

2 Cleaning Processes

The list of metals and their features explains how raw materials have natural surface conditions that interfere with coating adhesion and performance. In the process of being stored, handled and worked they will pick up additional some contamination on their surface. On metals, some of the probable contaminants are oily soils including petroleum products, animal fat, or vegetable oils, deposited during manufacturing operations for rust protection, drawing, machining and forming. There may also be heavy duty drawing compounds and lubrication greases or waxes and some solid soils such as carbon, graphite smuts, metal shavings, polishing products, metal oxides, welding scale, die release products, and red or white oxidation. Removal of soils prior to powder coating is essential to the successful life of the product. It affects the initial adhesion and the ultimate performance in the field.

Soils that are present on metal parts can be removed by a variety of mechanical and chemical methods. What method should be used in a given situation is determined by the part to be coated (size, configura-

tion, material), the type of soil to be removed (dust, wax, oil, salt crystals, etc.) and the performance requirements of the finished product.

Mechanical Cleaning

Soils may be organic substances such as oil or they may be inorganic materials such as mineral type rust inhibitors. Both types of soils can sometimes be effectively removed by mechanically abrading the surface. Mechanical methods, including wire brushing, abrasive blasting, grinding and sanding are used to smooth as well as clean surfaces. Mechanical cleaning using a hand held tool involves considerable labor. Automated processes include vibratory polishing and blasting. Mechanical cleaning is sometimes the only way to remove excessive dirt, rust or scale.

Abrasive Blasting

Blasting with a suitable media can remove dirt, mill scale, rust or previous coatings from a substrate, providing a surface profile that gives good coating adhesion. The blast media will vary dependent on the surface to be blasted and the quality requirements of the blasted product. Typically used media includes sand, steel shot, grit and glass bead. The media is delivered to the part surface at high velocity to impact the soils and cut them away from the metal surface.

The blasting equipment used to deliver the media may be air-blast or turbine-blast. Hand held air-blast systems are very dependent on the concentration of the operator and quality may vary. Blast cabinets are often suction-feed systems that draw particles into the spray gun by induced vacuum and accelerate the media it with a metered stream of compressed air. There are also pressure-blast systems that use a pressurized vessel to deliver the media. Pressure systems are capable of higher nozzle velocity that can provide much faster cleaning of the surface than a suction system.

Blast cabinets' function similar to any booth designed for containment of oversprayed material. Negative pressure within the cabinet is maintained with a fan that draws air into the enclosure through a suitable filter. Typically, this exhaust system will use a cyclone separator to remove the dust and fine particles from the air stream and recover the media for reuse. The scrap material that is separated out of the air-

stream is collected for disposal in a container attached to a dust collector. This scrap material should contain a small percentage of the heavier, reusable media to indicate that the fan pull is sufficient to prevent the build-up of fines in the recovered blast media. A vibratory screener can be added to the process to further refine the recovered material and maintain consistent particle size.

Turbine-blast systems use high-speed turbine wheel with blades. The media is metered to the center of the wheel where it is fed onto the blades, which sling the particles at the surface being blasted. These systems are more energy efficient than air-blast systems because they do not use compressed air for delivery.

Abrasive blasting is most often used for preparation of metal surfaces of heavy structural parts, particularly HRS weldments. It is a very good way of removing the encrustations and carbonized oils that are characteristic of this type of product.

Blasting operations can be manual or automated and they can be installed as part of a conveyORIZED powder coating system or as a batch process. The blasting device may be a nozzle type or a centrifugal wheel type. As previously stated, nozzle blast systems require compressed air for delivery of the media while a wheel system uses centrifugal force. Even though the compressed air is an added cost, it may be necessary to direct nozzles into hard to reach areas of a part. The blast area must be enclosed to contain the blast media and dust.

In addition to cleaning, a blasted surface can create a very good anchor pattern for a coating. Different blast media can be used to vary the profile created on the metal surface. Less aggressive media will remove most soils without cutting too deeply in the metal and leaving a visible texture on the metal surface. More aggressive media can be used to cut stubborn encrustations, such as red oxides, but it will leave more texture on the surface.

A blast system does not require as much space as a spray washer that uses chemical cleaning and it does not generate any wastewater. For these reasons, mechanical cleaning may be the only treatment required for finishes where initial paint adhesion is required. However, mechanical cleaning alone will not provide undercoat corrosion resistance or extend the life of the finished product.

Blast cleaning standards depend on the quality requirements of the surface. Published documents clearly define quality grades of blast-cleaned steel surfaces. Pictorial standards were originally developed by the Swedish Corrosion Committee and later adopted by the Steel Structures Painting Council (SSPC) and other organizations. The principal four grades of blasting endorsed by the SSPC are:

- **White Metal Blast:** Removal of all visible rust, mill scale, paint, and foreign matter. Used for conditions where corrosion resistance is very important and the environment is highly corrosive.
- **Near White Metal Blast:** Blast cleaning until at least 95% of all surface area is free of all visible residues. Used for harsh environments where product is exposed to heavy usage.
- **Commercial Blast:** Blast cleaning until at least two-thirds of the surface is free of all visible residues. For applications where tightly adhering contaminants are allowable on the surface; for products with lower quality standards and non-corrosive environments.
- **Brush-off Cleaning:** Blast cleaning of all except tightly adhering residues of mill scale, rust, and old coatings, exposing numerous evenly distributed flecks of underlying metal. Acceptable in non-corrosive environments where long-term coating life is not expected.

The Swedish Standards Association (SIS) includes very good equivalents of these standards. The British Standards Institute (BSI) also includes very close equivalents of the first three of these standards. The National Association of Corrosion Engineers (NACE) has also devel-

Table 2- Metal Blast Standards

Degree of Cleanliness	SSPC Standard	NACE Standard
White metal blast	SSPC-SP 5	NACE No. 1
Near white metal blast	SSPC-SP 10	NACE No. 2
Commercial blast	SSPC-SP 6	NACE No. 3
Brush of blast	SSPC-SP 7	NACE No. 4

(For additional information on blast cleaning standards: Plaster, H.J., Blast Cleaning & Allied Processes, Vols. I and II, Industrial Newspapers Ltd., London, 1973 and Fuchs, F.J., "Abrasive Blasting Systems," Metal Finishing Guidebook & Directory, Vol. 95, No. 5A, Metal Finishing, Hackensack, N.J., 1997, p49)

oped a set of encapsulated steel coupons that simulate the four degrees of cleanliness.

The texture of a blasted surface will vary with different media. The film thickness of the coating over a blasted surface must be thick enough to cover the peaks and valleys of the pattern created by the abrasion, typically around 1 mil above the peaks of the pattern.

Blast Media

In selecting a specific media it is helpful to understand some of the materials used and how they compare. Blast media can be made of natural material such as silica, sand, mineral sand, flint, garnet, zircon, and other mineral products. It can be made of some natural byproducts such as walnut shell or corncob. And it can be manufactured of a variety of metal and non-metal compositions such as steel, iron, aluminum oxide, silicon carbide, plastic, wheat starch, and glass bead.

In selecting a media, the comparative features that are the most important size of the product, how well it will cut, how well it will recycle and how much it cost. It is also important to know if there are any health and safety issues, such as lung problems associated with silica,

Table 3 – Comparison of Blast Media

Material	Mesh size (US)	Shape	Density Lb/ft³	Mohs (hardness)	Friability (break-down)	Init. Cost	No. of Cycles	Per Use Cost
Silica sand	6-270	■ ●	100	5.0-6.0	High	Low	1	Med
Mineral slag	8-80	■	85-112	7.0-7.5	High	Med.	1-2	Med
Steel grit	10-325	■	230	8.0	Low	High	200+	Med
Steel shot	8-200	●	280	8.0	Low	High	200+	Low
Aluminum oxide	12-325	■	125	8.0-9.0	Med.	High	6-8	Med
Glass bead	10-400	●	85-90	5.5	Med.	Med.	8-10	Low
Plastic	12-80	■	45-60	3.0-4.0	Low/med.	High	8-10	Med
Wheat starch	12-50	■	90	2.8-3.0	High	High	10-12	High
Corn cob	8-40	■	35-40	2.0-4.5	Med.	Low	4-5	Low

■ Angular ● Spherical

and if the media will leave by-products on the surface, such as oils from walnut shells. The chart shows some common blast media and their comparative characteristics.

In addition to the comparative issues listed in the table above, it is a good idea to test different media to have a visual idea of the effect that they will have on the part.

Hard grit media such as aluminum oxide will cut faster and deeper than soft, angular media such as plastic or agricultural grit. Mineral, ceramic, or metallic grit media are used in air-blast systems. Iron and steel media are more often used in turbine-blast equipment.

Materials that are more prone to fracture, reflected in the chart by friability, are not good materials for recycling. Recirculation of these materials will produce wide variations in the surface condition.

Ultrasonic Cleaning

Ultrasonic cleaning combines the chemical cleaning capability of a detergent or solvent solution with the mechanical action of ultrasonic waves. Transducers located at the bottom or sides of the cleaning solution tank generate the ultrasonic waves. The ultrasonic energy causes a cavitation process to take place at the part surface. The agitation of the solution at the part surface creates a scrubbing action that lifts and removes soils from the surface.

Ultrasonic cleaning is used in small systems with a series of immersion tanks. The equipment is fairly expensive but it can enhance the level of cleaning on parts that require special processing. It is often used to process brass parts prior to application of a clearcoat. The ultrasonic waves help remove soils that are hard to get out of the porous surface of the brass.

Parts that are dipped into an ultrasonic cleaner should provide good access to all surfaces to allow the cavitation to work. If parts are grouped too tightly together the process will not be effective.

Vibratory or Tumbling Cleaning Methods

Vibratory systems use an abrasive media in a cleaning solution to remove burrs, rough edges and surface contamination. They are very useful to prepare castings for coating. They will remove the rough-

ness and dried-on compounds that are often present on a cast surface. It is usually a good idea to alkaline clean and phosphate parts after the vibratory polishing so that and residual cleaning compound is removed before coating.

Chemical Cleaning

The first step in the chemical pretreatment process is the removal of oils, dirt, and other soils that will interfere with the development of a good quality phosphate coating, good coating adhesion, or cause surface defects. Chemical cleaning can be accomplished by subjecting a part to an aqueous spray or dip cleaner. The cleaner may be alkaline, acidic, neutral, solvent, or emulsion. The particular cleaner used will depend on the soils to be removed, the size and type of part, the type of coating to be applied, and the substrate material.

The mechanisms for cleaning processes are generally recognized to include solubilization, saponification, emulsification, sequestration and deflocculation. In each of these processes the action requires surface wetting of the metal by the cleaning solution. Solubilization, the dissolving of soils into solution, can occur when the soils have very similar polarity and chemical affinity for the cleaning media. Emulsification, the suspension of soils in solution, requires that the soils dispersible in the cleaning media. Saponification, turning the soils into soap, applies specifically to those soils that contain carboxylic acid and ester functionality that can react with alkaline cleaning media. Sequestration involves the deactivation of metallic ions in the soil to prevent them from interfering with the detergent action of the cleaner. Deflocculation is a process that breaks up large particles of aggregate soils into a finely divided material that is held in suspension in the solution to prevent redeposition on the part surface. The last two processes generally operate in conjunction with the first three processes mentioned.

While alkaline cleaners are the most common, there are also acid cleaners and emulsion cleaners used for industrial applications. The cleaner selected must have the ability to remove a wide variety of soils, prevent redeposition, provide cleaning even when contaminated, provide foam control, be easily rinsed and be cost effective.

Proper cleaning of some parts may require a combination of spray and immersion stages. The spray stage combines the chemical properties of the cleaner with the mechanical impingement of the solution applied under pressure. Immersion penetrates areas of the part that may be inaccessible to the spray.

Spray or immersion processes can be used in manual batch operations or in automated systems with overhead conveyor. Batch systems will use a hand-held spray wand or small dip tanks. Conveyorized systems will use an in-line spray washer that has the proper number of stages. Batch systems are suitable for smaller volumes with less stringent quality standards. The list below shows some of the types of hand held systems and how they compare. Larger volumes or products with demanding quality standards will probably require a spray washer.

Spray Wand Phosphatizing – Best suited for large bulky parts where dip tanks or conveyor systems would require more space and cost.

Steam Cleaning – For small volume of heavily soiled parts. Melts grease.

High Pressure Hot Water – Best for cleaning large bulky parts; should have 4-5 GPM, 1,000 PSI plus heat capacity at the nozzle of 160-200 °F (71-93 °C).

Cleaners may be classified according to their pH, a reference to the measurement of the relative alkalinity or acidity. pH is a measure of the ratio of hydrogen ions in solution to the number of hydroxyl ions in solution. If there are more hydrogen ions the solution will be acidic, if there are more hydroxyl ions the solution will be alkaline.

On the pH scale, pure water is neutral and has a pH of 7. A pH of 0 to 7 is acidic and 7 to 14 is alkaline. Caustic soda has a pH of 13 or 14 while hydrochloric acid has a pH of less than 1.

Cleaner pH varies with different products and substrate materials. Cleaner pH will typically range from 4.5 to 10.5.

- alkaline cleaners – mild, pH 9 - 10.5
 - medium, pH 10.5 - 11.5
 - high, pH >11.5
- neutral cleaners – pH 6.5 - 9
- acid cleaners – pH 1.0 - 5.5

Alkaline Cleaning

Alkaline cleaners are the most common method of soil removal for metal preparation prior to the application of powder coating. Cleaners based on sodium hydroxide (caustic) are very economical where cleaning by saponification is desirable. Caustic cleaning media are highly reactive on non-ferrous surfaces and they can cause over-etching on aluminum and zinc surfaces, possibly creating smut and adding zinc to the solution. Caustic cleaning residues are also difficult to rinse away, especially if the solution temperature is in the high range.

Alkali silicates are excellent for cleaners that are used on non-ferrous surfaces. Silicates can provide good cleaning with minimal chemical attack and they do a good job of soil emulsification. They are a little more costly than alkalis and not easily rinsed.

Synthetic detergents and surfactants offer many variations in composition. In some cases they cost a little more than alkalis but they provide a longer bath life which offsets the higher raw material cost. With good performance, easier handling and disposal, and superior effectiveness over a wider array of metals, these products are a good solution for many systems.

Typically, a mild alkaline cleaner (pH of 9 to 10) will provide better soil removal and longer bath life than a high caustic solution. Residues of alkali salts will kill the free acid, drop out metal salts and kill the phosphate bath. A mild alkaline cleaner, prior to the phosphate stage, will aid in the formation of a more uniform, dense phosphate coating, leading to better paint adhesion and corrosion protection.

If the cleaning is not adequate, it is usually better to increase the time in the cleaner rather than the concentration. Two mild alkaline cleaner stages are better than one high caustic stage. Mild alkaline cleaners are good for multiple metals and they can be run at a wide variety of temperatures. A higher pH cleaner may be necessary on occasion for very difficult soils.

An alkaline cleaner is typically comprised of:

- alkaline base
- surfactant/detergent package

- additives for
 - defoaming
 - minimizing attack on substrates
 - coupling agents
- water conditioners

Alkaline Cleaner Component Functions

- **Silicates** - (sodium metasilicate, sodium orthosilicate) High alkalinity, good saponifier and dispersant, softens water by precipitation, inhibits dissolution of zinc and aluminum. May leave a whitish residue on parts if not properly rinsed.
- **Phosphates** - (trisodium phosphate, sodium tripolyphosphate, tetrasodium pyrophosphate, disodium phosphate) Softens water alkalinity, improves rinsing, saponifier.
- **Carbonates** - (sodium carbonate, sodium bicarbonate) Alkalinity, good buffering.
- **Hydroxides** - (sodium hydroxide, potassium hydroxide) High alkalinity, saponifier.
- **Nitrites** - (sodium nitrite) Minimizes oxidation of metal due to cleaner drying.
- **Chelants** - (versene) Softens water, changes form of precipitation.
- **Surfactants** - Provide water/oil solubility. Enables cleaners to work more efficiently by reducing surface tension at the metal surface. Also, prevents part from drying between stages or the spray washer.
- **Defoamers** - Control foam.
- **Inhibitors** - Minimize attack on metal.

The alkaline cleaner is added to water (typically 2 - 10%) and applied hot. Solutions of this type have low surface tension, which means they can easily penetrate beneath and between dirt particles. In addition, the soap or detergent present can often combine with dirt, oil or grease and emulsify them in water to remove them from the surface of the part. When used with pressure spray or mechanical scrubbing, hot alkaline cleaning for 1-2 minutes is a very effective cleaning method.

Surfactants used for cleaners are usually anionic or nonionic, poly-addition products of ethylene oxide and/or propylene oxide with al-

cohol's, amines and phenols. Sometimes the surfactants used in dip cleaners are sulfonates.

The purpose of the surfactants is to break oil and grease from the surface of the parts and emulsify it in the solution. Oils will rise to the surface when the circulation pump is turned off and they can be removed by skimming or overflowing.

A typical spray cleaner stage in a washer is 60 to 90 seconds, while a dip stage may be anywhere from 3 - 5 minutes with temperatures ranging from 120 to 180 °F (49 to 82 °C). Times, temperatures and cleaner concentration vary depending on the cleaner used and the condition of the substrate.

The rinse stage following the cleaner is ambient tap water to remove any residual alkaline cleaner or loosened soil. Rinse stages are overflowed with fresh water as parts are processed.

Cleaning and rinsing alone prior to painting is sufficient as a stand alone pretreatment in a limited number of situations. As with mechanical cleaning, it will provide initial adhesion only and offers no long-term protection.

When cleaning prior to conversion coating, it is important to consider how the cleaner and its effect on the substrate may interfere with the formation and deposition of the conversion coating. Will the cleaner drag-out adversely affect the conversion coating solution? Will the cleaner alter the surface (etching, smutting, etc.)? In a pretreatment process, the cleaner should not be viewed as a separate process but as an integral part of the total pretreatment process that can effect the quality of the conversion coating.

Cleaner Performance Factors

Over time, the soils that are removed from the parts will build up in the cleaner solution. Solid particles will settle to the bottom of the tank as sludge and oils, grease and some floating debris will float on the top of the solution. There is a limit to the amount of contamination that a cleaner bath can tolerate before it will cease to clean and need to be dumped and recharged. Overflowing the solution can help to reduce the accumulation of floating debris but solids can still cause a problem and overflowing the solution will create a need for more chemical. Oil skimming and sludge removal can extend the life of the

cleaner. Techniques for this are explained in the discussion on washer design.

Control Parameters

The parameters for process control of a cleaning solution are process time, chemical concentration, temperature, spray pressure, drain time,

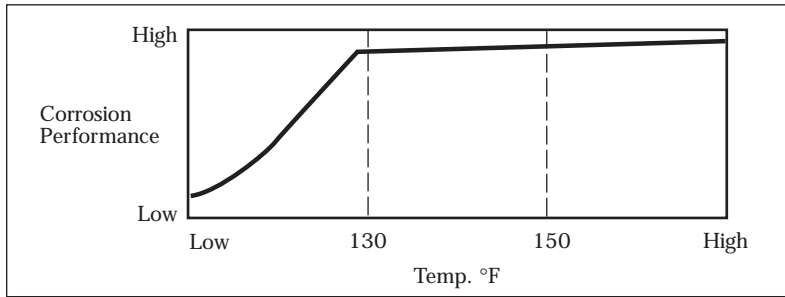


Figure 2-2 - Temperature vs. Corrosion Resistance

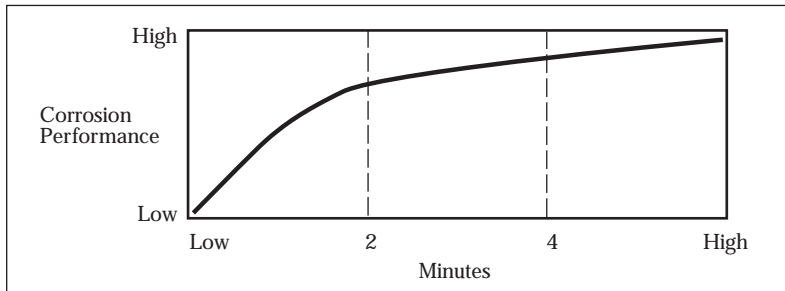
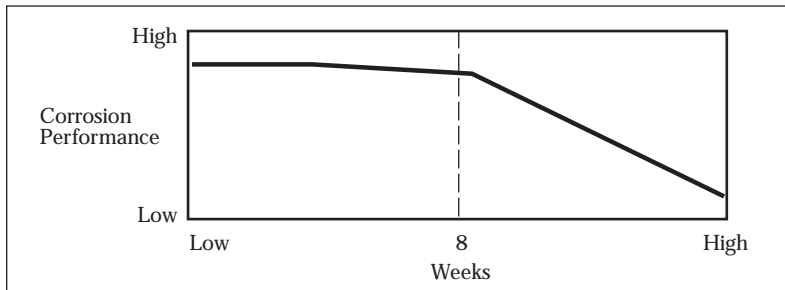


Figure 2-3 - Cleaning Time vs. Corrosion Performance

Figure 2-4 - Cleaning Solution Life vs. Corrosion Performance



and the volume of contaminants in the solution. These are the items that must be monitored, recorded, and maintained within proper ranges in order to achieve predictable cleaning performance. The set of charts shows how some of these control items can affect performance.

There are many variables that affect the length of time that a solution will remain effective, such as the number of shifts, the volume of metal processed, the type of metal processed, and the types of soils removed. This chart shows a typical relationship between time and performance.

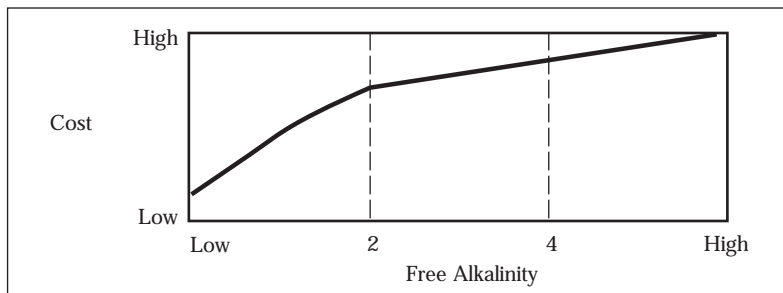


Figure 2-5 – Cleaner Alkalinity vs. Cost

Acidic Cleaning

Acidic cleaning is based on attack of the metal surface by sulfuric, hydrochloric, nitric, phosphoric, hydrofluoric, fluorboric, or chromic acids and the various acid salts of these acids. They generally include a surfactant package, metal ion sequestrants, alcoholic solvents, and an inhibitor to prevent excess attack of the metal. They can be useful for removal of light oxides, organic residues, persistent salts and other soils that are readily dissolved in acid.

Because of the fact that acids are corrosive and therefore more difficult to pump and handle, and because in some cases they are inferior to alkaline cleaners on organic soils, they are much less commonly used. For metals that are prone to hydrogen embrittlement, such as alloy steels and high-carbon grades of steel, acid cleaning is not an option. Acids can also react with some metals to form insoluble byproducts that interfere with subsequent processes.

In a three-stage washer, the first stage combines the cleaning and iron phosphating. These solutions will typically be made up of phosphoric acid, a wetting agent, and an activator.

Acid solutions may also be used to remove scale or oxides in pickling solutions. These solutions are relatively strong mineral acid solutions, using sulfuric, hydrochloric, phosphoric and nitric acid. This type of solution can be useful for removal of stubborn inorganic contamination. One particularly good use is the removal of laser cut scale. Laser cutting of steel will form an oxide layer that is resistant to alkaline cleaning.

Pickling rates increase with higher acid concentration and higher temperature. Excess concentration should be avoided because of the corrosive nature of the solution and the risk of an overly aggressive attack on the metal.

Phosphating

Phosphating, or conversion coating, is the application of an iron or zinc phosphate coating to the substrate. Conversion coating can be a very critical part of the pretreatment process, adding significantly to the performance of the finished coating. A phosphate coating converts the metal substrate to a uniform, inert surface, which improves bonding, minimizes the spread of oxidation if the coating is scratched and improves the overall corrosion resistance of the final part.

A conversion coating can be iron, zinc, polycrystalline, chromate, or manganese phosphate film. They are developed on both ferrous (iron based) and non-ferrous surfaces (zinc, aluminum, terne and manganese). Parts are subjected to an acidic bath and a chemical conversion forms a complete film on the part surface, changing the chemical and physical nature of the metal surface.

Iron Phosphate

Iron phosphate is the thinnest of phosphate films. In the application process, an iron oxide base is developed, followed by a flat or amorphous metal phosphate topcoat. The treated metal surface will typically have a gray to blue iridescent or blue-gold iridescent color, depending on the coating weight and the base metal. A typical iron phosphate consists of:

- phosphate acid base
- accelerators/oxidizers
- surfactant package (optional)

In an iron phosphate solution, the metal surface is etched, releasing some iron into the bath. When metal ions are etched from the part surface, the surface becomes positively charged. The metal ions in the bath are converted to iron phosphate, negatively charged. A pH rise occurs at the interface of the solution and the part, causing the iron phosphate ions to deposit an amorphous coating on the metal surface.

The acid salt content, type and amount of accelerator, and the type and amount of acid etchants varies from one compound to another. These compositions are all moderately acidic. Although crystal site activators are not typically required prior to application of iron phosphate coatings, formulations commonly contain oxidizers and/or accelerators. The oxidizers, such as nitrite or chlorate, act to initiate attack on ferrous parts, providing the iron for the iron phosphate coating. Accelerators, such as molybdate or vanadate, provide active sites for iron phosphate deposition. Choice of oxidizer or accelerator in a particular product may affect the performance or appearance of the final coating.

In a three-stage iron phosphate treatment process, the cleaning and coating are combined by incorporation of a detergent surfactant package in the iron phosphate solution. A source of fluoride ions may be added if aluminum is also being processed to increase the etching effect on the oxide surface of the aluminum.

Iron phosphate coatings can be applied by hand wiping, with a hand-held spray wand, immersion, or a spray washer. The number and type of process stages is directly dependent on finished part requirements. A cleaner/coater combination followed by a rinse is the typical minimum chemical cleaning and phosphating process used. The addition of stages in the process can provide enhanced performance.

The most effective and commonly used method is a multi-stage spray washer. Spray washers are built with as few as two stages and as many as eight.

II

- Two Stage: clean/coat, rinse
- Three Stage: clean/coat, rinse, rinse/seal
- Four Stage: clean/coat, rinse, rinse/seal, DI rinse*
- Five Stage: clean, rinse, phosphate, rinse, rinse/seal
- Six Stage: clean, rinse, phosphate, rinse, rinse/seal, DI rinse
- Seven Stage: clean, clean, rinse, phosphate, rinse, rinse/seal, DI rinse
- Eight Stage: clean, rinse, clean, rinse, phosphate, rinse, rinse/seal, DI rinse

* Deionized water; water that has been filtered to remove negative and positive ions.

Stages	Two	Three	Four	Five
Metals Treated	Steel	Steel Zinc Aluminum	Steel Zinc Aluminum	Steel Zinc Aluminum
Soils Removed	Average Soils	Average Soils	Moderate Soils	Heavy Soils
Degree of Cleaning	Light	Light	Moderate to Heavy	Heavy to Extra Heavy
Coating Weights				
MG/FT ²	25	25	25-45	25-90
G/M ²	0.27	0.27	0.27-0.49	0.27-0.97
Paint Adhesion	OK	Good	Good	Excellent
Corrosion Resistance	Low	Acceptable	Acceptable	Good

Table 4 - Pretreatment Quality From Spray Washers

Phosphate Coating Weight

Iron phosphate is measured in mg/ft², or grams per square meter. Coating weights vary with the different levels of pretreatment. The quality of paint adhesion and corrosion resistance will be affected by the phosphate coating weight. The chart above shows typical results of additional steps in the process.

To determine the coating weight, test panels should be run through the washer with all of the process variables under control. After a clean, fresh panel is run through the washer, it should be removed and tested with the procedure described below. Clean cotton or surgical gloves should be worn to avoid contamination of the sample with skin oils.

Determine the square feet of the panel:

1. Weigh the panel, correct to three places (.000), and record the first weight.
2. Immerse the panel in 10 % chromic acid (CrO_3) in water by weight at 160 °F (71 °C) for 10 minutes.
3. Rinse with tap water or D.I. Water if it is available.
4. Weigh the part again correct to three places and record the second weight.

Then complete the formula:

$$\frac{1^{\text{st}} \text{ wt. in grams} - 2^{\text{nd}} \text{ wt. in grams} \times 1000}{\text{Area in square foot}} = \text{mg./sq. ft.}$$

$$\frac{1^{\text{st}} \text{ wt. in grams} - 2^{\text{nd}} \text{ wt. in grams}}{\text{Area in square meters}} = \text{grams/sq. m}$$

Iron Phosphate Controls

In addition to the number of process stages, the factors that will affect the weight of an iron phosphate coating are time, temperature, concentration, acid consumed (pH), the condition of the substrate and the spray pressure.

- Time in Process - The more time that the chemistry has to work, the more work it will do. The process must be long enough to allow the chemistry to form to a uniform coating on the surface.
- Temperature of the Solution - Soils become more reactive in a heated solution and the chemicals become more aggressive.

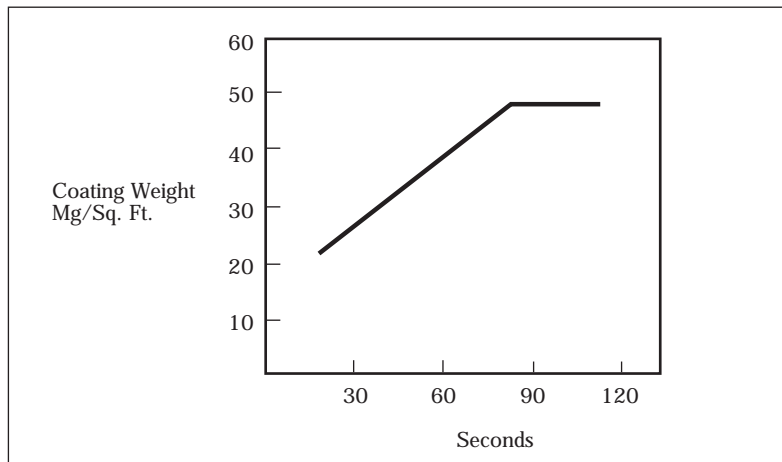
- Concentration - A higher concentration of chemical will provide more total acid, more accelerators and it can provide more coating weight.

Acid Consumed - A higher pH will give less acid and less coating, while a lower pH will give better cleaning and more acid. More acid gives more pickling, providing heavier coatings. Excessive acid can cause too much pickling and the excess acid can dissolve the phosphate coating. The pH that works best for iron phosphate is between 3.5 and 6.0, with most running around 5.0. If the pH is too high (above 6), the parts will not get enough coating weight and they may flash rust. If the pH is too low (below 3.5), the parts will be cleaned and pickled but they will not have any phosphate coating.

The "blueness" of the phosphate coating is related to the coating weight. An iridescent blue indicates a coating weight of 30-35 mg/sq.ft. As the coating weights go up the color will change from blue to blue-gray to gold.

The following graphs show the relationship of each of these control factors to the phosphate coating weight.

Figure 2-6 - Time in Process



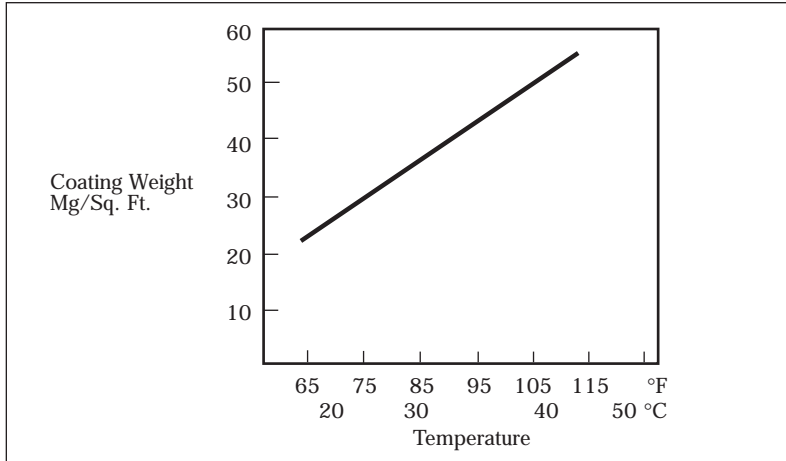
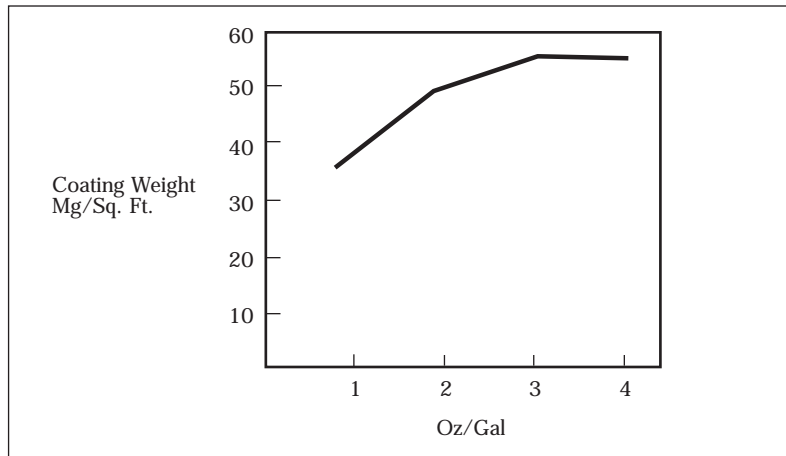


Figure 2-7 – Temperature of Solution

Figure 2-8 – Concentration of Phosphate Chemistry



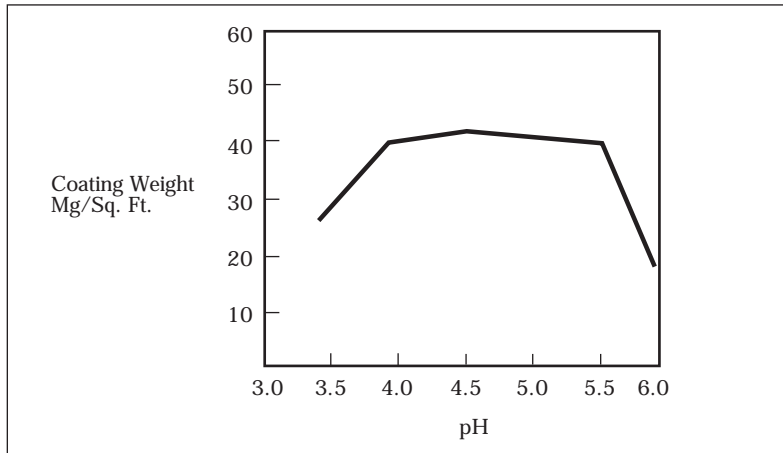


Figure 2-9 - pH of Phosphate Solution

It is also important to maintain consistent control of these variables. Alkaline solutions can be carried into the phosphate solution and cause a rise in pH, or the operators may not make the adjustments to the solution often enough to maintain consistency. This can cause the coating to be spotty in some areas and flash rusting can occur. Solutions should be monitored frequently (3 times per shift) to make sure that they are in good condition.

Zinc Phosphate

Zinc phosphate is a non-metallic, crystalline coating that chemically adheres to the substrate. Zinc coatings are extremely adherent, they provide a uniform coating with improved coating adhesion properties, better coating in recessed areas and better corrosion resistance. A typical zinc phosphate consists of:

- phosphoric acid base
- accelerators
- zinc salts

Zinc phosphate comes from the solution itself, not from the part surface like an iron phosphate coating. Crystals begin forming at anodic

sites on the part surface and stop forming when they hit another crystal. The more origination sites the better the density of the coating. For powder coating, it is best to keep the and densely packed. Powder does not stay in the flow stage for very long. Larger phosphate crystals may not allow the powder material to completely wet the surface and a capillary layer may form under the coating. Moisture will penetrate the coating and cause corrosion that will lift the coating from the surface.

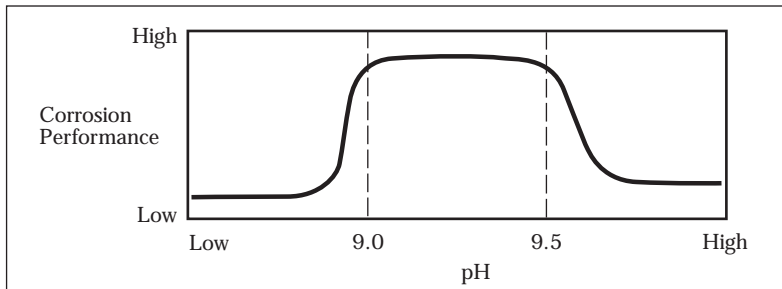
Unlike the iron phosphate, a zinc phosphate can not clean and coat simultaneously in a three-stage process, a separate cleaning stage is required.

Activating (Prior to Zinc Phosphate)

When zinc phosphating, the metal surface is activated by an additive in the cleaner bath or in a conditioning rinse prior to phosphating. Conditioners are mild alkaline suspensions of specialized active titanium salts that adhere to steel, zinc and aluminum surfaces. The conditioner will set up a network of uniform acceptor sites for zinc crystals to deposit. This will increase the number of zinc phosphate crystals, decrease the size of these crystals and generally improve the quality of the zinc phosphate coating. The small crystal size will be more uniform and lower weight, helping to promote adhesion, control the cost of phosphating, and generating less sludge.

Proper pH range, concentration, temperature and bath life are shown in the following series of figures.

Figure 2-10 – Conditioner pH vs. Corrosion Performance



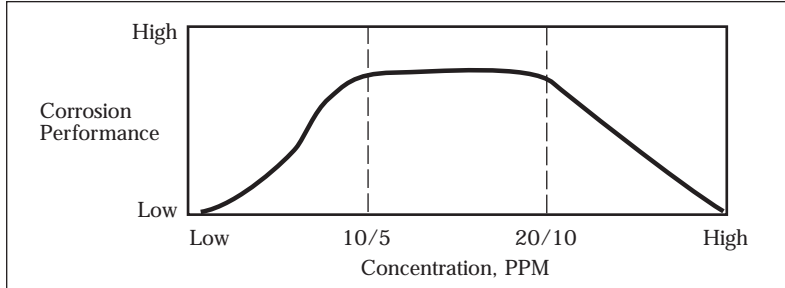


Figure 2-11 – Conditioner Concentrations vs. Corrosion Performance

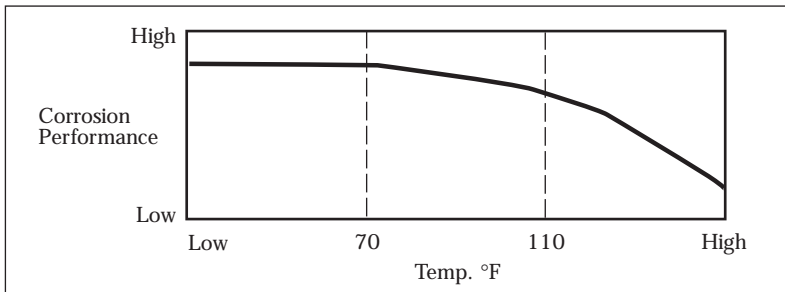
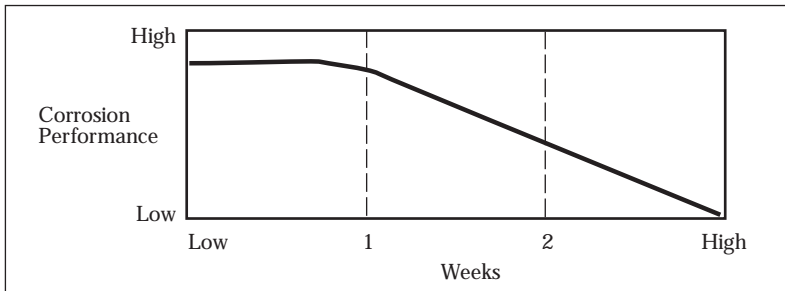


Figure 2-12 – Conditioner Temperature vs. Corrosion Performance

Figure 2-13 – Conditioner Age vs. Corrosion Performance



The crystal size of the phosphate coating has an impact on paint bonding capacity and corrosion resistance. A large crystal structure is more porous, has poorer corrosion resistance, and requires more paint to achieve a complete film. A fine-grained, tight, uniform coating will provide the best performance. The conditioner in the rinse preceding the phosphate stage can assist the development of this fine-grained phosphate coating.

Adding oxidants such as nitrate, chlorate, or nitrite controls the rate of coating formation. The proportion of these various ingredients can control the coating weight and phosphate crystal size. Fluorides are added if aluminum must be processed.

The reactions at the surface of the part during phosphating are:

1. Pickling attack on the metal and oxidation of hydrogen to water
2. Increase of the pH at the interface of the metal and the phosphate solution
3. Over-saturation of the film with coat forming substances
4. Nucleation on the metal
5. Growth of phosphate coating
6. Oxidation and precipitation of iron as sludge

The composition of the bath, the temperature, exposure time and the previous cleaning process will affect the phosphate composition and crystalline phase.

Zinc and polycrystalline phosphate solutions do require more careful attention to produce consistent high quality results. Additions of zinc phosphate and nitrite accelerator to the bath to maintain the proper concentration should be made by automatic feed pumps to ensure good quality and minimum chemical consumption. The improper concentration of these materials that results from bulk adds will produce coatings that are soft, too heavy, and create excessive sludge. If the materials are allowed to run too low, the coating will be coarse and spotty, resulting in poor adhesion and corrosion resistance.

Like other pretreatment processes, time temperature and concentration (total acid, free acid, accelerator and fluoride) will affect the outcome.

Zinc phosphate is the preferred conversion coating used by the automobile industry because of the superior corrosion resistance. The coating is firmly attached to the metal by ionic bonding, the porous crystalline structure provides an extended surface for paint bonding, and if the paint surface is scratched, the inorganic coating protects against corrosion "creepage."

A zinc phosphate solution will continually produce sludge through oxidation of soluble iron to an insoluble state that precipitates. A sludge removal system must be used to provide constant removal of this sludge.

The rinse stage following the phosphate should be ambient tap water. Phosphate salts are more soluble in cold water. The overflow volume should be sufficient to keep the rinse clean and reasonably cool.

Comparison of Iron Phosphate to Zinc Phosphate

From an environmental standpoint, iron phosphate is preferred because it does not generate large quantities of heavy metals that require waste treatment. In some municipalities, an iron phosphate solution can be neutralized and released to drain. Some coaters have waste treatment for iron phosphate and many coating facilities choose to have it waste hauled by a licensed hauler.

Zinc is listed by the United States Environmental Protection Agency (USEPA) in the Resource Recovery and Reclamation Act (RCRA) as a hazardous substance that is subject to waste regulations. It must be treated prior to discharge and the sludge must be waste hauled.

In terms of performance, zinc phosphate with a chrome sealer will typically provide far superior corrosion resistance. Iron phosphate is satisfactory for almost all indoor applications where corrosion resistance is not critical. Zinc is required for outdoor product with superior corrosion resistance requirements. Almost all automotive-specifications call for zinc phosphate.

Table 5 on page II/33 compares the two phosphate processes in more detail.

When deciding between zinc and iron phosphate, the end use of the product is the most important factor. For indoor use in non-corrosive environments, iron will work well and it has several economic and environmental advantages. Zinc will provide the undercoat protection

	Iron Phosphate	Zinc Phosphate
Avg. Coating Weights	15 to 90 mg/ft ² 0.16 to 0.97 g/m ²	50 to 500 mg/ft ² 0.54 to 5.4 g/m ²
Surface Structure	Amorphous (requires less powder to cover)	Crystalline (requires more powder to cover)
Paint Adhesion	Very good (normally will not fracture, even if the metal is bent)	Very good to excellent (crystalline layer may fracture if the metal is bent)
Salt Spray Resistance (5%)	200 to 500 hrs.	600 to 1000 hrs.
Cost in US Dollars	Average \$4.00 to \$8.00 per gal. (one product)	3 to 4 components needed a. zinc phos. \$8.00/gal b. accelerator \$8.00/gal c. activator \$7.00/lb d. pH adjust \$3-5.00/gal
Sludge Formation	Moderate	Heavy
Solution Life	Very good	Very good
Control of Solution	Very easy to control, 1 or 2 tests, 1 product	Difficult to control, 3 to 4 tests, products must be balanced, pre-conditioning stage must be controlled
Equipment	Same as other spray washer stages	Tanks should be stainless steel, sludge separation equipment is needed
Maintenance	Minimal	Higher (sludge handling)

Table 5 – Comparison of Iron & Zinc Phosphate

needed for the more demanding product used outdoors or in highly corrosive environments. The quality of the cleaner and the finish coat must also be considered. Part of the attraction of powder coating is the durability of the film. Good cleaning and high quality powder may allow the use of a less resistant conversion coating.