

TECHNICAL DATA SHEET

GIBONOL S - 34

BLACKENING PROCESS FOR IRON AND STEEL

Gibonol S-34 is a process for blackening iron and steel by simple immersion. Uniform, black coatings of ferroferric oxide, $\text{Fe}_3(\text{FeO}_2)_2$ are produced which are an integral part of the basis metal; that will withstand severe deformation without flaking. Beautiful, shiny, black coatings are obtained on buffed or polished surfaces, and flat non-reflective coatings are obtained on etched or sand blasted steel. Gibonol S-34 is a powdered product which is dissolved in water to make-up the blackening solution. The process blackens a wide range of alloys, low and high carbon steels, and low alloy steels, some cast and malleable irons, depending upon their composition. However, it will not blacken stainless steel.

Gibonol S-34 contains complexing agents that eliminate the effects of copper and iron contamination in the blackening solution. In other words, red smut formation on the work, caused by copper and iron contamination of the solution, is eliminated. It also contains a surfactant that reduces surface tension thereby lowering drag-out and reducing spray. The amount of sludge formation in Gibonol S-34 solution is less, thus affording a substantial saving in the amount of heat required to maintain the operating temperature.

In general, the lower the carbon content of the steel, the higher the corrosion resistance. Gibonol S-34 coatings will withstand from 2 to 6 hours in 20% salt spray, which is sufficient for most indoor use. For outdoor use, additional protection should be provided by oiling, waxing, or lacquering the work after blackening.

The dimensional changes involved in blackening are extremely small, being less than 0.01 mil. the thickness of the black oxide coatings range from 0.06 to 0.1 mil

HOW TO USE GIBONOL S-34

OPERATING CONDITIONS

Concentration	840 to 900 gm/ltr
Temperature	138°C - 142°C.
Time	3 - 25 minutes

MAKE-UP

Clean the tank thoroughly before making up the solution. Remove any rust and scale, otherwise it will dissolve in the solution and complex with the additives, thereby reducing the blackening effect.

Fill the tank about half full with cold water. While stirring with an iron paddle, slowly add 840-900 gms. of Gibonol S-34 for each litre of final solution desired. Continue stirring until the salts are almost completely dissolved.

Add water to raise the solution level to within about two inches of the final solution level which should be at least 6 inches below the top of the tank. **Do not fill to the mark because the solution expands on heating.** Heat the solution to boiling which should start at 138°C. If boiling occurs below 138°C., slowly add salts during heating until the desired boiling point is obtained. Lower the heat input, so that the solution boils gently.

TEMPERATURE

The blackening solution must boil within the recommended operating temperature range - 138°C -142°C. Such temperatures can be achieved without boiling if the concentration of salts is too high, but successful blackening will not be obtained.

As water evaporates from the bath, the solution will become concentrated and the boiling point will rise. When the boiling point reaches 142°C, water must be added to dilute the solution and thereby lower the boiling point to 138°C. The temperature of the solution should never be allowed to rise above 155°C. Overheating will destroy the complexing agents present in the solution and cause a pronounced deterioration in the quality of blackening obtained.

Do not try to adjust the temperature by means of the heat input. The temperature of a liquid in an open vessel cannot exceed its boiling point. Control of the boiling point is done only by additions of water (or salts if the boiling point is too low), and the heat should be adjusted so that the solution always boils gently.

SINCE THE BLACKENING BATH OPERATES ABOVE THE BOILING POINT OF WATER, ADDITIONS OF WATER SHOULD BE MADE VERY CAREFULLY TO AVOID SPLATTERING AND ERUPTION OF THE SOLUTION (SEE "EQUIPMENT" AND "CONTROL").

Note that control of the boiling point by addition of water automatically controls the concentration. Thus an automatic temperature controller which regulates water input instead of heat input will maintain both the correct boiling point and the correct concentration of Gibonol Salts (see "Equipment").

OPERATION

Component/Job to be blackened must be clean and free of rust and scales. The following cleaning cycle is recommended :-

1. Clean in Steelex K-20, 60 gm/lit at 80°C for 3 to 5 minutes as required.
2. Rinse in running cold water.
3. Remove rust in Gindox 214 Alkaline derusting solution, 250 gm/lit at 90°C for 1 to 5 minutes as required.
4. Rinse in running cold water.
5. Blacken in Gibonol S-34 at 138-142°C; 3 to 25 minutes.

The job may be put in baskets or barrels, or may be placed on racks or hooks. When immersing the job into the boiling Gibonol Solution, agitate it slightly to make sure that all surfaces are wetted by the solution. Work being blackened in baskets or barrels should be tumbled occasionally to expose contact surfaces to the solution. The time required for blackening may range from 3 to 25 minutes depending on the alloy and the volume of the work in tank. After twenty minutes, however, no further increase in the thickness of the oxide coating occurs.

RINSING

After blackening, the work is normally rinsed in running cold water. However, if the part contains recesses that are difficult to rinse, it may be best to rinse in warm or hot water to prevent precipitation of salts in the crevices, which may be difficult to rinse out by successive rinses. If a hot water rinse is used directly after blackening, followed by running cold water and then with a final hot water rinse with Gintek NR 37 may be used in the final rinse to promote stain- free drying and impart additional corrosion protection.

CHROMIC ACID DIP

A dip in 4.5 to 5 gm/lit of chromic acid for a few seconds is recommended to neutralize the alkaline film whenever the work is to be oiled, waxed or finished with lacquer or enamel. The chromic acid neutralization usually greatly increases the salt spray resistance afforded by the subsequently applied organic coating. The more dilute chromic acid rinse may be the final cold or hot rinse. A running rinse maybe used after the stronger chromic acid rinse.

DRY

Small parts are best dried in sawdust. A higher luster is imparted in sawdust drying due to the removal of any surface dust which may be on the work. If the work is to be oiled with soluble oil or is waxed, it can be transferred, after reasonable draining directly from the running cold water rinse or chromic acid bath to the hot soluble oil or wax. Also wax coated sawdust may be used for simultaneous drying and waxing of small parts. Simply add the wax emulsion into the sawdust barrel.

EQUIPMENT

No metal other than steel or stainless steel should come in contact with the Gibonol solution. All racks, baskets, treating cylinders, and tanks must be made of welded steel and not soldered or brazed steel.

Exhaust ventilation is recommended.

TANKS

The Gibonol S-34 tank should be made of steel or stainless with sturdy, oxide-free double welds. The solution weight is about 1.7 kgs. per litre, as such steel of sufficiently heavy guage to hold this weight must be used; for example, tanks from 200 to 800 litre capacity with solution depths up to 36" require 1/4" thick iron plate and a bottom of 3/8" thick plate. The depth of the tank should be such that the solution level will be at least 6" below the top of the tank, and the solution should be deep enough to allow the solution level to drop at least 2" without exposing the work. This is important because it will permit less frequent additions of salts.

HEATING

The tank may be heated with gas, high pressure steam, or electric immersion heaters. Gas is usually the most economical and practical method. For gas heating, use pipe burners placed underneath the tank. Approximately 500 BTU are required to heat one litre of the Gibonol S-34 solution from room temperature to operating temperature. After the operating temperature is reached, only a fraction of the heat input required; is merely enough gas is used to make the solution boil gently. When gas heating is used, the tank should be insulated on four sides by a wall of 1" thick magnesia held together by steel casing. The insulation should be placed about 1" from the wall of the tank to allow heat to rise along the tank walls from the burners at the bottom.

Heating can be done with closed steam coils if high pressure steam (80 lts. per square inch) is available. Stainless steel coils are recommended since steel coils will undergo continuous oxidation in the bath thereby causing leakage of high pressure steam and eruption of the solution. Place the steam coils on one side and on one end of the tank - not on the bottom. Insulate the four sides and bottom of the tank with asbestos of 1" magnesia insulation holding it on with a steel casing.

If electric immersion heaters are used approximately 60 KW hours are required to heat 400 litres of solution from room temperature to the operating temperature. To avoid very high amperage during heating up of a cold solution, 220 or 440 volts power should be used. Stainless steel sheathed units are recommended.

CONCENTRATION AND TEMPERATURE CONTROL

There are two methods of supplying water to replace loss by evaporation. One method is automatic and the other is manual. The automatic system involves the use of a water valve which is actuated by a temperature control when the boiling point rises above 142°C. The valve passes water from the

feedline to the operating tank. It is strongly recommended that the automatic mechanism be used because it will assure reasonably trouble-free operation.

The manual method requires a water valve for manual operation and a dial thermometer which must be watched closely by the operator.

With both methods of control, a 1/2" or 3/8" water pipe should run just above the normal solution level. The end of the pipe should be capped and a 1/8" hole drilled in the pipe on the side facing the tank wall. Also, the end of the pipe should be covered with a splash guard to minimize the spread of solution spray when the cold water comes in contact with the solution.

Since no design of splash guard can eliminate spray completely, it is important that the splash guard be located on a tank wall away from the loading side or end of the tank. At the same time, it must also be kept away from the end of the tank where the automatic temperature controls are mounted. The motor-operated water valve should be located at least six feet away from the splash guard and any other part of the Gibonol tank. Similarly all manual valves, by-pass valve, strainer, and electrical equipments should be at least six feet away from the tank. A shut-off valve in the water line should be so located that the operator can reach quickly should such ever be necessary. DO NOT, under any circumstances, mount valves and electrical equipment behind the tank in any unprotected location.

The introduction of water into the Gibonol tank under high pressure or in large volume may cause strong eruption so that the splash guard will be of little help. Therefore, a needle valve should be placed in the line between the automatic valve and the splash guard, and the needle valve should be adjusted so that water is added slowly when the automatic valve opens. Also, if water line pressure is greater than 40 psi, the installation of a pressure reducing valve to reduce the pressure to 25 psi is recommended.

CONTROL

The only additions required for Gibonol S-34 solutions are water and salts. Water is lost by evaporation and drag-out, and salts are lost by drag-out. Water is added either automatically or manually (see "Equipment") to maintain the boiling point within the recommended temperature range. When the solution level falls below the desired operating level, even though sufficient water has been added to maintain the proper boiling point, an addition of salts is required. The addition of salts will cause the boiling point to rise, thereby necessitating additions of water and resulting in a rise in solution level. As a thumb rule, addition of 14 gms. per litre of Gibonol S-34 salts will raise the boiling point by 1°C.

If water is added, either manually or automatically, and the solution is not boiling, the water may not mix readily with the solution. Instead, the water may form a clear layer on the surface of the solution and, when it suddenly mixes in it, it will partially turn into steam causing an eruption which may force some of the solution out from the tank. To prevent this, make certain that the heat is adjusted so that the solution boils gently when water is added. The boiling action will cause the water to mix rapidly. Addition of a large amount of salts at one time may cause the solution to stop boiling until the solution heats up to the higher boiling point. Therefore, smaller and more frequent additions of salts are recommended. If water is added when the solutions not boiling, it should be stirred mechanically by means of a steel or iron paddle until proper boiling point is reached.

ESTIMATION OF CONCENTRATION OF GIBONOL S 34 IN BATH SOLUTION :

Pipette out 10 ml of bath solution in 100 ml standard volumetric flask. Add distilled water to make the level to 100 ml. Mix well.

Pipette out 5 ml of this 10% solution in a 250 ml conical flask. Add about 25-30 ml distilled water and few drops of Phenolphthalein indicator. Titrate with 1 N sulfuric acid to colourless end point.

Calculation :

$$\text{Rdg} \times \text{N Sulfuric Acid} \times 114 = \text{gms/ltrs concentration of Gibonol S 34.}$$

TROUBLE SHOOTING

If the work fails to blacken, it is usually due to one of the following causes:-

1. The work contains too high a percentage of alloying elements.
2. Large amounts of cold work were placed in a relatively small volume of solution, causing the solution temperature to fall. In such cases, the work becomes passive and requires a dip in dilute muriatic acid prior to reblackening. Reduction in the amount of work entering the tank and use of a hot water rinse before entering the blackening solution will help to eliminate drop in solution temperature.
3. The boiling point of solution may be below required temperature. Frequently thermometers fail. If the solution starts to turn red, it indicates that the amount of iron in the bath is too high. This may occur when there is an excessive drag-in of iron salts. Improved rinsing will minimize this condition. Hence, alkaline pickling in Gindox 114 is preferable to acid pickling. Excess iron salts may also occur, if there is a large amount of high alloy steel in the work being blackened. In either case, the addition of Gibonol S-34 additive to the bath, will effectively correct this condition.

If the solution turns yellow, it is an indication that the complexing agents needed to eliminate the effects of copper and iron contamination have been destroyed by overheating (see "Temperature"). If these complexing agents are not replaced by the addition of Gibonol S-34 additive, some of the advantages of the Gibonol S-34 solution will be lost.

CAUTION :

Gibonol S-34 salts contain caustic alkali together with the strong oxidizing agents. Solutions containing these materials or the solid salts should not be allowed to come in contact with any organic matter, such as cloth, oil, wood, rubber, paper and sawdust and definitely must not be allowed to come in contact with any chemical reducing agents such as sulphur, phosphorous or sulphides because there may be danger of ignition or explosion. Use clean steel or stainless steel shovels, containers, scoops and paddles. Keep the salts away from fire, heat, sparks or flame.

Use protective clothing when making additions of salts or when operating the bath, including face shields and neoprene protective clothing. In case of contact with salts or solution, flush skin with large quantities of water; for eyes, flush with water for at least 15 minutes and obtain medical attention.

There is a certain amount of caustic spray generated when the Gibonol S-34 bath is boiling. Therefore, exhaust ventilation is recommended.

Due to the fact that the operating temperature of the solution is above the boiling point of water and the danger of eruption of the bath exists if water is added improperly. It is important that the entire contents of these operating instructions be thoroughly read as Gibonol S-34 blackening bath.