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2981 Independence Road
Cleveland, Ohio 44115
(216) 271-1601
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SECTION 1: INTRODUCTION

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<thead>
<tr>
<th>ZACLON</th>
<th>PRIMARY</th>
<th>USE IN:</th>
<th>RELATIVE</th>
<th>FOAMING</th>
<th>AVAILABLE</th>
</tr>
</thead>
<tbody>
<tr>
<td>F</td>
<td>General Purpose</td>
<td>Yes/Yes</td>
<td>300 F</td>
<td>3</td>
<td>Yes</td>
</tr>
<tr>
<td>K</td>
<td>General Purpose</td>
<td>Yes/Yes</td>
<td>300 F</td>
<td>3</td>
<td>No</td>
</tr>
<tr>
<td>CS</td>
<td>General Purpose</td>
<td>Yes/Yes</td>
<td>400 F</td>
<td>2</td>
<td>No</td>
</tr>
<tr>
<td>2N</td>
<td>Top Flux</td>
<td>No/Yes</td>
<td>250 F</td>
<td>4</td>
<td>Yes</td>
</tr>
<tr>
<td>2NNF</td>
<td>Top Flux</td>
<td>No/Yes</td>
<td>250 F</td>
<td>4</td>
<td>No</td>
</tr>
<tr>
<td>START UP</td>
<td>Top Flux</td>
<td>No/Yes</td>
<td>350 F</td>
<td>1</td>
<td>Yes</td>
</tr>
<tr>
<td>A</td>
<td>Low Fume Top Flux</td>
<td>Yes/Yes</td>
<td>350 F</td>
<td>1</td>
<td>No</td>
</tr>
<tr>
<td>AF</td>
<td>Low Fume Top Flux</td>
<td>Yes/Yes</td>
<td>350 F</td>
<td>1</td>
<td>Yes</td>
</tr>
<tr>
<td>AB</td>
<td>Inert Kettle Cover</td>
<td>Yes/Yes</td>
<td>450 F</td>
<td>0</td>
<td>No</td>
</tr>
<tr>
<td>W</td>
<td>Continuous Galv.</td>
<td>Yes/No</td>
<td>400 F</td>
<td>1</td>
<td>2</td>
</tr>
<tr>
<td>W-HT</td>
<td>Continuous Galv.</td>
<td>Yes/No</td>
<td>600 F</td>
<td>1</td>
<td>2</td>
</tr>
<tr>
<td>AMMONIUM CHLORIDE</td>
<td>Additive for Activity</td>
<td>Yes/Yes</td>
<td>250 F</td>
<td>4+</td>
<td>4+</td>
</tr>
</tbody>
</table>

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NOTE: BECAUSE OF FUMING, HYDROCHLORIC ACID PICKLES ARE NOT HEATED UNLESS ADEQUATELY VENTILATED.
FIGURE 2: Ratio, Galvanizing Quality, and Zinc Waste

In this chart, there are four substrates--heavy pipe (LP), Medium weight pipe (SP), 10 gauge sheet steel (SP), and light gauge sheet (Q). The 0 ratio represents straight ammonium chloride, and the 25.00 represents straight zinc chloride. All of these fluxes are at 20% by weight for comparison.
FIGURE 3: Kettle Zinc to Ash vs. Ratio
FIGURE 3: Kettle Zinc to Ash vs. Ratio

![Graph showing the relationship between Kettle Zinc to Ash and ratio.](image-url)
FIGURE 4: Ratio and Dross with Dry Time

Ratio vs. $\%$Zn to Dross, 90 minute Dry Time
FIGURE 5: Preflux Concentration and Density

Preflux Concentration and Density

Pounds of ZACLOHO Fr K per Gallon

Preflux

Hydrometer Reading (Baume)
FIGURE 6: Dross and Coating Weight from Iron Carryover
A flux system should contain 10% to 15% dissolved ammonium chloride to function effectively. This concentration can be achieved by reducing the flux temperature, moving along the equilibrium line toward higher ammonium chloride content in solution in the liquid flux.
An artist's concept of a cross section of the kettle flux shown here relates the effect of temperature on ammonium chloride content. A foamed ZACLON® kettle flux will have a temperature at the zinc metal interface of 850 F (454 C) but the insulating foam blanket will drop the temperature to as low as 650 F (343 C) at the outer surface. The phase relationship shown here shows the equilibrium concentrations of ammonium chloride for each temperature. This ranges from 2 to more than 30%.
### TABLE 2: Density Conversions

**Density of Liquids Heavier Than Water**

**BASIC CONVERSION:**

Conversions for Degrees Baume to Specific Gravity for Liquids at 60 F

Specific Gravity = \(145/(145 - \text{Degrees Baume})\)

Degrees Baume = \(145 - (145/\text{Specific Gravity})\)

<table>
<thead>
<tr>
<th>Degrees Baume</th>
<th>Specific Gravity</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>1.000</td>
</tr>
<tr>
<td>2</td>
<td>1.014</td>
</tr>
<tr>
<td>4</td>
<td>1.028</td>
</tr>
<tr>
<td>6</td>
<td>1.043</td>
</tr>
<tr>
<td>8</td>
<td>1.058</td>
</tr>
<tr>
<td>10</td>
<td>1.074</td>
</tr>
<tr>
<td>12</td>
<td>1.090</td>
</tr>
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<td>14</td>
<td>1.107</td>
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<tr>
<td>16</td>
<td>1.124</td>
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</tr>
<tr>
<td>20</td>
<td>1.160</td>
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<td>22</td>
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<td>1.218</td>
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<td>32</td>
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<td>34</td>
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<td>36</td>
<td>1.330</td>
</tr>
<tr>
<td>38</td>
<td>1.355</td>
</tr>
<tr>
<td>40</td>
<td>1.381</td>
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</tbody>
</table>

<table>
<thead>
<tr>
<th></th>
<th>Pounds Per Gallon</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>3.33</td>
</tr>
<tr>
<td></td>
<td>3.45</td>
</tr>
<tr>
<td></td>
<td>3.57</td>
</tr>
<tr>
<td></td>
<td>3.69</td>
</tr>
<tr>
<td></td>
<td>3.82</td>
</tr>
<tr>
<td></td>
<td>3.95</td>
</tr>
<tr>
<td></td>
<td>3.08</td>
</tr>
<tr>
<td></td>
<td>3.22</td>
</tr>
<tr>
<td></td>
<td>3.36</td>
</tr>
<tr>
<td></td>
<td>3.51</td>
</tr>
<tr>
<td></td>
<td>3.66</td>
</tr>
<tr>
<td></td>
<td>3.82</td>
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<td></td>
<td>3.99</td>
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<td>10.15</td>
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<td>10.32</td>
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<td>10.88</td>
</tr>
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<td></td>
<td>11.08</td>
</tr>
<tr>
<td></td>
<td>11.29</td>
</tr>
<tr>
<td></td>
<td>11.50</td>
</tr>
</tbody>
</table>
FIGURE 9: Typical Galvanized Coating Cross-Section
SECTION 1: INTRODUCTION

GENERAL

Zaclon Incorporated presents this Galvanizing Handbook as a basic reference for the galvanizing industry. It is concerned primarily with hot dip batch galvanizing of work in manual and semi-automatic operations. We hope the following pages will serve as a helpful guide for use of ZACLON® Galvanizing Fluxes.

Galvanizing is the practice of coating clean, oxide-free iron or steel with a thin layer of zinc or zinc alloy to protect the surface against corrosion. The zinc coating provides protection to the iron or steel in two ways: (1) it shields the base metal from the atmosphere and (2) because it is more electronegative than iron or steel, the zinc gives cathodic or sacrificial protection. Even if the surface becomes scratched and the base metal is exposed, the zinc is slowly consumed while the iron or steel remains protected from corrosion.

The hot dip process is adaptable to coating nearly all types of fabricated and non-fabricated products such as structural assemblies, hardware, chain, hollow ware, wire, pipes and tubes, fittings, tanks, sheets, strip, and wire cloth.

Galvanizing consists of four fundamental steps: (1) surface preparation, (2) fluxing, (3) galvanizing, and (4) finishing.

The preparation step consists of cleaning and pickling operations that free the surface of dirt, grease, rust and scale. The fluxing step serves to dissolve any oxide that may have formed on the iron or steel surface after pickling and prevents further rust from forming. This cleaning can be accomplished with either a preflux solution, a top flux on the kettle, or both. Clean, oxide-free work is galvanized by immersion into molten zinc. Finishing operations include removing excess zinc as well as quenching and passivating post-treatments.

ZACLON® Galvanizing Fluxes are used in the prefluxing and galvanizing steps. In addition to ZACLON® Fluxes, Zaclon supplies many other products used by the galvanizing industry. These include Zaclean Concentrate, ammonium chloride, Wetting Agent A, and non-chromate White Rust Preventative (ZACLON® WRP).

Table 1 summarizes the characteristics and versatility of the most widely used ZACLON® fluxes. The broad range of flux grades permits galvanizing applications such as Aqueous preflux solutions, molten top flux on the kettle, and molten but inert kettle covers.
Different levels of flux activity to meet specific operational requirements can be achieved in the proper selection of ZACLON® flux:

**High activity fluxes** facilitate high quality galvanizing. These fluxes should be considered especially when base metal surfaces can be given only a minimum amount of surface preparation or are difficult to prepare for galvanizing. An example is ZACLON® 2N.

**Intermediate activity fluxes** will allow high quality galvanizing where the base metal has been given adequate surface preparation. Examples of this would be ZACLON® F and K.

**Low activity or minimal fuming fluxes** will make good quality galvanizing possible after painstaking base metal surface preparation has been provided or when “easy to galvanize” work is processed. When minimal fume galvanizing is required, the galvanizer must provide careful preparation of the base metal surface to compensate for the lower activity of these fluxes. They may also be used as inert kettle covers, such as ZACLON® A or AB.

The grades of ZACLON® offered by Zaclon are based on zinc ammonium chloride but differ in fluxing activity, physical form and additive content. Some grades used as molten top fluxes are available with and without foaming agents. Also, several grades of ZACLON® provide a choice of physical form to accommodate specific needs, including solution versions for easier preflux tank start-up or swap-out. The table below is a quick reference guide for the various ZACLON® fluxes.

**TABLE 1: Zaclon Flux Selection Guide**

![click on icon or table name to display this table](image)

The grades of ZACLON® offered by Zaclon are based on zinc ammonium chloride but differ in fluxing activity, physical form and additive content. Some grades used as molten top fluxes are available with and without foaming agents. Also, several grades of ZACLON® provide a choice of physical form to accommodate specific needs, including solution versions for easier preflux tank start-up or swap-out. The table below is a quick reference guide for the various ZACLON® fluxes.

**GENERAL PURPOSE FLUXES**

**ZACLON® F:**

- Is the basic galvanizing flux used in the United States and Canada.
- Is also known as a “triple salt” flux.
- Is available in a variety of physical forms, both solid and solution.
- Used as a preflux, it assures uniform zinc coverage and continuous activation of kettle top flux, when present, through carry over of preflux film on the work.
- Improves prefluxing by faster activation of base metal and better dispersion of contaminants such as oils or lubricants which can nullify preflux action.
- Improves the drainage of preflux solution and promotes faster drying, increased productivity, and reduced flux consumption.
- Can be added directly to kettle top flux, and is the flux of choice for “wet kettle” operation.
- Produces a top flux foam blanket which reduces fuming and zinc spatter; prolongs flux life and lowers energy requirements.
- Also available in a higher viscosity formulation where a more tenacious foam blanket is needed.
**ZACLON® K:**

- Is recommended for preflux use.
- Is essentially the same composition as ZACLON® F except it does not contain foaming or wetting agents.
- Has essentially the same prefluxing performance as ZACLON® F, especially when used with Wetting Agent A additive.
- Produces a thin fluid top flux cover when added directly to the galvanizing kettle.
- Is available in both solid and solution forms.

**ZACLON® CS:**

- Is a “double salt” flux, such as commonly used in European shops.
- Has slightly lower fume, and somewhat lower activity than ZACLON® F or K.
- Produces a thin fluid top flux cover when added directly to the galvanizing kettle.
- Improves prefluxing by faster activation of base metal and better dispersion of contaminants such as oils or lubricants which can nullify preflux action.
- Improves the drainage of preflux solution and promotes faster drying, increased productivity, and reduced flux consumption.
- Is available in both solid and solution forms.

**ZACLON® C:**

- Is essentially the same as ZACLON® CS without wetting agents.
- Is a “double salt” flux, such as commonly used in European shops.
- Is available in both solid and solution forms.

**TOP FLUX MAINTENANCE/HIGH ACTIVITY FLUX**

**ZACLON® 2N:**

- Provides the most active flux of any ZACLON®.
- Can be used alone as top flux or for maintenance additions to another ZACLON® flux to improve fluxing activity.
- Foaming grade produces a blanket which reduces fuming, zinc spatter and prolongs flux life. Also available in a non-foaming grade which produces a thin fluid top flux cover.
- Is available in rod form which melts slowly in the top flux blanket to significantly decrease fume evolution.
- Replaces ammonium chloride for producing top fluxes and effectively eliminates costly zinc losses from the kettle.
- Is available in formulations which create higher viscosity top flux blanket.

**Ammonium Chloride:**

- Used to increase activity of virtually any top flux or preflux.
- Creates large amounts of fume when used alone.
- Is available in rod form which melts slowly in the top flux blanket to significantly decrease fume evolution.

**START-UP FLUX**

**ZACLON® Start Up Flux:**

- Is specially formulated to permit building an active zinc ammonium chloride top flux without generating excessive smoke.
- Produces a foam blanket which effectively suppresses fuming while a new top flux is being formed.
- Other foaming fluxes with more activity can be used for maintenance of the top flux after the blanket is first formed with Start-Up Flux.

**MINIMAL FUMING FLUXES**

NOTE: Minimal fuming fluxes have low activity levels and generally require careful surface preparation to assure acceptable galvanizing quality.

**ZACLON® AF:**

- Can be used as an aqueous preflux or added directly to the kettle top flux.
- Produces a top flux foam blanket which can further reduce the amount of fumes generated.

**ZACLON® A:**

- Is essentially the same composition as ZACLON® AF except it does not contain a foaming agent.
- Gives essentially same performance as ZACLON® AF except that thin fluid top flux cover is produced.
- Can be used as an aqueous preflux, molten salt bath preflux or top flux.

**ZACLON® AB:**

- Contains no ammonium chloride, so creates essentially no fume.
- Can be used as an aqueous preflux, molten salt bath preflux, or top flux.
- Provides a thin fluid top flux cover.
- Can be used to “work” or recover some zinc values from ash on the kettle.
SPECIALTY FLUXES

ZACLON® W:

- Designed for Continuous Sheet Galvanizing.
- Lower fume material with low activity but preheat-stable up to 400°F.
- Available as a concentrated solution.
- HT version preheat-stable up to 600°F.

ZACLON® Sulfate Control:

- Designed for jobs shops with filtered flux tanks and Sulfuric Pickle Acid.
- Removes some or all of the sulfates carried over from the pickle tank.
- Has same prefluxing activity as ZACLON® F.
- Reduces need for periodic flux purification down-time.

OTHER INFORMATION

Galvanizing fluxes have potential for causing irritation or chemical burns. They should be used carefully and with due regard for the associated hazards.

A discussion of health hazards, safety precautions, first aid, personal protective equipment and site facilities, in addition to storage, handling, and shipping information, is found in Material Safety Data Sheets for ZACLON® products. Users should read this bulletin and the Material Safety Data Sheet carefully before using any chemical. Contact Zaclon for the most recent MSDS.
SECTION 2. SURFACE PREPARATION

BASIC INFORMATION

Good surface preparation of the metal parts is essential for the production of high quality galvanized coatings. Surface preparation usually involves a combination of methods for producing a steel surface which is clean and oxide-free.

A variety of methods is available and more than one sequence of surface preparation steps may be used successfully in a particular situation. The selection of a specific sequence will depend on:

- Operating cost
- Type of work and steel
- Pollution concerns
- Production rate
- Safety and health factors
- Storage considerations

The information in this Section is based on Zaclon experience and other sources. It covers a wide range of mechanical and chemical surface preparation methods with emphasis on the most widely used methods: alkaline cleaning and acid pickling. Additional details can be obtained from appropriate suppliers, as well as excellent reference books such as *Metal Finishing's* “Guidebook and Directory” issue, which is published annually.

Several chemicals will be mentioned in this section. It is important that, before using these chemicals, supplier product literature be studied carefully on safety matters.

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PART 1: MECHANICAL SURFACE PREPARATION

General Information

Mechanical surface finishing methods include:

- Abrasive blasting
- Barrel and vibratory finishing
- Brush and hand finishing
- Grinding and polishing

One or more mechanical finishing methods may be used to remove rust, scale, mold sand, slag from welding operations, and paint or lacquer coatings. Several of these methods are particularly suitable for cleaning welded areas or to clean areas with defective galvanizing so that the work can be re-galvanized.
Mechanical finishing methods are most suitably used as the first surface preparation step. In many applications, the work can be prefluxed and galvanized after mechanical surface finishing.

Work that cannot be galvanized shortly after mechanical surface finishing may begin to rust quickly because the surface is so reactive. In such cases, the work may be immersed in a ZACLON® preflux solution at 3-4 lbs/gal (360-480 g/L) immediately after mechanical surface finishing, then allowed to dry. The dried concentrated preflux will offer protection against rusting for several hours if coated work is stored under dry conditions.

If very long storage periods are necessary or storage areas are quite humid or corrosive, consideration should be given to providing long term corrosion protection for the mechanically finished work. Corrosion protection with a film of water soluble oil may be suitable.

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Abrasive Blasting

Abrasive blast cleaning involves the forceful direction of abrasive particles against the work surfaces. The abrasives can include angular metallic grit, spherical metal shot, sand, or a variety of other media.

Two basic methods are used in dry blast cleaning:

- Mechanical blasting in which the abrasive is propelled by means of a power driven, rapidly rotating, bladed wheel.
- Air blasting in which the abrasive is propelled through a nozzle by compressed air.

Most dry blast cleaning is done using cabinet machines. The cabinet houses the abrasive-propelling mechanism, holds the work in position, and confines flying abrasive particles. Of particular concern when using silica sand for blasting is the danger of silicosis, a disease of the lungs that results from the prolonged breathing of fine crystalline silica particles of silica. Use of an abrasive sand with low free silica content reduces this problem.

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Barrel and Vibratory Finishing

Barrel finishing is a mass finishing operation for improving the surfaces of a large number of parts by tumbling or rolling the parts along with a finishing medium in rotating containers called barrels. This process is sometimes called tumbling. One third or more of the volume of the barrel should be left empty. The limiting dimensions of the parts which may be processed are determined by the barrel dimensions; during rotation, the part should remain covered by the media. The maximum rotational speed used is important so that centrifugal forces do not prevent tumbling. A very large part can be done in a barrel if it will fit inside and can be covered with a finishing medium. In this case, action is accomplished primarily by the shifting of the medium rather than by movement of the part. Extremely heavy parts are usually fastened to hold them stationary so damage is not done to the barrel or parts.

Vibratory finishing is another type of mass finishing. The vibratory machine imparts an oscillation motion to the parts and finishing medium. This motion produces a constant scouring or scrubbing action. Vibratory finishing can therefore finish hard-to-reach areas readily. Vibratory machines, in tub or barrel shape, may be loaded to about 90 percent of their volume. Usually vibratory finishing is completed in less time than barrel finishing.
Compounds such as soaps, synthetic detergents and acid or alkaline cleaners are used with water along with the finishing medium for both barrel and vibratory processes. Finishing media include a wide variety of materials and shapes and are employed to:

- Separate the workpieces and thus prevent them from being damaged.
- Provide the finishing action.
- Influence the tumbling characteristics of the parts in the case of barrel finishing.

Barrel finishing in the dry state is used to finish castings to remove fins and sand and other burned-on mold materials. Forgings are descaled this way. Media for dry finishing are usually made of steel.

**Brush and Hand Finishing**

Power driven rotary brushes may be used at various speeds for dry or wet surface cleaning operations. Brushes can be obtained filled with wire or nonmetallic fibers and are available in many dimensions and densities.

Mechanical finishing can also include hand operations with wire brushes, abrasive paper, files, etc. Usually only very small areas are economically cleaned by hand operations.

**Grinding and Polishing**

Grinding is an abrading operation which may be performed with either a power-driven belt or wheel to which an abrasive is bonded. Since grinding usually will produce a rough surface, it may be necessary to finish up with a polishing operation if the final surface appearance is important.

**PART 2: CHEMICAL SURFACE PREPARATIONS**

**Alkaline Cleaning**

**Introduction**

Removing soil from steel is crucial to good galvanizing. Failure to remove soils such as paints and lacquers can retard the dissolution of the iron oxide mill scale by pickling. This will inevitably slow the entire line down, and may cause galvanizing problems. Most metal cleaning is done in alkaline cleaning baths.

Alkaline cleaning baths are based on caustic material such as sodium hydroxide. They often contain one or more of a variety of rinsing aids, soil dispersing aids, and paint removers. Many have special chemicals for customized removal of user-specific soils. There are a number of galvanizers who rely on a simple caustic bath; additives are not an absolute requirement.
The soils which the galvanizer may see on incoming metal are of incredible diversity, from light mineral oils to heavy greases, baked on oil and soot, drawing compounds, mill scale, wax, paint, and various inks. It is nearly impossible to remove all of these soils with just one cleaning process. Alkaline cleaners come close to being universal metal cleaners, however, and they can remove most of these types of soils. Many of the proprietary alkaline cleaners are formulated for effective removal of a specific type of soil.

The main disadvantages of alkaline cleaners are in their corrosive nature, their need for elevated temperatures, and the fact that the cleaner will strip zinc from carriers over time. The zinc may reach levels over 8% as metal hydroxide, and may fall out of solution when the caustic level drops. There are also soils that caustic cleaners do not handle well, particularly “soap scum,” which is calcium or magnesium stearate. (Stearates are often used as drawing compounds.) Methods for removing soils like these are beyond the scope of this manual, and the galvanizer should contact a local cleaner supplier or consultant for help.

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**Equipment**

The tanks which are used for alkaline cleaning are best made from hot rolled mild steel plate, ¼” thick, or from a plastic capable of handling the high heat and alkalinity. Tanks over 6’ deep need more extensive bracing. All tanks should have an overflow dam for skimming oils and floating debris, and steam coils or other heating sufficient to heat the contents to 160°F. Some sort of insulation for the side walls may help keep heat in the tank. Perforated shields in front of the heating coils can help agitate the bath with convection currents. The bottom of the tank should be angled to allow easy clean out and drainage. An insulated cover for the tank would also be a useful addition.

It is important to agitate the bath; mechanical methods work well, as does recirculation through a pump or filter. Air agitation is not a good practice, as it will deplete the caustic from the bath rapidly; the air will cause the formation of sodium carbonate, which is a much less active cleaning material. A cover for the tank minimizes loss of cleaning activity when not in use. Use of a filter is recommended, but not absolutely necessary.

For small parts, the use of a rotating barrel cleaner is the preferred method; wire baskets do not allow all of the parts to contact the cleaning solution as well due to the lack of tumbling.

Alkaline cleaners can also be sprayed on, as long as they are low foaming materials. This can be an excellent enhancement for cleaning of certain soils.

There is other equipment used in cleaning such as ultrasonic cleaning devices and electrical equipment for electrolytic cleaning. These are very specialized devices and are beyond the scope of this manual.

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**Chemicals**

There are many chemicals involved with alkaline metal cleaners. Normally, the main component is sodium hydroxide (NaOH), also known as caustic soda. Most cleaners have about 10% by weight of this material. Many cleaners also have detergents and wetting agents, water softening components, buffers and alkalinity builders, and some level of less active builders.

The uses of detergents, wetting agents, and rinse aids are currently as much art as science. These materials are all
contained in the generic term “surfactant.” There are literally thousands of surfactants, and finding the best one for any given application is challenging. The chemicals used in surfactants may be “natural,” such as in many soaps, or they may be synthetics, such as ethylene oxide polymer derivatives. Surfactants used in cleaning of metals should be low foaming, stable at elevated temperatures in a highly caustic environment, excellent wetters to penetrate soils, good detergents to help remove the soil, and must rinse clean, all without attacking the metal. There are several classes used in metal cleaning; generally “anionic” materials and “nonionic” surfactants are popular, though some use of the “amphoteric” class is growing in the field for environmental reasons. Combinations of surfactants usually give the best results. For the highest surfactant activity, though, the water must be softened or the hardness nullified; hardness will deactivate many surfactants, especially of the “anionic” class.

The other active components, or builders, are silicates, primarily sodium silicate. These provide alkalinity and help “buffer” the cleaning bath, keeping it above a given pH or alkalinity. They also help disperse the soil. Nearly all commercial cleaners contain some silicate; for the galvanizer, however, silicates can present a problem, especially if the rinsing after cleaning is not thorough. Silicate left on the steel, when pickled, may coat the steel and thus cause galvanizing problems. If you use a silicate-based cleaner, you must rinse very well.

The other active builders are phosphates. These materials help to soften the water so the surfactants work at their best, and phosphates will add some alkalinity. In addition, they help disperse the soil once it is removed from the steel, thus helping to prevent redeposition. The use of phosphates in household cleaners has been curtailed recently due to environmental movement pressures, but there has been little action on industrial uses.

Less active or inactive builders include carbonates, mono-, di-, and triethanolamines, and borates. These all add some buffering, some water softening, and some alkalinity.

A basic formulation for an alkaline metal cleaner would be 10% NaOH, 1 to 5% phosphates, 1 to 5% surfactant blend, and up to 10% other builder, with 50% water. The specific formulation will ultimately depend on the soil to be removed, if there is a specific soil; the job shop operator will not have a specific soil, usually, so an all-purpose cleaner should be used. Many sheet, tube, wire, and nail manufacturers have consistent lacquers or oils, and can have a cleaner designed for their specific soils.

Zaclon makes an additive for simple caustic baths that will improve their activity by adding surfactants and active builders. It is designed to clean a wide range of soils well. The product, Zaclean® Concentrate, is used at 1 to 3 oz per gallon in caustic from 5 to 25%, and temperatures from 100-180°F. Zaclean will remove soil more quickly, decrease the temperature needed to clean, and can decrease the amount of caustic used in the bath for cleaning.

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Other Cleaning Methods

Other methods for enhancing the cleaning, often after or in an alkaline cleaner, include electrolytic cleaning and ultrasonic cleaning. These types of cleaning generally require specialized equipment and cleaning solutions to operate, but they are often necessary for ultra-clean surfaces needed for applications such as electroplating. Galvanizing does not normally require as clean a surface as electroplate, though some newer alloys demand extremely clean surfaces.

There are also other methods for cleaning which do not use the alkaline cleaner. These include vapor degreasing, solvent cleaning, emulsion cleaners, and acid cleaners. A discussion on acid cleaners is found below; other cleaning methods are likely to be very limited in use due to the large environmental concerns over air pollution and VOC’s, Volatile Organic Compounds. You can find more information about these methods in the ASTM Specification, “Standard Practice for Cleaning Metals prior to Electroplating,” ASTM B322-85.
Rinsing

Rinsing is a very important part of the cleaning process. To fully remove the oil and dirt, the rinse should be well agitated and skimmed; in addition it is best is to have brushes before the rinse and water sprays after the rinse. The water sprays can drain into the rinse tank. Monitoring the rinse is necessary—any oil built up in the rinse will redeposit on the work and thus cause galvanizing problems. Also, if it is a silicate-based cleaner, the problem described in Section III may occur if not properly rinsed. The rinse systems that use the least water and have the least discharge are the “cascading rinse” types, and are discussed later in this section.

Environmental

The disposal of cleaning baths and rinses must comply with federal, state, and local regulations. One common method for alkaline cleaners is to use spent pickle acid to neutralize used caustic; this produces sodium chloride or sodium sulfate, hydroxides and phosphates of zinc and iron, as well as organic materials like surfactants, inhibitors, and soaps or oils. This material might be easier to dispose of than the straight spent pickle or used caustic. Consult your local regulations and waste haulers for more information.

Acid Cleaning

Acid cleaning is a process in which a solution of a mineral acid, organic acid or acid salt, in combination with a wetting agent and detergent, is employed to remove light oxides, shop soils, oil, grease and other contaminants from the work. The distinction between acid cleaning and acid pickling is a matter of degree. In general, acid pickling refers to a more severe treatment for the removal of heavy layers of scale and oxide. Acid cleaning refers to final surface preparation of work which underwent some mechanical finishing treatment previously. It also is used for work which has only minor surface contamination.

Barrel acid cleaning at room temperature is often used for large quantities of small parts. Perforated barrels containing 500 to 1000 lbs (227-454 kg) of work are immersed and rotated in a tank containing the acid cleaner solutions. In many instances, abrasive media such as stones are added to the charges for more effective cleaning. The medium provides abrading action and helps prevent the workpieces from damaging each other. The concentration of the acid cleaner may be decreased to 0.13-0.50 lb./gal (15-60 g/L) for barrel cleaning. An anti-foaming agent may be used to suppress foaming if necessary.

Spraying the acid cleaner is practical when very large or bulky parts are to be processed. For this purpose, concentration of acid cleaner may be decreased to about 0.13-0.25 lb./gal (15-30 g/L). Provision should be made to do the spraying in such a way that the cleaner can be recirculated for reuse. Limited application can be achieved by wiping the acid cleaner on the work.

Alkaline Derusting

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Alkaline derusting is mainly of value to remove light films of rust or oxides. This method is chiefly used when an unetched metal surface is desired. There is essentially no loss of steel after the rust has been removed. Proprietary products available from various suppliers are used for this application. The solutions are used at 130-170° F (54-77° C).

**Salt Bath Descaling**

Salt bath descaling is used for removal of intractable oxides from stainless and or high tensile strength steels and corrosion-resistant alloys. While acid pickling alone is generally unsatisfactory, it can be quite effective when used for finishing-up after salt bath descaling.

Salt baths operate at 680-1000° F (360-538° C). Salt bath descaling may be practical when many items are to be processed or when a smoother metal surface than can be provided by mechanical finishing methods is desired. The baths are usually proprietary. They are not common in the after-fabrication galvanizing industry.

**PART 3: PICKLING**

**General**

Ferrous metals have a surface coating of metallic oxide or scale which must be removed before the zinc coating can be applied successfully. The most common process for removing this film is called pickling.

Pickling involves immersing the work in an acid to dissolve the scale or oxide film. Many acids can be used; the most common for galvanizers are sulfuric acid and hydrochloric (a.k.a. muriatic) acid.

**Action of the Pickling Acid**

**Sulfuric Acid:**
Sulfuric Acid, written H₂SO₄, acts on scale or oxide by penetrating the oxide layer and dissolving the oxidized region between the base metal and the outer oxide layers, which also generates hydrogen gas. The pressure of the gas combined with this dissolution loosens the scale and “blows it off”.

**Hydrochloric Acid:**
Hydrochloric or Muriatic Acid, written HCl, acts by dissolving each layer of scale from the outside in.
Both Sulfuric and Hydrochloric acid are used by U.S. galvanizers. European galvanizers use Hydrochloric almost exclusively in their plants. The choice of which acid to use requires the balancing of a number of factors, including:

- Type of work and steel substrate
- Cost and availability
- Pickling rate
- Volatility
- Energy requirements
- Work finish desired
- Storage requirements
- Effect of drag-out on other operations.
- Rework, recycle, or disposal costs.

Sulfuric acid is often more readily available at a lower cost than hydrochloric acid. Since much hydrochloric acid is produced as a by-product of chlorinated organic chemicals, its cost and availability depend on the markets for the primary chemicals.

Almost identical pickling rates can be obtained from sulfuric and hydrochloric acids by suitable selection of temperature and pickling bath strength. Hot, dilute solutions of sulfuric acid are generally used for pickling, while hydrochloric acid is mostly used at ambient temperature to avoid undesirable fuming. In common practice, faster pickling rates are obtained in hot sulfuric acid pickles than in room temperature hydrochloric acid baths.

Hydrochloric acid is highly volatile and generates fumes even at room temperature. Fuming becomes worse with increasing solution temperature. Fumes can cause severe corrosion of equipment and building structures in the vicinity of the pickle tank. Accordingly, hydrochloric acid pickles are not heated unless outstanding ventilation is available. Heated sulfuric acid solutions produce water vapor, which raises humidity and contributes somewhat to corrosion, though not nearly to the extent that hydrochloric acid fumes do.

Since sulfuric acid pickles are heated to 150-180°F (66-82°C), they consume more energy than hydrochloric acid pickles. Various methods of energy conservation can be employed. Pickle tanks can be insulated on the sides and tanks covered during idle periods. A surfactant that generates a foam blanket will reduce energy loss, in addition to trapping mist.

Pickling in sulfuric acid will produce a somewhat rougher surface than hydrochloric acid. However, smooth surface finishes will be produced by pickles of either acid if inhibitors are added to the baths.

Since hydrochloric acid is purchased at a fairly dilute concentration (31%) compared to sulfuric acid (98%), considerably more storage volume is required for inventory. More corrosion resistant materials are required for handling hydrochloric acid.

Drag-out from the pickling operation should be carefully rinsed off the work prior to prefluxing. If sulfate is carried into the preflux tank and allowed to build up, it will cause an increase in top flux viscosity. Drag-out of either sulfuric or hydrochloric acid pickles will contain soluble iron, which will contaminate the preflux if not rinsed away. Sulfuric acid pickle films are somewhat more difficult to rinse off.
The time required to remove oxide and scale depends not only on the thickness and type of scale, but also on:

- Temperature of the bath
- Acid concentration
- Iron concentration
- Agitation
- Inhibitor choice and concentration

**Temperature**

The effect of temperature on pickling rates with sulfuric acid and hydrochloric acid solutions is shown in Figure 1. The sulfuric acid reaction takes place very slowly at room temperature whereas hydrochloric acid pickles effectively at that temperature. The reaction rates for both acids increase as the temperature is increased. Faster pickling rates can be obtained with hot sulfuric acid than with hydrochloric acid at room temperature.

A temperature range of 150-185° F (66-85° C) is recommended for a sulfuric acid bath, but temperatures as low as 140° F (60° C) and as high as 200° F (93° C), may be used. Above 180° F (82° C), the steam and fog increase in the pickle room, unless proper ventilation is provided or pickle tanks are protected by ventilated covers. The normal temperature for a hydrochloric acid pickle is 70-100° F (21-38° C).

**FIGURE 1: Effect of Acid Strength and Temperature on Pickling Rate**

Acid Concentration

**Sulfuric Acid Concentrations:**
Figure 1 shows the effect of acid concentration. Low concentrations of sulfuric acid (0-4%) have a slow rate of attack on steel; medium concentrations (5-25%) react rather rapidly, whereas high concentrations (50-93%) are inactive to steel and scale. A good starting concentration for a sulfuric acid pickling bath is a 10 percent by weight solution. This is made up by adding 6.25 gal (23.6 L) of 66° Baumé acid to 94 gallons (356 liters) of water for each 100 gallons of pickle solution required.

It is wasteful of time to pickle with a bath containing less than 3-4% free acid and little is gained by using a bath containing more than 8-10% free sulfuric acid. In addition, use of more concentrated acid solutions is less economical due to increased drag-out losses. Another disadvantage of pickling solutions stronger than 8-10% is that more rinse water and time will be necessary to remove all the acidic residues.

**Hydrochloric Acid Concentration:**
While temperature is the primary method for controlling pickling rate for sulfuric acid, concentration is used to control the rate for hydrochloric acid. However, benefits from increasing the concentration above 15% are not significant for either sulfuric or hydrochloric acid. A hydrochloric acid pickling bath
is usually started by a 1:1 dilution of the 20-22° Baumé hydrochloric acid.

Iron Concentration

If a pickling bath is operated with repeated additions of acid to keep the concentration of free acid approximately constant, the pickling rate will decrease as the concentration of dissolved iron increases. When the iron concentration reaches 7 to 10%, the pickling rate is usually too low to be economical regardless of acid concentration with either acid.

Agitation

The rate of scale removal can be increased with bath agitation. A stationary film of acid next to the scale surface decreases in free acid concentration while it increases in dissolved iron concentration. Both changes decrease the pickling rate. Faster pickling will result if fresh acid is brought to the surface and the weak acid contaminated with dissolved iron is dispersed throughout the bath away from the metal. Agitation may be obtained by mechanical means or by blowing air or steam into the bath.

Inhibitor Choice and Concentration

Overpickling occurs when the pickling solution attacks and dissolves base metal after scale removal. It usually results because the scale layers are not uniform and the thicker or more deeply embedded material requires more time for removal. Proper selection and use of inhibitors will prevent overpickling. Quality problems due to overpickling also include possible hydrogen embrittlement and excessive surface roughness.

Inhibitors are recommended to retard the attack of acids on steel without greatly influencing the attack on scale. Thus, they lower acid consumption, reduce hydrogen formation and subsequent absorption by the steel, decrease acid fuming, and prevent “burned” and pitted steel. The use of an inhibitor will provide a better quality galvanized product as well as a more satisfactory and economical operation.

Inhibitors reduce the rate of attack of the acid on the steel by as much as 95%, and thereby reduce the amount of hydrogen evolution by the same amount. Since the evolution of hydrogen contributes to the formation of a fog of dilute acid around the pickle tank, inhibitors also improve working conditions. Some inhibitors are also available which will perform two functions: (1) reduce the attack of the acid on the steel and (2) form a blanket of foam on the pickle bath which aids in reducing the evolution of acid fog and steam around the pickle tank. Additional information about inhibitors can be obtained from various suppliers.

General Pickling Procedure

A uniform concentration of active acid should be maintained during the life of the pickling bath by making frequent additions of small amounts of acid. When the iron concentration reaches 4-5%, acid additions should be discontinued, allowing the free acid to work down to the point where pickling becomes too slow to be practical. When the iron concentration in the bath reaches about 7 to 10% and the free-acid content has been worked down to a minimum level, the solution should be discarded or regenerated.

The amount of sulfuric acid consumed will vary with the type and amount of scale and the metal surface being
handled through the bath, but normally it will be about 30 to 50 lbs (14-23 kg) of 66° Bé sulfuric acid per ton of steel. A similar guideline is about 45 to 90 lbs (20-41 kg) of 20° Bé hydrochloric acid per ton of steel.

Allowing zinc to build up in the pickle tank is undesirable for several reasons. First, this will cause zinc to appear in the pickle rinse water, which is subject to regulation by pollution control authorities; and second, some galvanizers believe that zinc adversely affects pickle performance. Zinc is introduced into the pickle by stripping of work carriers. Using the pickle for stripping defective coatings is not recommended, since this raises the zinc concentration of the pickle too rapidly.

Control Tests

In order to operate a pickling bath economically, it is necessary to determine when more fresh acid is required and/or when the bath should be discarded. It is recommended that the iron, zinc, and free-acid content of the pickle bath be determined by titration at regular intervals. This can also be easily done by using pickle pills or one of the assembled pickle control test kits and replenishment solutions which can be purchased from several sources.

A running record of the iron, zinc and free-acid contents, temperature, tonnage of work pickled, acid added, inhibitor additions, etc., will help control operating conditions and determine whether improvements can be made. An easy way to record these items is to plot them at regular intervals on a chart.

Equipment

Acid-resistant polymer (plastic) tanks with steel or other support are commonly used. Selection of polymer depends on acid used and temperatures expected. Steel or concrete tanks lined with plastic or acidproof brick with a lining of rubber or plastic between the steel and brick are found in some plants. Wooden tanks of tongue-and-groove construction, using Monel tie bolts are still occasionally used.

Acid pickling solutions may be heated with external or internal exchangers made of TEFLO® FEP Fluorocarbon Resins or “Karbate”. Open steam jets made of acid-and heat-resistant materials can also be used. Use of open steam heating causes dilution of the pickle solution. This can result in loss of acid through overflow and may lead to increased waste control costs.

To protect the flooring in the pickle-bath area from attack by spills and splashes, it is suggested that existing floors be covered with an epoxy resin flooring material. If a new floor is to be installed, it should be made of acidproof brick set in acidproof cement with an asphaltic membrane protecting the concrete base.

Pickling of small parts is often done in a tumbling barrel or basket suspended on chains that can be mechanically moved up and down. Baskets, hooks, tongs, and chains used to support work in the pickle bath are usually made of “Monel”, phosphor bronze or aluminum bronze. However, in aerated baths, the corrosion from “Monel” is increased, with subsequent copper flashing on the work in the bath which may adversely affect the galvanizing quality.
PART 4: RINSING

General

After the work has been processed in a cleaning or pickling solution, it should be thoroughly rinsed. Rinsing should remove the chemicals carried out on the work so as not to contaminate or chemically alter the next processing solution.

It is not difficult to justify improved rinsing when water costs are weighed against chemical savings (from prolonged life of baths in subsequent steps) and reduced galvanizing rejects. If drag-out is recovered, additional savings will result from reduced purchase of chemicals. A more detailed approach to rinsing can be found in T. Mooney's article “The Art and Science of Water Rinsing”, Metal Finishing, January 1996, volume 94-1A, pages 121 to 129.

Rinsing After Alkaline Cleaning

Rinsing after alkaline cleaning will prevent carryover of chemicals and prevent them from neutralizing free acid in the bath and weakening the pickling solution.

Rinsing after cleaning also removes emulsified soils carried out on the work in a film of cleaning solution. This prevents the oils and greases in the soils from being released in the pickling bath and floating on the surface. A film of oil on the pickle surface will coat the work going in, slowing or stopping the pickling action from taking place in some areas. Work removed through an oil film will carry oil into the preflux, interfering with its performance, too.

Rinsing After Pickling

Regardless of whether sulfuric acid or hydrochloric acid pickling is used, poor rinsing will:

- Introduce iron into the preflux solution. If iron is allowed to build up, the iron-contaminated film of preflux solution left on the work will carry iron into the galvanizing kettle, increasing dross formation. Iron carried into the kettle reacts with 24 times its own weight of zinc to produce dross.
- Drag acid values into the preflux solution. An excessively acidic preflux solution will cause corrosion not only in the preflux tank itself but also after the work leaves the tank. The net result will be additional iron salts carried into the galvanizing kettle, forming costly dross. There is also the possibility of the corrosion on the surface leading to uncoated areas especially when dry galvanizing is practiced.
- Inadequate rinsing after sulfuric acid pickling leads to sulfate contamination of the preflux solution. The sulfate contamination is carried into the top flux and causes increased viscosity, “gravel” formation and decreased flux activity.
The most effective way to improve rinsing is to minimize drag-out. Drag-out is the amount of solution retained on the work as it is removed from a bath. Approximately 0.5 fluid ounce (14.8 ml) of solution will be retained on each square foot of surface. The volume retained will be greatest on horizontal surfaces. Cup-like configurations will increase the retained volume of solution to a degree depending upon their dimensions and location. The amount of dragout can be decreased by:

- Holding the work over the processing tank for a short period to allow maximum drainage to occur. For example, a drainage time of 10 seconds will decrease the volume of retained solution by about 30-50%.
- Using wetting agents in the solutions to reduce surface tension and provide better drainage. This will decrease the volume of solution retained on the work surface by about 50-75%.
- Spray or fog rinsing while the work is suspended over a lot process tank.

After the maximum amount of alkaline cleaner or pickle solution has been removed from the work, that which remains must be rinsed off. Rinsing can be improved by:

- Providing vigorous agitation in the rinse tank. This can be done by use of air agitation or movement of the work in the rinse tank.
- Replacing some of the water in a non-flowing rinse tank periodically. The water removed can be returned to the process tank to replace evaporation losses.
- Using water nozzles to produce a fine water spray on parts going into a rinse will remove about 80-90% of the solution from outside surfaces of the work. This is a good way to provide the fresh water to a rinse tank.
- Using continuous flow rinse tanks with the water entering the bottom, flowing diagonally upward across the tank and flowing out the opposite side, with the water flow in the opposite direction of work flow.

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PART 1: FLUX CHEMISTRY

General

The primary purpose of a ZACLON® galvanizing flux is to dissolve surface oxides that form on the steel after the pickling operation. This flux can be put on the steel either by dipping the steel in an aqueous preflux solution of ZACLON®, as in “dry” galvanizing, or by passing the work through a molten salt top flux which floats on the surface of the molten zinc in “wet” galvanizing. These two approaches can also be used in combination. An additional property of the preflux bath is that it will prevent formation of oxides in the time before actual galvanizing step, allowing some delay between fluxing and dipping of work. A top flux will also minimize oxide formation on the molten zinc or zinc alloy and insulate the kettle surface.

When used in combination with top fluxes, the dip in the preflux bath will cover the entire surface of the work, including small crevices, with a fresh layer of flux; this sometimes cannot be done thoroughly when all of the fluxing is done by passing the work through a molten top flux. A preflux will also provide incremental activation of the top flux with the fresh flux carried over. This means that the use of a preflux of ZACLON® does not increase the over-all flux consumption for top flux users, since some ZACLON® from the preflux is carried over and becomes part of the top flux. This replaces all or part of the solid flux that would ordinarily be added to the top of the kettle. The advantage of adding flux to the top of the kettle by means of predipped coating on the work is that it continuously activates the top flux in small increments.

Hydrochloric acid has been used for the prefluxing step but this has the disadvantage of forming iron salts on the surface of the work. This increases dross formation when carried over into the kettle (see Table). Iron salts may also increase coating weights.

The use of ZACLON® instead of hydrochloric acid as a preflux permits a considerable time delay between the fluxing and the galvanizing without the build up of rust on the work. ZACLON® fluxes also permit preheating the work prior to immersion in the zinc kettle.

How Fluxes Work—Some Flux Chemistry

Fluxes work by removing the surface iron oxides (Fe\textsubscript{x}O\textsubscript{y}) from steel, giving a metallurgically clean surface for the iron/zinc reactions of galvanizing. The primary active ingredient which does this is ammonium chloride for both a preflux and a kettle flux. In a kettle flux, the ammonium chloride also works to keep the zinc surface free of oxides. The zinc chloride component also will remove some oxide, but its main purpose is to keep the flux stable to preheating, protect the steel surface from further oxidation, and keep the flux fluid in top fluxes. Zaclon rates its fluxes on the basis of “ratio”, which is the weight of zinc chloride divided by the weight of ammonium chloride in a flux. There are others who rate fluxes on “ammonium chloride number”, which is the exact opposite of weight ratio. The table below lists common fluxes with the ratio and ACN. Basically, activity is higher as the ratio DECREASES.
Flux Types, Ratios, and ACN's

The most common flux is ZnCl₂:3NH₄Cl, also known as triple salt. The actual weight ratio is about 0.85, as in ZACLON® K or F. A triple salt has 54% NH₄Cl, so is active. A flux of the formula ZnCl₂:2NH₄Cl is known as a double salt, with a weight ratio of 1.25. This has only 45% NH₄Cl, and is of lower activity. A flux such as ZACLON® W, used in the sheet industry, has a ratio of 2.5, with only 28.5% NH₄Cl. (This low activity has other advantages for sheet lines.)

The primary flux reactions are:

1) Iron oxide removal

Ammonium chloride + iron oxide = water + ammonia + iron chloride

\[2\text{NH}_4\text{Cl} + \text{FeO} = \text{H}_2\text{O} + 2\text{NH}_3 + \text{FeCl}_2\]

Reaction 1 is the desired reaction for a preflux, as well as the top flux.

2) Reaction of flux with base steel

Ammonium chloride + iron = hydrogen + ammonia + iron chloride

\[2\text{NH}_4\text{Cl} + \text{Fe} = \text{H}_2 + 2\text{NH}_3 + \text{FeCl}_2\]

3) Skimmings/Top flux reaction

Ammonium chloride + zinc oxide = water + ammonia + ammoniated zinc chloride

\[2\text{NH}_4\text{Cl} + \text{ZnO} = \text{H}_2\text{O} + \text{NH}_3 + \text{ZnCl}_2 : \text{NH}_3\]

Reaction 3 is desired with reaction 1 in the top flux.

4) Flux reaction with Zinc

Ammonium chloride + zinc = hydrogen + ammonia + ammoniated zinc chloride

\[2\text{NH}_4\text{Cl} + \text{Zn} = \text{H}_2 + \text{NH}_3 + \text{ZnCl}_2 : \text{NH}_3\]

The reactions which form ammonia or hydrogen (1 to 4) do not give off hazardous gases, since both zinc and zinc chlorides react quickly to prevent release of ammonia or hydrogen. In a top flux, these reactions help explain the occurrence of (a) zinc consumption, (b) hydrogen fires in the flux and (3) presence of ammonia odors. Once again, however, there is little or no gas released due to the very rapid reaction of ammonia with zinc and zinc chlorides.

5) Dross formation
Iron chloride + zinc = iron + zinc chloride

\[ \text{FeCl}_2 + \text{Zn} = \text{Fe} + \text{ZnCl}_2 \]

Reaction #5 is true for ANY iron chloride in the solution, whether from actual flux attack on the steel or by carryover of dissolved irons from the flux bath or pickle tank.

6) **Smoke and Fume Formation**

Ammonium Chloride sublimes at 640\(^\circ\)F and recombines within a few centimeters of the kettle surface (as soon as the temperature drops below 640\(^\circ\)F) to form white “smoke”.

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**Fumes and Smoke in Galvanizing**

The fumes or smoke evolved from a conventional hot dip galvanizing operation include the following:

- **Ammonium Chloride** - from sublimation of ammonium chloride, and reaction of any hydrogen chloride and ammonia evolved from the kettle. Reaction #6 above shows the primary reaction for flux smoke from a kettle. Note that in the preflux, the ammonium chloride and the zinc chloride form intermediate compounds which allow preheating, oxide protection, and smooth release from the work once it is dipped into the zinc. and in the top flux it would be lost far more rapidly if it did not form compounds with zinc chloride which are molten at the kettle temperature.
- **Aluminum Chloride** - from reaction of aluminum in the molten zinc with ammonium chloride in the flux.
- **Lubricants** - from improperly cleaned work which contaminates the process solutions and ultimately gets carried into the galvanizing kettle.
- **Steam** - from incompletely dried preflux film or water formed in chemical reactions in the galvanizing kettle.
- **Zinc Oxide** - from the open surface of a dry galvanizing bath and moisture from flux or wet work.

While all these sources are involved in generating fumes, the most significant source is ammonium chloride in both preflux and top flux systems. It is the active component in any zinc ammonium chloride flux system.

At about 660\(^\circ\) F (439\(^\circ\) C), ammonium chloride sublimes and breaks down to ammonia and hydrogen chloride gas. Hot hydrogen chloride gas is very reactive and will quickly remove metallic oxides. However, as the excess ammonia and hydrogen chloride gases rise above the zinc kettle, they cool and recombine to form solid ammonium chloride. The particles that form by this mechanism are extremely small—generally less than 1.5 microns in diameter. These particles create the smoke which may be objectionable.

Uncontrolled or excessive ammonium chloride evolution is the focal point of fume control problem. Only a small amount of ammonium chloride will create copious visible fumes. A handful of ammonium chloride on bare molten zinc can produce particles which will fill an entire working area with white fumes. More information on fume in galvanizing can be found in the section on Wet Galvanizing.
PART 2: PREFLUXING AND “DRY” GALVANIZING

General

The term “dry” galvanizing refers to the most common fluxing practice in the industry. There is no top flux on the kettle; instead, all of the fluxing activity comes from the preflux dip.

Successful production of high quality work by the dry galvanizing method is dependent upon the surface preparation of the work. The cleaning and pickling must be good, and the preflux concentration should be high enough to compensate for the absence of top flux activity. ZACLON® K is useful as a preflux for dry galvanizing because of its good drainage and fast drying characteristics, ZACLON® F can also be used.

Choosing a Preflux

The type of ZACLON® galvanizing flux to use as a preflux depends on the temperature to which the work is to be heated prior to dipping in the molten zinc, whether a top flux is to be used, and the level of cleaning or known difficulty in galvanizing a type of work. In most operations where there is little or no preheating, the preheating is more of a drying step with temperatures below 300°F/121°C. At these lower temperatures ZACLON® K is recommended when dry galvanizing or using a non-foaming top flux. If a foaming kettle flux is used, ZACLON® F should be used as the preflux.

If a lower fuming system is required, a preflux of ZACLON® A, AF or ZACLON® AB may be used. These fluxes will withstand preheat temperatures up to about 350°F. Because of lower activity, very careful cleaning and pickling is necessary when using ZACLON® A, AF or AB. The kettle may be operated without a flux, with a top flux of ZACLON® A or AB, or with a foaming top flux of ZACLON® AF. For preheating work to temperatures over 400°F, ZACLON® C, CS, W or W-HT will be needed.

If the work is difficult to galvanize, or the cleaning or pickling is poor for some reason, a flux (either preflux or top flux) may be activated by use of ZACLON® 2N or ammonium chloride. There will be more fume with these fortified baths, but they can give better coatings on difficult work.

The preflux concentration and ratio have direct impact on fuming for dry kettle galvanizing. The lowest practical concentration should be used, depending on the work to be processes, how well the cleaning and pickle operations work, and similar considerations. Use of a wetting agent in the preflux (such as Wetting Agent A) will also reduce the preflux fume by reducing the amount of flux carried on the work. The ratio of the preflux is also very important; the higher the ratio, the less fume evolved. However, higher ratios also mean lower activity of the flux, so the balance between fume and activity needed is going to be determined by each shop. More information on this is presented below. The ZACLON® K and F products, with a 0.85 ratio, are a good balance between higher activity and fume generation.
Ratio, Galvanizing Quality, and Zinc Waste

Galvanizing quality is reasonably constant over the common ranges of ratio--from the aggressive 0.25 ratio to the essentially smokeless 9.9 ratio.

Another factor to consider in choosing a flux is the influence of ratio on coating thickness. Figure 2 shows this consideration:

![FIGURE 2: Ratio, Galvanizing Quality, and Zinc Waste](image)

In the chart, there are four substrates--heavy pipe (LP), Medium weight pipe (SP), 10 gauge sheet steel (SP), and light gauge sheet (Q). The 0 ratio represents straight ammonium chloride, and the 25.00 represents straight zinc chloride. All of these fluxes are at 20% by weight for comparison.

The ratio has little effect on coating thicknesses. The differences are so small, in fact, that they can be considered statistically insignificant.

One reason to consider in choosing a flux, however, is the more active the flux, the higher the ash generation. Figure 3, which uses a calculation of kettle zinc lost by reaction of ammonium chloride carried in the flux, shows the zinc waste penalty for using lower ratios. The chart assumes an LME of about $0.46/# for zinc, and was derived from Hiscock's 1954 presentation to the 3rd International Conference on Hot Dip Galvanizing.

![FIGURE 3: Kettle Zinc to Ash vs. Ratio](image)

The other direct effect of ratio is in processing of material. The lower ratios, especially below 1.00, dry faster and more completely, giving less spatter, so processing can be done more quickly. If preheating is available, and good cleaning, then lower activity fluxes can be used.

There is also an indirect effect when considering drying of the work. While the flux layer dries on the work, it will pull some iron from the steel itself. Iron in the preflux layer generally goes to dross, so the longer it takes to dry, the more dross can be generated from flux interactions. The lower ratios dry in 30 minutes or less at 60% humidity and 70°F; ratios between 1.00 and 2.0 in about 90 minutes, and over 2.0 will not dry without a good heat source. Figure 4 below shows some Dross cost (per 1000 ft² of surface) vs. Ratio at a 90 minute time.

![FIGURE 4: Ratio and Dross with Dry Time](image)

As mentioned before, controlling the ratio to around 0.85 (ZACLON® K or F galvanizing flux) is a good compromise between dry time, activity, and ash generation.
Preflux Concentration

Considerable difficulty often arises from the use of flux solutions which are too low in concentration. Generally, the concentration should not be allowed to fall below 12° Baumé in order to prevent galvanizing problems resulting from inadequate oxide removal, rusting after prefluxing, or poor cleaning of the work before fluxing.

The concentration of ZACLON® which will prevent rusting between the preflux step and the kettle will depend upon the time delay between the two operations, the humidity of the air to which the prefluxed work is exposed during this period, and the pH of the preflux bath. The longer the holding time and the higher the humidity, the higher the specific gravity or concentration of the preflux solution should be. The pH must be controlled as described below, as well. Where the work is to be preheated, a change in the composition of the preflux itself may be required as discussed above.

A 15° to 20° Baumé solution (measured at room temperature) of ZACLON® flux is often used for the preflux. A solution of this strength can be made by dissolving in water 2 to 3 pounds (0.91-1.36 kg) of ZACLON® F or K per gallon of solution. Flux is added to the preflux as needed during use to maintain this concentration range. (NOTE: Galvanizing fluxes may cause irritation or burns. For safe handling information on ZACLON® Galvanizing Fluxes, see product literature in Section 8.)

There are advantages in using higher concentration preflux solutions over 20° Baumé, particularly when there is also a top flux, since such solutions maintain top flux activity with frequent small additions over wide areas, thus reducing the potential for fuming. They also give additional protection from rusting prior to galvanizing and the best assurance that high quality coatings will be produced.

Preparing the Preflux

A fresh preflux tank is easily made using concentrated ZACLON® flux solutions available in bulk tank trucks. The incoming solution is diluted in half or more with water to give the desired concentration of preflux.

The preflux can be prepared by adding dry ZACLON® flux to the preflux tank 75 percent full of hot water. Use Figure 5 to determine how much ZACLON® flux is required. Follow the line across from the hydrometer reading column (degrees Baumé) until it intersects with the sloping line for ZACLON® flux to be used. From that point, follow the line straight down to find the amount (pounds) of ZACLON® flux required for each gallon of preflux solution to be prepared. (Note that temperature can change these numbers; for 100° to 140° F, add 1° Baume; for 140° to 180° F, add 2° Baumé.)

Add ZACLON® flux at many points around the tank to prevent settling in one area. At the initial make-up of a new preflux solution, the flux should be dispersed uniformly and the solution well agitated. Mechanical or air agitation may be used. After initial mixing, the balance of the water is added and the preflux mixed to a uniform solution.
Temperature

The temperature of the preflux is not important to the fluxing action; the bath should be kept hot for other reasons. For example, the use of a hot flux solution will improve wetting, accelerate drying of the prefluxed work, help to maintain kettle temperatures, and reduce spattering. The optimum temperature will be determined by the thickness of the metal and the speed at which the work passes from preflux to the zinc bath; i.e., the work should be just dry enough after fluxing to prevent spattering of molten metal when immersed in the molten zinc.

The work should remain in the preflux bath only long enough to heat the work to the same temperature as the preflux bath itself. A temperature of 130°-170°F (54°-77°C) is recommended if the work has a little time after fluxing to partially dry from its own heat.

Drying and Preheating

Articles to be galvanized should not be introduced wet into the molten zinc because of hazardous spattering from the instantaneous evaporation of the water. One of the advantages of the use of a preflux is that the work, protected with a continuous coating of ZACLON® galvanizing flux can be dried and held for a period, if necessary, without oxidation. As a general rule, the prefluxed work should not be allowed to stand any longer than necessary prior to immersion. A heavier coating of ZACLON®, obtained by using higher strength preflux solutions, will give additional protection from rusting or oxidation; however, the work may dry somewhat more slowly.

Atmospheric humidity is an important factor in the rate of rusting of prefluxed work. Laboratory work has demonstrated that the rusting rate can be decreased eight to tenfold by reducing the relative humidity from 75% to 32%. This suggests that prefluxed work should not be stored near preflux or cleaning tanks where the humidity can be expected to be high.

Preheating of the work beyond that achieved in a hot preflux tank is desirable in order to avoid the need to add excessive amounts of heat to the zinc kettle. This means that kettle capacity may be increased if preheating is done. To take maximum advantage of preheat, it is important that the work pass as rapidly as possible into the zinc without cooling.

Flux layers formed by dipping in prefluxes of ZACLON® F or K can be preheated to temperatures around 300°F without significant degradation, as long as the preheat time is not excessive. Fluxes with lower activity, such as ZACLON® C, CS, or W can tolerate preheats up to 450°F, and there are also special ZACLON® fluxes which can tolerate temperatures up to 600°F. These less active fluxes do require better cleaning and pickling, as mentioned elsewhere in the handbook.

Equipment

Commonly, heavy-duty polyethylene tanks are used for preflux solutions. They may be supported by steel or
concrete when they are large. The same type of tanks can be used as suggested for the sulfuric or hydrochloric acid pickling operation as well. See Section 2.

Normally, preflux solution is heated; it is recommended that steam coils be used for this purpose. Impervious graphite (subject to mechanical breakage) or tantalum (expensive) are resistant materials for preflux tank heating. “Monel,” “Hastalloy 276”, titanium and heat exchangers of TEFLO® Fluorocarbon Resin can also be used. Any legs or braces required for metallic coils should be made of the same material as the coils to eliminate bi-metallic corrosion.

PART 3: CONTROL AND PURIFICATION OF FLUX SOLUTIONS

General

The quality of product, efficiency of production, and waste of zinc pounds can be directly influenced by control of flux parameters such as density (or concentration) and acidity or pH, as well as contaminants such as iron and sulfate. Measurement and control of these solution properties will help ensure the highest quality galvanizing with the least wasted zinc. In this section you will find methods for determining the most important flux parameters.

Sampling the Preflux and Pickling Baths

It is most important that the sample represent the bath composition under normal operating conditions. If necessary, adjust bath to normal operating level and temperature.

- Agitate to ensure uniform solution.
- Take at least a one quart sample for your use. Samples should be taken in a clean container at several different bath locations. Use a sampling tube to get a cross section of bath.
- Caution: If a hand held container is used to take the sample, proper protective equipment must be used.
- Federal regulations require that solutions be handled in properly labeled, approved containers and shipped if necessary according to Department of Transportation (DOT) regulations and be delivered by the appropriate carriers. Zaclon will supply properly marked containers addressed to Zaclon for a small fee.

Measuring Flux Concentration by Density of Flux Solution

Check the concentration of the preflux bath of ZACLON® galvanizing flux daily using the following method. The concentration is directly related to the density of the solution. In this case, density is measured in degrees Baumé (Bé) with a hydrometer for liquids heavier than water. For some calculations, however, the units of grams per milliliter (g/ml) are needed; others will use lbs/gallon as the units. A conversion from Baumé to g/ml is listed below, and from g/ml to lbs/gal. Also note that the terms density and specific gravity are used interchangeably.
Equipment

- Thermometer
- Hydrometer of appropriate range, certified
- Hydrometer jar or large graduated cylinder, usually about 12” high by 3” diameter.

Procedure

1. Fill hydrometer jar to within 1-1/2” of the top with the solution under test.
2. Warm or cool the solution as required to bring it to a temperature of $60^\circ + 0.5^\circ F$ $(15.5^\circ + 0.3^\circ C)$.
3. Immerse a standardized hydrometer, that has been brought to the same temperature and dried, to a point slightly below the equilibrium level and allow it to come to a rest floating an equal distance from the sides of the hydrometer jar.
4. Read the hydrometer at the point where the plane of the liquid surface cuts the hydrometer stem. Recheck the solution temperature and repeat the test if temperature has not remained within the range of $60^\circ + 0.5^\circ F$ $(15.5^\circ + 0.3^\circ C)$.
5. Record Baume at $60^\circ F$ $(15.5^\circ C)$ to the nearest 0.050 Bé reading. Table 2 converts Baumé to specific gravity and density.
6. For control purposes, measurements are sometimes taken at higher temperatures. To correct the density add $1^\circ$ Bé if the temperature is $100^\circ - 140^\circ F$ $(38^\circ - 60^\circ C)$; add $2^\circ$ Bé if the temperature is $140^\circ - 180^\circ F$ $(60^\circ - 82^\circ C)$.
7. Correct the concentration to the recommended level, mix well and recheck the density. As an approximation, add 20 lbs of ZACLON® for each 100 gallons (24 g/L) of bath to raise the specific gravity 1° Bé.
8. To convert Bé to g/ml: $145/(145-o \text{ Bé}) = \text{ specific gravity in g/ml}$
9. To convert g/ml to lbs/gal: $\text{g/ml} \times 8.345 = \text{ lbs/gal}$

**TABLE 2: Density Conversions**

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Acidity and pH in Flux Solutions

In spite of careful rinsing, some acid from the pickle bath may carry over into the preflux solution. The build up of free acid in the preflux bath has an objectionable action on the steel, increases corrosion of the heating coils, and increases the amount of soluble iron in the preflux bath and thereby adds to the dross in the zinc kettle.

Acidity of the preflux bath should not be permitted to fall below 3.5 pH. The pH can be adjusted upward by additions of ammonium hydroxide or by putting zinc slabs in the tank. Continued use of zinc slabs for pH control tends to produce zinc build-up in the preflux solution, and maintenance addition of ZACLON® 2N rather than ZACLON® F or K may be desirable to keep the solution in balance. A rule of thumb is to add 4 pounds of ZACLON® 2N for every pound of slab zinc dissolved in the preflux tank. Required corrections of solution density can still be made with ZACLON® F or K.
The best practice is to rinse well after pickling so that the pH of the preflux does not require frequent attention.

Measuring Free Acid Concentration in Flux Solutions

**Principle**

Free acid concentration is determined by titration with standard sodium carbonate solution to an indicator end point near pH 4. Methyl orange, which changes from red to yellow in the pH interval 3.1 to 4.4, is most commonly used. At pH values near and above 4.5, Fe(OH)\(_2\) begins to precipitate, can mask the end point color change of other indicators, and causes serious error in free acid titrations.

**Procedure**

1. Transfer by pipette twenty milliliters (20 ml) preflux bath solution into a beaker containing 100 ml distilled water. Add 3 to 4 drops methyl orange indicator solution.
2. For sulfuric acid, titrate the sample with 0.408 normal (N) sodium carbonate solution to the indicator color change. NOTE: For hydrochloric acid, titrate the sample with 0.548 N sodium carbonate solution to the indicator color change.
3. Read the acid concentration in grams/100 ml directly as the number of millimeters of sodium carbonate solution used divided by ten.

Preflux Contaminants: Iron and Sulfate

Over time, the preflux solution will become contaminated with iron salts; some are soluble, giving a clear reddish-brown flux solution, but most iron salts common to a flux tank are not soluble, and show up as the “mud” found in almost all preflux tanks. This iron mud is usually a brown-black material which makes the preflux tank appear muddy and opaque. To prevent the sediment in the solution from becoming excessive, the preflux solution can be (a) continuously circulated through a small filter (recommended procedure for large installations) or (b) allowed to settle out during a shutdown period. When a shutdown period occurs, it is suggested that the clean solution on top be siphoned or pumped to a clean tank, leaving the precipitate behind. The bottom portion with precipitates should be removed, the tank washed out and the clean liquor siphoned back into the original tank. The preflux should then be adjusted to the proper concentration.

In operations using a sulfuric pickle there will be a build-up of sulfate contamination in the preflux bath over time. Excessive sulfate will eventually lead to galvanizing problems. Use of ZACLON® Sulfate Control flux (with the appropriate filtration) or periodic treating of the bath for sulfates will minimize this potential problem. Treatment of the preflux bath, or changing out the bath entirely, is important when the sulfates exceed 1%. If the preflux is used in combination with a top flux, the sulfates should not exceed 0.5%; at this level, the top flux will start to become crusty and stiff as sulfates are carried over.

Costs of Iron Contamination in the Preflux
Soluble iron salts in the preflux bath will increase the costs of operation by increasing both dross generation and coating weights. Again, assuming a 0.46/# zinc price, Figure 6 below shows how carryover creates dross, and the increase in coating weights is also shown graphically in the following chart for 1000 square feet of surface coated.

**FIGURE 6: Dross and Coating Weight from Iron Carryover**

The bath should be treated out whenever the soluble iron exceeds 0.3% (3000 ppm) for best operation.

If the sulfate or iron content regularly exceeds 0.5%, it generally indicates that rinsing after pickling is inadequate and steps should be taken to improve that phase of the operation. Chemical purification of the preflux bath is a standard practice for many operations when the preflux tank is contaminated.

It may be desirable to discard the contaminated solution from a small tank. To do this, discontinue the addition of ZACLON® galvanizing flux to the tank and use the solution until its specific gravity becomes so low (8-12° Bé) that it fails to keep the steel from rusting or poor galvanizing results. It should then be dumped and a fresh, full-strength solution prepared. This can be done by dissolving solid flux or using concentrated flux solutions available from Zaclon. Disposal should be made in accord with local regulations; in some cases Zaclon will take spent flux for recycling.

When large preflux tanks are involved, the removal of impurities by chemical treatment of the solution is sometimes preferred to preparing a new solution. (Or, in many cases, Zaclon can offer a flux swap-out for larger preflux tanks, taking the spent solution and returning clean, fresh flux solution concentrate. Contact your local representative for more information.) The treatment is usually done before a weekend or shutdown period so that time can be allowed for settling precipitates, transferring treated preflux solution to and from an empty tank, and rinsing out the preflux tank. No heat will be required during this operation.

An alternative to periodic purification is to use a continuous purification. Operations with a good bag or cartridge filter, or a filter press, which filter down to 5 microns, can circulate the flux through the filter continuously. This will remove most of the “mud”. An air sparge, added to this system, will remove most of the soluble iron as well. For those with a sulfuric pickle, use of ZACLON® Sulfate Control Flux will enable the filter to remove some or all of the sulfate contamination on a continuous basis as well. Filter muds must be handled according to local, state, and Federal regulations.

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**In-place Chemical Purification Treatment of Flux Solutions**

The following procedure is based on sulfate precipitation as barium sulfate and iron precipitation as ferric hydroxide.

**Purification Methods:**

1. Determine the iron and sulfate content of the preflux bath by one of the methods listed below.
2. Calculate the pounds of sulfate as $H_2SO_4$ and the pounds of iron as Fe to be removed by the following formulas:
a) lbs sulfate = tank vol(gal) X soln density(lb./gal) X sulfate conc(%)  
b) lbs iron = tank vol(gal) X soln density(lb./gal) X iron conc(%)  

3. Calculate the weight of treating chemicals required as follows:  
   lbs of barium chloride (BaCl₂ 2H₂O) = 2.5 X lbs of sulfate to be removed  
   lbs of 35% hydrogen peroxide = 1.3 X lbs of iron to be removed  

4. Remove any zinc slabs from the tank that might be used to neutralize acid build-up.

5. First precipitate the sulfate by adding barium chloride to the warm preflux solution.

6. Check bath pH and adjust to between 2.0-3.0. If the bath pH needs to be adjusted downward use hydrochloric acid.

7. To remove the iron, cool the solution, add the calculated amount of hydrogen peroxide, and raise pH to 4.5-5.5 with ammonium hydroxide solution. The pH control is very important during iron removal. At a pH below 4.5 the iron may not completely precipitate and above 5.5 zinc in the solution may precipitate. (An alternative to the peroxide treatment is to air sparge for several hours once the pH is adjusted; this method is slower and less controllable, however.)

8. Mix the solution for a 3-4 hour period to allow the precipitating reaction to reach completion.

9. Allow the precipitate to settle; use of polyelectrolytes is frequently beneficial. Pump the clear liquid to a clean tank. When precipitate starts to be picked up in this operation, filter the remaining portion of the preflux bath.

10. Make hydrometer readings in degrees Baumé and adjust bath to preferred strength.

Example:

- Assume the following data have been obtained:
- Tank volume = 6000 gallons
- Solution density = 9.6 lbs/gal or 1.150 g/ml (19.50° Bé)
- Iron 1.3% as Fe
- Sulfate 1.5% as H₂SO₄

Calculate as follows:

   Total weight of solution = 6000 X 9.6 = 57,600 lbs  
   Total sulfates to be removed = 57,600 X 0.015 = 865 lbs  
   Total iron to be removed = 57,600 X 0.013 = 765 lbs  
   Barium chloride required = 2.5 X 865 = 2170 lbs  
   Hydrogen peroxide (35% soln) = 1.3 X 750 = 975 lbs

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Measuring Iron Contamination in Flux Solutions

Take about 100 ml of preflux bath and add 5 ml of 35% hydrogen peroxide that has been diluted 5:1 with distilled water. Mix and adjust pH to 4.5-5.0 with ammonium hydroxide. If the solution turns rust colored (or reddish), iron contamination is present and an analysis of the bath should be made.

Potassium Permanganate Test Method for Iron

General Method: - Iron existing in acid pickles or galvanizing preflux baths is found in the ferrous (Fe++) state. A direct titration with KMnO₄ is the simplest method for its determination. No indicator is necessary since the highly colored permanganate serves as its own indicator.
Chemical Basis: The oxidation of Fe++ to Fe+++ by the permanganate ion, MnO$_4^-$ 
(Fe++ + MnO$_4^-$ >>> Fe+++ + MnO$_2$) is the basis of this procedure.

Reagents - Potassium Permanganate (KMnO$_4$) 0.100 N solution: prepared by dissolving 3.1608 g of potassium permanganate crystals in distilled water and diluting to 1 liter. Solution should be stored in amber bottle and aged 24 hours before using. Prepared 0.1 N KMnO$_4$ solutions are available from most chemical supply houses.

Equipment:
- 50 ml burette
- 10 ml pipette
- 250 ml Erlenmeyer flasks
- 50 ml graduated cylinder
- rubber bulb

Procedure:
1. Pipette exactly 25 ml of sample solution into 250 ml Erlenmeyer flask using rubber aspirator bulb and add about 25 ml distilled water.
2. Add 10 ml of 1:1 Sulfuric acid.
3. Titrate with standard 0.1 N potassium permanganate solution to the first pink color which persists for 20 seconds.
4. Record titer (ml of 0.1 N KMnO$_4$).
5. Calculation ( ml titer X 0.005585 X 100)/( ml sample X sample density (g/ml))= % Fe

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**Measuring Sulfate Contamination in Flux Solutions**

The presence of sulfate in a preflux bath can be determined qualitatively by simply taking a small portion of bath (100 ml), adding 5 ml hydrochloric acid, and 10-15 ml 30% barium chloride solution. If sulfate is present, a white precipitate of barium sulfate will occur; if no precipitate forms, the bath is free of sulfate contamination.

**Centrifuge or Settling Test Method for Sulfate**

Discussion - Routine centrifuge determinations of the sulfate concentration of ZACLON® preflux baths aid in efficient bath maintenance. This method, based on precipitation of barium sulfate, gives sufficiently accurate results for day-to-day control, especially when two sulfate determinations are run simultaneously in matched, calibrated centrifuge tubes.

**Equipment**
- 5 ml graduate
- 10 ml pipette
- 20 ml pipette
- rubber bulb
- centrifuge
• centrifuge tubes - sulfate analysis (matched pairs 0-0.6 ml)
• small rubber stopper

Reagents

• Hydrochloric acid, 5N - Dilute 415 ml reagent grade concentrated HCl to 1 liter.
• Barium Chloride solution, 30% - Dissolve 300 g BaCl$_2$ in distilled water and dilute to 1 liter.

Procedure

1. Sample should be at room temperature and free of solids. Filter if necessary.
2. If a hand centrifuge is used, fasten it very securely to a solid table. This is an important safety precaution.
3. By graduate, add 5 ml of 5 Normal HCl to each of the two matched tubes.
4. Pipette exactly 20 ml of the bath sample into each tube.
5. Place rubber stoppers in each of the centrifuge tubes and mix contents thoroughly by shaking.
6. Centrifuge the sample for 30 seconds. If a hand centrifuge is used, turn the handle at a rate of 60 rpm. Be careful to keep all parts of the body and clothing away from the revolving tubes. If an electric centrifuge (sulfate-analysis type) is used, set the rotation speed at 900-1000 rpm (usually #1 setting).
7. Remove the tubes and check for solids in the bottom of each tube. Tap the stem until the solids have a flat surface. Record tube readings, if any. Each numbered division is equal to 0.1 oz/gal (0.75 g/L) and each subdivision is equal to 0.02 oz/gal (0.15 g/L). This gives a correction for solids in the flux sample.
8. Add 5 ml of 30% barium chloride solution to each centrifuge tube to precipitate barium sulfate. Stopper the tubes and shake thoroughly for one minute. Replace in same position and allow to stand for 5 minutes.
9. Centrifuge the samples for 30 seconds as described in 6 above. Remove the tubes, tap the stem until precipitate has a flat surface, and note the level of the precipitate. If the level of precipitate has fallen, repeat the centrifuging until the precipitate remains at a constant level. Record the reading for each tube. If any solids were found in Step 7, subtract those readings. Average the readings. This gives the sulfate content in oz/gal.

NOTE: If the level of the precipitate is above the highest mark on the centrifuge tubes, repeat the test with the following changes:

• In step 4, using a 10 ml pipette add 10 ml of flux bath sample and 10 ml of water to each tube.
• In Step 9, the average of the two readings must be multiplied by 2 in order to obtain the oz/gal of the sulfate in the flux bath.

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PART 4: KETTLE OR TOP FLUX AND “WET” GALVANIZING

General

Galvanizing is accomplished by passing clean, pickled, and fluxed work into molten zinc or zinc alloy. Some types of work are more easily processed without a top flux. However, as described later in this section, there are advantages
to having a molten top flux on the kettle. This section will describe how this top flux is started and maintained.

A **flux blanket** on the galvanizing kettle serves a number of important functions. It serves as a preheating and drying medium to reduce spattering in the molten metal. A flux blanket keeps the zinc free of oxides that, if included in the coating, tend to dull it and retard drainage of zinc from the work. The top flux also provides additional fluxing action to assure the removal of fresh surface oxide from the iron or steel as it contacts the molten zinc; this more readily imparts a tight, complete, bright coating on all parts of the work. Finally, the kettle flux reduces the cost of the galvanizing operation by minimizing degradation of the spelter to skimmings or oxide and reduces heat loss at the surface of the molten metal.

**Activity in Wet Galvanizing: The Phase Relationship and the Flux Blanket**

Fumes in wet galvanizing when using a zinc-ammonium chloride top flux are greatly influence by the ratio of the flux and the temperature of the top surface of the kettle flux.

The phase relationship of zinc chloride and ammonium chloride in ZACLON® flux systems is shown in Figure 7. The solubility of ammonium chloride in molten zinc chloride is a function of temperature.

**FIGURE 7: Flux Systems' Phase Diagram**

The line that separates the liquid zone from the liquid-gas zone indicates the maximum amount of ammonium chloride that will stay in the melt at any given temperature.

At the typical **kettle temperature** of 850°F (454°C), the maximum concentration of ammonium chloride which will be soluble in molten zinc chloride is only 2-3%. Larger amounts of ammonium chloride simply vaporize until the concentration in the melt is reduced to the 2-3% equilibrium level.

A flux system should contain 10% to 15% dissolved ammonium chloride to function effectively. This concentration can be achieved by reducing the flux temperature, moving along the equilibrium line toward higher ammonium chloride content in solution in the liquid flux.

ZACLON® flux systems containing an appropriate foaming agent will generate an insulating foam blanket which will reduce the outer surface temperature and consequently the fume evolution.

An artist's concept of a cross section of the kettle flux shown in Figure 8 will help relate the effect of temperature on ammonium chloride content. A foamed ZACLON® kettle flux will have a temperature at the zinc metal interface of 850°F (454°C) but the insulating foam blanket will drop the temperature to as low as 650°F (343°C) at the outer surface. The phase relationship shown in Figure 8 shows the equilibrium concentrations of ammonium chloride for each temperature. This ranges from 2 to more than 30%.

**FIGURE 8: Foam Blanket Cross Section**

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**Minimal Fume Wet Galvanizing**

“Minimal fume galvanizing” simply refers to what has long been established as good galvanizing practice with special emphasis on minimizing fume evolution. ZACLON® flux systems will not contribute significantly to air pollution when used carefully. This has been demonstrated to the satisfaction of many air pollution control authorities by galvanizers practicing minimal fume galvanizing.

The problem of fuming should not be associated only with the flux system, as almost every operation associated with galvanizing can contribute to fuming.

Some factors which should be considered when excessive fuming or smoking occurs are:

1. **Surface Preparation**
   - Cleaning
   - Rinsing
   - Pickling

2. **Prefluxing**
   - Preflux Concentration and Ratio
   - Wetting Agents
   - Oil, Grease, other soils carried over from step 1.

3. **Galvanizing Operation for Wet Galvanizing**
   - Top Flux Start-Up
   - Use of Kettle Dam
   - Top Flux Ratio, Depth, and Viscosity
   - Top Flux Addition Techniques
   - Galvanizing Techniques
   - Minimal Fume Fluxes

3. **Operators**

**Surface Preparation**

A chemically clean, oxide free surface can be galvanized without flux. For example, some steel strip is galvanized continuously without flux in totally enclosed lines in a reducing or neutral atmosphere. The use of such a totally enclosed controlled atmosphere system would be difficult and impractical for batch hot dip operation.

Poor surface preparation places a greater burden on the flux, increasing flux consumption with a greater potential contribution to fumes.
**Cleaning**

Surface films of oils, greases, drawing compounds, lubricants, paint, markings, etc., must be completely removed by an alkaline cleaning solution. Inadequate removal of these soils will cause contamination of the pickling and preflux solutions. Such contamination will not only downgrade the performance of the pickling and prefluxing operations, but will also cause the carryover of these soils to the galvanizing kettle where they will create smoke.

**Rinsing**

Sulfate contamination of the preflux solution (due to poor rinsing after a sulfuric acid pickle) directly contributes to the fuming problem in wet galvanizing because it increases top flux viscosity, reducing the ability of the flux to flow around the work being galvanized, promotes formation of “gravel” which needs to be frequently skimmed from the top flux layer. (This disturbs the thermal insulating foam blanket and creates fumes.) Sulfate carryover from pickle or preflux also decreases top flux activity, requiring more additions of flux which creates potential for fuming to occur. This also results in increased flux consumption.

**Prefluxing**

The preflux concentration and ratio have direct impact on fuming, particularly for dry kettle galvanizing. The lowest practical concentration should be used, depending on the work to be processes, how well the cleaning and pickle operations work, and similar considerations. Use of a wetting agent in the preflux (such as Wetting Agent A) will also reduce the preflux fume by reducing the amount of flux carried on the work. The ratio of the preflux is also very important; the higher the ratio, the less fume evolved. However, higher ratios also mean lower activity of the flux, so the balance between fume and activity needed is going to be determined by each shop. The ZACLON® K and F products, with a 0.85 ratio, are a good balance between higher activity and fume generation.

To minimize fuming, small frequent additions of the active flux should be made to the kettle top flux rather than larger infrequent ones. The most efficient addition method is the use of an aqueous preflux of sufficient concentration to bring the required amount of flux in with the work. The use of a preflux does not increase the overall flux consumption but rather will permit the flux product to provide double service, by preventing rusting as well as maintaining the top flux.

The following example shows how high preflux concentrations are cost effective as the dried preflux layer is released in the zinc bath and serves as a maintenance addition to the top flux. Assume that a top flux requires 200 pounds of ZACLON® flux for maintenance addition during a given time period and that 10,000 square feet of surface is processed during that time. Table IV shows the quantity of flux transferred to the zinc bath on the 10,000 square feet of work surface using various preflux concentrations. This Table also shows the additional quantity of flux required to maintain the top flux is reduced as the preflux concentration increases. High preflux concentrations plus small amounts of flux added directly to the top flux actually cost no more than low preflux concentrations with most of the flux added directly to the top flux blanket.
Top Flux Composition and Choosing a Top Flux

Smoke from wet kettle hot dip galvanizing operations is due to the release of visible ammonium chloride particles when the equilibrium solubility of ammonium chloride is exceeded; these solubility limits can be increased by maintaining a thermal insulating cover flux having an outer surface temperature of approximately 650°F (343°C) that will allow an average 10-20% soluble ammonium chloride content. A kettle flux meeting this condition will effectively depress both visible and gaseous fume evolution to the atmosphere and produce quality galvanizing.

The choice of flux depends upon cleanliness of steel, rate of throughput and the amount of fuming that can be tolerated. Activity of a top flux is primarily dependent upon the concentration of ammonium chloride and upon the quantity of oxides and “dirt” the flux has taken up. The amount of ammonium chloride that will remain in a molten zinc ammonium chloride top flux is dependent on the temperature of the flux. Above the equilibrium concentration for that temperature, fuming occurs until the ammonium chloride level decreases to steady state conditions.

The different grades of ZACLON® Galvanizing Fluxes are formulated to provide a choice of products to activate the top flux.

ZACLUSION® 2N Galvanizing Flux has a very high concentration of ammonium chloride and therefore provides activity similar to adding ammonium chloride to the top flux. However, fuming is less than if ammonium chloride is used. The flux is available with or without a foaming agent. The foaming grade is available in 2 forms: granular for fast melting and rods which sink into the flux layer and slowly melt to minimize fume evolution. ZACLON® 2N rods are recommended for supplemental additions because when used properly there is less chance for fuming.

ZACLUSION® F Galvanizing Flux is an active zinc ammonium chloride based flux which is less active than ZACLON® 2N. It is probably the standard kettle flux in use today as it provides a balance between activity and fuming. ZACLON® F produces an excellent foam blanket.

If a non-foaming kettle flux is desired, ZACLON® K should be considered. Its activity is the same as ZACLON® F.

In building a zinc ammonium chloride kettle flux with ZACLON® 2N or F Galvanizing Flux, it is recommended that ZACLON® Start Up Flux be used to start the melt. The heel of an old top flux may be used if it is fluid and active. This minimizes the smoke which would occur by melting the active flux directly on the hot zinc surface. ZACLON® F or 2N is then added to build up the activity. This is discussed further in the next section.

ZACLUSION® AF Galvanizing Flux is a non-fuming cover for molten zinc. It produces a foam blanket and is recommended where complete fume abatement is necessary. ZACLON® AF is less active than the zinc ammonium chloride fluxes. To produce acceptable galvanizing, the work must be well cleaned, pickled and prefluxed in a 20-25° Bé density solution of ZACLON® A or AF. Sometimes a preflux of ZACLON® K or F is used with a top flux of ZACLON® AF but some fuming will still result due to the ammonium chloride in the preflux.

ZACLUSION® AB is a non-fuming flux; it forms a thin flux layer on the kettle to inhibit ash formation. It is also used to “work” ash to minimize the zinc metallic content of the ash.

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Top Flux Foaming Agents and Top Flux Depth

ZACLUSION® 2N, AF, F and Start-Up Galvanizing Fluxes have efficient foaming agents incorporated in their
formulation. (Top flux foaming agents are NOT the same as wetting agents discussed elsewhere.) Foaming agents create a long lasting foam, sustained activity, excellent fluidity and reduced smoking. The advantages include:

- Slower loss of ammonium chloride by vaporization because a foaming blanket remains cooler. This means that less flux will be used and the kettle flux will remain active with lower viscosity for a longer period of time. A slower loss of ammonium chloride also means less fuming.
- Greater drying action because the work takes longer to pass through the flux blanket. This reduces chances of spattering.
- Less heat loss from the zinc because of the insulating effect of the foamed flux layer.

Glycerin has been used to supplement frothing action of the blanket. However, its action is short-lived and leads to a rapid increase in viscosity of the top flux.

The top flux blanket should be as deep and frothy as the operation will permit. While a 2 to 3 inch (5.1–7.6 cm) thick depth is effective, a greater depth is desirable. Use of a foaming agent in the flux is necessary to achieve the optimum top flux depths.

A deep, frothy flux acts as an insulator and keeps most of the flux well below kettle temperature. New flux can thus dissolve readily yet with minimum decomposition. Thin, non-frothy fluxes tend to approach kettle temperatures, and this results in high fuming and high flux consumption.

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Top Flux Viscosity

A top flux under conditions of use is not stable; its chemical and physical properties are changing continuously. Ammonium chloride is lost both through vaporization and is used up in its reactions with iron oxide, iron, zinc oxide and zinc. A kettle flux becomes less active with time and use, and is practically inactive in iron oxide removal when the ammonium chloride content decreases to about 5%. Fresh ZACLON® galvanizing flux must be added continuously to keep the flux active. In addition, the viscosity or fluidity of the kettle flux is an important property. A fluid top flux contributes to production of high quality coatings by closing quickly around the work as it enters the kettle. It also helps reduce smoke or fumes.

Gradually a kettle flux will become more viscous because of the buildup of high-melting zinc and iron compounds. The addition of fresh ZACLON® flux containing ammonium chloride will reduce the viscosity by converting the high-melting zinc compounds to ammoniated zinc chloride.

The viscosity of the top flux can have a significant effect on fume control and flux consumption. Thick, viscous fluxes result in inferior or difficult galvanizing and increase flux consumption. They also tend to resist foaming and approach kettle temperature throughout the depth. This produces copious fuming. The viscous top flux will also generate large gas bubbles which eventually burst releasing clouds of fumes. Factors that can contribute to inordinately high top flux viscosities include:

1. **Heating Time:** The top flux will become more viscous as the period of heating increases. The viscosity is quite low until the top flux has been heated for several hours. After that, the viscosity increases at a faster rate with each additional hour of heating.

2. **Ammonium Chloride Concentration:** With ammonium chloride concentrations above 9 percent, the viscosity of the top flux is quite low. The viscosity increases rapidly as the ammonium chloride concentration falls below 6 percent.

3. **Frequency of Flux Additions:** Considering the points above, it is advisable to maintain the top
flux at a constant low viscosity by making frequent small additions of flux rather than infrequent large additions. This can be achieved effectively by using the proper flux product at an adequate concentration in a preflux tank.

4. **Excess Aluminum in Zinc Bath:** Concentrations of aluminum over 0.01 percent in the zinc at the flux-zinc interface cause the flux to cease frothing and change from a light fluffy flux to a very thick and viscous flux. This is due chiefly to a reaction of the aluminum chloride which sublimes at about 360°F (182°C). These aluminum chloride fumes contribute to the overall fume or smoke problem. Excess aluminum can be oxidized by agitating the zinc bath mechanically or with air or steam.

5. **Excess Sulfates in Top Flux:** Excess sulfate in the top flux generally results from preflux contamination. Sulfate contamination increases the viscosity of the top flux. Severe cases cause the top flux to become like tar. Sulfate contamination in the preflux solution over 0.5 percent indicates that rinsing after pickling is inadequate. Steps to improve rinsing should immediately be taken and the preflux solution purified.

6. **Influence of Unsatisfactory Foaming Agents:** Use of unsuitable foaming agents such as glycerin, bran, wood flour or sawdust will noticeably thicken top fluxes and should be avoided.

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**Top Flux Galvanizing Techniques**

### Establishing the Kettle Flux

A typical zinc ammonium chloride flux contains more ammonium chloride than can exist in the molten salt system at galvanizing kettle temperatures. When building a fresh top flux on the galvanizing kettle with the zinc ammonium chloride flux, clouds of white smoke are given off until the flux has “cooked down” and is ready for galvanizing.

In building a new top flux, ZACLON® Start Up Flux should be used as a start-up flux to absorb fresh flux and reduce loss due to fuming. A heel of reasonably active flux from a previous operation may also be used. The composition of ZACLON® Start Up Flux is similar to that which results after the smoke subsides upon start-up with a typical zinc ammonium chloride flux. A foaming agent has been included which will suppress smoke, insulate the kettle and dry incoming work. ZACLON® HV and AF can also be used in start-up operations.

Probably the most troublesome procedure with regard to smoke emission is the building of a fresh molten flux on the galvanizing kettle. Generally, if the top flux is not started up using good technique, dense clouds of white smoke will be given off until the flux has “cooked down” and is ready for galvanizing. This is because some ZACLON® fluxes, when improperly used, will release more ammonium chloride than can exist in the molten salt system at galvanizing kettle temperatures. The smoking will be considerably worse if the top flux is started up using ammonium chloride alone.

A fresh top flux can be started using ZACLON® Start Up Flux. It is specially formulated to permit building an active top flux without generating smoke. The formulation includes an efficient foaming agent to provide an insulating blanket which further assures suppression of smoking.

To build a new kettle flux with ZACLON® Start Up Flux:

1. Start with a clean zinc surface.
2. Pour or shovel ZACLON® Start Up Flux into a small area to allow it to melt. Use of a restricted area behind a kettle dam is recommended.
3. Continue adding more ZACLON® Start Up Flux until an adequate depth is achieved after
which the area covered by the flux blanket can be increased.

4. Galvanizing can begin as soon as the ZACLON® Start Up Flux has melted and the kettle is covered with at least a 2 to 3 inch depth of flux. There is no need to wait until the flux “cooks down” as with start-up using typical maintenance fluxes.

A top flux can also be built starting with some heel of active flux previously removed from the kettle. At the end of the working period, the active working flux to be used as a heel for the next start up should be transferred to shallow trays. The flux for the heel will lose its activity if permitted to remain on the kettle for a long period.

The reactivated kettle top flux should be started up by remelting enough of the heel to give a two inch depth held in place behind the kettle dam. The top flux is then built up to the desired depth by slowly adding fresh ZACLON® flux. Consideration should be given to addition of ZACLON® 2N, which is available as rod-shaped pieces. These will sink below the flux surface and slowly release ammonium chloride to be dissolved in the flux. It is important that the operator disperse the ZACLON® 2N rods so as not to exceed ammonium chloride solubility in any one area.

Top Flux Additions

A galvanizing flux under conditions of use is not stable; its chemical and physical properties are changing continuously. Ammonium chloride is lost through vaporization and is used up in its reactions with iron, zinc, and their oxides as well as aluminum. Thus, a kettle flux becomes less active with time and use, and is practically inactive when the ammonium chloride content decreases to about 5%. Even though continuous additions of an active ZACLON® product are made by means of the preflux film carried on the work, supplemental additions may be needed at times.

Addition of active fluxes to build up the top flux or for increasing the activity of the top flux is a source of smoke when done improperly. This occurs when relatively large additions are made in a local area. As a result, a high concentration of ammonium chloride is built up thus exceeding the maximum solubility for ammonium chloride in that region of the top flux and causing formation of smoke.

Active fluxes should be added frequently in small increments rather than in larger infrequent additions. A good way to do this is by means of flux on the work carried in from the preflux step, which reduces the amount of flux that must be added to the kettle directly. Sometimes, however, small supplemental additions are desirable. The small additions should be made at many points in the top flux rather than at one point. Agitation after adding fresh flux should be avoided except to disperse localized areas of high ammonium chloride content.

As mentioned, consideration should be given to use of ZACLON® 2N rods for supplemental additions. These sink below the surface of the top flux and slowly release ammonium chloride to be dissolved in the flux. The ZACLON® 2N rods should be well dispersed when an addition is made.

Use of Kettle Dam

When starting up a top flux, it is advisable to use a dam to restrict the area covered by the flux. The top flux can be built up more quickly behind a dam, with less risk of smoking.

When a sufficient volume of top flux has been made in a restricted area of the kettle, the dam can be moved out to increase the working area. The dam should be let out gradually to avoid undue flux agitation. It is particularly
important that operators be trained not to over-extend the working area to a point where there is insufficient flux depth to provide the needed thermal insulating cover. Sufficient active flux should be added to achieve the desired top flux depth as the dam is gradually moved into working position.

Flux should not be allowed to flow past the dam onto the bare zinc area, since it is quickly overheated causing loss of ammonium chloride and excessive fuming. Flux residues should be skimmed behind the dam or taken off the exit end of the kettle as they occur, to avoid unnecessary fuming.

Work should be properly scheduled to avoid the need for continually changing the flux dam position.

After an effective top flux is established and the work has been cleaned, pickled, and prefluxed, it must be processed through the zinc kettle without creating excessive smoke. The key here is to minimize agitation of the top flux.

The methods to minimize agitation of the top flux include:

- A prefluxed, dry article should be lowered through the top flux as rapidly as possible consistent with safe practice.
- The kettle dam should remain stationary so that the flux is not moved back and forth. Work should be scheduled to avoid the need for continually changing the flux dam position. Properly prefluxed work can be held for up to 24 hours without affecting galvanizing performances.
- The galvanized article should be moved out from under the top flux and withdrawn through a clean zinc surface. Withdrawing through flux would cause smoke by exposing ammonium chloride to a hot surface.
- Flux residues should be skimmed behind the dam or taken off the exit end of the kettle as they occur, to avoid unnecessary fuming. Flux spills should be cleaned promptly from hot metal surfaces where they would contribute needlessly to fume evolution. Hooks, chains, and galvanizing tools should be kept from contributing unnecessary smoke by cooling in the water quench tank. Spent flux deposits should be moved away from the work as it is withdrawn from the kettle.
- Another detail requiring attention when practicing minimal fume galvanizing is the kettle temperature. Besides shortening kettle life, excessive kettle temperature overheats the top flux, causing premature exhaustion and excessive smoking.
- Unnecessary kettle agitation should be avoided, since this breaks up the thermal insulating barrier, causing the flux to overheat and fume. Crust formations on the flux surface are caused by excessive cooling and can be remelted by stirring into hotter flux without disturbing the total flux thermal barrier.
- The use of Fines White Ammonium Chloride as a galvanizing sprinkle should be avoided, since this produces considerable smoking. To avoid the need for sprinkling, the work should be withdrawn slowly and the kettle skimmed very carefully to keep oxides away from the galvanized surface. Rapid withdrawal causes lumpy heavy coatings because of oxidation before the zinc drainage is complete. Sprinkling is sometimes used to eliminate voids in the zinc coating. This usually can be traced to inadequate metal preparation which should be improved.

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**Minimal Fume Fluxes (Low Activity Fluxes)**

Some situations may require the use of less active “smokeless” fluxes. These include:

ZACLON® A - non-foaming grade
The “smokeless” fluxes do not contain substantial amounts of ammonium chloride. Therefore, the smoke emissions from such fluxes are extremely low.

“Smokeless” fluxes must be used in conjunction with good metal preparation practice, because they provide a very low level of fluxing activity compared to traditional fluxes. Properly cleaned work is prefluxed in the “smokeless” preflux solution. The prefluxed work should be reasonably dry before galvanizing to avoid excessive zinc splatter. The purpose of drying is to prevent trapped steam from destroying the protective flux film or splashing zinc oxide on the work surface.

Work being galvanized with “smokeless” flux requires a slightly longer time for the preflux film to be released. This is due to a lower level of ammonium chloride gas evolution and surface agitation as compared to zinc ammonium chloride fluxing agents.

Work withdrawal should always be made through a clean, freshly skimmed zinc surface. The somewhat slower spent flux release places greater importance on the kettle operator's skill in skimming residues away from the work as it is withdrawn from the kettle.

Even with the low fluxing activity of a “smokeless” flux, when work is carefully cleaned and pickled and the kettle flux is maintained properly, quality galvanizing can be performed.

Operators

Efforts to minimize fuming will be more successful if the operators:

- Know the problem and what factors affect fumes.
- Use optimum practices that minimize air pollution.
- It may be helpful to implement an operator educational program to:
  1. Impress operators with the importance of fume control.
  2. Show the operator where they are involved and where they can contribute to improvement.
  3. Develop good practice and work habits that will minimize fumes. Good operating instructions will be a useful tool.
  4. Encourage ideas and cooperation that can lead to better fume and air pollution control.

An educational or training program (which can be provided by Zaclon or the American Galvanizers Association) may provide other benefits, in that it can:

- Improve galvanizing practice.
- Produce better quality work.
- Reduce costs.
- Show good intent to local authorities.
- Reinforce operator awareness of good chemical handling and equipment safety practices.
PART 5: COMPARISON OF WET AND DRY GALVANIZING

General

Either method for fluxing can produce a high quality galvanized coating when proper operating practices are followed. Often both preflux and top flux are used in combination. This section will briefly present some comparisons of the two methods.

Surface Preparation

The surface preparation operations used before either wet or dry galvanizing are qualitatively the same. However, the limitations of the dry galvanizing method require that each of the operations, including alkaline cleaning, pickling, rinsing, and prefluxing, be carried out in a more painstaking manner to compensate for absence of the cleaning and fluxing action provided by the molten top flux used in wet galvanizing. When active top fluxes are used in wet galvanizing, high quality galvanizing can be produced on base metal surfaces which have been given a minimum amount of surface preparation.

Many galvanizers depend on the wet galvanizing process to provide strong cleaning and fluxing action as the metal passes through the molten top flux blanket. Oils, oxides, or other impurities will be removed, resulting in better galvanizing.

Drying

Prefluxed work must be thoroughly dried before dry galvanizing. This often requires additional equipment and energy consumption. The drying may be done in ovens, by passing between open gas burners, or by placing small pieces on the edge of the kettle. Some galvanizers use exhaust gases from the kettle burners to dry the metal. The drying temperature should not exceed 250-300°F (121-149°C) or flux decomposition may occur. Work prefluxed in ZACLON® can be stored at suitable locations for up to 24 hours to permit complete drying before dry galvanizing.

Fuming

Fume control is becoming increasingly important for good community relationships and operator comfort. Fuming can be minimized by careful operations in wet or dry galvanizing.

Dry galvanizing does generate fumes from the preflux film which is carried into the kettle on the work. The flux on the metal will melt off of the work in the kettle. In this case, the thin layer of flux can only hold 2 to 3% of ammonium chloride. Excess ammonium chloride will be vaporized, forming fumes and smoke.
To operate a galvanizing plant with minimal fumes, use may be made of ZACLON® A, ZACLON® AF, or ZACLON® AB as alternatives to dry galvanizing. These fluxes contain low amounts of volatile ammonium chloride, the component which usually creates objectionable fuming. However, with less ammonium chloride, the degree of activity as a kettle flux is significantly decreased. It is vital, therefore, that good metal preparation be provided to compensate for the low level of flux activity.

Active top flux can be used with minimal fuming when good galvanizing procedures discussed in the previous section are followed. This enables operators to take advantage of the benefits of active top fluxes while keeping fumes at an acceptable level.

Skimmings

Use of a top flux in wet galvanizing provides many benefits during the period the flux is active. When the flux becomes inactive, it is skimmed from the zinc surface. These skimmings may then be sold as a by-product.

The additional area of the zinc surface which is left exposed when dry galvanizing is practiced generates a considerable volume of zinc ash (zinc oxide). Dry galvanizing requires that the zinc surface be skimmed before the work is introduced as well as when the work is withdrawn. The zinc ash will more than double when dry galvanizing is practiced, as compared to wet galvanizing. The loss in zinc yield to zinc ash is estimated to be about 20% for dry galvanizing. While zinc ash may be sold to recover a portion of the value of zinc metal consumed, the zinc ash does not contribute any utility but, on the contrary, creates an increased labor cost factor for skimming.

Dross

While probably not the major factor in dross formation, the strong cleaning action of the molten top flux blanket on the steel being galvanized will result in more iron removal than when dry galvanizing is practiced. The iron will react with zinc in the kettle to form dross which later must be removed.

Coating Thickness

When dry galvanizing, the preheating step used to dry preflux films can allow work to be withdrawn sooner than is possible under wet galvanizing conditions, where preheating is not necessary. This feature can be useful where thin coatings are desired. However, the same effect can be obtained by preheating followed by wet galvanizing. This should result in similar kettle immersion times and consequently coating thickness.
SECTION 4. GALVANIZING

GENERAL BACKGROUND

Hot-dip galvanizing is basically the coating of steel with a protective barrier of zinc and zinc-iron alloy. It is accomplished by immersion of the work in molten zinc or zinc alloy at a temperature of 820-860°F (438-460°C) for a period of one to five minutes, depending upon the thickness, configuration, and type of alloy for the pieces to be coated. As noted in previous sections, the work must be metallurgically clean for the reaction to occur evenly; therefore surface preparation including fluxing (either wet or dry processes) is critical.

The pieces should be withdrawn at the opposite end of the galvanizing kettle, through a clean zinc surface. For carrying the articles to be galvanized into, through and out of the molten zinc, steel tongs, baskets or various types of hooks and chains are used. These may be handled with an air, electric, or even mechanical hoists.

PLANT LAYOUT

The layout of a galvanizing shop is important for efficient use of space as well as processing speed and environmental considerations. A full discussion of this is beyond the scope of this Handbook; one good reference is the Zinc Handbook, by Frank Porter (published by Marcel Dekker, New York, 1991), as well as publications by the American Galvanizers Association.

KETTLE CONSTRUCTION, EQUIPMENT, AND WORK FLOW

General Comments

The design and installation of a galvanizing kettle with proper heating facilities is very important and should be handled by companies experienced in this work. More information on the topics discussed below is available from vendors and the American Galvanizers Association.

Sizing the Kettle

The size, steel alloy, type of zinc alloy to be used, and quantity of parts to be galvanized will determine the size and
shape of the kettle. There must be sufficient heat capacity in the zinc bath to compensate for cooling when large work loads are immersed. Normally the weight of zinc in the bath should be at least 2 times the weight of parts that are to be galvanized in one hour. When very high production rates are required, this ratio may be doubled. The minimum surface area possible should be used to reduce heat loss and produce less surface oxidation. However, sufficient area should be provided to contain the top flux when it is pushed back for removal of large work items.

The kettle wall thickness is usually between ¾ and 2 inches (2-5 cm), depending upon how it is reinforced. The most widely used material is boiler plate of flange and firebox quality, and is made in special heats with specifications set by the kettle manufacturer. This material is sufficiently strong and has excellent ability to resist the corrosive attack of molten zinc. This same material should be used to construct dams to be used in the zinc bath.

Galvanizing kettles can be heated by combustion of oil or gas, by electrical resistors or by electromagnetic induction. The heating installation should provide efficient and uniform heating to maintain a temperature range of 820-860° F (438-460° C) throughout the zinc bath. Currently, burners with very flat flames, and end-fired kettles using high-velocity burners, are among the most common types.

The kettle wall should be protected from direct contact with burner flames by using linings of fire clay, brick or tile on the outside of the kettle wall near the bottom. Heat should be applied along the sides of the kettle at least 12 inches (0.30 m) and preferably 18-20 inches (0.46-0.51 m) above the bottom of the kettle. This provides a quiet zone in the bottom of the kettle for the settling of zinc dross and avoids undue kettle corrosion at the dross line.

When special galvanizing processes require temperatures above 900° F (482° C), the standard metal kettle will corrode rapidly, causing early failure of the kettle and excessive dross formation. These effects also occur when temperatures rise considerably above normal due to failure of temperature controls. A kettle must be lined with ceramic materials when temperatures of 900° F (482° C) or above are needed; often, ceramic kettles are top-fired.

The kettle should be deep enough so that the articles to be galvanized will be at least 12 inches (0.30 m) above the bottom of the kettle. This provides space for the dross to settle.

Starting New Kettles

The recommended procedure for starting up a new galvanizing kettle is to stack it as full of closely packed zinc slabs as possible, the several layers of slabs next to the outer walls of the kettle preferably being set up on edge. This insures better contact of the zinc slabs with the kettle walls and also promotes dropping down of the top layers as soon as the lower layers of the slabs melt. Some operators then fill in the “holes” with zinc shot or flake, while others will use water to allow the whole stack to reach the boiling point of water evenly.

From this point on, external heat should be applied very slowly. For an average-sized kettle, 20-40 hours will be required for complete melting.

Zinc Bath Temperature

In general, galvanizing should be carried out at the lowest temperature consistent with free drainage of zinc from the work during withdrawal. A low galvanizing temperature helps to keep the formation of ash and dross to minimum,
besides safeguarding the kettle and conserving fuel.

Almost all work can be galvanized within the temperature range of 820-860°F (438-460°C); a common working temperature is 850°F (454°C). A galvanizing bath should never be heated to 900°F (482°C) or above due to the high reaction rate between steel and zinc, causing high dross formation and early failure of the kettle.

**Movement of Work Through Kettle**

The work should be submerged into the molten zinc as rapidly as possible consistent with the operator's safety.

One danger is that preflux solution has been trapped in some hard-to-drain area. This liquid will undergo instantaneous evaporation of the water as soon as it contacts the molten zinc, with a resultant eruption and spattering of molten zinc for some distance from the kettle.

The uniformity of the zinc coating will depend a great deal on the rate of work immersion into the bath. Hoists capable of providing two speeds are useful since they permit fast immersion and slow withdrawal. When long parts are to be galvanized, the uniformity of the coating depends very much upon the skills of the operator since considerable immersion time may elapse between the start and end of entry. Many parts can undergo a lengthy immersion period with no harm other than non-uniformity of coating; other shapes may suffer severe distortion.

The period of time the work is left in the zinc bath varies with the thickness of the steel, the amount of preheat and the thickness of coating desired. The reaction between the clean steel and the molten zinc to give an alloy layer proceeds rapidly for the first one or two minutes and then continues at a decreased rate. The longer the immersion time, the heavier and more brittle the alloy layer becomes. The practice of immersing articles in batches and withdrawing them singly yields articles of varying coating thickness, and is wasteful of zinc because of excessive alloying.

Another factor that influences immersion duration is the nature of the preflux film. Inactive films take longer to release; galvanizing action does not begin until the film has released.

The rate of withdrawal determines, to an important extent, the thickness of the unalloyed zinc layer left on the work. An outer unalloyed zinc layer, over the alloyed layer, is desirable for a bright appearance and absence of discoloration in later use. The withdrawal rate should be slow enough to prevent excessive thickness and irregularity of the coating, but fast enough to yield an outer layer of bright zinc.

**HOT DIP ZINC COATINGS AND ALLOYS**

**Typical Coating Cross-Section**

Within the first half minute of reaction time, the first three layers have already been formed. During the next several minutes, these layers grow at a decreasing rate.

When the steel is removed from the molten zinc bath, the bright Eta layer is retained on the work. The thickness and
surface characteristics of this layer depends upon withdrawal conditions. The growth of the three alloy layers will continue as long as the steel is at elevated temperature converting some or all of the Eta layer, the steel temperature must be cooled substantially such as by quenching. Figure 9 shows a typical cross section for a PW alloy.

**FIGURE 9: Typical Galvanized Coating Cross-Section**

More detailed discussions about the coating structure and composition, as well as alloy additions to the zinc bath, can be found in Porter's Zinc Handbook, as well as numerous industry publications.

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**Zinc Bath Alloys**

**General**

ASTM B-6 defines the common grades of Zinc used in the after-fabrication industry. The zinc used should be of quality corresponding to ASTM B-6 or better, with a guaranteed assay. Impurities such as iron contribute to the formation of wasteful dross and excessive aluminum reacts with the flux to form volatile aluminum chloride, other impurities can alter coating appearance and thickness.

**Metallic Additives**

A small concentration (0.005%) of aluminum is sometimes added as a brightening agent. Aluminum also improves the tightness of the coating, gives thinner coatings and improves the run-off on withdrawal. However, aluminum can make galvanized articles more susceptible to white rusting and may adversely affect the top flux if too much is added. Amounts over 0.01% are detrimental to the kettle flux. The flux becomes very viscous and stiff due to the difficulty of dissolving the aluminum oxide which forms on the zinc and due to the depletion of active ammonium chloride as a result of the reactions of aluminum with the flux to form volatile aluminum chloride. This level of aluminum may also lead to excessive fuming in dry kettle operations, as well as black spots on the work.

Aluminum is conveniently added as a zinc-aluminum alloy, with a wide range being available from zinc suppliers. Consideration should be given to making all the aluminum alloy additions at the exit area of the zinc bath since this is where aluminum will do the most good. Regular and frequent small additions of aluminum should be made by submerging the zinc-aluminum alloy units inside of suitable containers at one or more points in the exit area of the kettle to compensate for loss of aluminum in the coating. Experience will dictate the best alloy concentration and frequency to give optimum conditions.

Periodic checks should be made on the aluminum concentration in the zinc bath. This is especially helpful while experimenting to find the optimum addition rate. In case of severe trouble, excessive aluminum in the zinc bath can be reduced by agitating the zinc bath by mechanical stirring or by bubbling with air. This will cause the aluminum to oxidize and rise to the surface where it can be skimmed off. Another option is to add extra ammonium chloride to the flux; the aluminum will go off in the fume as aluminum chloride. (This is only an option for shops with excellent fume hoods.)

Nickel is added to thin coatings and control some reactive steel reactions; this is discussed below.

The presence of lead is useful for reducing the surface tension of the molten zinc, thus aiding drainage, in
concentrations of at least 0.5%. It also improves the wetting of the substrate. Lead is soluble up to about 1.4%. Additions beyond that settle to the bottom and are sometimes added to aid in the removal of dross. For this purpose, sufficient lead should be added to form a layer several inches thick on the bottom of the kettle if this can be done without raising the dross into the upper work zone. The actual level of lead in the coated product is small.

**Bismuth** has been suggested by recent patents as a possible substitute for lead.

**Magnesium** has been reported to significantly improve the corrosion resistance of zinc coating. It is usually found as an additive to higher aluminum level baths, and can lead to unfavorable flux interactions. Magnesium is also difficult to maintain in the bath, and is not used much in after-fabrication shops.

**Tin** has been added to give an improved spangle and brightness to the galvanized coating. Tin concentration must be kept below 1%, since tin adversely affects corrosion resistance and ductility of the coating.

The presence of copper is detrimental since it increases the thickness and speed of formation of an undesirable brittle alloy layer.

**Antimony** can also be a problem, as it may lead to the formation of color in the galvanizing and form highly poisonous stibene gas under certain conditions from skimmings and ash.

**Cadmium** was once used to increase spangle size; in addition, it helps fluxing action in “wet” kettles. This is no longer a common additive, but may be found in standard Prime Western.

For more information about alloys and various effects, see the references listed at the end of this Handbook.

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**ALLOY STEELS**

High tensile strength alloy steels can be galvanized successfully if certain precautions are followed. Troubles arise because many alloy steels are readily attacked by both the acid pickle and molten zinc, causing them to be overpickled and to take on an exceptionally thick coating of zinc due to rapid alloying with the zinc. These coatings can be brittle, non-adherent and have a somewhat dull appearance. With alloy steels the mildest treatment possible should be used in all the galvanizing steps:

- The time in the pickle should be just long enough to remove the scale and rust and no longer. To minimize the pickle time, the work should be well degreased. The acid pickle should be well inhibited and not unduly hot or strong.
- The use of a hydrochloric acid preflux is particularly detrimental because it attacks alloy steel so rapidly.
- The molten zinc bath should be maintained between 820° F (438° C) and 850° F (454° C). The time of immersion in the zinc should be carefully controlled and held to a minimum. As soon as the work is heated enough for the preflux to release and a coating to adhere, the work should be removed.
SILICON-KILLED AND REACTIVE STEELS

General Background

Steels which are not fully “killed” and which are low in silicon exhibit “normal” coating growth in the galvanizing kettle; the rate of coating growth decreases with time as the work is immersed in the molten zinc bath.

Steels can be “killed” with aluminum or silicon. Silicon is somewhat cheaper, so many companies are using more silicon for this purpose. This leads to the galvanizing problem of “reactive steels”. This is further complicated by the recycling of steel, which can increase the phosphorous content of the steel. High silicon or phosphorus steels are known as “reactive steels”.

If steel contains silicon and/or phosphorus, the growth of the zinc-iron alloy layer is linear with time. Very thick coatings result, consisting of only intermetallic compounds with no outer layer of unalloyed zinc. The coating will have a gray or mottled appearance. Brittle coatings may be experienced because of the structure and thickness of the inter-metallic layers. These types of steels are often called Sandolin steels, and they have 0.08 to 0.30% or higher silicon. The worst reactivity occurs around 0.1% silicon in the steel. Also known as reactive steels, they present a significant problem for galvanizers because the higher silicon steels have become more common in the past decade.

Three methods used to minimize these undesirable results follow; nothing has been developed yet which will completely eliminate the problem for all silicon and phosphorous levels.

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Processing Methods for Reactive Steels

Low Temperature Galvanizing

The silicon effect can be minimized by short dip times, low galvanizing temperatures, and very rapid quenching to prevent the accelerated reaction of steel and zinc. Galvanizing temperatures as low as possible are used. The concentration of flux should also be lower in this method, to enable fast dips. Use of a preheating system for the fluxed work is also a good idea—this will allow even shorter dips and lower kettle temperatures. Most ZACLON fluxes will tolerate preheating to 250° or 300° F.

Kettle temperatures of about 825°F will help slow the reaction, though not as much as an 800°F kettle; for these lower temperatures, preheating is necessary to prevent zinc freeze-up.

Molten Salt Baths have also been used for preheating and fluxing. High preheat temperatures can be obtained with molten salt prefluxes instead of the usual aqueous solutions. This kind of processing helps to control the rapid buildup of coating thickness associated with silicon steels.

The molten salt bath may be prepared using ZACLON® A or ZACLON® AB. Both products are suitable since they provide suitable prefluxing characteristics with minimal fuming. They are chemically stable and do not corrode the salt bath container excessively. Keys to molten salt or preheated work include:

1. The work should be quickly transferred to the zinc bath so that chilling does not take place. Fast transfer will minimize the possibility of caking the flux from the molten salt bath. Since these fluxes have very low activity, a top flux is recommended for use on the zinc bath to assure high quality galvanizing.
2. The molten salt bath permits decreasing the zinc bath temperature to 825°F (441°C) or below, which is desirable when galvanizing both silicon and standard steels.

3. The molten salt bath can accept work which is not completely dry without spattering. This is because the salt bath transfers heat slower than a molten zinc bath does. This feature permits fast production rates.

High Temperature Galvanization

At temperatures above 986°F (530°C), the catalytic effect of silicon is eliminated. This is related to the Zeta coating phase (94% zinc, 6% iron) being unstable above 986°F.

At the required temperatures, ceramic kettles are necessary. Also, iron does not form dross but remains in solution. Lead promotes good coating adhesion and should be present at about 1%. At this concentration, lead is below the solubility limit, so no free lead layer is present at the bottom of the bath as in standard galvanizing.

Aluminum additions are helpful as in standard galvanizing. However, because of the high temperatures at concentrations above 0.03%, iron-aluminum floating dross is formed; this requires constant removal to prevent coating problems.

Inhibited pickling baths should be used following suitable cleaning. A ZACLON® preflux is used at higher concentrations than normal if the work is to be galvanized in a dry kettle. Standard zinc ammonium chloride top fluxes have short life at the high temperatures used and also fume considerably. “Non-fuming” fluxes such as ZACLON® A are suggested.

The operating costs for high temperature galvanizing are significantly increased compared to standard galvanizing. Heat input is about 25% more. The rate of dross formation will be at least double, requiring more frequent removal. To accomplish this, the kettle must be cooled down and reheated with a resultant loss of production.

High temperature galvanizing produces a coating on standard steel thicker than those produced at normal temperature. This can create problems on assemblies using both metals.

Special Alloy Baths for Reactive Steels

General

The use of alloying elements in zinc to control reactive steel has been the focus of much research in the past 20 years. Two approaches have had some commercial success.

Nickel Additions

A process developed in Europe to deal with the silicon steel problem is called “Technigalva”, and it involves the use of nickel at 0.05 to 0.5% in the zinc bath. This suppresses the silicon effect up to about 0.2% silicon, by suppressing intermetallic growth. The nickel additive is either via master alloy or by patented addition devices such as Cominco’s Direct Alloying device.
The nickel process is works with both lead-free and leaded zinc, and requires no special fluxes. It will give a generally thinner coating thickness. It does change the character of dross and somewhat of ash generated. The zinc nickel coating is slightly more difficult to strip when recoating is needed.

Aluminum Additions

Another European method involves addition of 0.04% aluminum and some other modifiers. The patented zinc alloy is called “Polygalva” and a galvanizing process using this special alloy zinc bath has been described. Polygalva uses aluminum to modify the growth of intermetallic compounds. Aluminum, when used alone, can result in uncoated spots; magnesium is required to assure a continuous coating. Other constituents include lead to decrease the surface tension of zinc on steel, and tin to improve the coating appearance and to enhance lead and magnesium functions. Controlling all of these alloy additions, and making sure the zinc bath is well mixed and correctly alloyed, is a drawback for this process.

Another drawback is the need for extra surface preparation prior to galvanizing is essential for good results. After cleaning, the work must be pickled in hydrochloric acid with an inhibitor, followed by rinsing. The work must then be immersed in stronger, uninhibited hydrochloric acid. After rinsing, the work is given a standard zinc ammonium chloride preflux and dried. The steel may then be galvanized in the “Polygalva” bath.

GALVANIZING BY-PRODUCTS

In the operation of a galvanizing kettle, several by products are formed. These are dross, zinc oxide or ash, and flux skimmings. Generally the quantity of these products generated can be controlled to some extent. The three by-products do have some sale value; however, it is more profitable to restrict their generation.

Dross

Dross contains approximately 96% zinc and 4% iron. Dross crystals sink to the bottom of the zinc kettle and must be removed periodically. Due to the high zinc content of dross and the entrainment of additional pure zinc in the dross crystals, the development of dross represents an uneconomical loss of zinc and should be minimized. Depending upon specific plant practices, 10-35% of the zinc consumed in a plant may be removed as dross.

Dross is produced from the following sources:

1. **Iron salt carryover**: Iron salts, formed in the pickle solution and/or the preflux tank and carried over to the galvanizing bath on the work, react with the zinc and top flux to form dross. A preflux of ZACLON® Galvanizing Flux results in considerably less dross from this source than a hydrochloric acid preflux.
2. **Flux-steel reaction**: The reaction between the flux and the steel being galvanized produces iron salts which form dross. The amount of dross from this source increases with acidity in the flux, drying time of the work, and in top flux the contact time, activity, and temperature of the flux blanket.
3. **Zinc-steel reaction:** The amount of dross formed by direct reaction of the zinc with the steel depends on (a) the temperature of the zinc, (b) the time of immersion and the surface roughness of the steel. For example, the rate of dross formation at 875°F (468°C) is twice that at 840°F (449°C). Dross formation increases with the time of immersion, but the rate of attack for lower silicon steels falls off as the alloy layers on the steel thicken. The effect of surface roughness is to increase the area of steel exposed to the action of the zinc. Causes of surface roughness are overpickling and sandblasting.

4. **Reaction between zinc and the kettle:** This is usually only a small part of the total dross. The steel plate of the kettle quickly develops an inner lining of protective alloy layers, which slows further reaction between the kettle itself and the molten zinc.

In summary, these are the recommendations for minimizing dross formation:

- Avoid overpickling.
- Rinse work well after pickling to prevent carryover of iron salts to preflux solution.
- Keep the iron content of the preflux solution below 0.5%.
- Pass the work through the top flux quickly to minimize the time of contact between the steel and flux.
- Use the lowest galvanizing temperature and the shortest immersion time in the galvanizing kettle consistent with good galvanizing.
- Avoid excessive concentrations of ammonium chloride in the top flux.
- Keep the zinc temperature within close limits by automatic control. Avoid localized overheating.

Dross should be removed before it builds up to the point where the work, in passing through the kettle, disturbs the dross which has settled on the bottom of the kettle. When the dross is stirred up into the molten zinc, rough coatings will result. Before and after dross removal, allow the kettle to stand idle for a few hours.
Some flux skimmings are also created in the dry galvanizing operation. All the flux product in the dry preflux film carried on the work surface will be released when the work is immersed in the molten zinc bath. As the flux accumulates on the surface of the bath, it will quickly decompose due to the high temperature. Considerable smoke will be evolved as the contained ammonium chloride is decomposed. Aluminum added to the zinc bath also reacts with the flux releasing smoke. The flux should be skimmed off the kettle surface as soon as a buildup occurs.

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**HOLDING THE LINE ON GALVANIZING COSTS**

Good galvanizing is the result of carefully maintaining the proper surface on the work pieces during each step of the process. Careful attention to operating conditions also leads to economical galvanizing with minimum losses and maximum profits.

Zinc is usually the single largest material cost factor in a hot dip galvanizing operation. Ideally, all the zinc purchased will be converted to zinc on the work pieces being galvanized. But in many plants, only 50 to 60 percent of the zinc consumed actually ends up on the work. Even this figure contains a certain amount of zinc loss due to zinc applied over and above that specified as the coating thickness. The 40 to 50 percent not used in coatings is accounted for by the following sources of zinc loss:

- Dross (contains about 96% zinc)
- Zinc Oxide or Ash (contains about 70% zinc)
- Flux Skimmings (contain about 40% zinc)
- Spatter (100% zinc)
- Zinc stripped off work carriers
- Additional zinc entrained during careless removal of dross, ash or flux skimmings.

Identifying and monitoring each source of zinc loss are the first steps toward improved profitability. Ways to avoid producing excessive coating thickness may include:

1. Reduce excessive surface roughness due to overpickling and/or mechanical finishing involving grit or shot blast.
2. Avoid cooling prefluxed pieces which then require increased immersion time.
3. Avoid removal of work too rapidly to permit adequate zinc drainage.
4. Optimize angle of withdrawal to get best zinc drainage.
5. Be certain silicon steel pieces are properly identified and galvanizing conditions adjusted to adequately process them.
6. Keep the zinc bath temperature below 900°F (482°C), preferably at 850°F (454°C).

In actual practice, compromises are often made to improve one processing step at the expense of another. These “trade-off” decisions are best made only when the galvanizer has developed current cost data for each step of the process. He knows the cost of each source of zinc loss and which sources can produce income through resale value of the by-product. Stripped zinc not only has no resale value but also represents an added cost penalty where pollution control procedures require removal of zinc from waste solutions. One galvanizer may reduce zinc losses by improving his rinsing operation, thus reducing dross formation due to iron in the pickle solution. Another galvanizer may use abrasive blasting to save time in removing lacquer coatings. He knows he has an appreciable zinc loss due to excessive coating thickness but he carefully controls this loss by preheating the work pieces so that immersion time
can be kept to a minimum.

The selection of a galvanizing flux also affects costs and profits. The two most important functions of galvanizing flux are 1) to dissolve zinc and iron oxides which interfere with galvanizing and 2) to protect clean zinc and iron surfaces from oxidation. The kettle flux protects zinc surfaces while the preflux protects freshly pickled steel.

Hydrochloric acid has been used as a preflux. Iron chloride salts form on the work and react with zinc to produce dross. This zinc loss together with the cost of periodic dross removal can be reduced by switching to ZACLON® galvanizing fluxes. ZACLON® protects the steel, reduces dross formation and provides better insurance of good galvanizing than does HCl. (See Table III on page 3-2).

Ammonium chloride (NH₄Cl) effectively dissolves oxides because it is an active source of HCl at galvanizing temperatures. But ammonium chloride top flux is usually not the most effective and least expensive material to use. The ammonium chloride in a zinc ammonium chloride top flux is consumed and lost too rapidly at galvanizing kettle temperatures to be really cost effective. In order for NH₄Cl to remain on the kettle as a molten salt, it must be dissolved in molten zinc chloride. Ammonium chloride can indeed be reacted with zinc from the kettle to form enough ZnCl₂ to provide an equilibrium system but this is a costly, time consuming process. Even after sufficient zinc chloride is generated on the kettle, there are still large ammonium chloride losses as evidenced by the continuous smoky fumes. ZACLON® galvanizing fluxes are designed to keep losses to a minimum and thus are more economical and easier to use.

The information presented in this section is based on Zaclon experience and other sources. Additional technical information on galvanizing is available from the organizations listed in Section 9.

The proper use of ZACLON® fluxes together with good galvanizing techniques and controls will produce “on-spec” coatings, improved zinc yields, and improved profits.
SECTION 5: FINISHING AND POST-TREATMENTS

SHAKING OR CENTRIFUGING

After the work emerges from the molten zinc, the excess zinc should be shaken off. This can be done with a vibrator on the hoist, by striking the work or carriers, or by centrifuging where practical.

QUENCHING

The art of quenching is gaining more attention as high-silicon/phosphorous “reactive” steels become more common. The use of a rapid quench after dipping can stop the formation of the gray or alligator coating, if this has not already formed on the work as it is removed from the molten zinc. In some operations, and some zinc alloys, the phenomenon of peeling can also be seen; if work cools too slowly, such that the temperature remains above 550°F for periods over 10 or 15 minutes, the pure zinc layer may peel off of the work. The quench will also wash off any flux residues that may be on the work from top flux or poor skimming.

The quench bath should be fresh water, unless a product such as ZACLON® WRP or other passivation treatment is used. Water quenching will build up both corrosives and inert salts in the quench; this will build-up in an over-used tank and may leave white residues on the work; for this reason, the quench tank should be changed out often.

Keeping the quench tank above 150 degrees has the benefit of drying the work faster, which makes it easier to handle and store.

WET STORAGE STAIN (WHITE RUST)

General

The normal corrosion products of zinc are zinc oxide and alkaline zinc carbonate; the zinc is solubilized by atmospheric moisture such as dew or rainwater in combination with galvanic currents into a zinc hydroxide within hours of galvanizing; this then reacts with oxygen to form oxide in a few days or months, and the oxide then reacts with CO₂ to form the carbonate. Zinc carbonate is a cohesive and slightly porous film; since film is considerably more inert than the underlying zinc, it also forms a barrier that slows the attack of water and air on both the zinc and the iron beneath it. The carbonate gives the characteristic dull gray appearance to older galvanized steel; the oxide, which is less useful as an inert coating, is a white material that is less adhesive. This change in the zinc surface over
time is one reason that galvanized steel varies in paintability.

Under certain conditions, however, this normal corrosion of the zinc is accelerated greatly, such as in marine environments or in acid rain areas; the protective zinc carbonate/zinc oxide layers never fully form, and the zinc is dissolved more rapidly. If the work is stored wet and closely nested, zinc hydroxides will form, again without the normal conversion to protective carbonate or oxide; this is the phenomenon known as “wet storage stain” (WSS). This stain is also called white rust. It will also form if the closely stacked work is rained on, or is under conditions promoting formation of dew or condensation on the work.

Methods to prevent white rust are important for two reasons: first, the corrosion products give an unsightly, mottled surface; secondly, the loose powder can make subsequent coating (such as painting) more difficult and expensive. Finally, severe white rust may damage long-term protection of the base steel, as the corrosion makes some of the zinc unavailable for sacrifice.

The methods used to control white rust to date have several aspects in common. All involve the formation of a barrier coating of some sort, which can be formed by reacting directly with the zinc to form “conversion” or passivation coatings. A barrier can also be developed by using a coating that adheres to the zinc, as in light oils, paints or primers. The methods used by galvanizers are varied, and depend upon processing and storage considerations, chemical treatments and disposal costs, and the final use of the product, as well as customer requirements and, of course, cost effectiveness.

Processing and Storage Considerations

One of the best ways to minimize or prevent the formation of white rust is in the initial galvanizing process. Bablik noted that work held at an elevated temperature for ten or fifteen minutes after galvanizing had better resistance to white rust simply due to the more rapid formation of oxides and carbonates at temperature. Of course, it is difficult to do this in a busy shop, and can lead to variations in spangle size and surface smoothness. As mentioned above, reactive silicon or phosphorous steels require rapid quenching to prevent the characteristic alligator or graying patterns. A quenched work should be allowed to dry before storing.

Proper storage of the work is helpful. Use of wide-spaced racks with wood between layers, and angled storage to allow moisture to drain off the work will minimize wet storage stain; covering the work or storing it inside prevents some staining as well. Unfortunately, proper storage is not always practical for an average post-fab galvanizer, and he has no control over how his customers store product.

There has also been a little work with zinc alloys to prevent white rust. Certainly, higher than normal aluminum can promote white rust; in very high levels (over 1%) a “black” rust can occur. The effects of other alloying materials is not yet clear.

General Mechanism

There are several chemical treatments for the prevention of white rust. We will look in some detail at those that form a “conversion” coating, or “passivate” the surface of the zinc. This means that they react with the zinc to form a more
inert zinc compound; in a sense, they mimic the long-term weathering of galvanized to zinc carbonate in a rapid reaction. Some treatments are an improvement over the carbonate over the short term, providing a more hydrophobic surface and having “self-healing” properties. Other treatments convert the surface to a more paintable form as well as providing the necessary barrier to moisture.

ZACLON® WRP and Metal Silicates

The use of metal silicates is a newer approach to forming conversion coatings on zinc; the use of silicate based zinc-rich primers has been known for many years, but the idea was not applied to conversion coatings until the late 1980's. DuPont, and its LBO Zaclon, obtained a general use patent in 1989 which covers the use of metal silicates on hot-dipped zinc surfaces. ZACLON® WRP is the current state of the art product from Zaclon for protection of zinc using silicate-based conversion coatings.

Silicate Chemistry

Silicates are solutions or solids of the general formula \( M_2SiO_2 \). The M can be just about any metal, or a combination of metals. The most useful picture of a silicate solution is as a solution of colloidal silica \( (SiO_2) \), which is a low molecular weight polymer, stabilized by an alkali; hence the practice of using weight ratios of \( M_2O \) (another way of expressing the alkali level) to \( SiO_2 \) is common in the silicate industry. Most of the metal silicates, such as calcium, magnesium, aluminum, and zinc are water insoluble; these are the materials we want to create to form the conversion coating. Many of the minerals found in nature, including precious stones such as emeralds and sapphires are specific forms of zinc, aluminum, and traces of other elements combined with silicate anion, and are very stable, inert materials.

Silicate Mechanism

The conversion coating using silicate is one that forms by the reaction of a fresh zinc surface with the silicate to form a zinc silicate layer over the zinc; this slightly opaque layer is quite insoluble, especially in acidic solutions, and water-resistant. It also appears to promote the formation of alkaline zinc carbonates to provide more protection; these are the same carbonates desired on normal passivated galvanized articles.

Advantages to Silicate Treatment

The primary advantage to the use of silicate technologies is the fact that they are very “green”; silicates are not hazardous to the environment especially at the dilute levels used in ZACLON® WRP, and have no air emissions.

A second advantage to the silicate system is that they are quite resistant to acid conditions; preliminary Kesternich (Acid Rain) results show three times the protection of chromate or untreated galvanized panels.

Silicate treatment should also leave the surface compatible with all major paint vehicles, cements, and concrete, and may improve adhesion.

Disadvantages to Silicate Solution

The primary disadvantage to silicate systems is that they require more monitoring and control than chromates; work temperature, concentration, and filtration all must be controlled for proper protection.
The silicate process generally leaves the zinc less reflective than untreated or chromated work. Also, the coating is also slightly permeable to and will absorb some moisture; while it controls wet storage stain and white rust, neutral salt spray readily penetrates and corrodes the underlying zinc. (Chromates do not allow this corrosion in salt spray.) The tendency to absorb water can cause silicated work to become more opaque in high humidity, and may even look like it has white rust. This white deposit (usually a streak) is hydrated zinc silicate, tightly bound to the surface, not the loose grit of white rust. The opacity will disappear as the work dries out.

**Environmental Concerns**
Silicates are alkaline materials, and in the concentrated forms are somewhat corrosive. (See the MSDS sheet in this section.) ZACLON® WRP is not, however, DOT Corrosive. There are no air emissions to be concerned about, and disposal is relatively easy. In fact, the dilute use solutions usually can be disposed of as nonhazardous (assuming no large concentration of zinc has built up); some of it can be added as make-up to caustic cleaning solutions (if there is adequate rinsing to prevent silicate carry-over into pickle tanks.) All of the health effects of silicate solutions described in the MSDS are for concentrated solutions; even in the concentrate, these materials are far less hazardous than the chromates.

**Use of ZACLON® WRP**

**Equipment**
The WRP product is designed for use in existing quench tanks; a 5 micron filter and pump are recommended to keep the bath clean, as is a cover for the tank for when it is not in use. A heat exchanger is also suggested to maintain bath temperatures.

**Use Levels**
WRP is used at 2.5 to 4 weight percent, with the optimum being 2.8 to 3.2%. There is a Hach Test Kit modified by Zaclon to monitor the level of WRP in the quench tank. The quench tank should be filtered, and for the best results the water should not be too hard. (Hard water will use up a little of the WRP to form precipitate, which is much like soap scum; this can lead to more opaque coatings.)

**Processing**
Work to be treated should be freshly galvanized and must be at a temperature of 160-235°C (320-450°F). Temperatures exceeding 235°C (450°F) can lead to mottled, streaky coatings, though the wet stain protection is still present and the mottling is not loose as in wet storage stain. (The bath will also become spent very rapidly if high quenching temperatures are used.) Temperatures under 325°F will not form the protective coating. This temperature restriction is the main drawback of ZACLON® WRP. The reaction is almost instantaneous, and the work can be removed within 30 seconds. For the best appearance, excess WRP solution should be shaken off. In addition, keeping the quench tank temperature between 50 and 75°C (120-160°F) will enhance the appearance of the finished product. The coated product will not be as bright and shiny as a chromated work; it will have a slightly dull surface.
Chromates

The use of chrome salts (also called chromates) for passivation is the most common method used on zinc, and has been proven effective for many years.

Chromates are the salts of chromic acid, \( \text{CrO}_3 \). The chromates have the general formula \( \text{X}_2\text{Cr}_2\text{O}_7 \), and the chrome is in the plus 6 oxidation state, written \( \text{Cr} \ (\text{VI}) \). In metal finishing applications, the \( \text{X} \) is usually sodium (\( \text{Na} \)) or potassium (\( \text{K} \)). (There are other oxidation states of chrome, including \( \text{Cr} \ (\text{III}) \) and \( \text{Cr} \ (\text{II}) \), but salts of these are either unstable or ineffective for direct passivation.)

**Chromate Use**

Chromates are used in concentrations ranging from 0.25% to 5% in water, depending on the level of protection needed and the appearance of the work requested. They can be used either in the quench tank, or later in the process on cooled work. Low levels of chromate give little color to the work, leaving it a bright, shiny surface which is lightly protected. The higher levels of chromate will make the surface turn yellow to brown in color, and will protect the galvanized very well. In all cases, the chromate bath is acidified, usually with sulfuric acid. For zinc-aluminum alloys such as continuous grade, and for aluminum and its alloys, HF or another fluoride is often added as an activator. Proprietary formulations may have additional activators, surfactants, and miscellaneous additives to improve performance or provide a variation on the yellow color. (Zinc electroplate can be tinted yellow, olive, blue, black, and red-orange using variations on chromate solutions.)

**Chromate Advantages**

The advantages of chromate passivation are: (1) ease of use; (2) efficacy in most accelerated and long-term testing, especially neutral salt spray; (3) low cost raw materials; (4) leave attractive appearance; (5) reasonably paintable; and (6) well accepted by most customers.

**Chromate Disadvantages**

The major disadvantage to chromate treating is the environmental and safety impact of \( \text{Cr} \ (\text{VI}) \), which is one of the nastier inorganic chemicals. (This will be discussed in following sections.) Chromate is also subject to problems in alloy composition; for example, the need for fluoride activation with continuous grade alloys and alloys with magnesium, and the problems occurring with various zinc alloys with lead and antimony. In addition, chromated work is not tolerant of acid rain conditions, and will fail rapidly under Kesternich testing. Finally, chromated surfaces cannot be primed with phosphate primers or vinyl-wash type paints.

**Environmental Concerns**

Hexavalent chrome (\( \text{Cr} \ (\text{VI}) \)) has been identified as a major health concern in our environment and the workplace. Stringent laws concerning landfill, water and air emissions of hexavalent compounds have been passed in many states. This is due to the toxicity and carcinogenicity (cancer-causing properties) of these chromates, as well as suspected mutagenicity and teratogenicity. Chromates are also poisons and significant skin and eye irritants. Hexavalent chrome has also been targeted for new OSHA regulations in workplace air emissions, with some groups asking for extremely low TLV/PEL limits. The increasing regulation has made chromates very unattractive compounds, but no suitable “drop in” replacement has been found.

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SPECIFICATIONS FOR GALVANIZED WORK

ASTM (American Society for Testing and Materials) is the primary source for standard specifications of galvanized work in the U.S., and has both the specifications and test methods used for checking of work. There has been considerable revision of many of the specifications during 1993 and 1994.

AASHTO and MIL specs also exist for galvanized products, and there are other specific requirements given by certain customers.

A good reference for inspection of galvanized products can be obtained from the American Galvanizer's Association; it is titled “Inspection of Products Hot Dip Galvanized After Fabrication.” For more information relating to specifications, you can contact the American Galvanizers Association, or the Galvanizers Association (for Sheet, Pipe, and Wire).

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- Dull Gray Coatings
- Rough Coatings
- Uneven and Lumpy Coatings
- Blisters
- Uncoated Areas
- Flux Spots, Flux Inclusions
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- Excessive Smoking
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  - Top Fluxes/Wet Kettle
- EXCESSIVE DROSS FORMATION
  - Dry Kettles/General Operation
  - Top Flux Operations

GALVANIZING COATING DEFECTS AND SOLUTIONS

Poor Adhesion

<table>
<thead>
<tr>
<th>POSSIBLE CAUSE</th>
<th>SOLUTIONS</th>
</tr>
</thead>
<tbody>
<tr>
<td>Alloy to layer too thick</td>
<td>- Shorten immersion time</td>
</tr>
<tr>
<td></td>
<td>- Reduce zinc bath temperature</td>
</tr>
<tr>
<td></td>
<td>- Adjust alluminum concentration</td>
</tr>
<tr>
<td></td>
<td>- Quench after galvanizing</td>
</tr>
<tr>
<td></td>
<td>- Eliminate copper contamination of zinc bath</td>
</tr>
</tbody>
</table>
### Dull Gray Coatings

<table>
<thead>
<tr>
<th>POSSIBLE CAUSE</th>
<th>SOLUTIONS</th>
</tr>
</thead>
<tbody>
<tr>
<td>Alloy to layer too thick</td>
<td>• Shorten immersion time; increase preheat</td>
</tr>
<tr>
<td></td>
<td>• Reduce zinc bath temperature</td>
</tr>
<tr>
<td></td>
<td>• Quench after galvanizing</td>
</tr>
<tr>
<td></td>
<td>• Silicon/Reactive steel substrate; see silicon steels in Section 4</td>
</tr>
<tr>
<td>Finished work exposed to pickling fumes</td>
<td>• Store work away from pickle</td>
</tr>
<tr>
<td></td>
<td>• Improve exhaust of pickling fumes</td>
</tr>
<tr>
<td>Insufficient aluminum in zinc</td>
<td>• Increase aluminum concentration</td>
</tr>
<tr>
<td>Oxide occluded in the coating</td>
<td>• Skim withdrawal area</td>
</tr>
</tbody>
</table>

### Rough Coatings

<table>
<thead>
<tr>
<th>POSSIBLE CAUSE</th>
<th>SOLUTIONS</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rough steel surface from overpickling</td>
<td>• Use less aggressive pickle conditions</td>
</tr>
<tr>
<td></td>
<td>• Add inhibitor to pickle</td>
</tr>
<tr>
<td>Sandblasting; shotblasting</td>
<td>• Change method of surface preparation</td>
</tr>
</tbody>
</table>
### Uneven and Lumpy Coatings

<table>
<thead>
<tr>
<th>POSSIBLE CAUSE</th>
<th>SOLUTIONS</th>
</tr>
</thead>
<tbody>
<tr>
<td>Inadequate drainage during withdrawal</td>
<td>- Raise kettle temperature</td>
</tr>
<tr>
<td></td>
<td>- Shake or strike part to remove excess zinc</td>
</tr>
<tr>
<td></td>
<td>- Add aluminum to zinc bath</td>
</tr>
<tr>
<td></td>
<td>- Check lead level--add if below 0.5%</td>
</tr>
<tr>
<td></td>
<td>- Articles in contact on withdrawal</td>
</tr>
<tr>
<td></td>
<td>- Delayed run-off</td>
</tr>
</tbody>
</table>

### Blisters

<table>
<thead>
<tr>
<th>POSSIBLE CAUSE</th>
<th>SOLUTIONS</th>
</tr>
</thead>
<tbody>
<tr>
<td>Surface and Subsurface Steel Defects</td>
<td>- Obtain or Specify Higher Quality Steel</td>
</tr>
<tr>
<td>Absorbed Hydrogen</td>
<td>- Shorten Pickle Time</td>
</tr>
<tr>
<td></td>
<td>- Lower Pickle Temperature</td>
</tr>
<tr>
<td></td>
<td>- Use Inhibited Acids</td>
</tr>
</tbody>
</table>
## Uncoated Areas

<table>
<thead>
<tr>
<th>POSSIBLE CAUSE</th>
<th>SOLUTIONS</th>
</tr>
</thead>
</table>
| Inadequate cleaning            | • Change cleaner  
   • Use higher cleaner temperature                                          |
| Inadequate pickling            | • Pickle for longer time  
   • Purify pickle  
   • Increase acid concentration  
   • Increase pickle temperature                                            |
| Inadequate prefluxing          | • Increase preflux Baumé  
   • Purify preflux  
   • Adjust preflux ratio by adding 0.5 lb./gal (60 g/L) ZACLON® 2N           |
| Build-up of spent flux ash, and dirt | • Skim the zinc bath                                         |
| Spattering                     | • Deeper flux blanket  
   • Improve drying  
   • Add Wetting Agent A to preflux bath  
   • Check ratio; make sure ratio below 1.0; adjust with ZACLON® 2N if necessary |

## Flux Spots, Flux Inclusions

<table>
<thead>
<tr>
<th>POSSIBLE CAUSE</th>
<th>SOLUTIONS</th>
</tr>
</thead>
</table>
| Inactive top flux              | • Keep sulfate in preflux low  
   • Add more active flux (ZACLON® 2N)  
   • Keep work in kettle longer to release flux  
   • Add flux more frequently  
   • Add flux more frequently                                   |
<p>| Oxides in kettle top flux      | • Replace top flux                                                        |</p>
<table>
<thead>
<tr>
<th>POSSIBLE CAUSE</th>
<th>SOLUTIONS</th>
</tr>
</thead>
<tbody>
<tr>
<td>Parts stacked before dry</td>
<td>• Let parts dry</td>
</tr>
<tr>
<td></td>
<td>• Use warm or hot quench tank</td>
</tr>
<tr>
<td>Moisture on parts</td>
<td>• Keep out of rain or snow</td>
</tr>
<tr>
<td></td>
<td>• Avoid dew, condensation</td>
</tr>
<tr>
<td></td>
<td>• Store indoors if possible</td>
</tr>
<tr>
<td>Parts not post-treated</td>
<td>• Dip in ZACLON® WRP White Rust Inhibitor</td>
</tr>
<tr>
<td></td>
<td>• Dip in chromate solution</td>
</tr>
<tr>
<td></td>
<td>• Coat with oil or lacquer</td>
</tr>
<tr>
<td>Too much aluminum in the zinc</td>
<td>• Reduce aluminum addition</td>
</tr>
<tr>
<td></td>
<td>• Oxidize excess aluminum with air sparge</td>
</tr>
</tbody>
</table>
### Excessive Smoking

Any Operation

<table>
<thead>
<tr>
<th>POSSIBLE CAUSE</th>
<th>SOLUTIONS</th>
</tr>
</thead>
<tbody>
<tr>
<td>Kettle temperature too high</td>
<td>• Reduce temperature</td>
</tr>
<tr>
<td>Inadequate ventilation</td>
<td>• Improve ventilation</td>
</tr>
<tr>
<td>Use of ammonium chloride</td>
<td>• Change of ZACLON® F or 2N</td>
</tr>
</tbody>
</table>

### Prefluxes/Dry Kettle Operations

<table>
<thead>
<tr>
<th>POSSIBLE CAUSE</th>
<th>SOLUTIONS</th>
</tr>
</thead>
<tbody>
<tr>
<td>Too much flux on work</td>
<td>• Decrease flux concentration</td>
</tr>
<tr>
<td></td>
<td>• Increase flux bath temperature</td>
</tr>
<tr>
<td></td>
<td>• Add Wetting Agent A</td>
</tr>
<tr>
<td>Flux too active</td>
<td>• Add zinc slab</td>
</tr>
<tr>
<td></td>
<td>• Add fresh ZACLON® K or F</td>
</tr>
<tr>
<td></td>
<td>• Add ZACLON® C</td>
</tr>
<tr>
<td>Inadequate preheat</td>
<td>• Increase flux bath temperature</td>
</tr>
<tr>
<td></td>
<td>• Improve preheating/drying techniques</td>
</tr>
<tr>
<td>Work not clean</td>
<td>• Improve cleaning (especially for grease and oil)</td>
</tr>
</tbody>
</table>

### Top Fluxes/Wet Kettle
<table>
<thead>
<tr>
<th>POSSIBLE CAUSE</th>
<th>SOLUTIONS</th>
</tr>
</thead>
<tbody>
<tr>
<td>Inadequate foam thickness</td>
<td>• Increase ZACLON® F or 2N additions</td>
</tr>
<tr>
<td>Top flux too active</td>
<td>• Maintain with less active grade</td>
</tr>
<tr>
<td>Localized overheating</td>
<td>• Provide for uniform heating</td>
</tr>
<tr>
<td>New top flux too active</td>
<td>• Start-up with ZACLON® Start-Up</td>
</tr>
<tr>
<td>Foaming agent exhausted</td>
<td>• Skim off dead flux and add ZACLON® F</td>
</tr>
<tr>
<td>Top flux too viscous/High Sulfate in Preflux</td>
<td>• Purify the preflux solution&lt;br&gt;• Improve rinsing after pickling&lt;br&gt;• Use ZACLON® Sulfate Control</td>
</tr>
<tr>
<td>Top flux too viscous/Excessive aluminum in zinc bath</td>
<td>• Reduce aluminum additions</td>
</tr>
<tr>
<td>Top flux too viscous/Build-up of refractory zinc and iron compounds</td>
<td>• Skim compounds out of flux and add fresh ZACLON®</td>
</tr>
<tr>
<td>Top flux too viscous/Insufficient flux additions</td>
<td>• Increase maintenance rate&lt;br&gt;• Use preflux&lt;br&gt;• Increase preflux concentration</td>
</tr>
<tr>
<td>Too much aluminum in zinc</td>
<td>• Reduce aluminum additions</td>
</tr>
<tr>
<td>Excessive top flux agitation</td>
<td>• Immerse work carefully&lt;br&gt;• Withdraw through skimmed zinc&lt;br&gt;• Resist stirring top flux</td>
</tr>
<tr>
<td>Large maintenance additions</td>
<td>• Use of ZACLON® 2N rods&lt;br&gt;• Increase preflux concentration&lt;br&gt;• Use frequent small additions</td>
</tr>
</tbody>
</table>
### Dry Kettles/General Operation

<table>
<thead>
<tr>
<th>POSSIBLE CAUSE</th>
<th>SOLUTIONS</th>
</tr>
</thead>
<tbody>
<tr>
<td>Iron salts carried into kettle</td>
<td>• Replace pickle</td>
</tr>
<tr>
<td></td>
<td>• Improve rinsing</td>
</tr>
<tr>
<td></td>
<td>• Put Zn or ammonium hydroxide in preflux tank to raise pH</td>
</tr>
<tr>
<td>Kettle temperature too high</td>
<td>• Reduce kettle temperature</td>
</tr>
<tr>
<td>Localized overheating</td>
<td>• Provide uniform heating</td>
</tr>
<tr>
<td>Steel surface too rough</td>
<td>• Add ZACLON® C</td>
</tr>
<tr>
<td>Inadequate preheat</td>
<td>• Change surface preparation</td>
</tr>
<tr>
<td></td>
<td>• Add inhibitor to pickle</td>
</tr>
<tr>
<td></td>
<td>• Avoid overpickling</td>
</tr>
</tbody>
</table>

### Top Flux Operations

<table>
<thead>
<tr>
<th>POSSIBLE CAUSE</th>
<th>SOLUTIONS</th>
</tr>
</thead>
<tbody>
<tr>
<td>Immersion too slow</td>
<td>• Speed up immersion</td>
</tr>
<tr>
<td>Top flux too active</td>
<td>• Move work through the top flux faster</td>
</tr>
<tr>
<td></td>
<td>• Maintain with less active grade</td>
</tr>
</tbody>
</table>
GENERAL INFORMATION

The galvanizing operation generates waste or pollution in several ways. Many of these are well beyond the scope of this Handbook, and the regulations local, state, and federal levels is changing rapidly; therefore, only a cursory look at pollution as it relates to some galvanizing chemicals is presented here. For more information on environmental concerns, contact the American Galvanizers Association.

AIR POLLUTION

Galvanizing operations generate fumes from the kettle itself, the flux as it is burned off, from the pickle and caustic cleaning operations, and possibly from the chromate tank. The composition of all of these fumes is beyond the scope of this Handbook, however, some general information on the composition of flux fumes is below.

Fumes from the Kettle and Flux Interactions:

The kettle itself generates a certain amount of particulate fume, especially when skimmed. These fumes consist primarily of Zinc Oxide, with a little Aluminum Oxide. The amount of fume depends slightly on the alloy being used, but more on the way the kettle is skimmed and how skimmings themselves are handled.

Fumes from the flux reactions depend upon the ratio of the flux, the concentration of the flux, and how good a cleaning job is being done. The fume is primarily ammonium chloride, with zinc oxide and a little zinc chloride in the particulate. The cleaning operation will determine the levels of carbon from greases and other soils carried over. An example from one job shop galvanizing operation had the following composition (in an EPA study):

<table>
<thead>
<tr>
<th>Component</th>
<th>Composition, wt %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ammonium Chloride</td>
<td>68.0</td>
</tr>
<tr>
<td>Zinc oxide</td>
<td>15.8</td>
</tr>
<tr>
<td>Zinc chloride</td>
<td>3.6</td>
</tr>
<tr>
<td>Zinc</td>
<td>4.9</td>
</tr>
<tr>
<td>Ammonia</td>
<td>1.0</td>
</tr>
<tr>
<td>Oil</td>
<td>1.4</td>
</tr>
</tbody>
</table>
As mentioned, the composition of the fumes can vary widely depending on the plant operation. For example, in a chain link fence operation oil can be about 40 percent of the fumes. In plants using the zinc bath for thermal paint stripping, the fumes may also contain heat decomposition products from the organic paint components.

The fume particles are typically less than 5 microns in diameter. The average particle size is 1.5 micron with about 90 weight percent being over 0.8 micron. For comparison, a 10 micron black spot on a white background is just barely visible to the naked eye. If plants use the practice of sprinkling with fine white ammonium chloride, the fumes will contain about 10 weight percent of particles in the range of 0.4 to 0.5 microns in diameter.

### Fume Control around the Kettle

In the event that galvanizing practices cannot be modified sufficiently to produce satisfactory air quality, equipment can be used to separate the fume particles from the air. Usually, fume control in a galvanizing plant may best be accomplished by local exhaust ventilation.

To control the emissions from a galvanizing kettle, the fumes generated must be conducted to an efficient control device. The headroom needed in batch galvanizing plants makes it necessary to use either high-canopy or room-type hoods. The amount of ventilation volume required increases considerably with the height of the hood; therefore, the size of the collector must be large enough to accommodate the large volumes of air required.

Slot hoods may be used for operations where the area of fume generation is small. The slot velocities needed to overcome the thermal draft for the entire surface of a large kettle are high, and large volumes of air movement across the kettle cool the surface of the zinc bath. When a slot hood can be used, the amount of ventilation required is smaller than that required with high-canopy hoods, and control devices are correspondingly smaller.

Low-canopy hoods may be suitable on a kettle when headroom is not required. These hoods permit lower ventilation rates for adequate fume capture, and smaller control devices can be used.

For the typical hot dip galvanizing shop using monorail hoist facilities to move work in and out of the kettle, consideration should be given to installing fireproof dry curtains to act as a hood or fume container over the galvanizing kettle. These should be hung as close to the side of the kettle as is practical, consistent with the size work that will require turning during processing at the kettle. The curtains may hang within 6-7 feet (1.8-2.1 m) of the floor and can be pulled back temporarily when unusually large work is being handled.

With collection ducts well designed to remove fumes and air from the trapped space over the kettle, an average air velocity of 50-70 ft/min. (0.25-0.36 m/s) under the edges of the hood will generally be sufficient provided there are no strong drafts caused by open doors or windows nearby.

When it is necessary to remove the fine fume particles from the air, various types of high efficiency gas cleaning or control devices may be considered. These include fabric filter devices usually known as bag filters, particle agglomeration devices, electrostatic precipitators and scrubbers with pressure drops in the range of 50 to 100 inches.
of water (12.4-24.9 kPa).

The efficiency of bag filters may be 99+ percent for removal of particles between 0.5 and 1 micron diameter. Electrostatic precipitator efficiency is about 90 percent for 0.5 micron particles and improves to about 96 percent for 1 micron particles. Scrubbers with pressure drops less than 10 inches of water (2.5 kPa) are ineffective. The efficiency at 60 inches of water (14.9 kPa) is about 96 to 98 percent for the above particle sizes.

Wet scrubbers will produce an acidic solution in addition to the solids removed from the air. The acidic solution will create another disposal problem.

Heating the total air flow to 150°F (66°C), will prevent moisture pick-up by the collected particles, enabling the bag houses to operate successfully at efficiencies over 99 percent. The bag house inside atmosphere must be kept above the dew point continuously, even during shutdown periods. Since the practice of heating so much air is a significant cost factor, use of lime dust addition instead of heat is employed to keep the bag filters from blinding.

The design considerations and specific operating conditions needed for the various control devices are beyond the scope of this handbook. The specific details may be obtained from suppliers of the control devices.

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**WATER POLLUTION**

Pollution abatement technology options include the following:

**Disposal Without Treatment**

**Contract Disposal**
Disposal of waste pickle solutions by contract is one method. Care is recommended since the company disposing of the solution is still liable for damages if the solution is disposed of improperly under RCRA. The procedures used by the hauler should be investigated thoroughly to be sure they are acceptable to regulatory agencies.

**Deep-Well Injection**
In many areas of North America, contract disposal in approved deep-wells is possible. Privately owned wells will require large volumes of waste pickle solutions before they are economically feasible. Furthermore, considerable study must be made by qualified geologists to be sure underground waters will not be polluted eventually.

**Miscellaneous Disposal Methods**
Arrangements may be made with certain municipal sewage districts who might find the waste acid solution useful even considering the zinc concentration. Local enforcement regulatory agencies should be consulted to establish to what extent such solutions may be released to sewer systems after they have been diluted with other plant water discharges.

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**Disposal With Treatment**

**Neutralization and Precipitation**
The most common and simplest way to treat waste acids is to neutralize with some alkaline chemical such as caustic soda or lime. An economical neutralizer would be residual chemicals in alkaline cleaning solutions which must be discarded.

Neutralization of the waste acid solution forms iron and zinc hydroxides which are gelatinous and slow to settle out. Gravity settling techniques may be employed to remove the large volumes of precipitates from the treated liquid. The settling rate depends upon the types of chemicals used in prior treatment and the conditions with which they were applied. The solids removal is a function of the retention time, particle size and density of the solids and the surface areas of the sedimentation catchment.

Organic polyelectrolytes enhance coagulation and significantly speed up complete precipitation. Specific flocculating agents and their concentrations can be selected to be compatible with the effective charge on the suspended particles. The time of addition in the processing cycle is critical since the agglomerated complexes may be broken up and settling effectiveness diminished if too much mixing occurs before the treated solution enters the settling device.

Filtration equipment or centrifuges may be considered to initially remove the precipitates or to polish the solution after the bulk of the metallic hydroxides have been removed by sedimentation.

**Solidification**

The term solidification collectively defines disposal technologies that fix or encapsulate waste in a solid matrix end-product. Fixation chemically and physically binds the waste with a solidification agent. Encapsulation methods physically surround the waste with a solidification agent. Both techniques reduce waste permeability and produce an end-product having significant shear strength. The fixed waste generally can be disposed of in an approved sanitary landfill. A number of companies are actively involved in fixation services and/or research.

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**Acid Recovery**

**General**

Various methods of regenerating both sulfuric and hydrochloric acid are available. The ones presented below should be suitable for typical galvanizers. Some methods have been successfully used in other metal finishing operations and may have application for recovering acids from pickling solutions.

**Purification**

Sulfuric acid solutions can be purified by using the basic principles of solubility and crystallization of iron and zinc salts. Efficient purification depends upon having the total impurity level as high as practical consistent with maintaining an acceptable pickling rate. Concentrations of impurities should be at or preferably above 8 percent. When the free sulfuric acid level is around 5%, it can hold about 14 to 16 percent metallic impurities in solution at 140° F (60° C) or higher. However, as the solution temperature is decreased to 68° F (20° C) only 8 percent is soluble. The metallic impurities formerly in solution at the higher temperature will have crystallized out. Addition of fresh acid after initial crystallization to produce a 15 percent free acid concentration will permit only 6 percent of impurities to remain in solution. Decreasing the temperature to 60° F (16° C) will only cause 4.5 percent metallic impurities to be soluble. There are commercial systems available which use this approach.

**Ion Exchange**

Metal ions can be removed from spent acid solutions in the same manner that hard water components are removed in water softening treatment. There are various concepts to accomplish purification. These involve use of ion exchange resins which can be selected to remove specific metals or all metals from the solution. The metals are freed from the ion exchange resin by a process known as regeneration chemicals. The purified acid may be returned to the
pickle tank. Ion exchange units are available from a number of vendors.

**Electrolytic Recovery (Electroplating)**
Electroplating techniques are applied to treating spent solutions to recover valuable metals in solutions or to "polish" the effluents before discharge. The process consists of passing the waste solutions through a cell (or tank) which contains insoluble cathodes and anodes. Direct current applied to the system through the electrolyte waste solution will cause metal ions such as iron or zinc to plate out on the cathode. At the same time, oxygen will be evolved at the anode. Metal collected on the cathode is disposed of in an appropriate manner.

**Electrodialysis**
Electrodialysis separates metal ions from waste pickle solutions. The equipment consists of three cells which are separated by semi-permeable membranes. Waste solutions are introduced into the center cell. Metal ions are positively charged and are drawn through the membrane into the cathode cell by electrolysis. These metal ions are then processed in an appropriate manner. In the case of sulfuric acid, sulfate ions are drawn into the anode cell after passing through the membrane and are then fed back into the sulfuric acid pickle solution is put back into the pickle tank. if hydrochloric acid solutions are treated, chlorine gas is generated in the anode compartment and needs special control.

**Disposal for Other Chemical Use**

**General**
Various companies may have interest in waste acid pickle solutions for the chemical value still available. Several possible considerations include the following: (Several may only have interest if there is little or no zinc present)

- Reducing agent value from the ferrous iron in either sulfuric or hydrochloric acid solution.
- Municipal sewage plant interest in ferrous chloride value.
- Wood preservative value of zinc content in hydrochloric acid.
- Agricultural interest in zinc value in either acid. Iron content will usually not present any problems.
- Animal feed value of zinc in either acid. Iron will not be a problem usually. Lead should be below 100 parts per million in most cases.
- Pesticide value of zinc concentration.
- Corrosion inhibition value of zinc for cooling water.
- Chemical manufacturing use of zinc in either acid.

**Zaclon Recycling Programs**
Zaclon, Inc., will often “swap-out” old, contaminated, used flux solutions for fresh flux. Costs for this will vary depending on distance from Zaclon, contamination level, and other factors. Contact your Zaclon representative for more details.

Zaclon, Inc., also recycles spent stripping acid from hydrochloric operations. This is acid from a dedicated stripping tank, not a pickle tank. There are advantages to a separate zinc stripping tank, including speed of strip, minimal zinc contamination in the pickle acid (allowing easier disposal of the spent pickle, usually), and possible recycle of this lower-iron stripping acid to Zaclon. Again, contact your Zaclon representative for more details on cost and availability.
Zaclon Incorporated, founded in 1987, supplies the galvanizing industry worldwide with top quality galvanizing chemicals. As leaders in innovation and customer service, Zaclon delivers a complete line of products to help galvanizers produce the highest quality galvanized products at the lowest cost.

For current data sheets and technical assistance selecting products, please call Zaclon Customer Service at 1-800-356-7327.

**DATA SHEETS:**

- **ZACLON® Grades:** C, CS, F, F, 2N
- **ZACLON® Grades:** A, AF, AB and Start-Up
- **ZACLON® Sulfate Control**
- **ZACLON WRP®**
- **ZACLON® Wetting Agent A**
- **ZACLON® W**
- **Zaclean® Concentrate**

**Zaclon Incorporated, Corporate Headquarters:**

2981 Independence Road  
Cleveland, Ohio 44115  
(216) 271-1601  

Customer Service: (800)-356-7327
APPENDIX A: TOTAL ZINC DETERMINATION

Discussion

Ethylenediaminetetraacetic acid is available as the free acid, disodium salt or tetra sodium salt. This method specifies the use of the disodium salt (dihydrate) because of its water solubility, favorable pH, and availability in pure form. The abbreviated term EDTA will be used here and refers to the disodium salt or its solutions.

The application of EDTA as a titrant for metal ions and as a metal complexing agent allows zinc in solution to be titrated directly. Use of the proper metal indicator, pH controls by buffering, and use of cyanide to prevent iron interference are required for this titration.

Direct titrations are based on the addition of a standardized solution of EDTA to a buffered solution of the cation to be determined until the equivalent point is reached.

Reagents

- EDTA Solution, 0.0575 M (molar): prepared by dissolving 21.4 g reagent grade disodium salt of EDTA in distilled water and diluting to 1 liter.
- Eriochrome Black T Indicator Mix: prepared by mixing 1 g dry indicator powder with 100 g dry sodium chloride.
- Buffer Solution: prepared by dissolving 67.5 g of ammonium chloride (C. P. grade) in approximately 200 mL distilled water, adding 570 mL of concentrated ammonium hydroxide (reagent grade) and diluting to 1 liter.

Equipment

- 50 mL burettes
- 10 mL pipettes
- rubber bulb
- 250 mL Erlenmeyer flasks
- 5 mL graduate
- 100 mL graduate
- 100 mL volumetric flask

Procedure (Run two titrations)

1. Pipette exactly 10 mL of bath sample into a 100 mL volumetric flask and dilute to 100 mL with distilled water.
2. Pipette exactly 10 mL of sample from volumetric flask into a 250 mL Erlenmeyer flask and add 50 mL distilled water.
3. Add 10-15 mL of buffer solution.
4. Add about 0.2 g of Eriochrome Black T indicator mix; sample should be dark purple in color.
5. Titrate with EDTA solution to a clear blue endpoint.

Calculations

1. Mi 0.0575 M EDTA = 3.759 mg zinc
2. oz/gal zinc = Mi EDTA X 3.759 X 0.1335
3. oz/gal zinc = Mi EDTA X 0.5018
4. g/L zinc = oz/gal zinc X 7.5
5. zinc X 2.0846 = zinc chloride
6. zinc chloride X 0.5203 = chloride
7. Record Mi EDTA solution needed to reach endpoint for each titration and take average of two readings and multiply by 0.5018.

Example: Two titrations 15.7 and 16.0 ml, average is 15.85 X 0.5018 = 7.95 oz/gal (60 g/L) zinc
ZACLEAN CONCENTRATE

DESCRIPTION

Zaclean Concentrate is a package of powerful cleansing agents and additives which activate the full cleaning power of a caustic bath against a wide variety of soils.

PROPERTIES

Appearance - Dark Blue Liquid

pH - 12.0

Density - 10 lbs/gallon

Odor - Citrus

USE

Zaclean Concentrate is designed for use in caustic baths having 5-15% NaOH concentrations and temperatures from 120 F to 185 F. The concentrate should be added directly to a prepared caustic bath at a rate of 0.022 gallons conc./gallon bath, or about 3 oz. per gallon. This is reflected in the following use table for "start-up".

Start-Up Table

<table>
<thead>
<tr>
<th>Gallons Bath</th>
<th>Gallons Concentrate</th>
</tr>
</thead>
<tbody>
<tr>
<td>100</td>
<td>5.5 (1 pail)</td>
</tr>
<tr>
<td>500</td>
<td>11.0 (2 pails)</td>
</tr>
<tr>
<td>1000</td>
<td>22.0 (4 pails)</td>
</tr>
<tr>
<td>2500</td>
<td>55.0 (1 drum)</td>
</tr>
<tr>
<td>5000</td>
<td>110.0 (2 drums)</td>
</tr>
<tr>
<td>10,000</td>
<td>220.0 (4 drums)</td>
</tr>
</tbody>
</table>

MAKE-UP

The make-up rates for Zaclean Concentrate are dependent on the soil type and heaviness, and to some extent on the type of steel involved.
good starting Point is the 3 OZ. per gallon level, just like the
start-up, measuring the gallons of liquid (whether water or caustic
solution) put into the bath. Light soil, or easily removed soil can
take as little as 1 oz./gallon make-up; heavy soils may take as much
as 4 oz./gallon.

Make-Up Table

<table>
<thead>
<tr>
<th>Gallons Added</th>
<th>Gallons Zaclean Concentrate</th>
</tr>
</thead>
<tbody>
<tr>
<td>100</td>
<td>0.75-2.2</td>
</tr>
<tr>
<td>250</td>
<td>1.890-5.5</td>
</tr>
<tr>
<td>500</td>
<td>3.7-11.0</td>
</tr>
<tr>
<td>1000</td>
<td>7.3-22.0</td>
</tr>
</tbody>
</table>

A clear, fresh caustic bath will turn blue when Zaclean Concentrate is
added, and will have a sweet odor. The color will fade as dirt levels
in the bath increase, but the odor will remain. The presence of the
odor and a little foam in an agitated bath are good indications that
Zaclean Concentrate is present and active.

Zaclean Concentrate at these use levels will clean a wide variety of
soils, but certain lacquers and dirts may require more caustic or more
Zaclean, or both, to give superior cleaning.

PACKAGING

Zaclean Concentrate is available in 550 pound, 55 gallon polyethylene
drums and in 55 pound, 5.5 gallon pails.

PRODUCT SAFETY

Read product MSDS Sheet. Wear normal protective gear - impermeable
gloves and safety glasses as a minimum. Follow standard industrial
hygiene practices, as well. Dispose of empty drums according to local,
state, and federal regulations. Addition of Zaclean to bath should not
effect disposal of bath via standard methods, but disposal companies
may ask for an MSDS sheet.
ZA CLON® GALVANIZING FLUXES

A ZACLON® Galvanizing Flux is available from Zaclon, Inc. for every galvanizing operation. Various grades of ZACLON® Flux have been developed based on extensive experience in the manufacture of zinc ammonium chloride fluxes, ammonium chloride fluxes and pickling acids and their application in the galvanizing industry.

ZACLON® Galvanizing Fluxes protect steel and zinc against oxidation, reduce dross formation, minimize ammonium chloride fume losses and insure good galvanizing. They are economical and easy to use.

Table 1 summarizes the characteristics and versatility of ZACLON® fluxes. The broad range of flux grades permits galvanizers to optimize flux performance for different galvanizing applications such as:

- Molten top flux in the galvanizing kettle.
- Aqueous preflux solution for hot dip applications.
- Aqueous preflux solution for continuous strip applications.
- Aqueous preflux solution for continuous wire line applications.
- Molten salt bath for prefluxing and preheating.

Different levels of flux activity allow the selection of a ZACLON® flux to meet specific operational requirements:

**Very active fluxes** facilitate high quality galvanizing. These fluxes should be considered especially when base metal surfaces can be given only a minimum amount of surface preparation or are difficult to prepare for galvanizing.

**Intermediate activity fluxes** will allow high quality galvanizing in applications where the base metal has been given adequate surface preparation.

**Low activity or minimal fuming fluxes** will make good quality galvanizing possible after painstaking base metal surface preparation has been provided or when "easy to galvanize" work is processed. When minimal fume galvanizing is required, the galvanizer must provide careful preparation of the base metal surface to compensate for the lower activity of these fluxes.

* Reg. U.S. Pat. & Tm. Off., Zaclon, Inc.
Zaclon® Fluxes are made only by Zaclon, Inc.

NOTICE: ZACLON® GALVANIZING FLUXES CAUSE SKIN & EYE BURNS.


The information set forth herein is furnished free of charge and is based on technical data that Zaclon, Inc. believes to be reliable. It
TABLE 1: Quick Reference for ZACLON® Galvanizing Fluxes

<table>
<thead>
<tr>
<th>ZACLON GALVANIZING FLUX</th>
<th>PURPOSE</th>
<th>USE IN: Preflux/Top Flux</th>
<th>RELATIVE FUME ACTIVITY</th>
<th>FOAMING TOP FLUX</th>
</tr>
</thead>
<tbody>
<tr>
<td>2N (Regular)</td>
<td>Top Flux</td>
<td>No/Yes</td>
<td>4</td>
<td>4</td>
</tr>
<tr>
<td>2N (Non-Foaming)</td>
<td>Top Flux</td>
<td>No/Yes</td>
<td>3</td>
<td>4</td>
</tr>
<tr>
<td>2N (Rods)</td>
<td>Top Flux</td>
<td>No/Yes</td>
<td>1</td>
<td>4</td>
</tr>
<tr>
<td>F</td>
<td>General Purpose</td>
<td>Yes/Yes</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>F-2</td>
<td>Preflux</td>
<td>Yes/No</td>
<td>NA</td>
<td>3</td>
</tr>
<tr>
<td>K</td>
<td>General Purpose</td>
<td>Yes/Yes</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>K-2</td>
<td>Preflux</td>
<td>Yes/No</td>
<td>NA</td>
<td>3</td>
</tr>
<tr>
<td>C</td>
<td>Preflux</td>
<td>Yes/Yes</td>
<td>3</td>
<td>2</td>
</tr>
<tr>
<td>CS</td>
<td>Preflux, When heating</td>
<td>Yes/Yes</td>
<td>3</td>
<td>2</td>
</tr>
<tr>
<td>HV</td>
<td>Top Flux Startup</td>
<td>No/Yes</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>A</td>
<td>Low Fume Top Flux</td>
<td>Yes/Yes</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>AB</td>
<td>Inert Kettle Cover</td>
<td>Yes/Yes</td>
<td>0</td>
<td>1</td>
</tr>
<tr>
<td>AF</td>
<td>Low Fume Top Flux</td>
<td>Yes/Yes</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>W Solution</td>
<td>Preflux</td>
<td>Yes/No</td>
<td>NA</td>
<td>2</td>
</tr>
<tr>
<td>W-HT</td>
<td>Continuous Galv.</td>
<td>Yes/No</td>
<td>1</td>
<td>2</td>
</tr>
</tbody>
</table>

The grades of ZACLON® Flux offered are based on zinc ammonium chloride but differ in physical form, fluxing activity and additive content. Some grades used as molten top fluxes are available with and without foaming agents. Foaming top fluxes may offer benefits to the galvanizer such as energy conservation and reduced evolution of flumes or smoke. Also, several grades of ZACLON® Flux provide a choice of physical form to accommodate specific needs which require either a small particle size or a larger rod shape.

THE GALVANIZING PROCESS

Clean, oxide-free iron or steel is galvanized by coating it with a thin layer of zinc. This protects the iron or steel by shielding it from the atmosphere, as well as providing cathodic or sacrificial protection. Even if the zinc coating is scratched and the base metal is exposed, the more electronegative zinc is slowly consumed while the iron or steel base is protected.

The most important galvanizing method used is the hot dip process which is adaptable to the galvanizing of nearly all types of fabricated and non-fabricated products such as wire, tanks, sheets, strip, pipes and tubes, fittings, hardware, wire cloth, hollow ware, and structural assemblies.

Hot dip galvanizing consists of these fundamental steps:
1. **Surface Preparation** -- The surface is cleaned, rinsed, pickled and rinsed to remove dirt, grease, rust and scale.

2. **Prefluxing** -- The work is immersed in a 15 to 25º Baumé solution of ZACLON® galvanizing flux. It is coated with a layer of flux to dissolve light oxides that may have formed since pickling and also to protect against any further oxidation.

3. **Galvanizing** -- Clean, oxide-free work is usually immersed through a molten layer of ZACLON galvanizing flux into the molten zinc. A thin coating of zinc is formed on the base metal.

4. **Finishing** -- Excess zinc is removed; the piece is quenched and inspected.

### GENERAL PURPOSE FLUXES

**ZAACLON® F:**

- Is the basic galvanizing flux used in the industry.
- Used as a preflux, it assures uniform zinc coverage and continuous activation of kettle top flux through carry over of preflux film on the work.
- Improves prefluxing by faster activation of base metal and better dispersion of contaminants such as oils or lubricants which can nullify preflux action.
- Improves the drainage of preflux solution and promotes faster drying, increased productivity, and reduced flux consumption.
- Can be added directly to kettle top flux.
- Produces a top flux foam blanket which reduces fuming and zinc spatter; prolongs flux life and lowers energy requirements.
- Is also available as ZACLON® F-2, a fine grade which may be used when faster dissolution in preflux is desirable; F-2 may cause excessive fuming if added directly to kettle top flux.

**ZAACLON® K:**

- Is essentially the same composition as ZACLON® F except it does not contain a foaming agent.
- Has essentially the same prefluxing performance as ZACLON® F.
- Produces a thin fluid top flux cover when added directly to the kettle.
- Is also available as ZACLON® K-2, a fine grade which may be used when faster dissolution in preflux is desirable; K-2 may cause excessive fuming if added directly to kettle top flux.

### TOP FLUX MAINTENANCE

**ZAACLON® 2N:**

- Can be used alone as top flux or for maintenance additions to another ZACLON® flux to improve fluxing activity.
- Produces a foam blanket which reduces fuming, zinc spatter and prolongs flux life.
- Is available in rod form which melts slowly in the top flux blanket to significantly decrease fume evolution and minimize consumption.
ZACLON® 2N (Non-Foaming):

- Has the same composition as ZACLON® 2N except it does not contain a foaming agent.
- Gives the same performance as ZACLON® 2N except that a thin fluid top flux cover is produced.
- Replaces ammonium chloride for producing top fluxes and effectively eliminates costly zinc losses from the kettle.

CONTINUOUS SHEET GALVANIZING FLUXES

ZACLON® W SOLUTION:

- Is specifically formulated as an aqueous solution for use as a preflux in continuous sheet galvanizing.
- Solution form allows bulk storage and handling with automated additions possible.
- Contains a superior wetting system which levels out the coverage of preflux solution, improved drainage as sheet exits preflux bath resulting in faster drying, increased productivity and reduced flux consumption.
- Protects steel against oxidation at the higher preheat temperatures which are required in this process.

ZACLON® CS:

- A fine grade granular flux specially formulated for use in an aqueous preflux in continuous sheet galvanizing.
- Improves prefluxing by faster activation of sheet and better dispersion of contaminants such as oils or lubricants which can nullify preflux action.
- Contains a wetting agent to assist with preflux drainage promoting faster drying and reduced flux consumption.

MINIMAL FUMING FLUXES

The following minimal fuming fluxes have low activity levels and generally require careful practice with regard to surface preparation to assure acceptable galvanizing quality.

ZACLON® AF:

- Can be used as an aqueous preflux or added directly to the kettle top flux.
- Produces a top flux foam blanket which can further reduce the amount of fumes generated.

ZACLON® A:

- Is essentially the same composition as ZACLON® AF except it does not contain a foaming agent.
• Gives essentially the same performance as ZACLON® AF except that thin fluid top flux cover is produced.

**ZACLON® AB:**

• Can be used as an aqueous preflux, molten salt bath preflux, or top flux.
• Provides a thin fluid top flux cover.
• Contains zinc chloride and potassium chloride, but no zinc ammonium chloride.

**SPECIAL PURPOSE FLUXES**

**ZACLON® HV -- Start Up Flux:**

• Is specially formulated to permit building an active zinc ammonium chloride top flux without generating excessive smoke.
• Produces a foam blanket which effectively suppresses fuming while a new top flux is being formed.
• Other foaming fluxes with more activity can be used for maintenance of the top flux after the blanket is first formed with HV.

**ZACLON® C - Compounding Flux:**

• Is generally used in the formulation of other fluxes for special tinning and soldering applications.
• May be used as an aqueous preflux when work must be preheated before galvanizing.

**PERSONAL SAFETY AND FIRST AID**

**HEALTH HAZARDS**

ZACLON® A, AF, AB, HV and W Solution galvanizing fluxes are acidic materials and can cause severe skin or eye injury. The principal hazard is to the eye since even brief contact with the undiluted product may produce permanent damage. ZACLON® C, CS, F, K and 2N galvanizing fluxes cause skin and eye burns.

When ZACLON® fluxes are heated to high temperatures, irritating zinc chloride fumes and gaseous hydrogen chloride may be released. Severe exposures may cause pulmonary edema. Heating may also release zinc oxide fumes which may cause metal fume fever.

The U.S. Department of Labor (OSHA) has rules that an employee's exposure to zinc chloride fumes in any 8-hour shift of a 40-hour work week shall not exceed the 8-hour time-weighed average of 1 mg/m\(^3\). (29 CFR 1910.1000, Air Contaminants.)* The limit for zinc oxide is 5 mg/m\(^3\) and for hydrogen chloride, it is 5 ppm or 7mg/kg (ceiling).

* Due to changing governmental regulations, such as those of the Department of Transportation, Department of Labor and U.S. Environmental Protection Agency, references herein to governmental requirements may be superseded. You should consult and follow the current governmental regulations, such as Hazard Classification, Labeling, Worker Exposure Limitations, and Waste Disposal Procedures for the up-to-date requirements for the products described in this literature.
SAFETY PRECAUTIONS
All persons handling ZACLON® fluxes should avoid contact with the solid materials or solutions. Do not get in eyes, on skin or on clothing. Avoid breathing dusts, mists or fumes. Wash thoroughly after handling. Contaminated clothing should be washed before reuse. Adequate ventilation should be provided.

FIRST AID
In case of contact, immediately flush eyes or skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Call a physician. Wash contaminated clothing before reusing and discard shoes.

If dust, mists or fumes are inhaled, remove person to fresh air immediately and call a physician.

If ingested, administer large quantities of water or milk. Do not induce vomiting. Never give anything by mouth to an unconscious person.

PERSONAL PROTECTIVE EQUIPMENT
Wear appropriate equipment to prevent any possibility of eye contact or reasonable probability of skin contact, such as: chemical splash goggles, rubber gloves, boots and apron, long sleeve shirt and pants. If considerable contact is likely, wear impervious (rubber) clothing or acid suit. Neoprene or PVC are recommended for the rubber items and accessories. If air concentrations exceed exposure limits, use OSHA permissible respiratory equipment.

SITE FACILITIES
The following safety facilities should be readily accessible in all areas where ZACLON® galvanizing fluxes are handled or stored.

- Safety showers -- or water hoses connected to spigots with quick-opening valves which stay open.
- Eye wash fountains -- or other means of washing the eyes with a gentle flow of filtered, tepid tap water.

STORAGE AND HANDLING
All solid grades of ZACLON® galvanizing flux should be stored so as to avoid moisture pickup. The grades containing higher amounts of zinc chloride, such as ZACLON® A, AF, AB and HV, should be stored in tightly closed containers in a dry place. An inventory turnover rate of 2-3 months is recommended to minimize caking.

Unheated solutions of ZACLON® flux slowly corrode steel. Rubber-lined steel tanks or fiber glass reinforced polyester tanks are recommended for storage.

SHIPPING CONTAINERS
Zaclon, Inc. ships ZACLON® C, CS, F, F-2, K, K-2 and 2N grades in 50 lb (net) paper bags; ZACLON® F and K are also available in 400-lb (net) fiber drums. ZACLON® A, AF, AB are shipped in 525-lb steel drums and HV in 200 lb. steel drums. Bulk solution shipments of certain grades are available. ZACLON® W solution is shipped in tank cars, tank trucks, 30 gallon and 55 gallon drums.

The information set forth herein is furnished free of charge and is based on technical data that Zaclon, Inc. believes to be reliable. It is intended for use by persons having technical skill and at their own discretion and risk. Since conditions of use are outside our
Due to changing governmental regulations, such as those of the Department of Transportation, Department of Labor, U.S. Environmental Protection Agency and the Food and Drug Administration, references herein to governmental regulations may be superseded. You should consult and follow the current governmental regulations, such as Hazardous Classification, Labeling, Food Use Clearances, Worker Exposure Limitations and Waste Disposal Procedures for the up-to-date requirements for the products described in this literature.

ZACLON, INCORPORATED

For placing orders or requesting additional product information, please use our convenient toll-free telephone number: (800) 356-7327.
From outside United States: (216) 271-1715

If you prefer, you can write to us.

Zaclon, Incorporated
2981 Independence Road
Cleveland, Ohio 44115

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ZACLON® SULFATE CONTROL FLUX

DESCRIPTION:

ZACLON® Sulfate Control Flux is a product designed for use in operations with Sulfuric Acid pickles where the flux bath is filtered. It contains components which will precipitate sulfate carried over to the flux bath, allowing the removal of some or all of the carry over by the filter.

BENEFITS:

Used properly, ZACLON® Sulfate Control Flux will reduce the need for full scale purification of the flux bath. In some cases, it may eliminate this time-consuming practice, especially if this product is combined with improved rinsing of pickled work.

BACKGROUND:

Sulfuric Acid pickling is common in the galvanizing industry. The process uses heated Sulfuric Acid to remove heavy oxide layers from steel; the steel is then rinsed in many shops. Depending on the effectiveness of the rinse, there are either large or small levels of Sulfuric Acid Salts, known as sulfates, left on the surface of the steel. These salts then build up in the flux tank, eventually leading to black spotting on the galvanized work. The level of sulfates in the flux which cause these problems varies with the zinc alloy used, the type of work, and other factors. In general, then, it is good practice to keep the sulfate levels as low as possible, preferably below 0.5% for most job shop galvanizers.

ZACLON® Sulfate Control Flux is a product with the same fluxing activity as ZACLON® K or F, but with additives to help remove some or all of the sulfates carried over into the flux tank. It also contains components which improve the drying of the flux layer and provide a small amount of foam on the flux tank, slowing evaporation of water from the tank and providing a small amount of insulation.

PRODUCT USE:

This product can be used in place of either ZACLON® F or K in a preflux, and operates at the same concentrations as already used.

It is strongly suggested that this product be used in operations which filter the flux tank. This allows the continuous removal of sulfates by the Sulfate Control as they are carried in, rather than the old method of periodic treating and settling. A 5 micron filter is best, though a 10 micron should work also; filter presses are the most common type
used, though bag or cartridge filters will also work.

Sulfate Control works best when it is regularly added to the flux tank over time; it is not designed for irregular or infrequent flux additions to the flux tank. Adding a few pound of flux every shift or every day will give the maximum control of sulfates possible.
ZACLON® W SOLUTION FLUX

ZACLON® W is a high purity solution flux used by large and small continuous sheet galvanizing lines all over the world. This 2.5 weight ratio zinc ammonium chloride flux provides the fast releases needed for high speed operations, while providing all of the necessary oxide removal and oxidation protection desired from a preflux.

The typical “cold line” hot dip galvanizing operation consists of several surface preparation segments, including hot alkaline degreasing, water rinse, one or two hydrochloric acid pickling stages, and then more rinsing. A hot preflux step is the last chemical step before the kettle, and this flux layer is often dried in some sort of furnace before entering the zinc alloy. The temperature of the zinc or zinc alloy is between 840º F and 860º F.

ZACLON® W flux is provided in solution form at about 43º Baumé. (There is also a dry crystal form, ZACLON® WD, available for some locations.) In use, the typical concentrations range from 12º to 20º Be, with heavier gauges of steel, or incompletely cleaned steel, requiring the higher concentrations for the highest quality zinc alloy coating.

Normally, the ZACLON® W preflux is heated to about 180º F in the preflux tank. ZACLON® W contains proprietary additives which give a thin, even layer of flux on the steel sheet, making the drying of the flux layer easier; these additives also minimize the foam in the preflux tank. The flux layer is then dried in a furnace; ZACLON® W can tolerate steel surface temperatures up to 400º F readily, and in some cases temperatures up to 500º F are possible for short times. Due to the excellent release characteristics of this flux, line speeds of 400 feet per minute or more are achievable with ZACLON® W preflux.

Zaclon, Incorporated is the world's largest producer of zinc ammonium chloride fluxes for galvanizing, with products for after-fabrication, sheet, wire, pipe, and tube galvanizing. Custom manufacture of fluxes for various applications is also available.

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ZACLON® Wetting Agent

ZACLON® Wetting Agent A is available from Zaclon, Inc. to suit most galvanizing operations.

ZACLON® WETTING AGENT A

ZACLON® Wetting Agent A is especially prepared for use in acidic surface preparation baths such as acid rinse water and preflux solutions. The optimum use concentration range is 0.01 to 0.03% by volume (0.1 gallon to 0.3 gallon Wetting Agent A per 1000 gallons [0.1L to 0.3L Wetting Agent A per 1000L] preflux or rinse solution). To assure better mixing of the viscous wetting agent, it should be dissolved in ten parts of hot water before adding to large galvanizing plant tanks. Excessive agitation will cause excess foam.

Used in the preflux, Wetting Agent A will assure complete coverage of the work immersed in the bath with a preflux film and better drainage when it is withdrawn. Complete coverage will help improve zinc coating quality while better drainage will reduce preflux consumption and, by assisting drying, reduce spattering at the kettle. A thin foam blanket on the preflux bath will insulate the bath to reduce heating costs.

Used in the acid rinse water Wetting Agent A will improve rinsing and reduce dragout of contaminants such as acid, sulfates and iron into your preflux solution.

ZACLON® Wetting Agent A is not recommended for use in hydrochloric or sulfuric acid pickling baths. Pickling and stripping action cause evolution of hydrogen gas which can be trapped in the foam bubbles of the Wetting Agent A making the foam flammable.

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PERSONAL SAFETY AND FIRST AID

HEALTH HAZARDS
ZACLON® Wetting Agent A causes irritation of the eyes, skin, nose, and throat. Overexposure to these products may cause liver and anaesthetic effects and may cause dizziness, headache, and nausea.

SAFETY PRECAUTIONS
Avoid contact with the eyes and skin. Avoid breathing mists or vapors.
FIRST AID
In case of contact, immediately flush eyes with plenty of water for at least 15 minutes. Call a physician. For skin, flush with water.

If inhaled, remove to fresh air. If not breathing, give artificial respiration, preferably mouth-to-mouth. If breathing is difficult, give oxygen, and call a physician.

If swallowed, do not induce vomiting. Immediately give two glasses of water, or activated charcoal slurry. Never give anything by mouth to an unconscious person. Call a physician.

INSTRUCTIONS TO PHYSICIANS
To prepare activated charcoal slurry, suspend 50 g activated charcoal in 400 ml water in plastic bottle and shake well. Administer 5 ml/kg, or 350 ml for an average adult.
ZACLON WRP®

ZACLON WRP® is a non-chromate white rust preventative for galvanized steel. ZACLON WRP® is an environmentally acceptable alternative to chromate treatment.

When ZACLON WRP® is applied under controlled conditions, it forms a clear reaction coating with the zinc coated substrate which prevents white rust formation on galvanized steel. Zaclon Inc. guarantees that the ZACLON WRP® will passivate the galvanized steel to prevent white rust formation, when used as prescribed.

ZACLON WRP® must be applied to the newly galvanized steel while the steel is at high temperature to obtain a conversion coating between the zinc surface and the ZACLON WRP®. After the galvanized steel is removed from the zinc kettle, it must be quenched in a ZACLON WRP® bath while the galvanized steel is still hot. The optimum temperature range of the galvanized steel going into the ZACLON WRP® quench tank is 325ºF to 450ºF. The conversion coating of the ZACLON WRP® to the zinc surface is complete within one minute. Applying ZACLON WRP® to zinc coated steel above the 450ºF degree temperature will give white rust protection but can yield a streaky and opaque appearance on the galvanized steel. The higher temperatures will also precipitate some of the ZACLON WRP® in the bath, thus the consumption rate will increase as well as the amount of precipitate. Application at steel temperatures below 300ºF will give insufficient protection of the galvanized steel and is not recommended. Zaclon also recommends maintaining the quench bath temperature. Bath temperatures from 120ºF to 180ºF offer the most consistent appearance and performance; exceeding 180ºF may degrade performance of the product.

The recommended ZACLON WRP® concentration range is between 2% and 4% by weight in water. As an example for a 4000 gallon tank, you would add 1000 pounds of ZACLON WRP® to obtain a 3% by weight. (8.34 lbs./gallon water times 4000 gallons times .03 equals 1000 pounds of ZACLON WRP®). In those instances where it is difficult to control the temperature below 450ºF, we recommend the lower range of concentration and more frequent analysis of the bath to ensure that the concentration does not get below the 1.5% level. The ideal concentration would have to be developed for that particular temperature and operating condition.

As with all controlled conditions and ranges, working in the middle of the ranges presented above will give the best results.

A colorimetric test kit and instructions are available that will analyze the % ZACLON WRP® in the bath. Approximately 20 minutes are needed to do the analysis including the sample preparation time. The frequency of analysis will be determined by the tonnage and use rate of the ZACLON WRP®.

We recommend a filtration system with a 30 micron or less filter to maintain a clean application of the ZACLON WRP® on the galvanized steel. Experience from the field has shown that it is best to keep the filter system recirculating or wet even when ZACLON WRP® is not being applied. Although the precipitate will be minimal, ZACLON WRP® can plug the pores of the filter if allowed to dry on the filter.

ZACLON WRP® is covered by one or more patents, including a process use patent. The purchase of the product
from Zaclon authorizes the consumer to its use under such patents.

Under the ZACLON WRP® guarantee, Zaclon will refund the purchase price of our product should any properly treated galvanized steel exhibit white rust.

**PERSONAL SAFETY AND FIRST AID**

**HEALTH HAZARDS**
ZACLON WRP® is an alkaline material that may irritate the skin and cause eye injury.

**SAFETY PRECAUTIONS**
Avoid contact of aqueous solutions of ZACLON WRP® with eyes, skin and clothing. Wear chemical goggles and rubber gloves when handling ZACLON WRP®.

**FIRST AID**
In case of eye contact, immediately flush eyes with plenty of water for at least 15 minutes, and call a physician. In case of skin contact, flush skin with water. Remove contaminated clothing and wash before reuse.

**STORAGE AND HANDLING**

The freezing point of ZACLON WRP® is very close to that of water. If the solution is exposed to temperatures below 32°F (°C), move the container to warm storage until thawed and mixed thoroughly before using.

All spills of ZACLON WRP® solutions should be washed away immediately to a chemical sewer with large volumes of water to avoid slippery footing.

**DISPOSAL**

After neutralization with acid, the ZACLON WRP® solution can be disposed of as you would any non-RCRA plant waste. Meeting Federal, State and local compliance regulations and approval, solutions may be flushed to sewer to waste treatment area.

**SHIPPING CONTAINERS**

ZACLON WRP® is shipped in 55 gallon non-returnable plastic drums containing a net weight of 550 pounds (249 kg).

ZACLON WRP® is not regulated as a hazardous material by the Department of Transportation.

**ADDITIONAL INFORMATION AND REFERENCES**