 ÷ 0.359 = 11.45 mols. of H_2O per mol. of H_2SO_4.

From curves on integral heats of solution of H_2SO_4 in water at 18°C for,

11.45 mols of water per mol of H_2SO_4, 15,500 cal. per mol.

1.53 mols of water per mol of H_2SO_4, 8,500 cal. per.mol.

Subtracting:

15,500 - 8,500 = 7,000 cal. per mol.

46.7 lbs. of H_2SO_4 will be diluted.

46.7 X 453 ÷ 98 X 7000 = 1,515,000 cal. per min.

(3) Heat taken up when the diluted acid will be built to 1.6 sp. gr. on final concentration as it leaves the tower (E) at (D) (Refer to blue print no. 4). It will be the same as in (2) above but 46.7 lbs., more of H_2SO_4 will be made. So there will be twice as much heat taken up as given off upon dilution, or 12,000 B.T.U. per minute.

(4) Total Heat To Be Removed From Towers (F).

Heat due to chemical **formation**, 110,556 B.T.U. per min.
Heat due to dilution formation, 6,000 B.T.U. per min.
Total positive heat formation, 116,556 B.T.U. per min.
Heat taken up by concentration,
of 1.2 sp.gr. acid to 1.6 sp. gr. - 12,000 B.T.U. per min.
Net total . . . . 104,556 B.T.U. per min.

(5) Area of Surface for Conduction of Heat From the towers (F) calculated in (4) above. As soon as the heat is conducted to the outside of towers (F), the circulating water will remove it from there. Eleven towers with 2 foot diameters and 40 feet high compose the total conduction area.

3.1416 dh — the surface area of each tower (F).
3.1416 X 2 X 40 250 sq. ft.
250 X 11 — 2750 sq. ft., or total conduction area.
Thickness of tower walls will be half an inch.

(6) Heat Transfer of Towers (F).

Conduction surface, 250 sq. ft. for each tower (F).

Conducting surface, 0.5 inches thick.

Inside temperature, 122°F.

Outside temperature, 80°F.

$Q\ \ K(t_2 - t_1)\ at - d$

Q, B.T.U.

K, 0.11 for Duriron, or specific conductivity factor.

t_2 inside temperature, 122°F.

t_1, outside temperature 80°F.

a, area, 250 sq.ft.

T, time in seconds.

d, thickness, 0.04 ft.

Q = 0.11 (122 - 80)250 X 60 ÷ .04

Q = 0.11 (42) 250 X 60 ÷ .04

Q 6,870 B.T.U. per min. heat transfer of each tower (F).

11 X 6,870 = 75,570 B.T.U. per minute or total heat transfer.

Per cent efficiency of heat transfer.

75,730 ÷ 104,556 X 100 = 72.5

(7) Gallons of Water Necessary to Remove the Heat Transferred in (6) per minute.

Inlet temperature of cooling water 70°F.

Outlet temperature of cooling water 86°F.

$Q = (t_2 - t_1)\ W$

Q, 75570 B.T.U. from (6).

t_2, 86° F.

t_1, 70° F.

W, weight of water in pounds.

$W = Q \div (t_2 - t)$.

$W = 75,570 - (86 - 70)$

$= 75,570 \div 16$

$= 4,740$ lbs. of water per minute.

(2) Space between towers (F) of unit (E).

The inter space will be based on the volume that 4,740 lbs. of water will occupy at the average temperature of inlet water or 77°F.

Cubic feet of water at 77°F weighs 62.2 lbs.

Dividing:

$4,740 \div 62.2 = 76^{+}$ cu. ft.

$76^{+} \div 11 = 7$ cu. ft. of cooling vol. per tower (F) per min.

Inside vol. of tower (F), 126 cu. ft.

Outside vol. of tower (F), 136 cu. ft. inculding wall thickness.

143 cu. ft. is the volume occupied by the tower (F) and water thickness sourrounding (F)

$143 = 3.1416\ R^2 h$

$R^2 = 143 \div 3.1416\ h$

$R^2 = 143 \div (40 \times 3.1416)$

$R^2 = 1.14$ feet

Square root of $1.14 = 1.07$ feet or radius.

$1.07 - 1.04 = 0.03$ feet

$0.03 \times 12 = 0.36$ inches or thickness of water circulating around (F).

Due to some heat that will be carried into (E) from (C) it will be safe to make the space 4 inches between 2 foot towers (F).

b. Design of (D) and (K) of tower (E).

In D the gross acids will drain into from each respective tower (F) and (E). A two foot depth appears to be sufficient basing on the amount of acid that will drain into (D) each minute. (K) is the space above (D) for gases to enter the towers (F).

c. Design of (J) and (L) and (E).

Section (J) will be the reservoir which will feed the water lines of each tower (F). Section (L) will be the reservoir which will feed the Gay-Lussac acid into the Gay-Lussac towers of (F). (J) and (K) will be six inches high or 1 foot in all.

d. Diameter of Tower (E).

There are 11 small towers (F) to be equally spaced 4 inches apart. By starting with three in the center and the remaining 8 to surround these about a circumference is about the best selection that can be made. An equilateral triangle whose sides are 2.5 feet long with the apexes as centers of three circles will place the first three towers. Each tower is to be two feet on the inside diameter and allowing half inch thickness for the walls. (Refer to blue print No. 4, the ground floor view). Taking the center of the equilateral triangle as the center of a circle whose radius is 4 feet and drawing this circle will be the circular line where the centers of the remaining 8 towers (F) will be. Equally spacing the 8 towers on this circumference concludes the spacing of 11 towers. Allowing quarter inch thickness of the wall inclosing the 11 small towers constitutes the water-jacket of tower (E). Adding all the radii and allowance for wall thickness (E), the diameter of (E) will be 10.5 feet.

e. .Final Dimensions of (E).

10.5 feet in diameter and 43 feet high.

f. Height of base (M) upon which the tower (E) will rest.

Considering the height of (G) and (C) so that the 60° acid can flow from (D) to (C) and from (C) to (G) by flow of gravity will be the same height of the base (M) which is seven feet.

g. Final height of (E) from the ground floor.

Forty-three is the height of (F) and seven feet the base (M) supporting (E).

Adding:

43 + 7 = 50 feet, or total height.

4. Nitrogen Dioxide Generator (O).

Basing on a 6 per cent loss of HNO_3 per ton of 60° acid made.

Ton of 60° acid contains 1560 pounds H_2SO_4.

Amount of NO_2 to convert enough SO_2 to SO_3 to make 1560 H_2SO_4.

$$NO_2 + SO_2 = SO_3 + NO$$

$$SO_3 + H_2O = H_2SO_4$$

Mol. wt. of NO_2. 46 grams

Mol. wt. of SO_3, 80 grams.

Mol. wt. of H_2SO_4. 98 grams.

1560 X (80 ÷ 98) = 1273 lbs. of SO_3.

46 : X = 80 : 1273

X = 1273 X 46 ÷ 80

X = 725 lbs. of NO_2

$2NaNO_3 + H_2SO_4 \rightarrow 2HNO_3 + Na_2SO_4$

$2HNO_3 + \text{heat} \rightarrow 2NO_2 + H_2O + \frac{1}{2} O_2$

46 : 725 = 63 : X

X = 725 X 63 ÷ 46 = 993 lbs. of HNO_3.

63 : 993 = 85 : X

X = 993 X 85 ÷ 63 = 1340 lbs. of $NaNO_3$.

1340 X .06 = 80 lbs. of $NaNO_3$.

But 1.8 tons of 60° acid will be made per hour.

1.8 X 80 = 144 lbs. of $NaNO_3$ must be fed each hour to make up for the loss.

Weight of H_2SO_4 used with $NaNO_3$.

Mol. weight of HNO_3, 63 grams.

Mol. weight of H_2SO_4, 98 grams.

Mol. weight of $NaNO_3$, 85 grams.

85 : 80 = 63 : X

X = 80 X 63 ÷ 85 = 60 lbs. of HNO_3.

98 : X = 2(63) : 60

X = 98 X 60 ÷ 126 = 47 lbs. of H_2SO_4.

47 X 1.8 = 85 lbs. of H_2SO_4 / hour or amount required each hour into (C).

Density of H_2SO_4, 1.83.

Density of $NaNO_3$, 2.30

Weight of $NaNO_3$, 144 lbs.

Weight of H_2SO_4, 85 lbs.

$(144 \div 62.5) \div 2.3 = 1.00$ cu. ft. that $NaNO_3$ will occupy.

$2.3 + 0.75 = 1.75$ cu. ft. or volume that both $NaNO_3$ and H_2SO_4 will take up.

A pan imbedded in (E) about six inches will make up the bottom of (C).

Volume $= 3.1416\ r^2h$

$1.75 = 3.1416\ r^2\ 0.5$

$r^2 = 1.75 \div (3.1416 \times 0.5)$

$r^2 = 1.11$

$r = 1.05$ feet, or radius of (O).

Allowing for air and evolved NO_2, the deminsions of (C) will be one foot high and 2.5 feet in diameter.

5. Design of tank (G).

Each minute tank (G) will receive 134 pounds of 60 acid from (C). $134 \div (62.5 \times 1.6) = 1.34$ cu. ft. or theoretical volume of (G).

Allowing for the expansion of the 60° acid from (C), a tank one foot wide, one foot high and two feet long will discharge 134 pounds per minute. Half of the acid will be pumped back into (L) on top of tower (E). The remaining half is to be removed for dilution from 60° to 53° acid.

6. Unit (M).

22 tons of SO_2 will be made in 24 hours.

$22 \times 2000 \div (24 \times 60) = 30$ lbs. of SO_2 / minute.

At 70° F, 30 lbs. of SO_2 are soluble in 300 lbs. of water at 20°C. 300 + 30 = 330 lbs. of H_2SO_3 and water will be discharged into (M) every minute from tower (N).

330 ÷ 63.2 = 5.2 cu. ft. volume discharge from (N).

It would be better to design (M) on an hour basis for capacity of (M), because when (M) is once filled with H_2SO_3 and water a small amount discharged every minute into it would quickly lose its SO_2 due to coming in contact with large amount of heat maintained in a large body of liquid in (M).

Then,

5.2 x 60 = 312 cu. ft., volume of (M).

Ten feet will be the length of (M).

312 ÷ 10 = 31.2 sq. ft. or cross sectional area.

Final deminsion of (M) will be four feet high, eight feet wide and ten feet long.

7. Design of absorption tower for SO_2 (N).

330 lbs. of H_2SO_3 and water will be discharged from the tower (N) every minute.

300 lbs. of water will be sprayed over the packing of (N).

30 lbs. of SO_2 will enter (N) through the bottom

60 lbs. of nitrogen will enter through the bottom of (N)

Thus there are 3oo lbs. of counter flow of liquid to 90 lbs. of gases passing upwardly.

It is assumed that an absorption tower of 100 cubic feet of volume with half-inch packing will handle 90 lbs.of gases and

and 300 lbs. of water.

20 feet will be the height of (M).

100 + 20 = 5 square feet or cross sectional area of tower (M)

Area = 3.1416 r^2

R^2 = A ÷ 3.1416

R^2 = 5 ÷ 3.1416

R^2 = 1.6

R = 1.27

Final inside dimensions of (M), 2.54 feet in diameter and 20 feet high.

C. Operation of H_2SO_4 Plant.

Sulphur is fed at one end of the sulphur burner by the conveyer (R) which fills the hopper((S). A worm-feed mechanism feeds the sulphur into the rotating cylinder (A). At the same end that sulphur is fed, primary air is also fed. The heat of combustion melts the sulphur within the cylinder (A). The rotation of the cylinder distributes this sulphur in a thin film on the inside of the apparatus, and also causes it to shower in drops through the hot gas. The discharge end of the cylinder (A) fits into a combustion chamber (B), and secondary air passes in to burn the sublimed sulphur. The following reaction takes place in the sulphur burner:

1. $S + O_2$ — SO_2 + heat

The hot gases, SO_2 and N_2, leave the combustion chamber (B) and pass through a coiled flue line in (C). As the hot gases pass through coil, portion of their heat is lost by transference into the 60° acid and dissolved NO_2 which surround the coil. The temperature of the hot gases is about 900°F and when they leave (C), the temperature is reduced to about 380°F. Next, the hot gases leave (C) with a temperature of 380°F and enter another coil in (H) where some more of their heat is transferred into the liquid containing dissolved SO_2 from the absorption tower (N). In (H) the heat of the hot gases is reduced to 200° F and from there they pass through the coolers (T) and finally are cooled to about 70°F. Leaving (T) the gases enter at the bottom of the absorption tower (N). They pass upwardly through the packing which is composed of half-inch packing material. The concurrent flow of water passes downward and absorbs the SO_2, thus separating it from N_2 which passes out through the top unabsorbed. As the water absorbs the SO_2, H_2SO_3 acid is formed with it.

2. $H_2SO - SO_2 \quad H_2SO_3$

H_2SO_3 and water collect at the bottom of (N) and pass as such over a suitable pipe line into (H). Thus (H) is filled with the sulphurous liquid from (N). As the hot gases from (C) pass through the coil in (H), the SO_2 is driven out of solution and carried out by the air bubbling through the liquid surrounding the coil in (H). The water leaving (H) is deprived of its SO_2 and enters the coolers (U) where its temperature is reduced from 200°F to 70°F before being pumped back into (N) by the centrifugal pump (V). The water pumped in (N) preforms its job over again in absorbing more SO_2.

Going back to (H) where the SO_2 was released by heat and air bubbling through the liquid, SO_2 is carried over a flue line into the tower (E) at the bottom (K). Just before the SO_2 and air enter the tower (E) they pass through a perforated pipe system (in K) which distributes an equal amount of SO_2 and air into each respective small towers (F) which are enclosed in (E).

NO_2 is generated in (C) from $NaNO_3$ and H_2SO_4. A suitable pan containing enough $NaNO_3$ and H_2SO_4 is heated by being imbedded in the top of the combustion chamber (D). The heat of combustion between sulphur and air heats the pan (C) and the following reactions take place in (C) --

3. $NaNO_3 + H_2SO_4 \longrightarrow NaHSO_4 + HNO_3$
4. $NaNO_3 + NaHSO_4 \longrightarrow NaSO_4 + HNO_3$
5. $2HNO_3 + \text{heat} \longrightarrow 2NO_2 + H_2O + \frac{1}{2}O_2$

The NO_2, water vapor, and oxygen are carried into (E) by air. Now as the NO_2 passes into each small tower (F) with the SO_2 coming over the pipe line from (H), both enter the oxidation zone (AA) of the Glover tower of (F), the following take place between SO_2 and NO_2.--

6. $SO_2 + NO_2 \longrightarrow SO_3 + NO + \text{heat}$.

The NO is carried up by the air through the packing of (F) leaving the SO_3 which is absorbed. As the NO passes upwardly, it is oxidized in H_2SO_4 to NO_2 in the presence of air. After the NO_2 leaves the Glover tower (AA), it keeps passing up into the Gay-Lussac tower (BB) where it is showered with 60° acid. The 60° acid makes its way in from (L) which feeds some sort of a sprayer mechanism in each tower (F) and in this fashion the 60° acid showers over the packing of the Gay-Lussac tower (BB) in (F), and the following reaction results --

7. $2NO_2 + H_2SO_4 \longrightarrow SO_5NH + HNO_3$

The spent air which contains practically N_2 escapes at the top of (E) as shown by the arrow pointing to N_2.

The SO_5NH, HNO_3 and H_2SO_4 trickle through the Gay-Lussac tower (BB) and pass into the Glover tower (AA). In the oxidation zone of the Glover tower the oxides are released and the H_2SO_4 collects in (D) of (E) from where it drains over the suitable line into (C).

Going back to reaction 6, the SO_3 is absorbed by the water which comes down the water line (Z). The water is fed in each water line (Z) from (J). This reaction results when water and SO_3 react —

8. $SO_3 + H_2O \longrightarrow H_2SO_4$ + heat.

While reaction 8 is taking place SO_5NH is also diluted by the water to release the NO_2. The made H_2SO_4 according to reaction 8 trickles down into D of tower (E). Since the tower was designed to make 60° acid, oxides of nitrogen will be absorbed by it and carry them into D. Such concentration of acid is desirous for the Gay-Lussac tower since half of the made acid has to be pumped back into the Gay-Lussac tower (BB). If 53° acid were made, then half of it would have to be concentrated to 60° strength. In the end it is cheaper to dilute acids than to concentrate them.

To remove the oxides of nitrogen which happen to be dissolved in the 60° acid collected in (D) of tower (E), the unit (C) was designed. 60° acid from (D) feeds (C) and the heat of the gases passing from (E) through (C) drive off the oxides of nitrogen because they can be liberated by heat. Since the temperature of the hot gases from (B) is 900° F, all the oxides of nitrogen will be released without much effort. The air is introduced to bubble through the 60° acid in (C) so that final assurance can be depended upon the bubbling of air through the acid to remove the NO_2. Air also conveys the NO_2

back in the tower (E) where it is used again as shown by reaction 6. The NO_2 and air are distributed evenly under all the small towers (F) by a perforated pipe system.

The 60° acid free from oxides of nitrogen drains at the bottom of (C) into a recepticle (G). Half of the 60° acid made per minute is pumped back on top of tower (E) in (L) from where it is fed into the 60° acid distributors of each Gay-Lussac tower (BB) of tower (F). A centrifugal pump is used to pump the 60° acid from (G) into (L). The remaining half of the 60° acid in (G) is drawn off to be diluted to 53° acid at which concentration it will be stored for marketing. This concludes the operation of the plant, and it is a continuous process.

D. MATERIAL BALANCE

INPUT

1. Sulphur.

50 tons of 53° Be' acid will be made / day.

Mol. weight of sulphur, 32 grams.

Mol. weight of H_2SO_4, 98 grams.

53° Be' acid contains 67% H_2SO_4.

Multiplying and dividing,

$50 \times 0.67 \times 32 \div 98 = 11$ tons of sulphur.

2. Air (Dry)

a. Input into sulphur burner.

$S + O_2 \rightarrow SO_2$

Mol. weight of S, 32 grams.

Mol. weight of SO_2, 64 grams.

$32 : 11 = 64 : X$

$X = 64 \times 11 \div 32 = 22$ tons of SO_2.

Air contains 21% of O_2 by volume and 79% of N_2.

11 tons of O_2 were consumed by the sulphur.

11 .21 52 tons of air.

b. Input in tower (E) for converting SO_2 to SO_3.

This would be half as much as in a., or 5.5 tons.

c. Total.

$21 + 52 = 73$ tons or theoretical amount.

Theoretical ratio is 3.62 of air to sulphur.

Actual ratio of 4 to 1 is sufficient.

$73 \times 4 \div 3.62 = 81$ tons of air.

3. Weight of water to produce 50 tons of 53° acid from 11 tons of sulphur.

22 tons of SO_2 made in sulphur burner.

Mol. weight of SO_3, 80 grams.

Mol. weight of SO_2, 64 grams.

$SO_2 + \frac{1}{2}O_2 \rightarrow SO_3$

$22 \times 80 \div 64 = 27.5$ tons of SO_3 made in tower (E).

50 tons of 53° Be' acid will be produced / 24 hours.

$50 - 27.5 = 22.5$ tons of water.

4. $NaNO_3$, basing only the 6% loss / ton of 60° Be' acid.

Ton of 60° acid contains 1560 lbs. of H_2SO_4.

$NO_2 + SO_2 \longrightarrow SO_3 + NO$

$SO_3 + H_2O \rightarrow H_2SO_4$

Mol. weight of NO_2, 46 grams.

Mol. weight of SO_3, 80 grams.

Mol. weight of H_2SO_4, 98 grams.

$1560 \times 80 \div 98 = 1273$ lbs. of SO_3.

$46 : X = 80 : 1273$

$X = 1273 \times 46 \div 80$

$X = 725$ lbs. of NO_2.

$2NaNO_3 + H_2SO_4 \longrightarrow 2\ HNO_3 + Na_2SO_4$.

$2HNO_3 + \text{heat} \longrightarrow 2NO_2 + H_2O + \frac{1}{2}O_2$

$46 : 725 = 63 : X$

$X = 725 \times 63 \div 46 = 993$ lbs. of HNO_3.

63 : 993 = 85 : X

X = 993 x 85 ÷ 63 = 1340 lbs. of $NaNO_3$.

1340 x .06 = 80 lbs. of $NaNO_3$ / ton of 60° acid.

43 tons of 60° acid will be made.

43 x 80 = 3440 lbs. of $NaNO_3$.

OUTPUT

1. 50 tons of 53° acid.
2. Air minus O_2 content.
    a. 81 tons of air were passed.
    b. 16.5 tons of O_2 were consumed by sulphur.
    c. 81 - 16.5 = 64.5 tons of spent air.
3. Loss of NO_2 as $NaNO_3$, 1.7 tons.

Summary Material Balance

INPUT

1.	Sulphur	11.0 tons.	9.50%
2.	Air	81.0 tons	71.00%
3.	Water	22.5 tons	19.35%
4.	$NaNO_3$	1.7 tons	0.15%
	Total	116.2 tons	100.00%

OUTPUT

1.	53° acid	50.0 tons	43.00%
2.	Spent air	64.5 tons	56.85%
3.	Loss of $NaNO_3$	1.7 tons	0.15%
	Total	116.2 tons	100.00%

E. PRECONSTRUCTION COST ACCOUNTING OF TOWER PLANT.

1. Cost of raw materials / accounting year.

   a. Sulphur.

      11 tons of sulphur are required.

      The price of sulphur is \$18.00 per long ton.

      11 x 18 x 2000 ÷ 2400 = \$165.00 cost of sulphur/day

   b. Amount of $NaNO_3$.

      From the material balance 3440 lbs. of $NaNO_3$ were employed to make up the loss of NO_2.

      Price of $NaNO_3$, \$23.50 / ton.

      23.50 x 3440 ÷ 2000 = \$40.42 cost of $NaNO_3$./day

   c. Total cost for a year's supply of raw materials.

      165. + 40.42 = \$205.42 / day.

      205.4 x 300 = \$61,626.00 per year.

2. Structure building costs.

   a. Foundation and excavation for a building 40 feet wide and 170 feet long.

      170 + 170 + 40 + 40 = 420 feet.

      The foundation will be 2 feet wide and 6 feet deep.

      420 x 2 x 6 = 5040 cu. ft. of excavated dirt.

      5040 ÷ 27 = 187 cu. yds. of excavated dirt.

      (1) Cost of excavating is \$2.00 / cu. yd.

          187 x 2 = \$374.00

      (2) Wooden forms for the foundation.

30¢ / sq. ft. or cost of setting forms.

420 x 6 = 2520 sq. ft.

2520 x 0.3 = $756.00

(3) Concrete pourings.

$10.00 / cu. yd.

187 x $10.00 = $1,870.00 or cost of concrete foundation.

b. Flooring.

Acid-proof tile on concrete base at 70¢ / sq. ft.

170 x 40 = 6800 sq. ft. of flooring.

6800 x .70 = $4760.00

c. Walls and roof.

Dimensions of building are 40 feet wide, 170 feet long and 25 feet high except where tower (E) passes through the roof and extends 25 feet above the roof of the building. The walls will be one foot wide.

(1) Sq. ft. of wall surface,

170 x 25 x 2 = 6800 sq. ft. area of side walls

40 x 25 x 2 = 2000 Sq. ft. area of end walls

Total,

6800 + 2000 = 8800 sq. ft.

Solid common brick wall costs $0.75 / sq. ft. and one foot thick (this includes the windows).

8800 x 0.75 = $6,700.00 (for walls and the windows.)

(2) Roof.

7500 sq. ft. of roof surface.

Tile roof costs 40 cents / sq. ft.

7500 x .40 — $3000.00

d. Cost of lighting equipment.

One 300 watt lamp will light up 100 sq. ft. of floor space.

There are 6,800 sq. ft. of ground floor that will need lighting equipment.

6,800 ÷ 100 = 68 lamps.

Cost of fittings, conduit, wire, panel boards, miscellaneous supplies, fixtures and switches, not including the lamp, is $12.70 / light.

12.70 x 68 — $865.00

e. Miscellaneous ....$5,675.00.

3. Equipment cost.

a. Belt conveyor for sulphur.

Width of belt will be 18 inches.

Belt speed, 2 feet per minute.

Capacity in tons per hour, $\frac{1}{2}$ ton.

Approximate cost $370.00

b. Glen Falls sulphur burner $8,000.00

c. 3 centrifugal pumps.

$200.00, cost of each pump.

" x 3 — $600.00

d. Cost of tower (E).

(1) 11 towers, 2 feet in diameter and 40 feet long. The wall of each tower is one-half an inch thick.

11 x 40 x 3.1416 x 2 x 0.04 — 110 cu. ft.

Cu. ft. of Duriron weighs 440 lbs.

110 x 440 — 48,400 lbs.

Cost of Duriron, 40¢ / lb.

48,400 x .40 = $18,560.00

(2) Casing for tower (E) which is made of $\frac{1}{4}$ inch steel plate. It is 10.5 in diameter and 43 feet high.

10.5 x 3.1416 x 0.02 x 43 = 28.6 cu. ft.

One cu. ft of steel weighs 450 lbs.

28.6 x 450 = 12,750 lbs. at 3¢ / lb.

12,750 x .03 = $383.00

(3) Packing material costs about $1200.00

(4) Inside piping of towers.

Calculated weight, 1475 lbs. of nichrome pipe at 50¢ per pound.

1475 x .50 = $737.00

e. Absorption tower (N).

(1) Acid proof brick will be used for its construction.

Its dimensions are 2.5 in diameter and 20 feet high.

20 x 2.5 x 3.1416 — 157.0 sq. ft. of wall surface.

Cost of acid-proof brick is $1.50 / sq. ft.

157 x 1.50 — $236.00

(2) Packing material.

$240.00 is the estimated price for packing this size tower.

(3) Concrete base.

The dimensions of the base are 3 feet in diameter and 5 feet high.

1.5^2 3.1416 x 5 ÷ 27 = 1.3 cu. yds.

Cost of concrete is $10.00 / cu. yd.

10 x 1.3 = $13.00

(4) Small attachments will cost $200.00

f. Units (C), (C), (G) and (H).

These units will be made if Duriron and the total cost is $2,640.00.

g. Piping, valves, fittings, etc., cost $200.00.

4. Installation cost of equipment.

Based on 10 - 20% of invoice cost of equipment.

Total equipment cost.

a.	Belt conveyor .1	$370.00
b.	Glen Falls sulphur burner	8,000.00
c.	Centrifugal pumps	600.00
d.	Acid tower	219.30
e.	Absorption tower	689.00
f.	Miscellaneous	8,411.00
g.	Storage tanks	10,000.00
	Total	$50,000.00

$50,000.00 x 0.15 = $7500.

5. Total cost H_2SO_4 plant.

a.	Land	$10,000.00
b.	Building	25,000.00
c.	Equipment	50,000.00
d.	Installation cost	7,500.00
	Total fixed cost	$92,500.00

F. Distribution of Plant costs % of selling price*.

Based on one year's operation.

1. Yearly production of 53 Be' acid is 15,000 tons at $12.00 per ton, and the gross sales amount to $180,000.00.

Raw Materials	$61,626.00	34.2%
Direct Labor	8,650.00	4.8%
Fuel, Power, & Water	3,600.00	2.0%
Repairs and Maintenance	1,800.00	1.0%
Depreciation	7,200.00	4.0%
Factory administration & Fixed Charges	18,000.00	10.0%
Taxes and Insurance	5,400.00	3.0%
Distribution and Profits	73,724.00	43.0%
Total	$180,000.00	100.0%

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*Based on Chem. Met. Eng., 39, 2-3 (1932).

G. PRECONSTRUCTION COST ACCOUNTING OF THE CHAMBER PLANT.

1. Raw Materials costs / accounting year.

a. Same as tower method, $31,626.00

2. Structure building costs.

Dimensions of chamber plant building are 100 feet wide, 600 feet long, and 50 feet high.

a. Foundation and excavation.

600 + 600 +100 +100 = 1400 feet or length of excavation.

Excavation will be 2 feet wide and 9 feet deep.

1400 x 9 x 2 ÷ 27 = 900 cu. yds. of excavated dirt.

(1) The cost of excavation is $2.00 per cu. yd.

900 x 2 = $1800.

Forms for pouring the concrete foundation.

600 x 9 x 2 = 10,800 sq. ft. or the area of the sides of the building.

100 x 2 x 9 = 1,800 sq. ft. or the area of the ends of the building.

10,800 + 1800 = 12,600 sq. ft. or the total surface.

Cost of forms is 30¢ per sq. ft.

12,600 x 30¢ = $3,780.00

(2) Cost of the foundation.

900 cu. yds. of concrete are required.

Cost of concrete per cu. yd. is $10.00

900 x $10. = $9,000.00

b. Flooring.

Acid-proof tile on concrete base costs 70¢ / sq. ft.

100 x 600 = 60,000 sq. ft. of floor space.

60,000 x 70¢ = $42,000.00

c. Walls and roof.

(1) The average price for a solid wall one foot thick, made of common brick, and windows, included is 75¢ / sq. ft.

1400 x 50 = 70,000 sq. ft. or wall surface.

70,000 x .75 = $52,500

(2) Roof.

100 x 600 = 60,000 sq. ft. at 40¢ / sq. ft.

60,000 x 40¢ = $24,000.00

d. Cost of lighting equipment.

One 300 watt lamp will light up 100 sq. ft. at $12.90 per cost of fittings, conduit, wires, panel boards, miscellaneous, supplies, fixtures and switches, not including cost of lamp.

60,000 sq. ft. of lighting space.

60,000 ÷ 300 = 200 lights.

200 x 12.90 = $2,580.00

e. Miscellaneous $10,000.00

3. Equipment cost.

a.	Belt conveyor	$870.00
b.	Glen Falls burner	8,000.00
c.	2 centrifugal pumps	400.00
d.	Chambers	

(1) Lead for the chamber wall lining.

Weight of lead in lbs., 153,000

Price of lead is 6¢ / lb.

153,000 x .06 = $9,180.00

(2) Steel scaffolding to support the chambers.

100,000 lbs. at 5¢ / lb.

100,000 x .05 = $5,000.00

e. Towers.

(1) Glover tower.

Tower surface, 1130 sq. ft.

Acid proof brick at $70.00 / M.

1000 bricks will cover up 41 sq. ft.

1130 ÷ 41 = 27.5 M.

27.5 x 70 = $1,920.00

Scaffolding, packing and minor attachments..... $3,000.00

(2) Gay - Lussac tower.

Tower surface, 1256 sq. ft.

Acid-proof brick at $70.00 / M.

1000 bricks will cover up 41 sq. ft.

1256 ÷ 41 = 30.8 M.

30.8 M x 70 = $2,156.00

Scaffolding, packing, etc., ... $3,000.00

f. Piping, valves, fitting, etc., $1,500.00

g. Miscellaneous, $3,000.00

h. Storage tanks, $10,000.00

4. Installation cost of equipment.

Based on 10 - 20% of invoice cost of equipment.

Total invoice cost $58,500.00

A 15% basis will be used.

58,500 x .15 = $8,775.00

5. Total cost of chamber plant.

a.	Land	$30,000.00
b.	Building	190,000.00
c.	Equipment	58,500.00
d.	Installation cost	8,775.00
	Total fixed cost	$287,275.00

II. Distribution of Plant cost % of selling price*.

1. Yearly production of 53 Be' acid, 15,000 tons, at $12.00 per ton selling price, or $180,000.00

Raw Materials	$61,678.00	34.2%
Direct Labor	9,000.00	5.0%
Fuel, power and water	4,680.00	2.6%
Repairs and Maintenance	5,220.00	2.9%
Depreciation	12,600.00	7.0%
Factory Administration & Fixed charges	18,000.00	10.0%
Taxes & Insurance	7,200.00	4.0%
Distribution and Profits	62,352.00	34.3%
Total	$180,000.00	100.0%

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*Based on Chem. Met. Eng., 39, 2 - 3 (1932).

I. Comparison of fixed cost of chamber & tower plant.

Fixed Costs	Chamber	Tower.
Land	$30,000.00	$10,000.00
Building	190,000.00	25,000.00
Equipment	58,500.00	50,000.00
Installation cost	8,775.00	7,500.00
Total fixed cost.......................	$287,275.00	$92,500.00

II. Advantages of the tower plant over the chamber plant.

1. Simplicity.
2. Same capacity of output on less ground space and unit plant.
3. Including housing cost, equipment and etc., it costs 1/3 as much to build a tower plant yielding an equal daily output.
4. Depreciation costs are lower on the tower plant because the most vital parts are made of Durison, and the estimated life of these under working conditions is about 250 years, where as the vital parts of the chamber plant last about 7 - 10 years.
5. It takes a smaller investment to finance the tower method, and a larger return on the original investment is possible.
6. Expenses and charges involved in operation are less than those of the chamber plant.
7. It takes less unit volume to convert one pound of sulphur to H_2SO_4 / 24 hours. The chamber plant needs 12 cu. ft. of chamber space to convert one pound of sulphur to sulphuric acid / 24 hours. The tower plant plant requires 0.13 of cu. ft. as derived from pilot plant results.
8. In the end, the cubic volume for producing H_2SO_4 was reduced at a ratio of 100 to 1.

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A study of the manufacture of sulphuric acid by the Tower method

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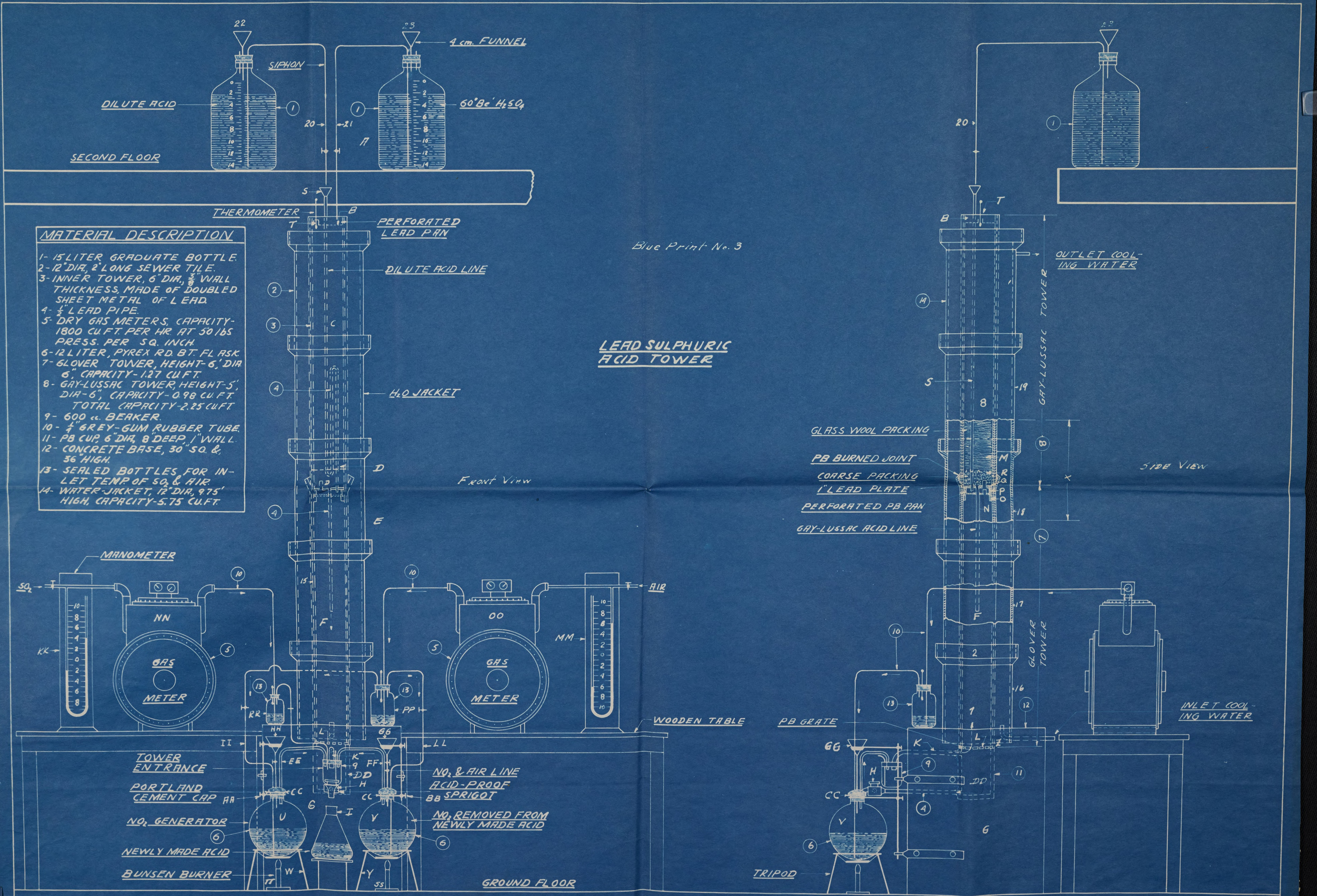
Rakas



MATERIAL DESCRIPTION

1- 15 LITER GRADUATE BOTTLE.
2- 12" DIA., 2' LONG SEWER TILE.
3- INNER TOWER, 6" DIA., 3/8" WALL THICKNESS, MADE OF DOUBLED SHEET METAL OF LEAD.
4- 1/2" LEAD PIPE.
5- DRY GAS METERS, CAPACITY- 1800 CU. FT. PER HR. AT 50 lbs PRESS. PER SQ. INCH.
6- 12 LITER, PYREX RD. BT. FLASK.
7- GLOVER TOWER, HEIGHT- 6,' DIA. 6", CAPACITY- 1.27 CU. FT.
8- GAY-LUSSAC TOWER, HEIGHT- 5', DIA- 6", CAPACITY- 0.98 CU. FT. TOTAL CAPACITY- 2.25 CU. FT.
9- 600 cc. BEAKER.
10- 1/4" GREY-GUM RUBBER TUBE.
11- PB CUP, 6" DIA., 8" DEEP, 1" WALL.
12- CONCRETE BASE, 30" SQ. & 36" HIGH.
13- SEALED BOTTLES FOR INLET TEMP. OF SO_2 & AIR.
14- WATER-JACKET, 12" DIA., 9.75' HIGH, CAPACITY- 5.75 CU. FT.

MICHIGAN STATE COLLEGE
CHEMICAL
ENGINEERING DEPARTMENT

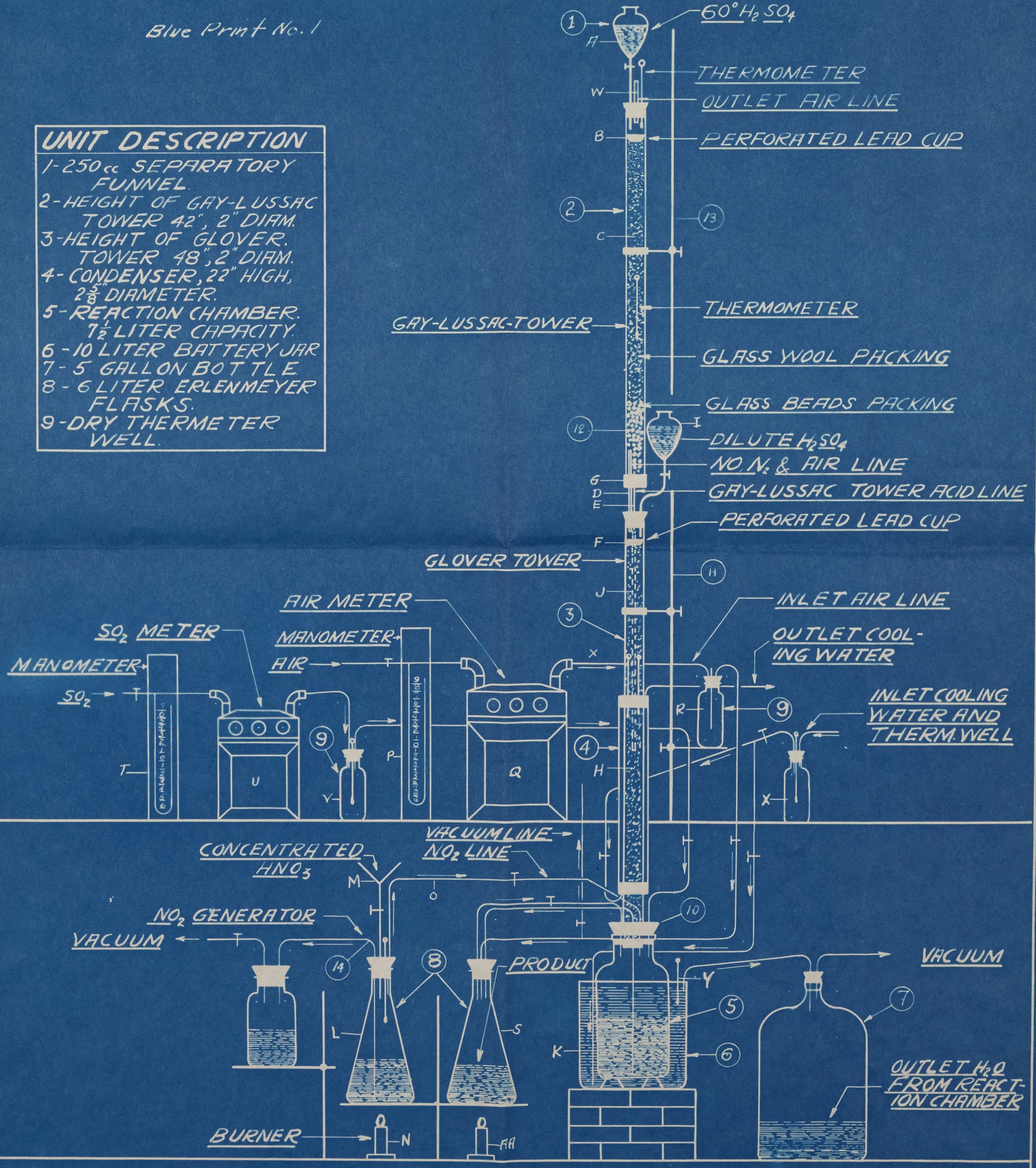
PILOT PLANT
SULPHURIC ACID TOWER

DRAWN BY:- N. Rakos
TRACED BY:- N. Rakos
DATE:- APRIL 27 1935, SCALE:- 1"=1'

Blue Print No. 1

UNIT DESCRIPTION

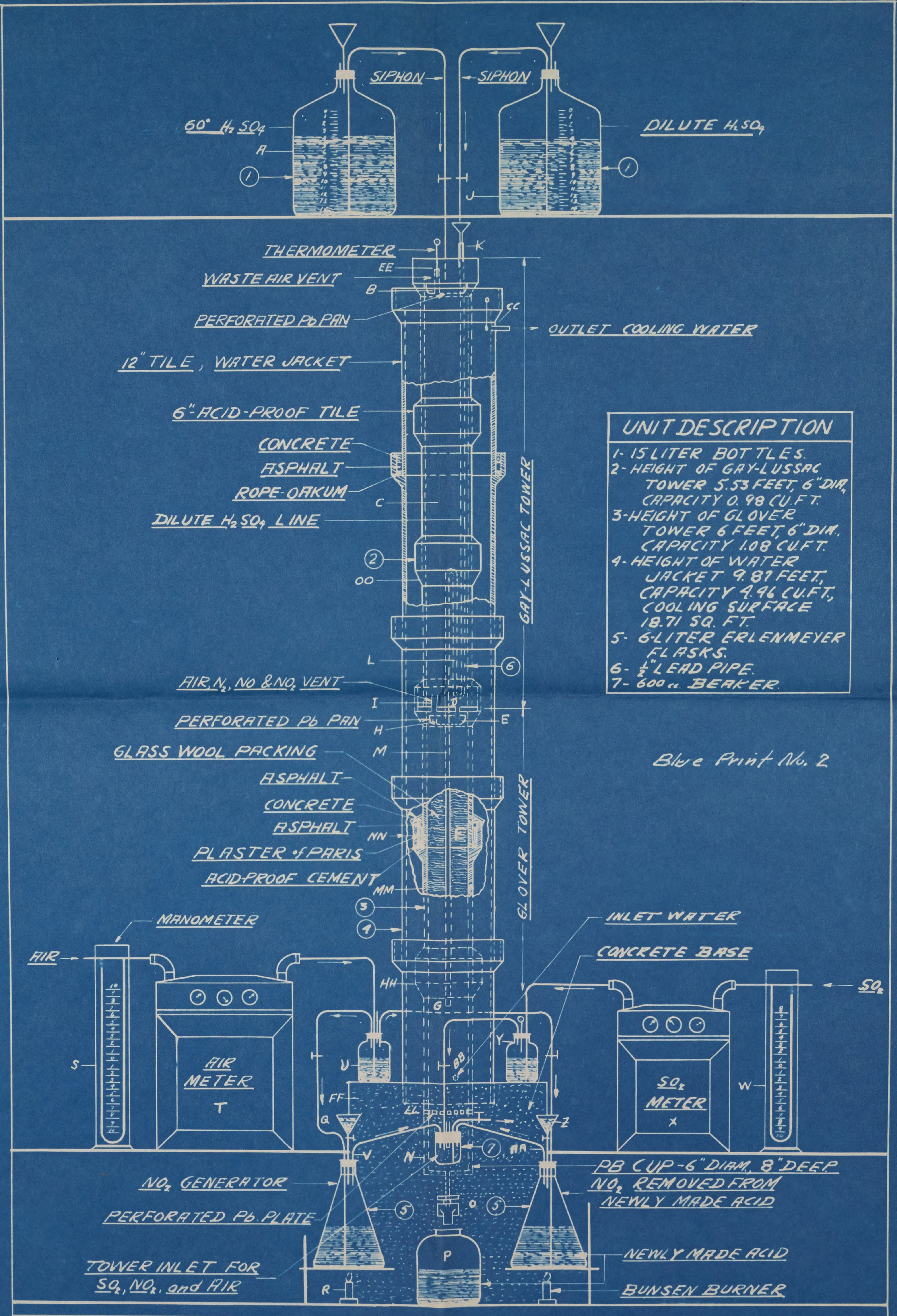
1-250 cc SEPARATORY FUNNEL.
2-HEIGHT OF GAY-LUSSAC TOWER 42", 2" DIAM.
3-HEIGHT OF GLOVER. TOWER 48", 2" DIAM.
4-CONDENSER, 22" HIGH, $2\frac{5}{8}$" DIAMETER.
5-REACTION CHAMBER. $7\frac{1}{2}$ LITER CAPACITY.
6-10 LITER BATTERY JAR
7-5 GALLON BOTTLE.
8-6 LITER ERLENMEYER FLASKS.
9-DRY THERMETER WELL.



MICHIGAN STATE COLLEGE
[redacted] CHEMICAL
ENGINEERING DEPART.

H_2SO_4 TOWER
LABORATORY SET UP

DRAWN BY:- Nick Rakas
TRACED BY:- Nick Rakas
NOV. 22, 1934; SCALE: $\frac{3}{32}$" = 1"

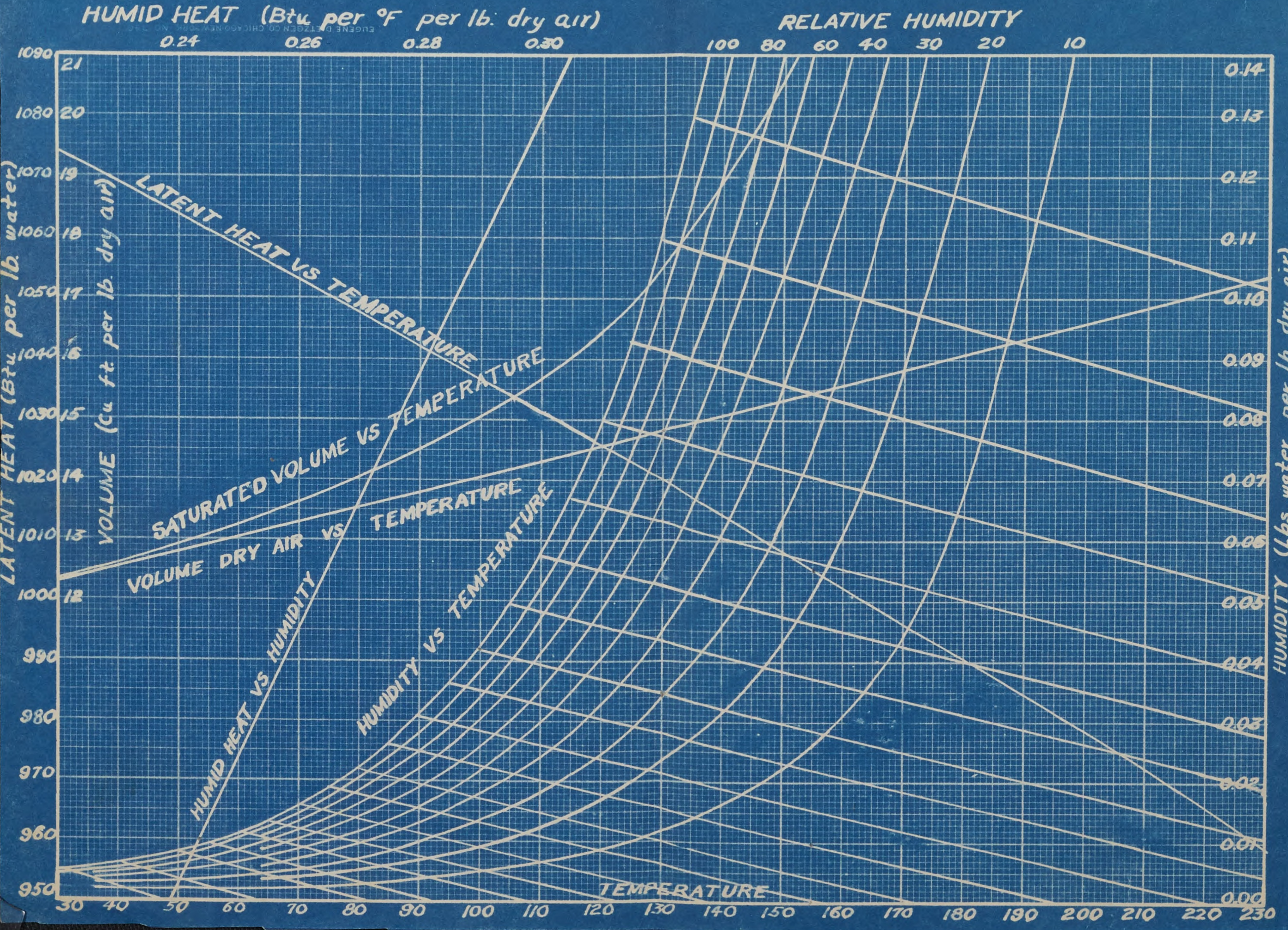


UNIT DESCRIPTION

1- 15 LITER BOTTLES.
2- HEIGHT OF GAY-LUSSAC TOWER 5.53 FEET, 6" DIA, CAPACITY 0.98 CU.FT.
3- HEIGHT OF GLOVER TOWER 6 FEET, 6" DIA. CAPACITY 1.08 CU.FT.
4- HEIGHT OF WATER JACKET 9.87 FEET, CAPACITY 4.96 CU.FT, COOLING SURFACE 18.71 SQ. FT.
5- 6-LITER ERLENMEYER FLASKS.
6- ½" LEAD PIPE.
7- 600 cc. BEAKER.

Blue Print No. 2

MICHIGAN STATE COLLEGE [redacted] CHEMICAL ENGINEERING DEPARTMENT	- PILOT PLANT - SULPHURIC ACID TOWER	DRAWN BY:- N. Pakas TRACED BY:- N. Pakas CHECKED BY:- N. Pakas	DATE:- FEB. 21, 1935 SCALE 1"=1'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0.26
0.28
0.30
RELATIVE HUMIDITY
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80
60
40
30
20
10
LATENT HEAT (Btu per lb. water)
1090
1080
1070
1060
1050
1040
1030
1020
1010
1000
990
980
970
960
950
VOLUME (cu ft per lb. dry air)
21
20
19
18
17
16
15
14
13
12
HUMIDITY (Lbs. water per lb. dry air)
0.14
0.13
0.12
0.11
0.10
0.09
0.08
0.07
0.06
0.05
0.04
0.03
0.02
0.01
0.00
LATENT HEAT VS TEMPERATURE
SATURATED VOLUME VS TEMPERATURE
VOLUME DRY AIR VS TEMPERATURE
HUMID HEAT VS HUMIDITY
HUMIDITY VS TEMPERATURE
TEMPERATURE
30
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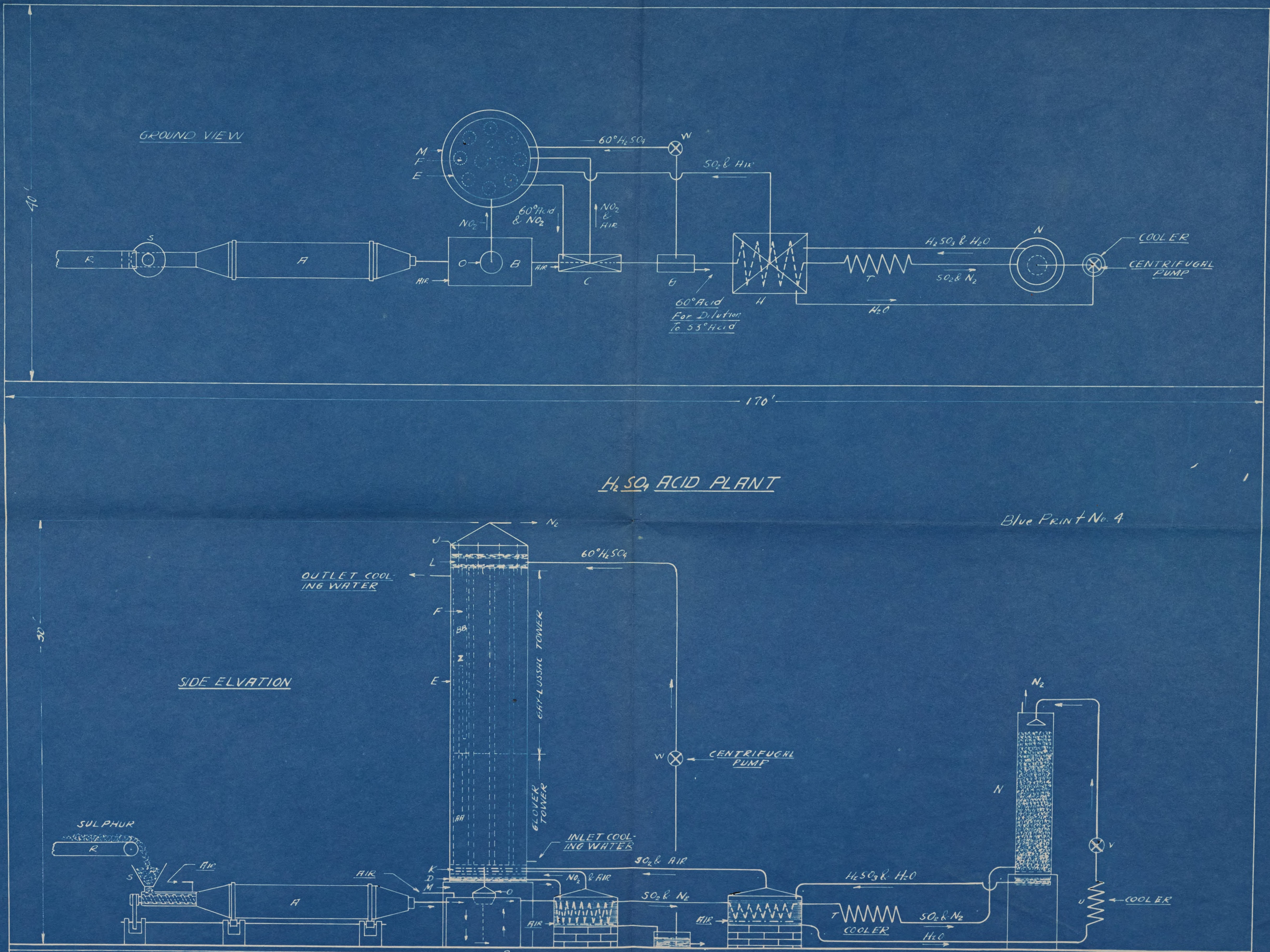

H_2SO_4 ACID PLANT

Blue Print No. 4

MICHIGAN STATE COLLEGE
CHEMICAL ENGINEERING DEP'T.
DRAWN & TRACED BY: N. RAKAS

DATE: MAY, 13, 1935
SCALE: 1" = 10'