[illegible]

[illegible] METALLIC LEAD [illegible]

[illegible]

A. [illegible]

In the process of washing silica gels of various types with dilute sulfuric acid, a [illegible] is useful up to [illegible] F. For wash temperatures in the [illegible] 140 – 175° F., [illegible] is unsuitable and lead linings are used with [illegible] trouble. However, the gel containers used during washing damage the lead [illegible] being [illegible] in or out of the lead lined wash towers. Misconstruction [illegible] resulted more from improper design considerations and unwillingness to reduce slightly the size of each basket or tray to allow timber runners or guide posts, placed [illegible]ally along the tank sides, to take the scraping effect of the wooden gel containers.

Lead pipe has given good service in this plant. However in valves they have replaced lead seats with rubber. Formerly lead lined weir boxes were used, however these failed as the one inch holes in the baffle plates enlarged by erosion and acid flow characteristics were disturbed.

B. INTRODUCTION

Silica gel is an absorbent prepared from the coagulation of a colloidal solution of silicic acid. It is a hard glassy substance, the term "gel" merely indicates the condition of the material during one stage of its manufacture. It resembles a clear quartz sand and has the chemical composition, SiO_2. Silica gel is a highly porous structure with uniform arrangement and size of the pores.

Principal use of silica gel is for the absorption of water, sulfur dioxide, acetone, benzine and other materials. It is estimated that 1 gram of silica gel has an exposed surface area of 6000 sq. ft. This feature enables it to act as a catalyst carrier for platinum used in the contact process for sulfuric acid manufacture. Petroleum refining, carbon dioxide purification and numerous other processes make use of the [illegible] properties of silica gel. It is interesting to note that most of the [illegible] in North America are made in rooms where silica gel is used for dehydration of air to reduce the humidity to as low as 1%.

C. PROCESS DESCRIPTION

A certain amount of economy secrecy prevents inquiry into the initial phase of the process where the production of silica gel by reaction of certain proportions of liquid sodium silicates with sulfuric acid takes place under carefully controlled conditions. The mixture is coagulated into a hydrogel which takes the form of a solid slug, normally 8 ft. in diameter by 8 ft. deep. This is broken up and washed to remove the Na_2SO_4 formed in the reaction. The hydrogel contains 80% water and 20% silica.

The wash towers are generally arranged in groups which act together as a unit. In the larger groups there are eight towers, one always empty, the other seven containing wooden boxes partially filled with gel in various stages of the 60 hr. washing cycle. The hot (160 - 175° F.) dilute H_2SO_4 wash solution (6 g.p.m.) is put through the first tower which contains gel that has undergone all the previous washing stages. The acid wash solution is still fairly clean and it is passed through the next tower which contains gel that has undergone all previous washing stages but one. This continues to the last tower where the nearly spent acid washes freshly made silica gel and is then discarded. In this way the clean, fresh acid first contacts the almost completely washed gel for 6 to 8 hours prior to its removal for drying and packing.

Two large wooden boxes are partially filled with unwashed gel and these are placed in the towers for washing (see drawing). Formerly 28 trays of gel were put in each wash tower, but the stacking of these was often uneven or became so in handling and an offset tray with its brass fittings in the corners could gouge or tear the lead lining as the trays were placed in or withdrawn from the tower. Very recently the change has been made from 28 trays to 2 boxes. The boxes have close clearance with the lead tower wall and are lifted out by means of brass rods in the form of an inverted "V" on two sides of the box. An overhead travelling crane transports the boxes to the unloading station.

Wash acid is prepared continuously in a large wooden open top tank by adding a trickle of concentrated sulfuric acid to inflowing streams of cold water and low pressure steam. This hot solution is pumped through steel pipe to a steel header tank from which it flows by gravity through lead pipe to a steel, rubber covered weir box where a series of 1 in. holes cut on the diagonal of a center plate control the flow. From the weir box, wash liquor flows through lead pipe by gravity to a lead lined flow distribution box and then into the wash towers as shown on the drawing.

A flow distribution box with connections as shown in the drawing is located at the working floor level near each wash tower. Dilute acid for washing is routed through these boxes from tower to tower and also from the weir box to the initial tower and to the drain from the final wash tower.

D. EQUIPMENT DESCRIPTION

The wash towers are made of 6 lb. chemical sheet lead supported by a cribbing of 3 x 8 in. yellow pine timbers. The drawing gives a general idea of the construction but does not attempt to illustrate all the pipe connections from towers to

distribution boxes. The towers are 4 ft. 6 in. square and 18 ft. deep and are provided with a light weight wooden cover. In the bottom corner of each tower there is a [illegible] angle offset or step which acts as a support for the wash boxes and allows circulation of the liquid through the center area of the box. A close clearance between boxes and tower sides is necessary to prevent a bypass flow of acid wash water. Damage to the lead, especially in the corners, has been excessive when withdrawing or putting in place the wooden boxes or the 14 trays which formerly took the place of each wash box. These trays had brass fittings in their corners which tore the lining unless they were handled with great care which is certainly unusual among workmen today. Before changing to the boxes it was necessary to lead and unlead 28 trays in the towers filled with wash solution keeping them aligned so that no edges or corners would project out to rip the lead. Two wooden boxes are now used as shown in the drawing and are handled by means of brass rods on two sides which are hooked to an overhead traveling crane. So far as could be seen, there were no sharp brass edges exposed on the boxes now being used which could damage the lead.

A slight decrease in damage to the lead was gained by using a double thickness of lead in the corners. However, this is not a solution to the problem, nor has the change from a number of trays to two boxes completely eliminated the damaging scraping effect.

The flow distribution box is also fabricated from 8 lb. sheet lead and is roughly 14 x 10 in. by 10 in. deep. One of these is located about two feet from each tower and also has a wood cover. Various routings of the wash liquid can be obtained by means of this box and thus eliminate several valves per wash tower. This is important when you consider valving each of the 68 wash towers used in this process.

The weir box was formerly of all lead construction but the 1 in. holes set on the diagonal of the center plate eroded with the flow of hot acid and became enlarged. This resulted in irregular acid flow and to correct it, steel boxes completely rubber covered were substituted. These have given excellent service and it is unlikely that lead can supplant rubber here.

Lead pipe of 2 and 3 in. sizes, 1/4-in. wall, gives good service in this plant. Hills McKenna lead valves have given good service except that the lead seats were out in 4 to 5 months. These seats were replaced with rubber and the same valves have since given good service. For pumps they have used Hastelloy C, Illium and stainless steel, 3/16 in. thick. Small pumps of Illium have given the best service in 10 years of operation, although said to cost $2200.

B. OTHER WASH UNITS

In a larger portion of the plant, silica gels of other grades are prepared. These do not require the wash acid to be over 120° F. and concrete tanks lined with a mastic material, said to be supplied by the Ralph Y. Rulon Company of Philadelphia, are giving very good service. Temperature is the governing factor here as the mastic will not stand up at temperatures over 120° F. These tanks are quite large (roughly 20 ft. sq.) and the gel is removed by means of a clam shell bucket. Damage to the mastic lining is prevented by a complete coverage of heavy timber. The tanks were built in 1943 and only an occasional timber is replaced, usually from damage done by the clam shell bucket.

F. CONCLUSIONS

1. Wash Towers

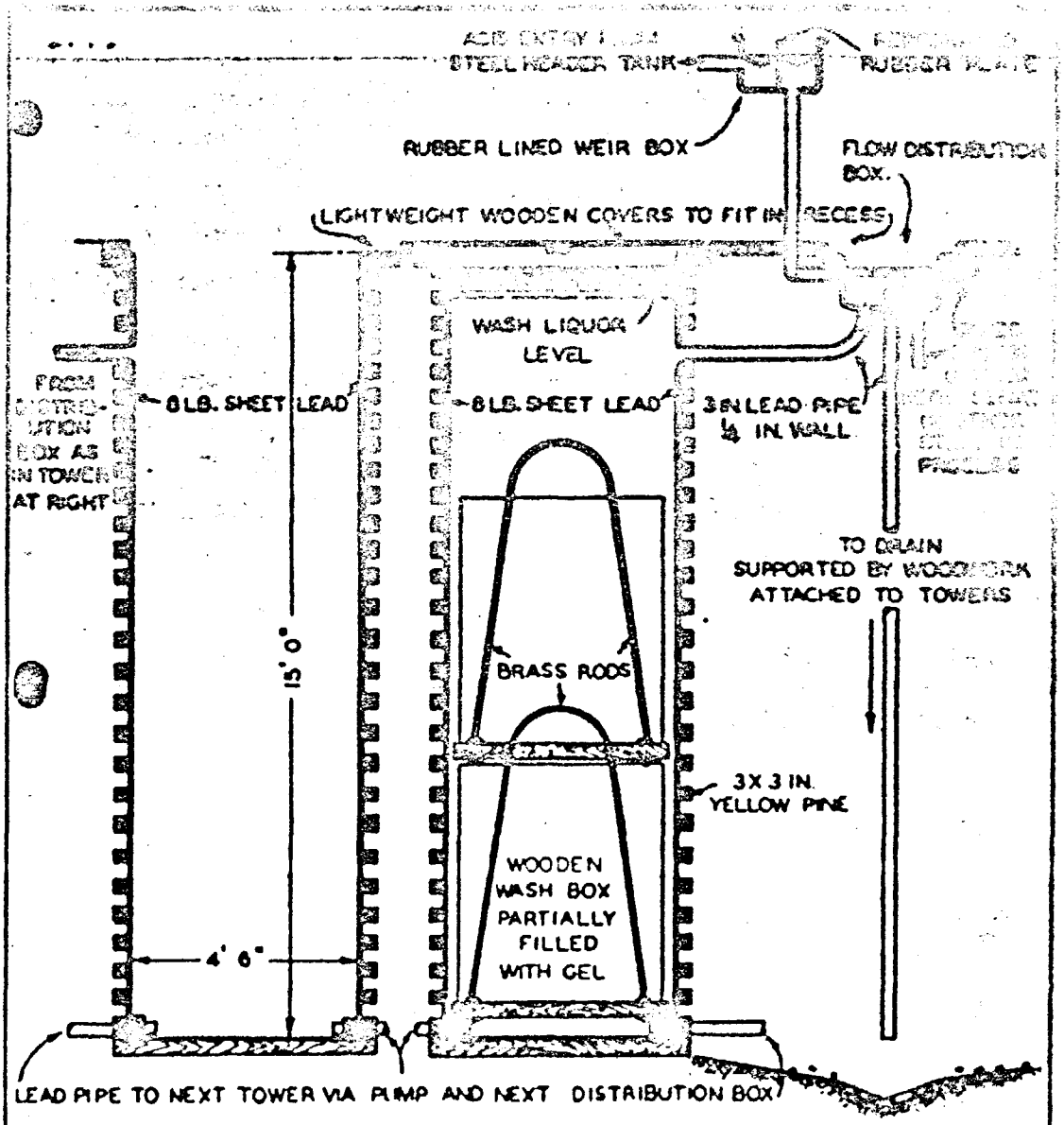
There is no problem of corrosion of lead involved. Dissatisfaction with lead results from its use where no provision has been made for protection against tearing of the lead wall — to correct this, buffers should be used, either wood runners in the corners or strips of rubber would seem to be a very simple means of protecting the lead.

2. Weir Boxes

While lead was formerly used, erosion of the holes in the center plate destroyed flow characteristics and the ensuing change to rubber has proved very satisfactory. These might still be lead units if designed properly using another material to form the edges of the holes where erosion later ruled lead completely out of the picture.

3. Pipe — Valves — Pumps

Two and three inch lead pipe has proved satisfactory here. Lead valves have given good service except that the seats were cut in three to four months. The same valves have given excellent service with the use of rubber seats. Few if any pumps in this plant are of lead. Hastelloy C, stainless steel and Illium have all given good service. Illium pumps are the most expensive but have given the best service in handling the acid wash water.



LEAD LINED WASH TOWER BY H.M. CHURCH JR. MAY 26, 1947	LEAD INDUSTRIES ASSOCIATION 420 LEXINGTON AVE. N.Y. 17, N.Y.	SILICA GEL WASH PROCESS CHEMICAL BULLETIN NO. 17