PICKLING OF STEEL

THESIS FOR THE DEGREE OF M. E.

D. F. Jones

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PICKLING OF STEEL

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THESIS

INDEX

PICKLING DEFINED

Pickling is the operation of removing sand, scale, oxide and other foreign material from the surface of metal by dipping it into acid solutions. Unless otherwise stated the following will deal with the pickling of steel in Sulphuric Acid.

HISTORY OF PICKLING

AS APPLIED TO IRON AND STEEL PRODUCTS:

Since the coating of iron was being successfully accomplished in the Ore Mountains of Saxony, Germany, during the sixteenth century, it is logical to believe that knowledge of the art of pickling existed at that time.

In the manufacture of galvanized steel sheets, tin and terne plate, the sheets are pickled in a sulphuric acid and water solution at temperatures only slightly higher than the atmosphere. The pickling operation follows the hot rolling and annealing operation wherein the elevated temperatures create the surface oxide of iron. The sheets are packed in cradles and submerged in the pickling solution until the oxides are removed. Continuous agitation is carried on to insure the contact with the pickling solution of 100% of the area of each sheet. After the pickling operation is complete, the sheets are cleansed with water and moved on to the next operation. Just before introducing the steel sheets into the coating metal, they are subjected to a bath of cut muriatic

acid, the temperature being only a little lower than that of the molten coating metal; this acid bath, or pickling, effects an etching action that promotes a better adhesion of the coating to the steel sheets.

The art of pickling has a very considerable effect on the character of surface carried by the finished product. Consequently, we find the steel mills employing this operation on the sheet bars from which automobile and metal furniture sheets are made. The operation is similar to that employed in connection with coated sheets except that the acid content is generally increased.

The steel industry has recently put into production numerous alloys of iron that have presented a pickling problem of considerable complexity to the metallurgical engineer. A representative composition in the so called stainless group of ferrous alloys is - 17% to 20% chrome, 7% to 10% nickel, the balance of the composition being iron, a familiar trade name for such a composition being "Allegheny Metal".

This popularly termed 18-8 alloy is remarkably resistant to acids; consequently, to thoroughly clean its surface, the following pickling solution is being employed:

25% by volume of 20° BE Muriatic Acid; or
10% by weight of 60° BE Sulphuric Acid
7% by weight of Rock Salt
Temperature of bath 140° to 160° F.

After pickling, a thorough rinse is employed, and then the alloy is dipped into a hot solution of 10-20% Nitric (38° BE strength). After again washing carefully in water, the above treatment produces a uniform clean gray surface on the piece pickled. Should a brighter finish be desired, the following formula will serve admirably:

20% by volume of 20° BE Muriatic Acid
10% by volume of 38° BE Nitric Acid
5% by volume of 85° Phosphoric Acid
1/2% of any standard inhibitor
Temperature of the bath, 180° to 200° F.

In the past it has been customary for the manufacturers and fabricators of the finished steel products, which require special coatings or finishes, to buy the steel from the rolling mills in the pickled and oiled condition. However, at the present time there are many manufacturers who choose to do their own pickling, for the following reasons:

That the manufacturers can equip for the pickling and handling of their special sizes more economically. The oiling process to prevent rust can be omitted as the steel can be pickled just before fabrication into the finished product, thereby eliminating the cost of oil and the expense of removing the oil. Rusting many times occurs after the steel is oiled, especially if the steel is stored for some time before using and the humidity and temperature conditions are favorable for rust formations. In the fabrication process it is many times necessary to heat the steel to

high temperatures to make it more easily workable or to anneal the steel after it has been formed. Scale is again produced and pickling in the manufacturer's plant is as necessary as before.

The pickling of steel at the rolling mill is an added operation that is only applied to certain classes of steel and during times of heavy production the pickling department may be so over-loaded with work that shipments are held up for much longer periods of time than would be required to do the pickling in the manufacturer's plant.

Different classes of work in the same plant with the same steel may require varying degrees of care and time in pickling to satisfy the demands of the finished product. The manufacturers cannot anticipate this varying requirement, so consequently it is necessary for them to have all steel pickled so that it will be satisfactory for the best class of finish.

Metal is pickled in order to give it a clean surface. A clean surface is necessary before the application of protective coatings such as produced by painting, galvanizing, tinning, plating, and enameling. While it is not necessary it is decidedly advantageous usually to clean metal by pickling before machining in order to save wear on tools and also before lacquering. Another advantage of pickling is that it facilitates more thorough inspection.

ACID

The most commonly used acid for pickling metals due primarily to its lower cost is sulphuric acid (H_2SO_4) or Oil of Vitriol, usually purchased at a concentration of 66° "Baume", approximately 95.5 pure straw-colored acid. The boiling point of this acid is approximately 590° F., and the present price is about $20.00 per ton in carload shipment. 60° "Baume" is also used, but this is required in greater volume; another disadvantage of 60° "Baume" is that more arsenic, a very detrimental element, is found in it.

Substances used in place of sulphuric acid are: Edis Compound - manufactured in the form of dry cakes, soluble in water; Kleanrite - powdered compound ordinarily used in a solution made up of 1 lb. Kleanrite to 4 lbs. or 5 lbs. water; Nitrate Cake - used like the compounds just described.

Sometimes Hydrochloric Acid is used for pickling where it can be obtained more cheaply than Sulphuric Acid.

Phosphoric Acid has been used with good results where the surface is to be cleaned before enameling.

When sulphuric acid is dissolved in water, a portion of the acid molecules dissociate, or separate into three electrically-charged parts called ions. The solution, as a whole of course, remains electrically neutral and the water undergoes no chemical change but merely serves as a piece of apparatus in the process. This breaking up into ions is called ionization. The effect is as follows:

$$H_2SO_4 = H^+ + HSO_4^- = H^+ + H^+ + SO_4^{--}$$

The whole basis of the pickling action is the displacement of the positively-charged hydrogen ion by the metal being pickled. If there is no hydrogen evolution there is little pickling. The following list shows the relative activity of various metals in displacing the hydrogen ions. All metals before hydrogen in this list displace hydrogen from the pickling solution, while those after it do not. The highest in the list are the most active in the order named:- Aluminum, manganese, zinc, chromium, iron, nickel, lead, tin, hydrogen, copper, silver, platinum, gold. The reaction with pure iron is as follows:

$H_2 SO_4 + Fe = Fe SO_4 + H_2$

The H being given off as a gas.

In pickling it is common to say the acid eats the rust or scale. Rust consists chiefly of a hydrated ferric oxide ($3Fe_2 O_3 H_2O$). This is porous, brittle, and adheres more or less loosely to the steel. Hammer or rolling mill scale is the same as magnetic oxide of iron, $Fe_3 O_4$. It adheres more firmly to the steel. Both are but slowly soluble in pickling acids, scale being the slower.

The acid actually penetrates in, around, and under the rust and scale. The hydrogen ion being displaced by the pure iron under the scale or rust, the hydrogen being set free. The evolution of the hydrogen exerts a prying action on the scale and rust; thus, the removal is mechanical as well as chemical. The reactions are as follows:

$Fe_3 O_4 + 4 H_2 SO_4 = FeSO_4 + Fe_2 (SO_4)_3 + 4 H_2O =$ (Scale)

$Fe_2 O_3 + 3 H_2 SO_4 = Fe_2 (SO_4)_3 + 3 H_2O =$ (Rust)

The iron is attacked and the slightly attacked scale or rust falls off.

Hydrogen brittleness caused by over pickling is the result of hydrogen entering the steel, probably between crystal boundaries, and can be largely removed by heating in boiling water.

With regard to the amount of sulphuric acid used per ton of steel pickled, this varies with the thickness of material and with the concentration of the acid.

Acid consumption is generally determined per ton of material pickled and not by the square feet of area pickled, because it is difficult to determine surface areas, while on the other hand, it is easy to determine the weight of material to be pickled.

Consequently, in comparing the acid used per ton of material pickled in different plants, we must compare acid consumption between only those plants that pickle material of similar shape or of the same thickness of plates.

Material Pickled	Average Pounds of Concentrated Acid per Ton of Material Pickled
Tin Plates	75 to 100 Pounds
Sheets	40 to 75 Pounds
Coils	50 to 100 Pounds
Drop Forgings	40 to 120 Pounds

(This table is based on the use of inhibitors and a temperature of between 175° and 195° F. in the acid bath.)

Agitation also materially hastens the time of pickling. The work may be agitated, or the solution in the tank, or both.

REMOVAL OF GREASE

Pickling acids have little or no effect on oil, grease, or lubricating compounds, so these must be removed, before pickling, in a bath of caustic soda or some similar commercial cleaning compound. Proper cleaning of the material before pickling has a marked beneficial effect on pickling results. At the same time as much as one-half of the acid otherwise required may be saved where proper cleaning for grease, and oil precedes pickling.

STRENGTH OF ACID SOLUTION

The solution of acid should be strong enough to remove foreign matter with a period of time short enough to prevent either over-pickling, and consequent loss of metal, or precipitation of combined carbon, and consequent formation of a graphitic coat on the surface.

Different materials require pickling baths of different acid strength. The following table shows the volume of acid commonly used to pickle different materials. The percentage given in the table means the percentage of concentrated sulphuric acid in the solution.

Material Pickled	Percentage of Acid by Volume in Pickling Bath
Tin Plate	3% to 10%
Sheets	4% to 10%
Drop Forgings, including Alloy Forgings	6% to 15%

(This table is based on the use of inhibitors and a temperature of bath between 175° and 195° F.)

Higher acid strengths may be used but since no faster pickling can be secured, the acid would be wasted to a much greater degree due to evaporation, acid and water carried away by the product, and leakage of tanks or vats, which is always a serious item of consideration.

MAINTAINING ACID SOLUTION IN PROPER CONDITION

It is necessary to refresh the acid solution at certain intervals; for example, if the tonnage is large and if pickling is begun with 8% acid by volume, that concentration is maintained through most of the turn by refreshing with additional concentrated acid; a few hours before the end of the turn the solution is allowed to drop to 2-1/2% to 3% acid by volume and the entire solution is drained from the tank at the end of the turn. If tonnage is small, every two or three days, or even once a week, will suffice for draining the tanks and for cleaning them of the ferrous salts collected. A range of from 3% to 40% acid by volume effects the pickling time but little, provided the temperatures are kept within the above stated limits (175° - 195° F.)

See Chart #1 to ascertain the pickling time with different strength of acid at the temperatures of 175° - 195° F.

The strength of a new acid solution can be satisfactorily determined by a hydrometer. But as the iron salts build up, other methods must be used, due to the increase in the specific

gravity of the solution by the iron salts.

As the pickling process goes on the constant evolution of hydrogen gives a decreasing concentration of ions with a decreasing rate of pickling. The addition of more acid will not help in proportion to the amount of acid added, because the percent ionization is lowered by the presence of the ferrous sulphate or iron salts. Increasing the temperature increases the percentage of ionization and also their activity.

But there is an economical limit to adding acid and raising temperature and the solution should be dumped when the percentage of ferrous salts becomes high enough to reach this limit. Chart #3 shows the percentage of acid lost when the solution is dumped under various conditions for a sulphuric acid solution used to pickle iron and steel.

Referring to the chart, if the solution contains 3% acid and 8% iron at the time of dumping, then 18% of the total acid put into the solution will be wasted when the solution is dumped or this 18% acid has never done any useful work. If the solution were used down to 1% acid strength and 9% iron the loss would be reduced to 6%. Longer time would be required to pickle at the reduced strength of acid, and this would have to be balanced in by the expense of production time.

Simple methods of chemically testing acid by titration may be employed to ascertain the acid strength. The acid strength is generally kept constant at the proper strength by the constant addition of acid until the iron content be-

comes heavy and slows the operation. The acid content in the solution is then reduced by not adding further acid and continuing the pickling process until the increased time factor prohibits the further use of the solution, at which time it is dumped.

The solution is generally dumped when the iron content is between 6% and 15%; the average being around 8%, depending on the conditions. It is always customary to dump the solution before the iron salt starts to crystallize out on the wall of the tank, due to the additional difficulty in cleaning. The effect of Iron Sulphate or Iron Salts on time is noted here by a test made by Bablik, using a 5% solution of sulphuric acid.

Iron Sulphate Percentage	Pickling Time in Percentage
Let 5 equal	100 %
Then 10 equals	160 %
Then 20 equals	230 %

INHIBITORS

Inhibitors, or "catalysers," are used extensively in the best pickling practice. They are compounds added to the acid solution in order to retard the action of the acid on the metal. They do this by depositing a molecular film on the surface of the bare metal. This film tends to prevent over-pickling without seriously interfering with the action of the acid on oxide and other foreign materials to be removed.

The use of inhibitors eliminates waste of steel by acid. Conversely, inhibitors prevent waste of acid in unnecessarily dissolving steel. They prevent pitting of metal surface; they overcome tendency of carbon or graphite in metal to dissolve and to form a coating on metal surface. In addition, inhibitors also lengthen the life of acid solutions.

Another reason why inhibitors are very beneficial in pickling is, because during the process of pickling, hydrogen is set free and follows microscopic crevices in the steel, giving rise to brittleness and formation of blisters in sheets and tin plate; certain inhibitors counteract this action and prevent brittleness and blisters.

Such organic substances as "rye flour", and "red dog", are used as inhibitors. Others are marketed under trade names, "Pickelette", "Picklerite", "Sumfoam", and "Rodine".

TEMPERATURE OF BATHS

In order to save acid and to increase the speed with which pickling is done, thus reducing the cost of pickling, it is advisable to keep the temperature of the pickling bath as close to the boiling point as is practical.

It has been found by experience that the best range of temperature lies between 175° F. and 195° F. The higher the temperature the more difficult it is to maintain proper ventilation of the pickling room, and the more acid is wasted, because at high temperature more acid is carried away with the water vapor.

The rinsing baths should vary in temperature with respect to the material being cleaned. For example, enameled ware should be rinsed in cold water; heavy gauge sheets on the other hand are rinsed in very hot water and dried by the heat absorbed while in the rinsing bath.

Pickled steel tends to rust quickly, sometimes within a very few minutes, unless the acid left in the pores of the steel is neutralized and any free iron (F_e) coated. After rinsing, it is customary for the steel to be dipped in oil if is to be shipped to others for fabrication. This treatment keeps out oxygen, thereby preventing rust to a great degree.

Where the pickling and fabricating occurs in the same plant, the steel after rinsing is often given an additional rinse in water saturated with lime. The lime not only coats and neutralizes any acid left, preventing rusting, but tends to make the steel more easily workable, acting as a slight lubricant; also a lighter color is sometimes produced in the steel when the reworking or fabricating temperatures run above 350° F. This lighter color is quite desirable in marketing certain drawn wire.

Steam is generally used for heating the tanks. The open jet may be used as the condensed steam helps to replace the water carried away by the work and the evaporation. Low pressure or exhaust steam is suitable if the steam pressure is greater than the pressure in the pickling solution at the jet location and there is not sufficient oil present to cause trouble. More steam will be required, however, with low pressure than with high pressure, and pipe sizes and the number of jets will have to be altered accordingly.

It is generally not difficult to satisfactorily control the temperature of the large tanks by hand valve operation. Automatic control equipment is available on the market, but is more or less subject to the action of the acid and, its installation may mean additional openings in the tank which are very objectionable.

PICKLING TIME

The time required to pickle material, as well as the capacity of a vat or tank depends upon: (1) concentration of acid; (2) temperature of pickling bath; (3) inhibitors; (4) iron salts in solution; (5) nature and thickness of scale; (6) the ease with which unconsumed acid comes in contact with the surfaces to be pickled.

The effect of the first four, concentration, temperature, inhibitors, and iron salts, has already been discussed.

The nature of the scale has a marked influence on pickling time, for it is easier and, therefore, quicker to clean material where the scale is not deep and does not tightly adhere to the material.

The ease with which contact can be made between the surfaces to be pickled and the unconsumed acid depends upon the temperature, the agitation, and the aeration produced by the pickling machine or in the solution.

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On account of the many factors, as described, influencing pickling time, it is impossible to definitely predict pickling time. The following data, however, have been collected from experience with and without pickling machines and represent fairly average pickling time.

	Length of Time in Pickling Bath	
Material Pickled	With	Without
Tin Plate	6 to 10 minutes	8 to 15 minutes
Sheets	8 to 15 minutes	10 to 20 minutes
Coils	10 to 15 minutes	12 to 20 minutes
Drop Forgings, including wide range of Alloy Forgings	10 to 40 minutes	15 to 50 minutes

Sheets pickled longer than 20 minutes generally develop objectionable pitting.

See Chart #1 for the influence of acid strength and temperature on time of pickling.

CRATES OR RACKS

Crates or racks constructed of acid resisting phosphor-bronze metal or monel metal, and adapted to the shape and weight of the material to be pickled, are used to carry the work in and out of the solution.

In placing the material to be pickled into the crate or rack it is necessary so to arrange it that the acid has free access to each and every square inch of surface to be pickled.

As an illustration of this statement: in pickling hollow ware, the pieces must not be nested too tightly; and in pickling sheets and tin plate, the material must be placed between pins, or other devices for separating the sheets. Avoid the use of brass in the construction of the racks as the battery action may plate the brass onto the steel. The voltage between the work and the monel tank, when the work is suspended in the solution but not in contact with the tank has been checked, in one instance, to be 475 millivolts, with a current flow of 3.9 amperes.

VATS OR TANKS

Their construction and maintenance constitutes one of the greatest problems in the pickling department. Constant experimentation is being carried on to develop longer life tanks with less maintenance cost and the elimination of expensive acid leakage.

California fir, white pine, cypress, birch, monel metal, concrete, anti-acid brick, and other materials are used in making vats or tanks for pickling. A steel tank with a rubber lining and also steel tanks with lead linings are in use at the present time.

The most desirable material for a tank, among the woods, is probably "number one" white pine, although this is somewhat expensive and difficult to obtain. However, tanks properly constructed from this material have an average life from two to four years.

Concrete tanks lead-lined, wooden tanks lead-lined, and anti-acid brick tanks are also giving excellent service in a number of plants. These tanks are more expensive than ordinary wooden tanks.

A very good method in the construction of a wooden tank is to use 6" x 6", 6" x 8", or 8" x 8" timbers. These are painted with thick white lead, or asphalt paint, rabbeted, (in a few places machines are available for planing timbers so accurately that rabbeting can be dispensed with), and held together with copper or monel bolts at the joints. A false lining on the walls and on the bottom inside of the tank is made of 1" matched white pine. Guards made of 3" x 4" or 3" x 6" lumber are placed vertically in the tank at different points to protect the lining. This lining must be removed and replaced from time to time, as it is subject to the hardest service from the action of heat, acid and wear. This lining is secured in place by the use of copper or monel metal nails.

When concrete tanks are used, it is best to line them with lead, in order to keep the acid solution from attacking the concrete. This also serves to prevent the concrete from chipping easily.

Monel metal tanks within the last six years have been experimented with by several concerns due to certain inherent qualities not possessed by any of the other types of construction. This metal and its use in the pickling department will be discussed here at some length as the Motor Wheel Corporation has pioneered this metal's use in

the installation of probably the first monel metal pickling tank placed in service.

Monel metal is the trade name of a natural ore mined at Sudbury, Canada, by the International Nickel Company, with offices in New York City. The ore is comparatively free from impurities. It contains approximately 67% nickel, 28% copper, and 5% of impurities and other metals, such as manganese, cobalt, and iron. Its tensile strength varies from 60,000 to 140,000 pounds depending upon the method of working. It has a density of 8.80, melts at 1315° C or 2460° F; has a hot working range of 1800° to 2100° F; is red-short between 1300° and 1800° F; and will not stand severe deformations between the last above mentioned temperatures; heat conductivity 6.6% as compared to copper at 100%; Brinell Hardness (500 Kg) 80 to 105. It does not rust or oxidize at low temperatures and but little at welding temperatures. It is very tough and hard to machine; will be weakened when heated by a fire containing sulphur, such as a coal fire. Natural gas, fuel oil heat, or other fuels free from sulphur should be used. It is impossible to produce a weld having the same analysis as the monel metal.

From the above it will be noted that extreme care must be used in the working, fabrication, and use of monel. The cost of monel tank sheets at the mill averages about 50¢ per pound.

Monel was first used by the Motor Wheel Corporation in the form of containers for holding small parts in the pickling solution. These containers were in the form of large pails having many small holes punched through the metal to allow the acid to flow easily through the work. Tight joints were

not necessary and leakage was not a factor. These original containers are still in use in good condition after seven or eight years.

The construction of the tanks presented an entirely different problem as they had to be liquid tight and could not be made of one piece as is a thimble. Then too, the acid was in continuous contact with the tanks where the pails were only in the acid for short periods of time throughout the working hours.

The first monel tank was installed October 23, 1936. This was a 3/16" thick welded tank having three pieces in the bottom and onepiece for each end and side. It was not anticipated that trouble at the welds would be encountered so no special effort was made to use as few pieces as possible in its construction. See Plate #1 for the over-all dimensions of this tank but not the construction. The outlet in the bottom was also welded in. The life of the tank after considerable repairing was 35 months; failure occurring at the welds. Certain important information was gained, however, which was made use of in later construction.

It was noted that the steam jets and acid agitation caused considerable vibration; that welded joints as employed were unsatisfactory; that the monel was eaten away a maximum thickness of 0.022" per year at the surface of the solution while the average below the surface was 0.015" per year. Later tests on other monel tanks run very close to these figures, a figure of 0.03" is being used for estimation purposes.

As it was decided that when a tank became thinner than 1/8" thick it would probably give trouble and would have to be scrapped, it became evident that increasing the thickness from 3/16" to 1/4" should lengthen the life of the metal 100%, with only 33-1/3% additional metal and less than 33-1/3% increase in the cost since fabrication cost would remain approximately the same.

The next tank was constructed with 1/4" monel having lap riveted joints. The heads of these rivets were inside the tank and so a lead fillet covering was used to protect the rivets and to make the tank water tight. The tank was constructed of three pieces, that is, two sides and the bottom and ends being in one piece. It was thought that the increased thickness would sufficiently reduce the vibration as well as increase the life. In time it developed that the vibration and eating action on the tin which was applied before filleting, loosened the fillet, allowing the acid to eat the rivets and cause the tank to leak. Very little eating produced some loose rivets which had to be replaced with larger rivets when new fillets were applied.

The next tanks were constructed as are the present ones, with an open joint so that the rivet heads would not be exposed to the acid. This simplified the fillet installation since the leakage at the seams only had to be considered. Fillets have been constructed by fluxing directly on to the monel without tinning. The shape of the fillets have been varied to provide a longer path for the acid to eat its way out and to give more flexibility to overcome vibration; all

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these with varying degrees of success. The average life of a fillet being less than one year, generally the original fillet standing up the best.

The last tank was constructed with extra reinforced vertical angles at the sides and wider angles at the top to decrease vibration which has now been reduced to such a point as is not considered seriously objectionable. See Plate #1 for present construction.

It is evident that the fillets are the greatest source of trouble and maintenance cost and further improvement should be expected. Asphaltic plastic compounds were tried for use as fillets but would only last a few days due to the high temperatures, about 150° F being the maximum temperature they would withstand. Experimental work is now being carried on with pure rubber gaskets for joints; the rubber apparently being about double the life of the lead fillet; but this rubber must be replaced in time and bolts must be substituted for rivets at increased cost and shorter life due to the rapid eating of fine threads, which are exposed to acid fumes and some dripping and spilling of acid. Monel inverted channel shaped strips are used to lay over the edges of two adjacent tanks to prevent the solution dripping from the work and coming in contact with the outside of the tanks and the rivets or bolts as this would cause eating on the outside of the tanks. The tanks are also kept painted or oiled on the outside as a further protection. Tests indicate that eating occurs more rapidly where air is present as would be the case if dripping

occurred as before mentioned.

Some of the relative advantages of the use of monel as compared to other tanks are:

(A) Eliminates the cost of a large percent of the acid lost by leakage. (approximately $200.00 per year per old wood tanks).

(B) Reduces maintenance costs. (Wood tanks require three to four linings per year. Monel - one lead fillet, approximately saving $135.00 per year).

(C) Same depreciation charge based on six years' life.

(D) Less time out of service for repairs.

(E) Elimination of Wood splinters in sewers.

(F) Elimination of acid leaks in sewer water which gives sewer trouble and may have to be treated.

(G) Excessive and varying leakage in wood tank makes constant acid strength more difficult to control.

(H) Saves steam, shorter heating-up period; less radiation loss, less steam used.

(I) Easier to clean.

(J) Allows for installing 6 tanks in the space where 5 wood tanks were formerly used, thereby increasing tank capacity.

(K) Monel has considerable scrap value.

(L) When out of service does not have to be kept full of water to prevent drying out.

(M) Lead lined tanks puncture easily and lead sags.

(N) Acid attacks concrete tanks rapidly. They are not quickly repaired or replaced, and are subject to crack-

ing and breaking.

(O) Can be made to fit racks more closely as guide or wearing rails on sides of tanks can be eliminated, thereby reducing heating-up time and cutting down on the amount of acid wasted at dumping time.

DISADVANTAGES:

(A) Harder to machine.

(B) Expensive metal - increased first cost.

(C) Must use extreme care in fabricating.

(D) Fillets may be very short and of un-uniformed life.

(E) Size somewhat limited by the size of sheets available.

(F) Extra precautions necessary due to vibration.

(G) More skill required to make repairs.

Chart #3 gives the time in service of 13 monel metal tanks, the majority of which are still in service.

Tank #A failed at joints - it was 3/16" metal.

Tank #B failed at joints due to welding.

Tank #2 failed because of serious mistakes in fabricating the joints, the metal was reworked for #11 tank, which is much smaller.

The remainder of the tanks are in good condition and at least double their present life may be expected.

OUTLETS IN TANKS

The drain or outlet in any type of pickling tank has always been a source of leaks and trouble so much so that many tanks are built without it, and the solution is either

siphoned or pumped out of the tank; this however, is slow, expensive, and renders the cleaning of the sludge or iron salts in the tank more difficult.

The present outlets in the monel tanks are made as shown in Plate #3 and are proving very satisfactory. It is expected that they will only have to be reworked about twice in the life of the tank, the flange being used for the next larger size hole and the plug for the next smaller, in reality making them as long lived as the tanks. The leakage is negligible. The inexpensive gaskets are replaced about once a month. The square jackscrew threads having very long life.

The original outlets were built in somewhat the same manner but gave endless trouble. The flanges were bolted on instead of sweat on with lead and leaks occurred around each bolt. The thread on the plug was standard pipe thread. The gaskets were lead. They set up a battery action that etched the tank beneath them; they offered too much resistance to tightening and did not stop the acid from leaking and became loose soon after being placed in service as the lead had little elasticity. This leakage allowed the fine threads to eat off on the bolts quickly. The nuts due to vibration would also loosen since no monel lock washers are available as yet.

Rubber gaskets were used but the grinding action in putting in the plug tore them badly and they sometimes leaked when first installed and could not be used over the second time. The gasket now being used is one manufactured

by the Johns-Manville Company and goes under the name of "Blue African Asbestos Fibre Gasket". Elimination of the bolts and their gaskets left a smooth bottom which facilitates easier draining and cleaning.

SUPPORTING BLOCKS IN TANK

Four acid resisting phosphor-bronze metal blocks 10" x 10" x 3" which are the same material as the racks are constructed from are laid in the tank bottom in such a manner as to support the four corners of the pickling rack. They are held in place by a light weight monel fabricated frame. These blocks distribute the load more evenly, prevent excessive wear at these points, and hold the rack out of the sludge or iron salts which would otherwise adhere to the rack and steel, preventing uniform pickling and clean work. They withstand the acid as well as the tanks. This cast metal costs approximately one-half that of monel, or about 35¢ per pound.

STEAM JETS FOR HEATING

Plate #3 illustrates the monel metal steam jet in its present form. This jet is light in weight, isn't subject to breakage, eliminates joints, takes up but very little room, and agitates as well as directs the heat in the solution. Various sizes of tubing and different arrangements have been used until vibration has been greatly reduced and agitation

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increased. The jet is designed so that one is sufficient for heating each tank. It has an average life of six months, wearing out rather than eating out. Increased tube thickness may be tried to prolong its life although difficulties may be encountered in making the short bends required.

The life of lead or brass which this jet replaced averaged two weeks. The above qualities mentioned could not be inculcated in their design. A commercial cast metal jet constructed of acid resisting metal was tried but was complicated in shape, expensive, heavy, caused greater vibration, and was finally accidentally dropped and broken.

BATTERY ACTION IN THE SOLUTION

The following measurements were made by connecting one lead to a piece of steel in the load in the monel metal crate and the other lead to the monel metal pickling tank. The crate and the load were suspended from the hoist so that the crate would not come into contact with the bottom of the tank or any other part of it. The load being pickled consisted of 119 pieces of steel, 55-1/4" long, 4-3/8" wide, 1/8" thick. The tank itself measured 90" long, 46" wide, 32" deep and was filled to a depth of 28". The pickling solution had a specific gravity of between 20-25° Be. and an acid strength of from 5 to 6%. The voltage developed between the monel metal tank and the load in the hot acid was 475 millivolts, the load being anodic to the tank. Under the same conditions, the current flow was found to be 2.9 amperes. In the cold acid

the voltage dropped to 400 millivolts with a current flow of 0.9 amperes, the direction of flow being the same as with the hot acid.

The calculated area of monel metal in the tank exposed to the acid was 11,756 square inches or 760 square decimeters. The area of steel being pickled was 59,381 square inches or 3,830 square decimeters. From these figures it can be shown that the cathode current density equals $\frac{2.9}{760}$ or 0.0038 amperes per square decimeter, which is equivalent to —

$\frac{3.8 \times 24 \times 60 \times 60}{1000} = 328.3$ coulombs per square decimeter per day. In a similar manner it can be shown that the anode current density equals $\frac{2.9}{3830} = 0.00076$ amperes per square decimeter or $\frac{0.76 \times 24 \times 60 \times 60}{1000} = 65.66$ coulombs per square decimeter per day. This flow of current would be equivalent to the corrosion of steel at a rate of 65.66 x .28394 equals 18.65 milligrams per square decimeter per day.

It is quite apparent then that the galvanic forces have very little effect on the rate of solution of the steel. It was not possible to measure the magnitude of the flow of current between the steel load and the pickling rack itself since they were in intimate contact. This flow of current must be appreciable and is probably greater than the portion flowing from the rack to the tank itself. Whether or not the flow of current towards the monel tank is sufficient to have any appreciable effect on the corrosion of the metal is a matter of conjecture, although it should be expected to be quite appreciable.

As a matter of interest it was calculated that if the flow of current towards the monel metal were in opposite direction, that is, tending to corrode the metal rather than to protect it, it would be equivalent to corrosion at a rate of 103 milligrams per square decimeter per day, which would result in penetration of a sheet to a depth of about .017 inches in a year of continuous contact with the pickling solution.

From the above it will be noted that what little current is flowing acts as a protection to the tank.

VENTILATION

An exceedingly important factor in the pickling department is the proper ventilation of the pickling room. It is necessary to remove the acid fumes with the least possible damage on their way out of the room.

Acid vapors are injurious to the life of the building structure, to the life of the machines in the building, and to the health of the workmen.

A positive air change of once every two minutes will give proper ventilation if the fumes do not pass the workmen at all, or the structural work before being diluted.

In the summer, fumes may sometimes be removed without difficulty through open windows and doors. When cold weather prevails, an additional factor enters, namely, the formation of a heavy fog by contact between the cold air and the vapors rising from the tanks. It is, therefore, highly desirable

to maintain a temperature of 60° to 70° for the air that comes into the pickling room to displace the vapors rising from the tanks. If such temperature is maintained, there is practically no fog formed. Exhaust or used air from other parts of the shop may be used for this purpose before being thrown away.

The ventilating equipment best suited for a pickling installation varies greatly with local conditions, such as location of surrounding buildings, location of windows and doors, height of room available, and still other factors.

It is impossible, therefore, to describe one ventilating system that will fit all conditions. Instead, three different systems, which have given excellent results in three different plants, will be described. At least one of these systems can be used with slight modifications to fit most any condition.

One of the most modern ventilating systems used in pickling rooms is to place suction fans at opposite sides of the room near the roof. In this way, the acid fumes are carried out in a simple manner. Suction fans are made of anti-acid metal, such as monel. The replacement air may be replaced by still other fans which force the new air in at floor levels so that it passes the workmen.

Another excellent method of ventilation is to take the cold air from the outside, passing it between hot steam pipes, and then forcing it into a large horizontal pipe. From this pipe, which is located about fifteen feet above the floor level, several downward outlets branch off, sending warm air into the room near the floor level. The acid fumes are

carried upward by the warm air entering, and they pass out of the building through a single row of windows at the top.

In the third method, air is forced upward through pipes projecting from the floor at an angle of 45 degrees, and on the side of the room which is opposite the tanks. By this method the room is heated in winter and cooled in summer, and the rising fumes are caught by the blast and carried out through a ventilator.

When a pickling department must be located in a large building containing machinery, a very good plan is to build brick walls around the tanks; these walls will direct the fumes upward twenty to thirty feet. A fan placed in the ventilator above will remove the fumes with comparatively little damage to equipment. If the fumes are objectionable as they leave the building, tall stacks can carry them to such heights that dilution will take place before they can do any damage.

HANDLING THE MATERIAL

The handling must be adapted to the type of product to be pickled. In small plants the work may be hung in the tank by hand by the use of hooks or baskets. Larger installations warrant the use of more elaborate equipment. "I" beams are used above the tanks with trolleys and hoists for moving the work along the "I" beam and raising or lowering the work into the solution. Either hand power, or mechanical power utilizing electricity or air may be

employed to operate the trolleys and hoists. Electric cranes are used for this purpose in some plants. The operator may ride and operate the crane either from the floor or from a ceiling location. The ceiling location subjects the operator to more heat and fumes and is objectionable. Conveyors through the solution are used but this is hard on the handling equipment.

In still larger departments, pickling machines which carry several racks in a rotating circle are in general use. The tanks are arranged in a circle to conform to the machine. The machine is arranged to quickly raise and lower the work through a small distance to produce agitation and more quickly pickle the steel.

COST OF PICKLING

Pickling costs are generally estimated per ton of material pickled. The cost of pickling a ton depends upon many factors, among which are:

1. Ratio of weight of material to surface area of material — since pickling is a surface action.
2. Payment of labor — by wages or piece work.
3. Method of buying acid — by carload or carboy.
4. Length of time of contract for purchasing acid.
5. Grade of acid used.
6. Location of boiler and cost of steam for heating.
7. Cost of water used.
8. Method of handling material.

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6. Location of boiler and cost of steam for heating.
7. Cost of water used.
8. Method of handling material.

9. Temperature of pickling bath.

10. Disposal system for spent acid solution.

11. Other but minor influences.

With so many factors influencing pickling costs, no general cost data can be given. However, large steel sheets have been pickled for less than $1.00 per ton, while small steel sheets under similar conditions run $2.60 to $3.00 per ton.

DISPOSAL OF THE ACID SOLUTION

AFTER IT HAS SERVED ITS PURPOSE:

At one time very little attention was given to this important phase of pickling. The spent acid was either dumped into the sewers or onto lower waste land where the water could evaporate or seep away in the ground.

However, it was found that sooner or later the acid would attack the sewers and render them inoperative. The concrete being consumed first, the vitrified brick or tile being effected but little by the action of the acid. Vitrified sewers are constructed with concrete joints so they are but little better than concrete sewers for carrying the acid.

Waste low land is not always available for acid disposal and in time the acid drainage from this land would travel to other sections where it would be found objectionable.

State governments have recognized the damage done in the way of stream pollution from factory waste, acid being

one of the major items, and have enacted laws governing the proper disposal or treatment of these wastes before they may be discharged in public waters. Their attitude being that public waters must not be polluted with any substance which is a menace to public health and comfort, or which will tend to stupify, injure, or kill fish or aquatic life. A copy of Michigan Laws can be found in Act 245 of the Public Acts of 1929, State of Michigan.

Municipalities have ordinances which prohibit factory wastes being discharged into sewers which will cause damage to them or give trouble in disposal plants. It is self-evident that acid wastes fall clearly within the class of wastes as above covered, therefore, it becomes necessary to so treat or dispose of this waste as to meet with the municipal and state requirements.

The method used will depend upon the conditions encountered as well as the cost involved. From an economical standpoint it is apparent that as little acid should be treated as possible. Generally it is sufficient to treat only the free acid. Of course, the ferrous sulphate which contains the large bulk of acid which has been consumed in pickling will have a part of this acid set free when it is diluted with river water but in most cases this combined acid is not required to be treated.

Tank leakage should be eliminated as far as possible to lessen the expense of treating the waste from the pickling department. This acid solution leakage has a much higher percentage of acid than is usually encountered in the dumped solution.

The acid in the solution should be consumed as far as possible before dumping. This is accomplished as stated under the discussion headed "Maintaining Acid Solution in Proper Condition" and as illustrated in Chart #3. It will be noted from this discussion that while the steel is being pickled through-out the major portion of the life of the solution the acid strength is held for example, at about 8% by volume while just before dumping the percentage should be lowered to 2% or 3% if possible.

The treating process may be accomplished in the pickling tank, in a pit just below the tanks, or in a specially constructed pond or reservoir at some distance from the pickling department and preferably on lower ground so that the liquid will flow by gravity to the treating pond. The discharge from these treating processes should be high enough if possible to again flow to the sewer by gravity. The location of the treating process should be as close to the pickling department as conditions permit, as expensive tight acid resisting sewer construction between the two will have to be maintained.

The acid wastes may be satisfactorily treated by one of the following methods:

(A) Recovery Method - where the acid is reclaimed before discharging to sewers.

(B) Killing or spending the acid - by allowing it to re-act with some element such as iron until the acid has been completely used.

(C) Dilution Method - with water to such a point that the percentage of acid becomes harmless.

(D) Neutralization Method - by use of an alkaline substance such as lime.

These different methods will be discussed in turn under the letter headings as given:

(A) Recovery Method - this method is not in general use. There is at least one concern who employes this method. It requires more elaborate equipment and the use of chemical engineers. The reclaimed acid is again used in the pickling tanks and the iron sulphate is reclaimed and disposed of by selling to others who have use for it.

(B) Killing or spending the acid - by allowing it to re-act with some element such as iron. This method is based on consuming the free acid in the same manner as it is used up in the pickling process. It has been previously explained that low percentages of acid in the pickling solution prolong the pickling time, also that the iron salts do likewise. Therefore, to completely use all the acid in the pickling solution would take more time than could be given and the steel would become pitted and damaged, so if this free acid is to be completely consumed it must be removed from the tanks to a reservoir in which inexpensive scrap iron is placed and the action allowed to continue until the acid re-action is complete. This will be a very slow process as the weak acid will re-act but slowly. It will not be economical to maintain high temperatures in this reservoir which will again further slow the action. More than one reservoir will be necessary as considerable time must be allowed after adding the last pickling solution so

that the re-action will be complete before dumping. The reservoirs must be of acid-proof construction. The iron salts that accumulate in the bottom of the reservoir must be removed from time to time.

(C) Dilution - This requires a large amount of water and a large pond, however, if plenty of free water from sewers or streams is available this process may be feasible. The pond must be large enough so that the dilution will be such as to render the acid strength harmless to sewers or to public waters when it is discharged. This method is seldom used alone but generally in conjunction with the neutralization method. Most waters are slightly alkaline and act as a neutralizing agent and will, therefore, be treated under that heading.

(D) Neutralization Method - this is the general and most accepted method used by most industries. Two systems of this type are in use at the Motor Wheel Corporation, Lansing, Michigan. The smaller system employs a concrete pit coated with asphalt just beneath the pickling tanks. This pit has a capacity of 8,250 gallons. The sewer outlet is located at the lower end of the pit and is provided with a tight plug. All overflow and dumped rinse and lime water is collected in this pit from the pickling and galvanizing departments. This waste rinse and lime water is sufficient to fill the pit by the time it is necessary to dump a 500 gallon pickling tank. By alternating, only one tank is dumped to each full pit of water. After allowing for neutralizing and dilution time the pit is emptied by removing

the sewer plug and the operation is again repeated. This operation may be repeated at half or full day intervals, depending on the amount of production which may run as high as 300 tons per 14 hour day.

The pit is not subjected to strong acid solution because it is first filled with water and lime solution and since the volume of the comparatively cold water is large to the volume of hot solution dumped the resultant temperature is low. Any tank leakage is taken care of in this pit also.

The concrete pit was constructed in 1923 with paving brick laid in asphalt for a lining. Wood tanks were then in use and were set in the pit. Two years ago the tanks which had been replaced by monel construction were raised above the pit and the pit was repaired and coated with asphalt, the brick being removed from the side walls, and has been in constant use since for treating the acid. The pit shows no evidence of disintegration.

The neutralizing may be carried as far as required by the addition of more lime. The lime should be added in the form of lime water such as is used in the liming operation of the pickling department. Lime added directly re-acts quickly on the surface forming a calcium sulphate; this forms a ball or lump and is then very slow to break up or mix in the solution. Calcium sulphate is insoluble in water and large amounts cannot be carried through the sewers without giving trouble.

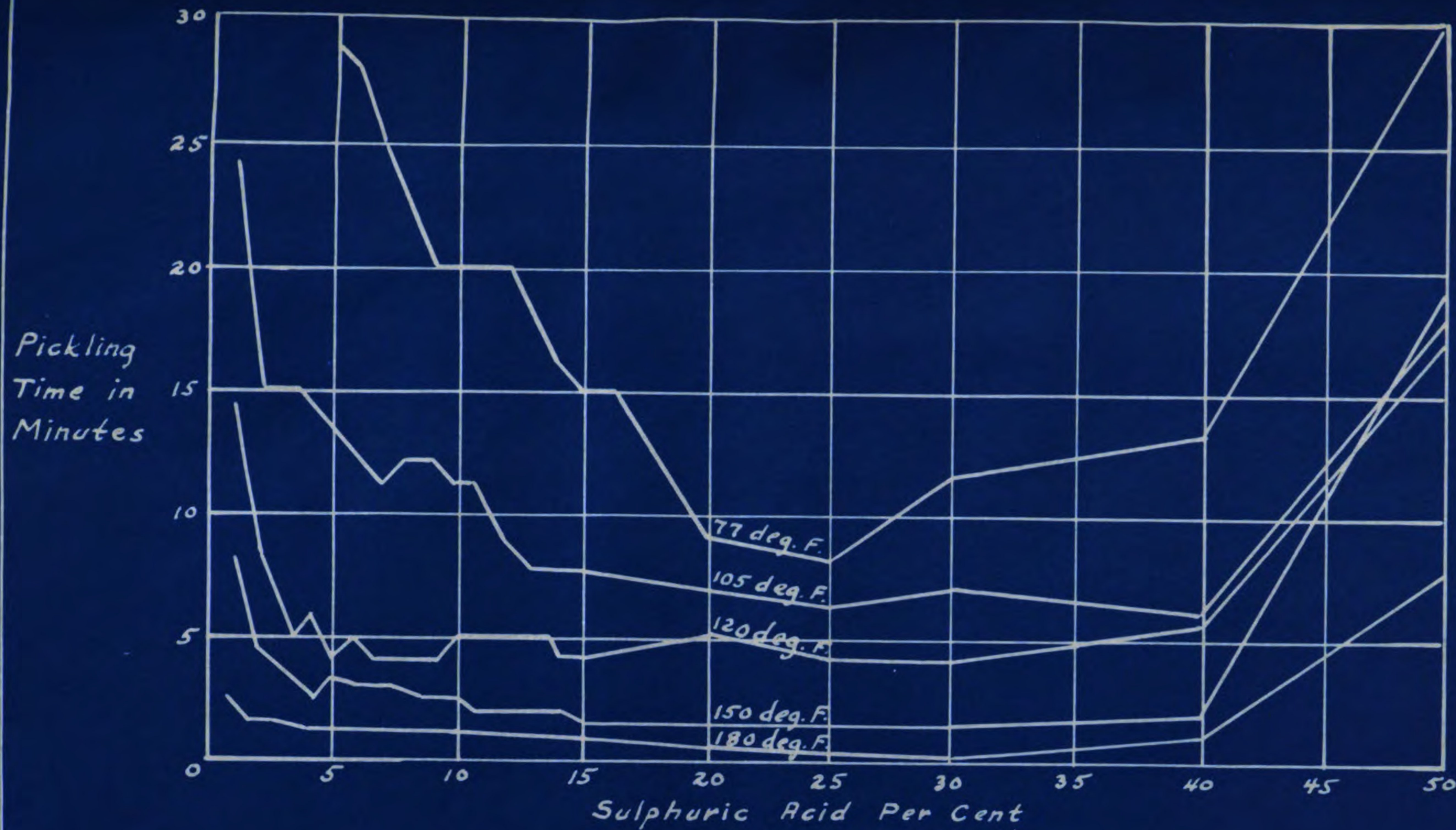
Tests taken from the pit when dumped show an acid strength of from .01% by volume to .31%, which is nearly 1/3 of 1%. The average being .08%. These results appear to be satisfactory to the Municipal and State authorities. Since the majority of sewer waters are alkaline this remaining acid will probably be neutralized before reaching the disposal plant or public waters. The larger system which has been in use two years takes care of a pickling department having a maximum capacity of 1,000 tons of steel per 24 hour day. The treating reservoir, costing approximately $13,000.00 to construct, is located on lower ground about 300 feet from the pickling department. This reservoir is constructed of concrete coated with asphalt, it has a capacity of 150,000 gallons of water. The total capacity of the 14 wood pickling tanks is 15,750 gallons or at 3% strength by volume would contain 472 gallons of ($H_2 SO_4$) Sulphuric Acid. All tanks are dumped at one time and if the reservoir were full of neutral water the dilution would be .0031% or $\frac{31}{100}$ %. To this is added the lime water and also all factory water containing alkali washing solution dumped from washers. The outlet to the sewer is constructed with a siphon overflow and also a method for completely draining the reservoir when cleaning is necessary. The normal operation is to use the siphon which prevents oil from leaving, thereby the reservoir serves the dual purpose of acid treating and oil removal. The oil on the surface is drawn off from time to time as required and used to lay dust on roads or burned as fuel in the boiler plant.

Actual tests taken from the continuous overflow outlet show the acid strength to run from .02% to .125% when the department is operating at approximately 70% capacity.

Tests conducted at the junction of the plant and city sewer before the installation of the reservoir showed the condition to vary from an alkaline percentage of .04% to an acid percentage of 5.9%. Other tests taken at the mouth of the city sewer at the river approximately three-quarters of a mile away, before the installation of the reservoir, showed the sewer water to run alkaline except at times when tanks were being dumped, the percentage running as high as .888%. Seepage from the sewer at this point at one time caused a gas line to be eaten through.

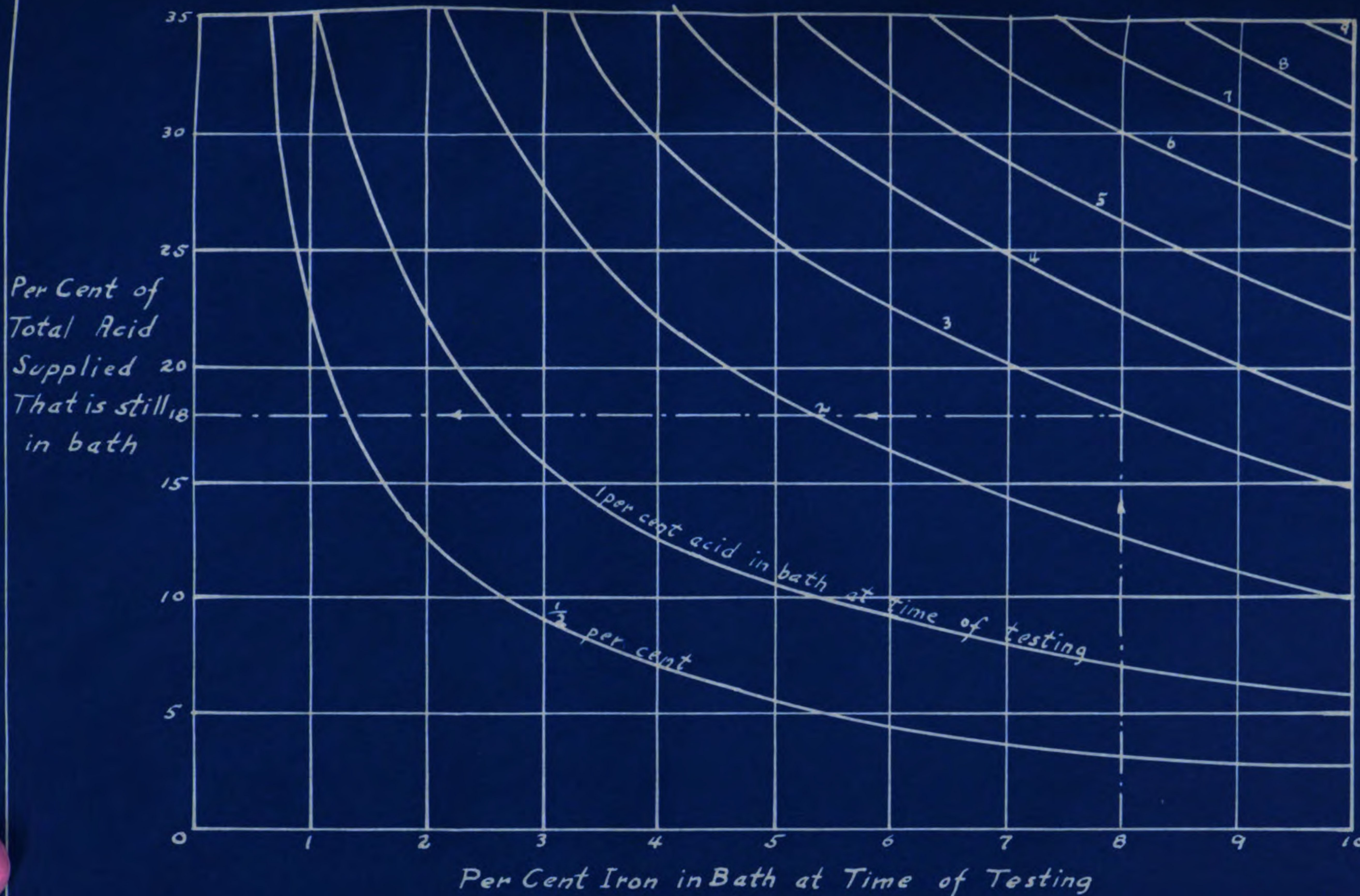
Other substances besides hydrated lime, which is used in the lime tanks, may be used for neutralizing if it is found necessary to add more neutralizing agents. Quick lime is generally inexpensive but requires a slacking process before using. Finely ground lime-stone or agricultural lime, or lime waste from a sugar plant may be used; several days being required to complete the process, but the cost of the lime is very low.

Soda Ash has been used for neutralizing and works very satisfactorily. The sodium sulphate formed in the re-action has the advantage of being soluble in water and does not form a sludge to be cleaned out of the pit or clog sewers. The objections to this being the cost which is several times that of lime and also the large amount of fumes generated which may prove objectionable.



Results of tests by J. T. Robson, Ph. D. on twenty gage sheet steel which had been given a light anneal. The tests were made with a rather fresh bath and are useful in showing the relative and not the exact influence of the factors of acid strength and temperature on pickling time.

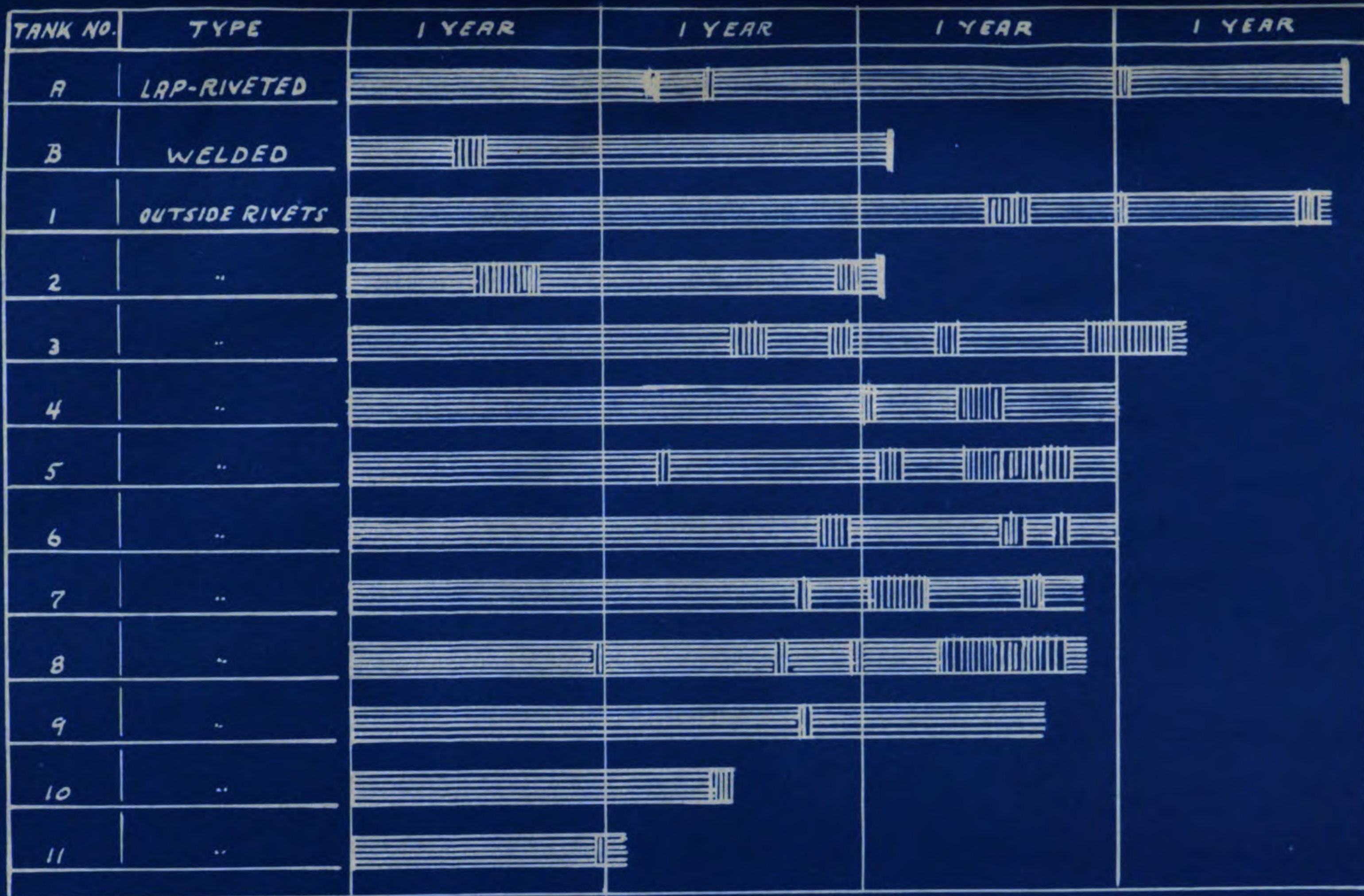
5-1-32 Chart #1



The example shows that 18 per cent of the total acid supplied to the original bath and by additions would be lost if the bath were dumped when it tested 8 per cent iron and 3 per cent acid. By W.H. Shipman, Asso. Ed., Am. Machinist.

5-1-32

Chart #2



MONEL PICKLING TANKS — TIME IN SERVICE (COMPILED 5-1-32)

NOTE :- TANK "A" SCRAPPED - MARCH, 1931
" B " - JANUARY, 1929
" #2 " - JULY, 1930
ALL OTHER TANKS ARE IN SERVICE

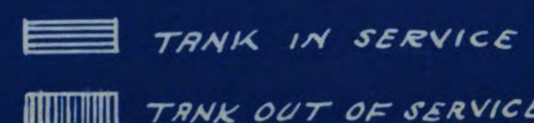
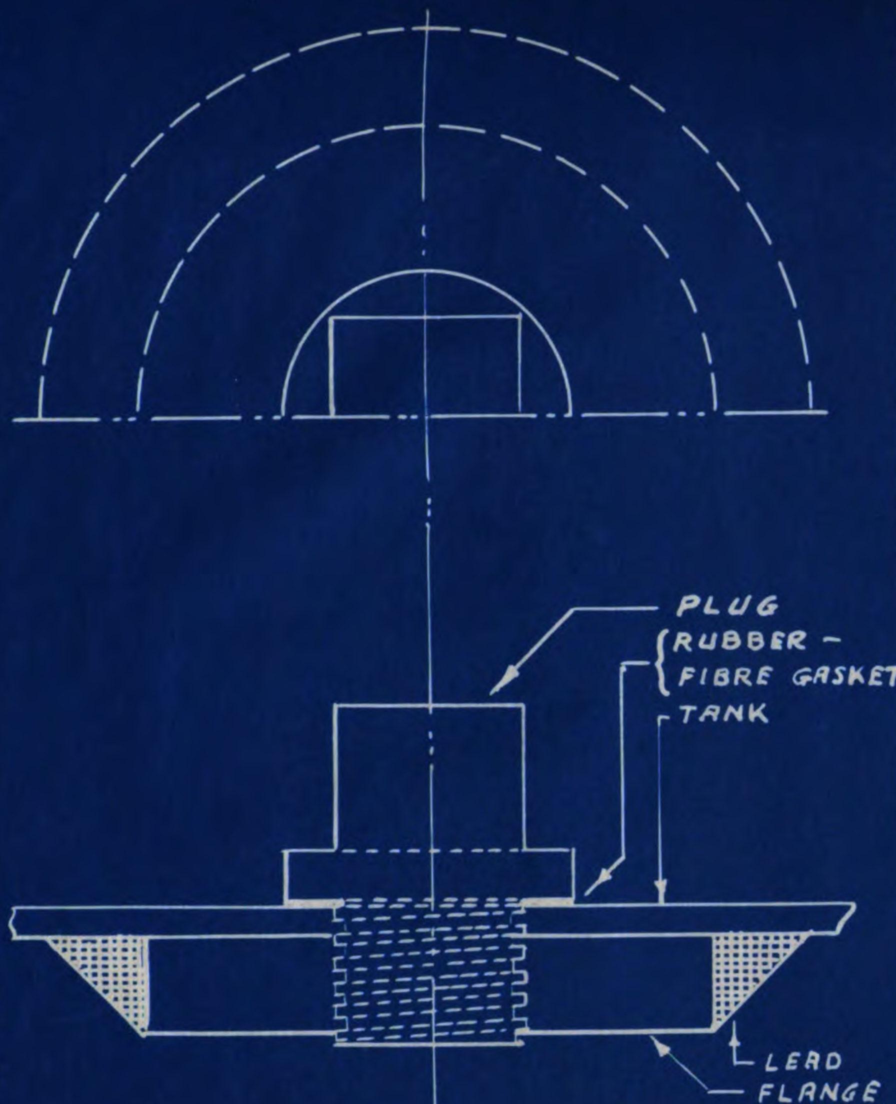
LEGEND -


Chart # 3

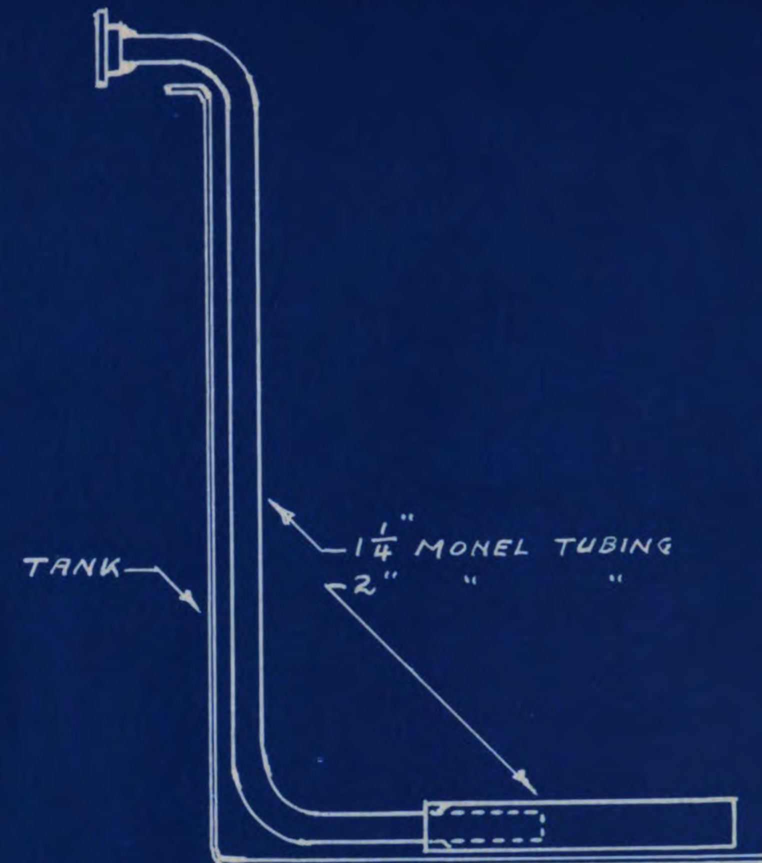
5-1-32



SECTION THROUGH ℄
½ SCALE

MONEL OUTLET PLUG & FLANGE ASSEMBLY

5-1-32 PLATE #2



MONEL PICKLING TANK STEAM JET
1/8TH SCALE

5-1-32 PLATE #3

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