

1994

## The corrosive effects of salt spray on electrolytic zinc plated mechanical fasteners: The case of clear chromate and yellow dichromate coatings on medium carbon plated bolts

Souleymane Boukari  
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**The corrosive effects of salt spray on electrolytic zinc-plated  
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dichromate coatings on medium carbon-plated bolts**

**Boukari, Souleymane, D.I.T.**

**University of Northern Iowa, 1994**

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THE CORROSIVE EFFECTS OF SALT SPRAY ON  
ELECTROLYTIC ZINC PLATED MECHANICAL FASTENERS:  
THE CASE OF CLEAR CHROMATE AND YELLOW  
DICHROMATE COATINGS ON MEDIUM CARBON  
PLATED BOLTS

A Dissertation

Submitted

In Partial Fulfillment

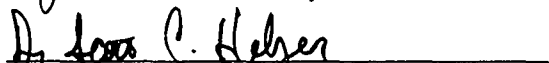
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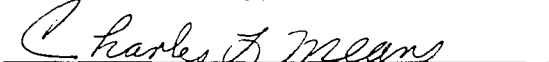
  
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May 1994

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## ACKNOWLEDGEMENTS

The writer wishes to praise God Almighty for providing love, light and guidance necessary for the completion of this work.

The doctoral degree means that the paramount in the mountains of academic fields is attained. However, it is important to mention certain individuals without whose contributions, the achievement of such an honor would not have been possible. It is the duty of the writer to recognize the efforts of the individuals who contributed directly or by any means to the completion of this degree as an appreciation of their generous and supportive assistance.

The writer is giving special thanks to the members of his doctoral committee: Dr. Mohammed F. Fahmy, Dr. John T. Fecik, Dr. Scott C. Helzer, Dr. Andrew F. Thompson, Dr. Radhi Al-Mabuk, and Dr. Charles Means.

His appreciation and gratitude also to the following persons for their help and moral support: Mr. Dan McIlhon, Mr. Edward McIlhon, and Joseph McIlhon all of whom represent their firms. A special thanks go to Doug Hundley, Jeff Jensen, and Mike Fisher for providing their friendly assistance. Thanks also are extended to Mrs. Lori Wurtz. The researcher also thanks Mr. Randy Aschbrenner for helping with the pictures. Special recognition goes to Susan Seelhammer who helped type the entire work. Special thanks



also goes to the Industrial Technology Department at UNI for providing their support.

My warm regards and thanks also are awarded to Tim and Jay of the midwestern plating company. Their support has been very instrumental for this study. My thanks also go to Mr. Phillip Martin for helping me with the electronic balance.

Finally, I would like to express my very special gratitude to my wife, Haoua, my daughters Zenabou, Myriam, and Nadia, and my son Adam, for their endless warmth and love and most of all for their patience and support throughout this study.

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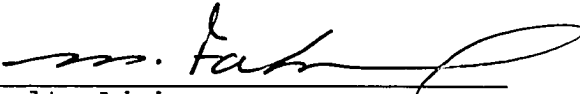
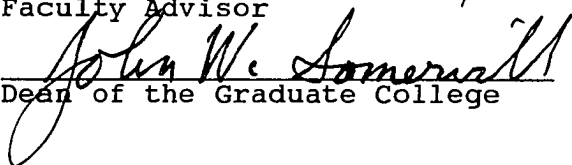
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THE CORROSIVE EFFECTS OF SALT SPRAY  
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AND YELLOW DICHROMATE COATINGS ON MEDIUM  
CARBON PLATED BOLTS

An Abstract of a Dissertation  
Submitted  
In Partial Fulfillment  
of the Requirements for the Degree  
Doctor of Industrial Technology

Approved:

  
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## ABSTRACT

The purpose of this study was to evaluate the relationship between plating thickness and corrosion resistance of zinc coated medium carbon steel bolts under salt spray moisture conditions. Two types of zinc coatings were chosen for the study: the clear chromate zinc and the yellow dichromate zinc. One hundred and eighty 3/8-16 x 5 3/4 inch medium carbon steel bolts were selected for the test and divided into groups of 18. Two types of salt solution were used in the study: the low salt and the high salt. Bolts with various coatings tested with low salt concentration were composed of groups of 18 with different coating conditions. Bolts with various coatings and tested with high salt concentration solution were composed of groups of 18 each.

An electronic balance was used to take the weight before and after for each sample tested. An electronic thickness tester was also used to measure the thickness before and after for each sample. The hypothesis was supported when P-value of the overall test was less than the set alpha value.

The percentage weight loss, thickness loss, and the analysis of variance or ANOVA were the statistics used to interpret the results of the test.

The conclusions drawn from these tests were:

1. Medium carbon steel bolts plated electrolytically with yellow dichromate zinc were more corrosion resistant than the ones plated with clear chromate zinc.
2. The yellow dichromate zinc appeared to be more protective than the clear chromate zinc.
3. Bolt coatings should be at least 0.0002" thick to guarantee a minimum protection on medium carbon steel bolts.
4. Yellow dichromate zinc was chosen to be the best coating on medium carbon bolts.
5. The clear chromate zinc does not provide a good protection for medium carbon bolts beyond 60 hours in the fog chamber of the salt spray apparatus.

Based on the findings of this study, the following suggestions were made:

1. Manufacturers should be aware of some geographical locations with high moisture content and high pollution in the atmosphere and provide thicker coating for fasteners used in their products.
2. Manufacturers should try to coat bolts with yellow dichromate zinc.
3. Manufactured products going to the ultra-dry desert climate of Nevada and Arizona could use fasteners coated with clear chromate zinc provided they are at least 0.0003" thick.

## CHAPTER 1

## INTRODUCTION

Metals play a very important role in the manufacturing of fasteners. Steel is the most widely used metal in making fasteners. According to Brandt (1992), steel is a material composed primarily of iron. He noted that most steel contains more than 90 percent iron. Steel is classified as either carbon steel or alloy steel (Pollack, 1988).

Carbon steel comprises by far the largest tonnage of all steels. It contains less alloys and less expensive ingredients. Carbon steel according to (Pollack, 1988) can be broken down into low carbon, medium carbon, and high carbon steel. It is noted in the Industrial Fasteners Institute Book that most fasteners are manufactured using carbon steel. This is due to the fact that steel has excellent workability; it offers the broadest range of attainable combinations of strength properties, and most of all, in comparison with other commonly used fastener materials, it is inexpensive. The type of steel used in this research was medium carbon steel.

Medium carbon steels are heat treatable, which means that through metallurgical treatments the tensile strength of the fastener after processing can be significantly greater than that of its original raw material. Since products made out of steel need to be protected from

corrosion, medium-carbon-steel bolts coated by electroplated zinc were chosen for this study.

According to Zaki (1988), zinc is more corrosive resistant than steel is. Hence, it is used extensively as a type of coating to protect steel components from premature failure. Groshart (1986) stated that 40% of all zinc used in the United States of America is for coating purposes. Zinc usage far exceeds any other metallic coating. Groshart also noted that zinc is anodic to steel in the atmosphere and in both salt water and fresh water. He also stated that zinc can be applied as a bright, decorative coating resembling chromium, but when exposed to relative humidities, the unprotected zinc surface forms oxides, hydroxides, and carbonates which will cause it to become dull. He added that zinc can also be colored not only by standard chromate conversion coatings (used for the corrosive protection of the zinc) that provide golden, olive drab, and black colors. Also, by adding commercial dyes to the conversion coatings, blue, green, and red colors can be provided.

This form of coating can be achieved through a method called electroplating. According to Moniz (1992, p. 435) electroplating (electrodeposition) is a process for depositing (plating) a thin layer of metal (zinc) on the surface of another metal such as steel by immersing the

fasteners in a plating bath and running current through them. Zinc travels through the electrolytic bath from the zinc anodes and attaches to the surface of the fasteners being plated. Electroplating is used to improve corrosion or wear resistance, increase surface hardness and provide an attractive appearance.

The corrosion protection of zinc plating is measured by a standardized ASTM test method called salt spray testing. Salt spray corrosion testing is done in an environmentally controlled chamber at 95 degrees Fahrenheit with continuous exposure to a 5% sodium chloride solution by means of an atomized fog of salt solution within the chamber. For chromated surfaces, the time in the chamber necessary to cause the chromate coating to fail and allow zinc corrosion (white and red corrosion products) is measured. In accordance to ASTM specifications, a clear chromate must pass 12 hours, and a yellow/bronze chromate must pass 96 hours without the appearance of white corrosion product. Accelerative corrosion in the laboratory equates a 24-hour exposure in the fog chamber to a one-year natural exposure. Since climates vary from one region to another, these estimations were adopted by the National Association of Corrosion Engineers (Atkinson & Van Droffelaar, 1982).

In addition to the protection of the chromate, the zinc plating will protect about 18 hours per .0001" thickness.

Salt spray test results provide a method of comparison testing of coatings in a controlled environment, but does not directly predict corrosion protection in real world conditions or under different conditions.

As the fight against corrosion is taking place in many industries in different forms, zinc electroplating appears to be the most prevalent as far as fasteners are concerned.

According to Groshart (1986), when stress corrosion occurs in a metal, it can fail at a load level far below its load carrying capability. Most often the corrosion is not visible or even suspected until the failure occurs. Looking at corrosion in fasteners, Bickford (1988) stated that corroded parts of a joint can cause serious damage to expensive equipment. Moreover, Raymond (1992) stated that corrosion associated with fracture of a stressed component usually involves an incubation period during which a pit develops and leads to the formation of a crack and a period during which a crack grows to a critical size. This failure mechanism, defined as stress corrosion cracking (SCC) is known to be costly in dollar terms to industries worldwide. It appears that this problem can be alleviated if fasteners get better coatings.

This study was focused on the corrosive effects of salt spray test on zinc plated mechanical fasteners. A case of clear zinc chromate and the yellow dichromate zinc were investigated. It is generally expected that thicker

coatings, especially the yellow dichromate zinc coating, would provide the bolts with a better corrosion resistance. Such findings regarding the use of mechanical fasteners in industries as well as in households would contribute to some existing technical literature. As stated by Brandt (1992), carbon steel fasteners constitute 90% of all fasteners used in industries. Results of this research may contribute to equipment failure prevention and better choice of coating for these types of fasteners.

#### Statement of the Problem

Corrosion of steel fasteners under industrial use such as conditions where parts vibrate, through stress and strain can lead to equipment, structural, and machine failure due to stress corrosion; this can be costly and even cause injuries. The problem of this study consisted of two parts:

1. To investigate the corrosive effects of salt spray on two types of zinc coated medium carbon steel bolts.
2. To determine the corrosive effects of yellow dichromate zinc coated medium carbon steel bolts under salt spray moisture conditions and their resistance to red corrosion products.

#### Purpose of the Study

The main purpose of this investigation was to evaluate the relationship between plating thickness and corrosion resistance of zinc coated medium carbon steel bolts under salt spray moisture conditions.

### Statement of Need

In today's modern industries, more than 200 billion fasteners are produced each year (Fasteners, 1984). These fasteners are used in automotive industries, aircraft, aerospace, non-electric machinery, road and farm equipment, construction, electrical equipment, appliances, computers, office equipment, telecommunications, etc. All together, these industries represent more than 30% of the consumption in terms of both dollars and percentage volume in the United States (Fasteners). Additionally, Atkinson and Van Droffelaar (1982) noted that corroded parts, especially fasteners, are known to cause disasters, thereby costing companies not only human lives but also millions of dollars. As an example, Atkinson and Van Droffelaar stated that a corroded part caused a blast furnace to blow up in Europe. Corrosion is like a cancer on metals; it is not predictable. Uhlig and Revie (1985) defined it as "the destructive attack of a metal by chemicals or electrochemical reaction with its environment" (p. 12). They stated that a better protection of metals, especially fasteners, could bring about improvements in three areas:

1. The first area of improvement would be economic and reflected in direct and indirect losses. The direct losses would be the costs of replacing damaged equipment due to the failure of a corroded bolt. The long wait for an ordered



part costs companies money indirectly. Uhlig and Revie (1985) estimated the total costs of combined losses at \$70 billion per year or 4.2% of the gross national product each year in the United States. Donovan (1986), in his assessment of the costs associated with direct losses to corrosion, asserted that approximately 3 to 4% of the GNP is spent to solve this problem.

2. Better coatings on fasteners can improve the safety of operating equipment. This would provide better attachment on industrial machinery, airplanes, tractors, and automobiles (Uhlig & Revie, 1985).

3. Coated fasteners can also preserve the longevity of equipment. A corroded bolt can loosen the attachment or part and cause vibration on equipment, thereby causing a breakdown.

#### Research Hypothesis

The research hypothesis for this study was as follows: The null hypothesis ( $H_0$ ) which stated that there is no correlation between plating thickness and corrosion resistance of medium carbon steel bolts.

The alternative hypothesis ( $H_a$ ) which stated that there is a correlation between plating thickness and corrosion resistance of medium carbon steel bolts.

Since most specific inquiries were necessary beyond the significance of the hypothesis test, the following research questions for this study were investigated:

1. Is coating correlated to corrosion resistance of fasteners?
2. Is clear chromate coating more protective than the yellow dichromate coating on fasteners?
3. In a salt spray test, does higher concentration of salt cause more corrosion on parts?
4. What is the effect of coating thickness on the corrosion resistance of zinc coated medium carbon steel bolts?
5. What is the appropriate coating thickness for medium carbon steel bolts?

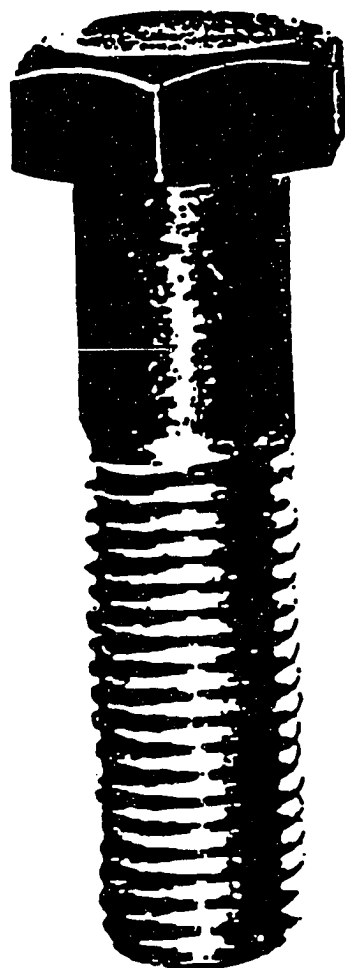
After these research questions, the researcher looked at the assumptions under which this study would be done.

#### Assumptions

The assumptions for this investigation were as follows:

1. If left unprotected, fasteners made of medium carbon steel would corrode and cause equipment failure.
2. The degree of humidity in the fog chamber of a salt spray test is similar to that of most coastal regions of the United States of America.
3. All the bolts used in this study had similar composition. (See Figure 1 for a hex head cap partially threaded bolt.)
4. Coatings are uniform on fasteners.

Figure 1. A hex head cap partially threaded bolt.  
(Courtesy of Bossard International, Inc.)



### Limitations

As is the case with all studies, this investigation was limited by the following factors:

1. Only medium carbon steel 3/8-16 x 5 3/4 bolts were used in this research.
2. The bolts used in this research were plated electrolytically using the inorganic coating (Nucor Fasteners, 1992).
3. A salt spray (FOG) testing apparatus was used in this investigation. It is composed of a fog chamber, a salt solution reservoir, a supply of suitably conditioned compressed air, four atomizing nozzles, specimen supports, provision for heating the chamber and necessary means of control of the apparatus as specified by the (ASTM, B117).
4. This study was limited to two types of coatings: the clear chromate zinc and the yellow dichromate zinc (Lake Erie Manufacturing, 1991).
5. An electric scale was also used to weigh each sample. Its precision level is within thousands of a metric grams.
6. A digital coating thickness tester was used to record the plating thickness of the bolt to be used in this study.

### Definition of Terms

The following terms were used frequently throughout this dissertation.

**Anodic Coating:** Conversion coating applied by the anodic oxidation of certain base metals (Moniz, 1992).

**Anodizing:** To subject a metal to electrolytic action as the anode of a cell in order to coat it with a protective or decorative film; used for non ferrous metals (Neely, 1989).

**Bolt:** A headed and externally threaded mechanical device designed for insertion through holes in assembled parts to mate with a nut and is normally intended to be tightened or released by turning that nut (IFI, 1988).

**Carbon Steel:** A carbon steel is a steel which does not contain any substantial amounts of alloying materials other than carbon (IFI, 1988).

**Chromate:** A salt or ester of chromic acid (Neufeldt & Guralnik, 1989).

**Chromium:** A metallic element with 24 atoms and 51.966 mass, not occurring freely in nature, but produced from chromate. It's special properties, e.g. hardness, resistance to corrosion and wear, are used in alloys with steel and nickel, and in electroplating (Neufeldt & Guralnik, 1990).

**Coating:** The application of some material such as metal, organic compound, etc. to the surface of a fastener (IFI, 1988).

**Conversion Coatings:** Surface treatment that converts the surface layer of a metal by oxidation into a constituent

of a coating, contributing to a strong bond between the metal and the conversion coating (Neely, 1989).

**Corrosion:** The deterioration of a metal by chemical or electrochemical reaction with its environment (ASM, 1988).

**Dichromate:** A salt of dichromate acid containing the divalent negative radical  $\text{Cr}_2\text{O}_7$ , (Neufeldt & Guralnik, 1989).

**Dichromic:** Chemical having two atoms of chromium per molecule; [designating an acid  $\text{H}_2\text{Cr}_2\text{O}_7$ , that exists only in solution, from which dichromate are formed] (Neufeldt & Guralnik, 1989).

**Electrolyte:** Liquid, most often a solution, that will conduct an electrical current (Moniz, 1992).

**Electroplating:** Process for depositing a thin layer of metal onto a metallic component that made the cathode in an electrical circuit and immersed in a solution containing ions of the metal to be plated (Moniz, 1992).

**Fastener:** A mechanical device designed specifically to hold, join, couple or maintain equilibrium of a single or multiple components. The resulting assembly may function dynamically or statically as a primary or secondary component of a mechanism or structure. Based on the application intended, a fastener receives varying degrees of built-in precision and engineering capability, insuring adequate, sound service under planned, pre-established environmental conditions (IFI, 1988).

**Ion:** A substance, or one of its component atoms or radicals, resulting from electrolytic decomposition or dissociation (Funk & Thomas, 1963).

**Plating:** Forming an adherent layer of metal or alloy on an object (Moniz, 1992).

**pH:** Hydrogen--Ion activity that denotes the acidity or basicity of a solution (Moniz, 1992).

**Salt Spray Testing:** Exposure of coating to various formulations of salt spray or fog, with and without cyclic immersion and exposure to highly actinic light, heat, and to various wetting and drying cycles is a common practice. There was, for example, rough correlation between the nitric acid test and salt fog exposures of ten coatings (NACE, 1984).

**Throwing Power:** The ability of an electrolyte to deposit metal of a uniform thickness across the surface of a cathode (Moniz, 1992).

**Yellow Dichromate:** Orange to red chromium salt containing the radical  $\text{Cr}_2\text{O}_7$ , (Neufeldt & Guralnik, 1989).

**Zinc:** A metallic element with 30 atoms and 65.37 mass, hard and resisting corrosion. It is used for coating metals to prevent corrosion (Neufeldt & Guralnik, 1989).

#### General Procedure

The procedure for this study consisted of the following information:

1. The review of the literature to determine and investigate the technical nature of testing, the appropriate apparatus to be used in this investigation, the accepted accreditation and standards, the recognized procedures, and the state-of-the-art information pertaining to the nature of this study.

2. The methodology projected to be pursued consisted of the following steps:

Random selection of sample steel bolts.

Selection of the types of plating and coating procedure.

Determination of the type of tests to be conducted.

Determination of the necessary testing instruments and/or equipment.

Design of this study which was done based on the pre-post experimental type of investigation.

3. The data analysis of the test results, which was composed of the following steps:

Collection of data and results.

Developing the results emanating from the collected data.

4. Finally, the last part of this procedure consisted of the following:

Reporting the conclusions resulting from the scrutiny of this investigation.

Writing recommendations for future studies.



## CHAPTER 2

### REVIEW OF LITERATURE

The literature review consists of seven parts: (a) an introduction; (b) an explanation of electrodeposition or electroplating process; (c) a description of the chromate conversion coating; (d) a definition and delineation of elements involved in corrosion formation; (e) elaboration on how instrumental electrolytes are in the formation of corrosion; (f) a description of salt-spray testing; and (g) a summary of the chapter.

#### Introduction

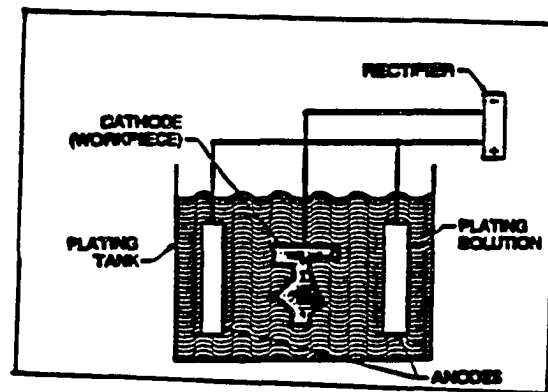
Zinc is a metal which is largely used in the form of zinc coatings on medium carbon steel and even alloy steel. Burns and Bradley (1967) wrote that 400,055 tons or 40% of the total consumption of zinc were used for metal coatings in 1963. They noted that zinc coatings are applied by several methods: hot dipping, electroplating, spraying, cementation, and vapor deposition. They added that hot dipping and electroplating are commercially predominant. It was also noted that electroplated zinc layers are relatively pure and ductile, and provides a coating of uniform thickness and, if applied by a plating technique, it provides a pleasing appearance. The coating thickness can be varied over a wide range to correspond to the degree of protection required. In their 1967 work, Burns and Bradley

explained how electroplating of iron and steel with zinc for protection against corrosion was developed. They stated that:

The electroplating of iron and steel with zinc for protection against corrosion was proposed in 1840. An alkaline zinc cyanide plating bath was patented as early as 1855, although baths of this type assumed little commercial importance until about 1916. The first patent of zinc plating granted in this country was in 1862. (p. 106)

The next section focuses on the process of electrodeposition. (See Figure 2.)

Figure 2. Plating process diagram.



### Electrodeposition or Electroplating

Fontana and Greene (1978) explained in a similar study that thin coatings of metallic and inorganic materials can provide a satisfactory barrier between metal and its environment. Describing how electroplating is performed, they wrote:

Electroplating consists of immersing a part to be coated in a solution of metal to be plated and passing a direct current between the part and another electrode. They added that the character of the deposit depends on many factors including temperature, current density, time, and composition of the bath. (p. 215)

Also, Fontana and Greene (1978) noted that electroplate can be a single metal, layers of several metals, or even an alloy composition such as brass. For example, an automotive bumper has an inner flash plate of copper (for good adhesion), an intermediate layer of nickel (for corrosion protection), and a thin top layer of chromium (primarily for appearance).

There are various ways to obtain zinc plating through electrodeposition. Traditionally, zinc plating can be carried out in cyanide zinc electrolytes because of their reliability and ease to control. In recent years, however, the environmental pollution caused by cyanide has prompted extensive efforts to find a non-cyanide electrolyte. Various low cyanide and non-cyanide solutions have been developed (Pushpananam & Shenoi, 1980).

According to Pushpananam and Shenoi (1980), there has recently been a tendency to replace cyanide solutions with weak acid or neutral zinc solutions in which high efficiency and plating rates can be combined with decorative appeal, low efficient cost, and reasonable throwing power. Pushpananam and Shenoi also conducted a research study and found out that recent development in neutral zinc

plating is the use of potassium chloride and boric acid in place of ammonium chloride. In addition, they stated:

While cyanide type electrolytes yield a cathode current efficiency of between 60 and 75%, the acid electrolytes achieve 80 to 98% efficiency. With throwing power comparable to that of the cyanide processes. (p. 69)

A similar study conducted by Atkinson and Van Droffelaar (1982) showed that cyanide electrolytes are becoming less common because of environmental considerations.

Computer searches through dissertation abstracts and through the on-line services at the library of the University of Northern Iowa did reveal information about zinc plating and chromate conversion coating. Books such as Corrosion Basics (National Association of Corrosion Engineers, 1984), Corrosion and Corrosion Control (Uhlig & Revie, 1985), Corrosion and Its Control (Atkinson & Van Droffelaar, 1982) provided information about chromate conversion coatings. In addition to these books, various midwest companies also provided the researcher with professional literature dealing with chromatic conversion, a topic to which we turn next.

#### Chromate Conversion Coating

Chromate conversion coating is also referred to as chromating. Brumer (1988) noted that chromate films are chemical conversion coatings. He explained that the layer of metal underlying a coating participates in the coating

reaction and becomes a component of the coating. He added that zinc and cadmium electroplates, zinc die castings, hot-dipped galvanized steel, aluminum (in almost all conceivable form) and sometimes, copper and silver alloys are the most commercially chromated metals. In his study of chromate conversion/coating, Brumer delineated three reasons why this final process is done on zinc plated parts:

1. Improve corrosion resistance.
2. Improve paint or adhesive bonding properties.
3. Provide a decorative or colored finish.

With respect to the chemistry involved in chromate conversion coating, Brumer noted that it involves reaction between the metal surface and an aqueous solution containing chromates and certain activator ions. According to the author, activators include: sulfate, chlorides, fluorides, phosphates and complex cyanides. Activation (pickling) is performed just before plating. The process consists of exposing the workpiece to an acid such as sulfuric, hydrochloric, nitric, hydrofluoric, or mixtures of these, which removes oxides and scale from the metal surface. After activation, the workpiece is rinsed with water and placed immediately in the plating solution. He also mentioned that a given chromate is designed to work on a particular metal, but in few cases there are solutions that will work on two or more. For example, certain chromates for zinc electroplate will also coat cadmium and may be

suitable for zinc coatings. The author noted that the solutions for chromating are acidic, therefore a simplified reaction proceeds along these lines:

1. Metal at the interface is dissolved by the acid and enters solution as metal ions.

2. There is a local rise in pH (lowering acid content) in the immediate vicinity of the interface.

3. Basic metal ions combine with chromate ions to form a compound that is insoluble at the local (higher) pH. This compound precipitates on the metal surface as an adherent coating.

4. Reaction by-products enter the main solution.

This piece of information would be very useful for this study because of its relevance to the topic. Given the above chemistry at chromate conversion coating, Zaki (1988) explained the protection mechanism of chromate conversion coating. According to Zaki, chromate conversion coatings afford protection to zinc surfaces through two mechanisms:

1. The chromium chromates complex presents a non-reactive barrier to humidity and air, thus retarding white corrosion.
2. The chromate film retains a water absorbing characteristic as long as it remains in a hydrated form. When scratched or mechanically damaged, enough water is absorbed by the film to swell and mend at the damaged areas. This is the so called "self-healing effects." (p. 7)

He also elaborated on types of chromates and their classification. He noted that chromate conversion coatings

are classified by color and in turn they are also denoted by the film thickness and chromate contents. Colors vary from clear to light blue, yellow, olive drab, and black. Zaki reported that the single-dip clear chromate films contain a lower content of chrome and offer less corrosion protection than the colored types. He conducted similar research on various parts and reported his findings which are shown in Table 1.

Table 1  
Chromate Types (Typical Values)

OPERATING RANGE	CLEAR (SINGLE DIP)	BLUE BRIGHT	CLEAR (LEACHED)	YELLOW BRONZE	OLIVE DRAB	BLACK SILVER
Cr <sup>+6</sup> in solution, g/l	2-6	0.1-0.5	30-60	1.0-5.0	10-30	10-30
pH	2.5-3.5	0.5-1.5	0.1-0.7	0.5-1.5	1.0-2.0	1.0-2.0
Temperature, °F	80-100	60-90	60-90	60-90	70-90	75-100
Salt spray resistance (to white corrosion), hrs.	3-10	20-30	70-100	100-160	100-300	12-48

Zaki (1988) showed that the formation of the chromate film starts from the metal partially being dissolved in the solution. A sufficient thickness of zinc must be available in order to produce an even, satisfactory coating. He reported that in general, an average of 0.00035

of zinc is dissolved during the chromating process. Therefore, a minimum of 0.0002" is required to produce satisfactory chromate films. Color inconsistencies in a well controlled chromating solution are often due to wide variations or insufficient zinc thickness, particularly in low current density areas.

Zaki (1988) also supported his findings by reporting three types of problems that can affect color and appearance, adhesion or corrosion resistance. With regard to color and appearance, he noted that color is directly related to film thickness and composition. The heavier the coating, the darker the deposit. For example, he reported that typical blue bright coating contains 3 to 5 mg/ft<sup>2</sup> chromium as Cr, yellow iridescent 35 to 50 mg/ft<sup>2</sup>, of which 7 to 12% is as Cr<sup>+6</sup> and olive drab around 100 mg/ft<sup>2</sup>. He added that rinsing could also affect color. He acknowledged that a good rinsing prior to chromating is critical. Residue from plating, alkaline, or acidic, as well as organic brightener, leaves the zinc surface inactive, resulting in uneven color, streaky or blotchy chromate coating. He also reported that loss of adhesion, commonly known as "slough off," may occur when the chromate film on drying would not adhere to the surface. Concerning the corrosion resistance, he noted that failure to meet corrosion resistance for the particular coating applied could be due to insufficient thickness for the required type



of chromate, contamination of the chromate film with metallic impurities. He recommended that chromate coatings be topped by a protective layer of clear lacquer to enhance overall corrosion resistance. These findings were of great importance in this study due to its relevancy of the topic. The researcher advised the midwest plating company of the shortcomings associated with the problems stated above.

Among other researchers, Mori, Miyawaki, and Matsushimor (1991) conducted a similar study in which they explained that:

The composition of zinc composite coating, which shows good heat resistance and high corrosion resistance was investigated with the use of different coating or electrogalvanized steel with various coating solutions containing different kinds of metal chloride compounds. Corrosion performance of the respective coatings was evaluated by salt spray test after heating. As a result, the coating with magnesium compound showed highest corrosion resistance after heat treatment at 300°C for 5 hours. Further, the relationship between heat resistance and solubility of compound of chromium, magnesium, and aluminum in the acid solution was investigated. It was found that the change in chemical property of composite coating by heating gave a big influence to corrosion resistance. Also it was confirmed that the driest composite coating impregnation with the solution of magnesium salt or porous zinc coating showed excellent heat and corrosion resistance.  
(p. 401/1)

Based on the above findings, a careful examination of the author's work, Mori et al. (1991) should be taken into consideration when determining what specific time and salt concentration should be used in this research.

Nelson (1963) conducted another study related to the electrodeposition of metals and coatings. In his dissertation study, Nelson addressed the process of electrodeposition and explored the correlation between the maturity of dilute zinc sulfate solutions and the activity of the zinc ion. Nelson used a method for determining the potentials of amalgam cathodes, and in his correlation of the topic with previously determined data, he indicated the experimental procedure to be acceptable. He also determined the nominal deposition potentials for a number of metals from dilute aqueous solutions using pure mercury cathodes. The values in Nelson's study were not absolute but are useful in predicting the possibilities of separation. Nelson used a constant for inclusion in his work and it was described for copper, cadmium, and zinc, allowing the metal content of the amalgam to be expressed as weight percentage. In addition, Nelson determined a constant to correlate the molarity of dilute zinc sulfate solution to the activity of the zinc ion.

Nelson's (1963) research is very helpful to investigators interested in the separation of the coating. It is a problem that needs to be dealt with in various industries as far as fasteners are concerned. Along the same idea, another investigation conducted by Cook, Griesser, and Beamish (1992) explored the various types of coatings on steel bolts. They found that the thickness and

uniformity of the coating are important because they determine bolt/nut thread fit, lifetime of the bolt/nut connection, and economical use of coating materials. Their findings were different from Nelson's due to the type of coating investigated. For example, the electroplated zinc was found to provide adequate protection for both nuts and bolts. However, if the coating is thin, bolts would not be suitable for long-term outside industrial use because they will corrode much faster.

From the above findings, the importance of and need for a good coating can be a major answer in the battle against corrosion. The next section focuses on the process of corrosion formation.

#### Corrosion

Corrosion was defined in The Industrial Fasteners Institute reference book (1988) as:

the destructive alteration of metal by chemical or electrochemical reaction within its environment which encompasses not only atmospheric exposure but all the interacting conditions associated with a service application. (p. B-30)

All mechanically-fastened joints are subject to corrosion of one type or another during their service life. This is caused by the growing aggressiveness of industrial atmospheres, longer service lives, higher operating temperatures, and increasing working stresses.

The corrosion of most fasteners, as noted by IFI, occurs by direct chemical attack. The other basic form of

corrosion is electrochemical in nature. It was noted in the IFI reference book that corrosion by direct chemical attack occurs when the base material is soluble in the corroding medium. Fasteners used in highly moisturized environments as well as in high temperature areas need to be protected against corrosion. For example, in the petroleum industry, crude oil normally contains sulfur, salt, and water. During distillation, cracking, and refining processes, all of which are performed at high temperatures, the salt hydrolyses into hydrogen chloride which, in turn, converts to hydrochloric acid when exposed to water vapor in the primarily fractionation step. Hydrochloric acid, even in weak concentrations, causes severe corrosion of most constructional materials. According to IFI (1988), unprotected carbon steel fasteners would literally disappear quickly by corrosion.

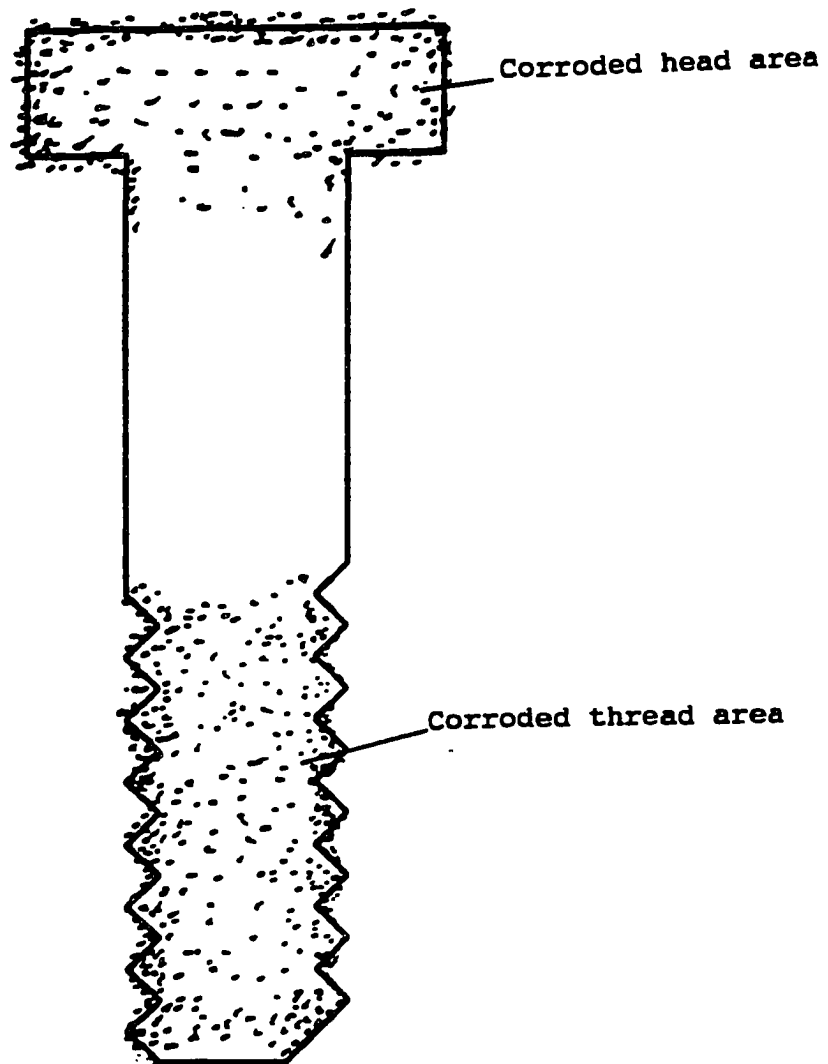
It was also noted in the IFI (1988) reference book that all metals have an electrical potential. When metals of different potentials come in contact with one another in the presence of an electrolyte, an electrical current flows from the metal of higher potential toward that of the lower and a chemical reaction occurs. Based on that phenomenon, it is apparent that electrochemical types of corrosion are practically exclusive to metals. It is also noted in the IFI reference book that the common forms of corrosion are:

galvanic corrosion, stress corrosion, fatigue corrosion, fretting, crevice corrosion, erosion corrosion, pitting, and cavitation.

It is important to know the mechanism of electrochemical corrosion for this study. As stated in the IFI (1988) reference book, the most common form of corrosion is the oxidation of steel. Oxidation is a chemical reaction. As explained by IFI, the iron content returns to its original state as iron oxide when mined as iron ore.

It was also explained by IFI (1988) that the oxidation of steel requires three constituents: an anode, a cathode, and an electrolyte. The cathode to anode electrical flow in steel occurs for several reasons, but all are generally attributable to a lack of homogeneity. The electrolyte is any surface moisture. A relative humidity about 30% and higher is enough to initiate the flow of minute electrical currents. These currents flow from particles of high electropotential (cathodes) through the steel to particles of lower potential (anodes) then through the electrolyte (the surface moisture) back to the cathode. At the point where the current leaves the anode and enters the electrolyte corrosion product forms. Corrosion products on carbon steel are coarse, flaky, and easily washed or brushed off. Corrosion continues because moisture and oxygen remain in contact with the base metal. (See Figure 3.)

Figure 3. Schematic drawing of a corroded bolt.



Further explanation noted in the IFI (1988) reference book enabled the researcher to consider the interacting influences of a medium carbon steel corroding electrochemically. However, the pH value of the water is very critical for this type of testing, and it is to further discussion of pH value that we turn next.

#### pH Value

Moniz (1992) noted that pH is the hydrogen-ion activity and denotes the acidity or basicity of a solution. It can be an important indicator of corrosiveness. In addition, Moniz stated that solutions of pH 0 to 7 are acidic, and solutions of pH 7 to 14 are alkaline. The lower the pH, the greater the amount free acid present and the more corrosive the solution. He also pointed out that the minimum pH for carbon steel applications, without excessive corrosion is 5. He added that metals such as zinc, aluminum, and lead are susceptible to corrosion in highly alkaline (high pH) solutions. A discussion of electrolytes follows.

#### Electrolytes

An electrolyte is a medium through which the electrical current flows. Electrolytes occur naturally in our everyday environments; they are: humidity, fog, dew, condensation, and rain. These electrolytes have a broad variation in their electrical conductivity. For example, the corrosion survival expectancy of steel is enhanced considerably when exposed in the high humidity of southern states such as

Florida and Texas as opposed to the ultra-dry desert climates of Nevada and Arizona. Similarly, structure and equipment survive longer in rural midwestern states than in either ocean bordering states or in heavily industrialized areas with their high impurity atmosphere.

It was also noted in the IFI (1988) reference book that distilled water is a poor conductor, tap water with its little chemical content is better. Industrial exposures with their pollutants and contaminants are even better; and salt water is exceptionally good in conducting electrical currents. Whenever the electrolyte is agitated, its conductivity is significantly magnified. The "dirtier" the electrolyte, the better its conductivity, and the faster the rate of corrosion (p. B-31).

The presence of oxygen accelerates corrosion. As most products of corrosion are oxides formed by the chemical reaction, the presence of oxygen is necessary. Without oxygen, most forms of corrosion would propagate at a much lower rate. Oxygen is the principal element of the air we breathe, and it is present, although to a lesser degree, in all areas where electrolytes are present.

Temperature is another factor that contributes to corrosion. While not a direct contributor to corrosion, temperature is ultimately a factor in the rate at which it occurs. Generally, heat accelerates chemical reactions. It is noted in the IFI reference book that corrosion happens



quicker and more remarkedly in southern climates than in those of the North. It also occurs more quickly in industrial plants where heat is inherent in the process as opposed to industry which conduct their operations at ambient temperatures or in the luxury of an air conditioned atmosphere.

#### Salt Spray Testing

Over the years, there have been many attempts to quantify corrosion losses and to assess the value of all possible remedial measures. Atkinson and Van Droffelaar (1982) noted that corrosion tests are necessarily as diverse as corrosion itself. They acknowledged that corrosion test pieces are immersed, buried, exposed to atmospheres, and exposed to known industrial or artificial environments that are judged to simulate particular conditions. They added that if mechanical factors are known to be significant, they are incorporated in the tests in a controlled fashion.

Atkinson and Van Droffelaar (1982) suggested that the best method to test corrosion is the salt spray testing. They also recommended that the most common criteria used in testing corrosion is the weight loss or gain of a sample of a known area. In this testing method they recommended that after a testing period in a fog chamber of the salt spray testing machine, the tested samples must be thoroughly cleaned to remove oxide films and grease. The National Association of Corrosion Engineers (NACE, 1984) also noted

that the proper technique that can be used to remove corrosion products and not the base metal from tested samples remains to be soap and water cleaning with a soft brush. Forshee (1991) agreed that salt spray testing is the best method for corrosion testing. (See Figures 4 and 5.) He acknowledged the limitations of this mode of testing, however, he maintained that it is the best form available for industries to simulate the very condition in which parts are exposed to pollutants and humidity. Atkinson and Van Droffelaar (1982) advised that any corrosion test be standardized. The American Society for Testing and Materials (ASTM) came out with its own standards in salt spray testing. It is referred to as the ASTM B117. This standard method sets forth the conditions required in salt spray (fog) testing for specification purposes. One method does not conform to the type of test specimen or exposure periods to be used for a specific product. The (ASTM B117) requirements for salt spray testing are listed in Appendix A.

#### Summary

From the literature reviewed no systematic or detailed studies were done on the relationship between various plating thickness and corrosion resistance on fasteners. Several publications have dealt indirectly with the area, but there appeared to be no information directed specifically towards these relationships.

Therefore, it was determined that further investigation into this area would contribute to the scientific study of a problem associated with fasteners and corrosion.

Figure 4. Salt spray apparatus.

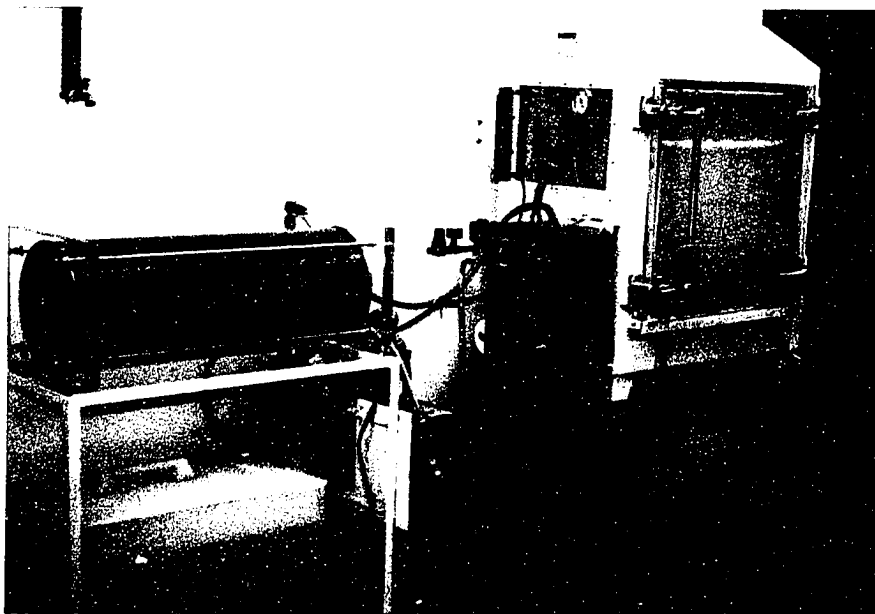
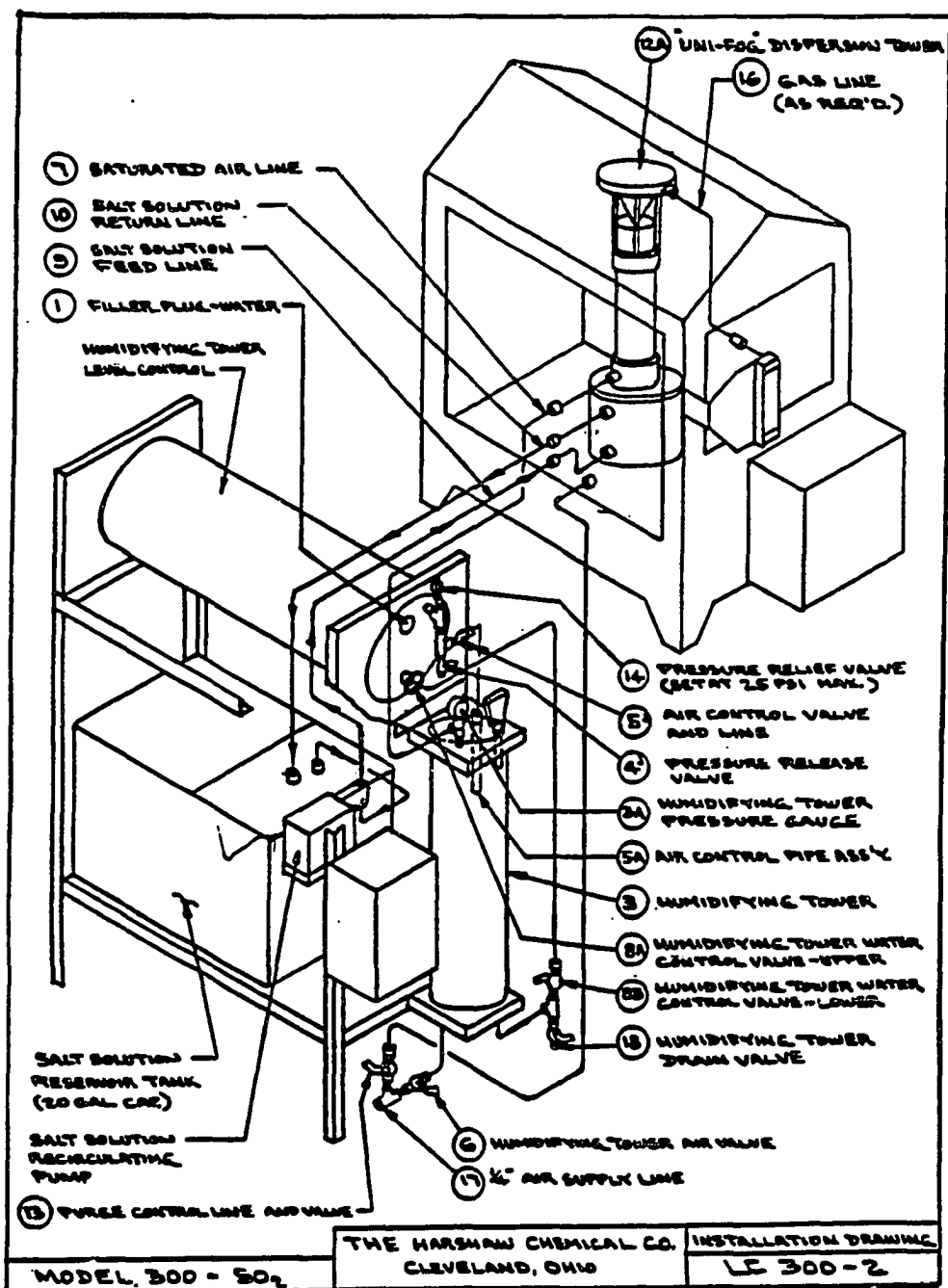


Figure 5. Schematic drawing of the salt spray apparatus.



### CHAPTER 3

#### METHODOLOGY

This chapter consists of three parts: (a) sample selection, (b) procedures to be followed and instruments to be used in this study, and (c) the statistical design of this study.

##### Selection of Samples

The samples used for this investigation consisted of 180 pieces of (3/8-16 x 5 3/4) medium carbon steel bolts randomly selected from a lot of 56,000 pieces. The rationale for this selection criteria was based on the Society of Automotive Engineers' Handbook (SAE, 1991) which specified that bolts, screws, studs, and seams should be made of steel conforming to the description and chemical composition requirements specified in Table 2 for the applicable grade. See Table 2.

According to their head marking, the samples selected are grade five bolts. It can also be said that these bolts are medium carbon steel, quenched and tempered. Each bolt contains between a minimum of 0.28% and a maximum of 0.55% of carbon. See Table 3. The phosphorus content of each bolt is about 0.048%, and that of sulfur is 0.058%. Brandt (1992) noted that "carbon provides the metal with hardness, strength and wear resistance; phosphorus also provides the metal with strength; and sulfur, although present in small amount allows the metal to be machinable" (p. 23). The

Table 2

Chemical Composition Requirements

Grade	Material and Treatment	Element, %					
		C		Mn Max	P Max	S Max	B Min
		MIN	MAX				
1	Low or medium carbon steel	—	0.55	—	0.048	0.058	—
2	Low or medium carbon steel	—	0.55	—	0.048	0.058 <sup>b</sup>	—
4	Medium carbon cold drawn steel	—	0.55	—	0.048	0.13	—
5	Medium carbon steel, quenched and tempered	0.28	0.55	—	0.048	0.058 <sup>c</sup>	—
5.1	Low or medium carbon steel, quenched and tempered	0.15	0.30	—	0.048	0.058	—
5.2	Low carbon martensite steel, fully killed, fine grain, quenched and tempered	0.15	0.25	0.74	0.048	0.058	0.0005
7	Medium carbon alloy steel, quenched and tempered <sup>d</sup>	0.28	0.55	—	0.040	0.045	—
8	Medium carbon alloy steel, quenched and tempered <sup>d</sup>	0.28	0.55	—	0.040	0.045	—
8.1	Elevated temperature drawn steel—medium carbon alloy or SAE 1541 (or 1541E steel)	0.28	0.55	—	0.048	0.058	—
8.2	Low carbon martensite steel, fully killed, fine grain, quenched and tempered <sup>d</sup>	0.15	0.25	0.74	0.048	0.058	0.0005

Note. (Courtesy of Industrial Fasteners Institute)

**Table 3**

**Mechanical Requirement and Identification Marking for Bolts, Screws, Studs, Sems, and U-Bolts.**

Grade Designation	Products	Nominal Size Dia, In.	Full Size Bolts, Screws, Studs, Sems		Machine Test Specimens of Bolts, Screws, and Studs				Surface Hardness	Core Hardness		Grade Identification Marking <sup>1</sup>
			Proof Load (Stress), psi	Tensile Strength (Stress) Min, psi	Yield <sup>A</sup> Strength (Stress) Min, psi	Tensile Strength (Stress) Min, psi	Elongation <sup>B</sup> Min, %	Reduction of Area Min, %	Rockwell 30N Max	Rockwell		
										Min	Max	
1	Bolts, Screws, Studs	¼ thru 1½	33,000 <sup>A</sup>	60,000	36,000 <sup>B</sup>	60,000	18	35	—	B70	B100	None
2	Bolts, Screws, Studs	¼ thru ¼ <sup>C</sup>	55,000 <sup>A</sup>	74,000	57,000	74,000	18	35	—	B80	B100	None
		Over ¼ to 1½	33,000	60,000	36,000 <sup>B</sup>	60,000	18	35	—	B70	B100	
4	Studs	¼ thru 1½	65,000	115,000	100,000	115,000	10	35	—	C22	C32	None
5	Bolts, Screws, Studs	¼ thru 1	85,000	120,000	92,000	120,000	14	35	54	C25	C34	⌵
		Over 1 to 1½	74,000	105,000	81,000	105,000	14	35	50	C19	C30	
5.1 <sup>D</sup>	Sems, <sup>H</sup>	No. 6 thru ¼	85,000	120,000	—	—	—	—	59.5 <sup>D</sup>	C25	C40 <sup>D</sup>	⌵
	Bolts, Screws	No. 6 thru ¼										
5.2	Bolts, Screws	¼ thru 1	85,000	120,000	92,000	120,000	14	35	56	C26	C36	⌵
7 <sup>E</sup>	Bolts, Screws	¼ thru 1½	105,000	133,000	115,000	133,000	12	35	54	C28	C34	⌵
8	Bolts, Screws, Studs	¼ thru 1½	120,000	150,000	130,000	150,000	12	35	58.8	C33	C39	⌵
8.1	Studs	¼ thru 1½	120,000	150,000	130,000	150,000	10	35	—	C32	C38	None
8.2	Bolts, Screws	¼ thru 1	120,000	150,000	130,000	150,000	10	35	58.8	C33	C39	⌵

**Note.** (Courtesy of SAE)

samples used for this study were provided by Iowa Industrial Products, Inc. These bolts were purchased from a major manufacturer in Canada. This Canadian manufacturer was certified by a midwest manufacturer. This assures the researcher that the samples were tested and certified for their quality. Extra care was taken to assure that the proper grade of the bolts selected was obtained.

The 180 bolts in the sample were divided into 10 groups: 8 experimental and 2 control. There were 2 groups in the control condition: 18 bolts tested with low-salt concentration solution (oL), and 18 bolts tested with high-salt concentration solution (oH). Within the experimental groups, the first one had 18 bolts coated with thin clear zinc and exposed to low-salt concentration solution (cL<sub>1</sub>). The second group had 18 bolts coated with thick clear zinc and exposed to low-salt concentration solution (cL<sub>2</sub>). The third group had 18 bolts coated with thin clear zinc and exposed to high-salt concentration solution (cH<sub>1</sub>). The fourth group was composed of 18 bolts coated with thick clear zinc and exposed to high-salt concentration solution (cH<sub>2</sub>). The fifth group consisted of 18 bolts coated with thin yellow zinc and exposed to low-salt concentration solution (yL<sub>1</sub>). The sixth group had 18 bolts coated with thick yellow zinc and exposed to low-salt concentration solution (yL<sub>2</sub>). The seventh group was composed of 18 bolts coated with thin yellow zinc and

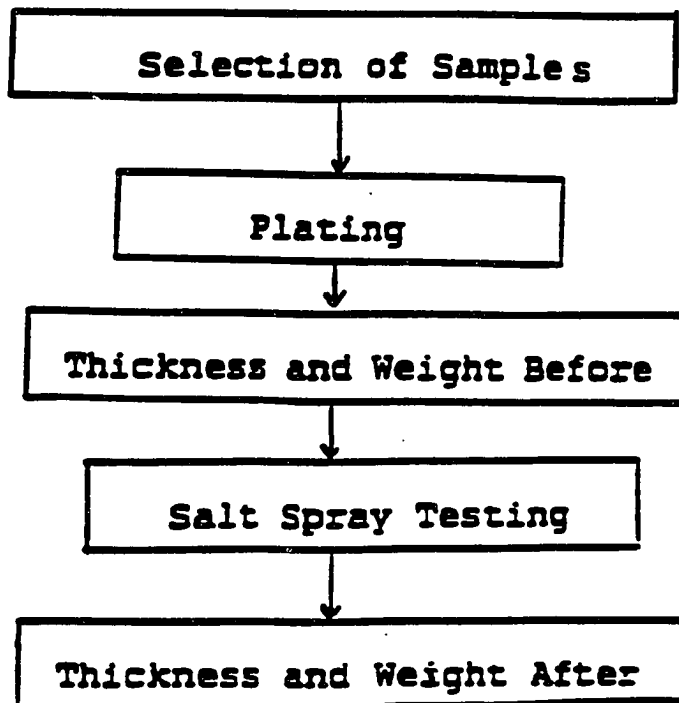


exposed to high-salt concentration ( $\gamma H_1$ ). The eighth experimental group consisted of 18 bolts coated thick yellow zinc and exposed to high-salt concentration solution ( $\gamma H_2$ ).

#### Testing Procedure

The testing procedure of this study consisted of five phases: (a) selection of samples, (b) plating phase, (c) thickness and weight measurement before testing, (d) salt-spray testing, and (e) thickness and weight measurement after testing. The five phases are outlined in Figure 6.

Figure 6. Phases of testing procedure.



### Plating

The selected samples were plated electrolytically by a midwest plating company as required by the ASTM Standard and noted in the Review of Literature. Plating is a process for depositing a thin layer of metal onto a metallic component; that constitutes the cathode in an electrical circuit, and it is immersed in a solution containing ions of the metal to be plated. The plating of the samples was conducted by the midwestern plating company which complies with ASTM Plating Standards and has been certified by a supplier for a large midwestern manufacturer's concern. They have been reviewed and audited and are awaiting the formal awarding of ISO 9000 certification. According to the design of the experiment, there are two groups of samples with different plating criteria to be done for the bolts--the clear zinc and the yellow zinc. Each group has a sub-group labelled as thin and thick. Each of these experimental conditions consisted of 18 bolts.

The clear zinc was labelled as cL. The yellow zinc was labelled as yL. The thin clear zinc was labeled as cL<sub>1</sub>. The thick clear zinc was labeled as cL<sub>2</sub>. Along with the same idea, the yellow zinc thin was labeled yL<sub>1</sub>, and the yellow zinc thick was labeled at yL<sub>2</sub>.

The plain bolts used in the experiment as control group were labeled as (0). There were 18 bolts in each category assigned to the low-salt concentration solution, and 18

bolts in each category assigned to the high-salt concentration solution. The midwest plating company subjected each group to certain steps in his task. These were:

1. Electroplate
2. Rinse (multiple rinse)
3. Dip in 0.25 - 0.50 pct. nitric acid
4. Chromate
5. Cold Rinse
6. Leach
7. Cold rinse
8. Warm air dry off

After these steps the plated parts were placed on a stainless steel basket. Though the plater stated that it was difficult to obtain a given plating thickness, a certain thickness was achieved within the range recommended by Zaki, 1988. He noted that a minimum of 0.0002" and an average of 0.00035" would be the appropriate plating thickness. In this study, the researcher called "low" any thickness in the range of 0.00020" and high any plating thickness of 0.00050" and above. The midwest plating company tried to carry on with the plating by applying the above procedures. These procedures were considered as the company's secret to maintain their competitive edge in the plating business.

### Thickness and Weight Measurements Before Test

The third stage in this experimental study consisted of weighing each part and also taking the plating thickness of each part. To achieve this goal the researcher used two different instruments: an electronic balance and a coating thickness tester. Here is a brief description of each instrument.

1. Electronic Balance. An electronic balance was used and was equipped with a read out display window where one can read out the weight of a given sample. It also has both the metric and the English Standard Units. The weight before and after in grams of each sample was reported in the table provided for data. (See Figure 7.)

2. Coating Thickness Tester. The coating thickness tester used in this research was the Kocour Model H10/2. (See Figure 8.) It is a non-destructive coating thickness measuring instrument based upon the magnetic induction principle. It is capable of measuring:

- (a) Non-magnetic coatings on ferro-magnetic substrates.
- (b) Conductive coatings on ferro-magnetic substrates.
- (c) Non-conductive coating on ferro-magnetic substrates.

The Kocour Model H 10/2 uses the magnetic properties of the base material as the basis for measurement. Large masses of ferrous materials and strong electromagnetic field

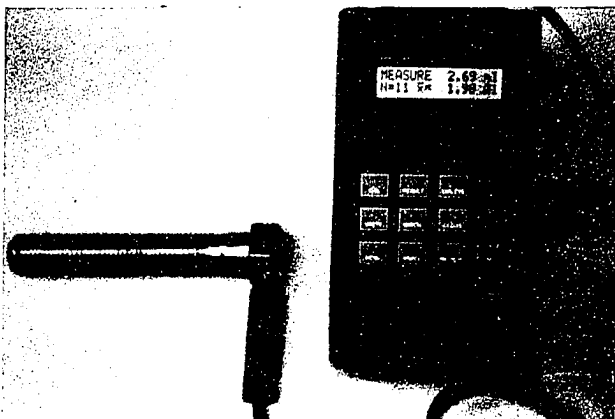
adversely affect these measurements. Therefore, the researcher used a wooden table to conduct the thickness measurements on selected samples.

The Bossard International, Inc. was referenced and followed in the Metric fasteners for advanced assembly engineering on p. T-58. It stated that when the plating thickness on thin and long bolts was not gauging properly, it was advisable to select the head area as a measuring point. The researcher respected this rule during the course of this experiment. All measurements were expressed in mils (ML).

Figure 7. Electronic balance.



Figure 8. The Kocour coating thickness tester model H10/2.



#### Salt Spray Testing

The most critical step in salt spray testing is the salt solution. The American Society for Testing and Materials in their publication Standard Method of Salt Spray (FOG) Testing (B 117-90, p. 21) advised that the salt solution be prepared by dissolving 5 (plus or minus 1) parts by weight of sodium chloride in 95 parts of water. The Society also noted that the salt used should be sodium chloride substantially free of nickel and copper and containing on the dry basis not more than 0.1% of sodium iodide and not more than 0.3% of total impurities.

This testing consisted of two parts: samples tested with low salt concentration in the main 20 gallon tank of water of the salt spray testing apparatus, and samples also

tested with high salt concentration in the main 20 gallon tank of water of the salt spray testing apparatus.

Using the Mead Corporation Conversion Table, the researcher determined the amount of salt that needed to be used in each test. It was noted in the Mead Conversion Table that one liter of water weighs one kilogram. The mass weight of 1 kilogram is equal to 2.20 pounds. It was also noted in the Mead Conversion Table that one gallon of water is equal to 3.785 liters. Using this information, the researcher determined the amount of salt to be used in the low salt concentration salt spray testing; 5 parts minus 1 part equals 4 parts. Translated in fraction this would be  $\frac{4}{96}$  if the total is 100% or a whole. The total capacity of the main water tank that was used in this project is 20 gallons. To determine the amount of salt needed for the low salt concentration salt spray testing, the researcher proceeded as follows:

It is known that 1 gallon of water is equal to 3.785 liters. It is also known that 1 kilogram equals 2.20 pounds and 1 liter of water weighs 1 kilogram, therefore, 1 gallon of water weighs  $3.785 \times 2.20$  pounds = 8.327 pounds. The water weight in the main 20 gallon water tank, therefore, was  $20 \times 8.327 = 166.54$  pounds. To compute the amount of salt needed for the low concentration salt spray testing, the researcher expressed the known and unknown parameters in an equation.

$$\frac{x}{166.54} = \frac{4}{96} \quad \text{solving for } x \text{ we got;}$$

$$96x = 4 \times 166.54$$

$$96x = 666.16$$

$$x = \frac{666.16}{96} = 6.9391 \text{ pounds}$$

To determine the amount of high salt concentration in the second part of the testing, the researcher proceeded in the similar way.

Five parts + 1 part = 6 parts. Translated in fraction this would be 6/94, with the above information. The equation became:

$$\frac{x}{166.54} = \frac{6}{94} \quad \text{solving for } x \text{ we got;}$$

$$94x = 6 \times 166.54$$

$$94x = 999.24$$

$$x = \frac{999.24}{94} = 10.630 \text{ pounds}$$

The American Society for Testing and Materials also noted in their book that the pH of the salt solution should be around 7.0; when atomized at 35°C (95°F) the collected solution was in the pH range of 6.2-7.2. The researcher kept a log of the solution collected (see Appendix E).

The specimens were hung in the fog chamber according to the (ASTM B117-90, p. 91) specifications. The position of



the specimens during the test should meet the following conditions as noted in (ASTM B117-90, p. 21):

1. Specimens were supported and suspended between 15° and 30° from the vertical and preferably parallel to the principal direction of horizontal flow of fog through the chamber, based upon the dominant surface being tested.

2. The specimens did not contact each other or any metallic material or any material capable of acting as a wick.

3. Each specimen was placed so as to permit free settling of fog on all specimens.

4. Salt solution from one specimen did not drip on any other specimens.

The researcher kept a daily log of the temperature inside the fog chamber. A nylon kite-thread was used to suspend the specimens in the fog chamber. Each specimen group was tested in the fog chamber for 192 hours or a total of eight days.

#### Thickness and Weight Measurement After Test

After 192 hours of testing in the fog chamber, the specimens were observed to be corroded with both the white and red corrosion products. In order to be able to take the thickness and the weight measurements on each specimen, the researcher followed the recommendation of the National Association for Corrosion Engineers' publication Corrosion Basics: An Introduction. Atkinson and Van Droffelaar (1982,

p. 112) noted that the researcher must use techniques that remove only corrosion products, and not the base metal. As stated earlier in the Literature Review, the simplest technique for removing corrosion products is soap and water cleaning with a soft bristle brush. After thoroughly washing each specimen according to the above recommendations, the researcher then proceeded with the measurements taking.

Using the Fairbanks electronic balance described above, the weight after for each specimen was taken and recorded (see Appendix C). The thickness measurements were also taken and recorded using the Kocour coating thickness tester Model H10/2 (see Appendix C).

#### Statistical Design and Analysis

This study employed the pre-post experimental design, with eight experimental conditions consisting of 18 bolts each which were then compared to two control conditions. The data collected was processed using the University of Northern Iowa mainframe VAX 6000 and analyzed with the statistical package SAS System. The results were interpreted by the researcher in a comparative way with the control groups.

#### Summary

In Chapter 3 the researcher outlined the methodology used for selecting samples, testing, and evaluating these samples according to the ASTM guidelines. The National

Association for Corrosion Engineers official publication entitled: Corrosion and Its Control was consulted while carrying out this experiment. The next chapter covers the analysis of the results of this study.

## CHAPTER 4

## ANALYSIS OF THE RESULTS

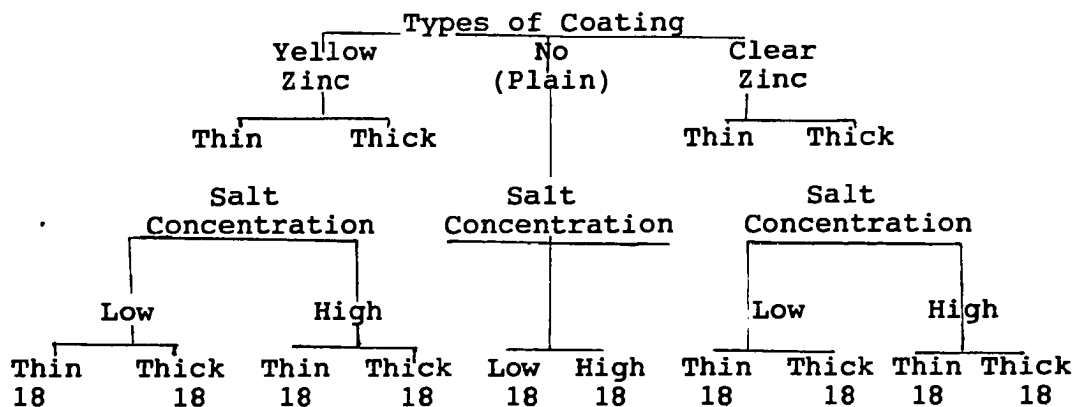
The main purpose of this study was to investigate the effects of various coatings on electroplated fasteners especially the 3/8-16 x 5 3/4 medium carbon steel bolts. The types of coatings involved in this experiment were the clear chromate and the yellow dichromate conversion coatings. The midwestern plating company assisted with the plating of samples in this experiment. The rest of the experiment was conducted at the manufacturing supplier in the midwest in accordance with the guidelines of (ASTM, B117). A daily log was kept from the beginning to the end of the test (see Appendix E). The data obtained were analyzed at the University of Northern Iowa computing center using the VAX 6000 mainframe computer. These results enabled the researcher to answer the research questions and subquestions. They also provided answers to a number of subquestions asked about the longevity, the degree of protection, as well as the safety issue involved with a given electroplated coating, particularly the clear chromate or yellow dichromate zinc.

There were five interrelated questions asked in this research. It was expected that the results of the data provided by the test would show the relationship between fasteners plated electrolytically with different types of coatings.

### The Data Presentation

The design of the experiment in Figure 5 indicates that there were 180 data points. The attributes involved in the collection of this data were: weight before and after and the thickness before and after.

Figure 9. Salt spray test design.



There were two dependent variables: (a) weight loss (Loss 1) and (b) thickness loss (Loss 2). There were also 3 categorical independent variables:

\* Salt with 2 categories: Low concentration (1) and high concentration (2).

\* Treatment with 3 categories: Plain (1), yellow (2), clear (3).

\* Thickness with 3 categories: Plain (0), thin (1), and thick (3).

The 180 data points (see Appendix C) were composed of the following attributes: the identity of each sample, the treatment and the category of salt concentration used to run the test. The weight before and after as well as the

thickness before and after of each sample unit were recorded. Using the SAS System, the statistics of the experiment were generated and the results analyzed. However, it was deemed important to describe the appearance of each group of samples right after the test.

#### Description of the Samples After Each Test

The test consisted of two parts: the first part was run with low concentration of salt solution and the second part was run with high concentration of salt solution. On July 20, 1993 at 3:35 p.m., the first part of the study was concluded. After the first run, the fog chamber of the salt spray testing equipment was emptied. It appeared that a visual description of the sample tested would be necessary in order to support some of the theory described in the literature review. This first part of the test was conducted with the low concentration of salt in the main tank of the salt spray machine. There were five groups of bolts tested during that period.

Based on the researcher's visual estimate, the following results were observed:

1. The bolts coated with thin clear zinc chromate (cL<sub>1</sub>) appeared to be covered at 80% with white corrosion product and 20% with red corrosion product on the threaded and head areas (see Figures 10 and 11).
2. The bolts coated with thick clear zinc chromate (cL<sub>2</sub>) appeared to be covered at 85% with white corrosion

products and few traces of red corrosion products in the thread and head areas (see Figures 12 and 13).

3. The sample coated with thin yellow zinc dichromate ( $yL_1$ ) were covered at 20% with white corrosion products and no traces of red corrosion products anywhere at all. However, there was a slight loss of color on both the head and the thread areas (see Figures 14 and 15).

4. The bolts coated with thick yellow zinc dichromate ( $yL_2$ ) appeared to be covered at 15% with white corrosion products and there was no red corrosion products anywhere at all. Here again, the head and the thread areas had a slight loss of color (see Figures 16 and 17).

5. Finally, the plain samples were all covered at 100% with red corrosion products with most of the thread gone on each one of them. The head areas also appeared to have more corrosion (see Figures 18 and 19).

On July 28, 1993 at 4:00 p.m., the second part of the test was completed. This test was conducted with high salt concentration in the main tank of the salt spray machine. There were also five groups of bolts tested during that period. The results, based on the experimenter's visual estimate, were as follows:

1. The bolts coated with thin clear zinc chromate ( $CH_1$ ) were covered with 95% white corrosion products and traces of red corrosion products on the head and thread areas (see Figures 20 and 21).

2. The bolts coated with thick clear zinc chromate ( $\text{CH}_2$ ) were covered at 90% with white corrosion products and a trace of red corrosion products on the thread area (see Figures 22 and 23).

3. The samples coated with thin yellow zinc dichromate ( $\text{yH}_1$ ) were covered with 15% white corrosion products and no red corrosion products on any part of the samples. However, their yellow color was fading out (see Figures 24 and 25).

4. The group of bolts coated with thick yellow ( $\text{yH}_2$ ) zinc dichromate were covered with 10% white corrosion products and no red corrosion products were found anywhere on the parts (see Figures 26 and 27).

5. Finally, the group of bolts with no coating was covered with red corrosion products at 100%. They appeared to be heavily corroded on the thread area as well as the head area (see Figures 28 and 29).



Figure 10. Eighteen medium carbon steel bolts plated electrolytically with thin clear chromate zinc: Appearance before test.

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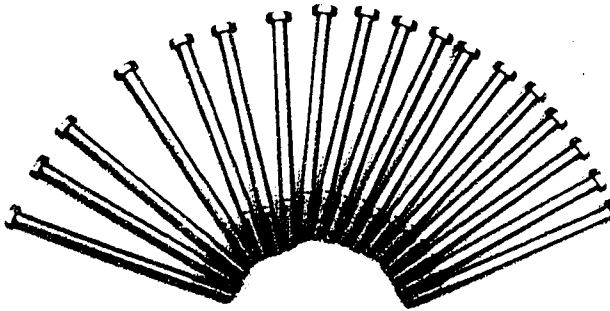


Figure 11. Eighteen medium carbon steel bolts plated electrolytically with thin clear chromate zinc tested with low salt concentration solution in the main tank of the salt spray machine: Appearance after test.

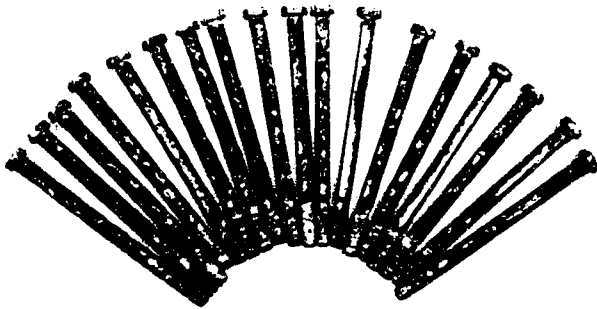


Figure 12. Eighteen medium carbon steel bolts plated electrolytically with thick clear chromate zinc: Appearance before test.

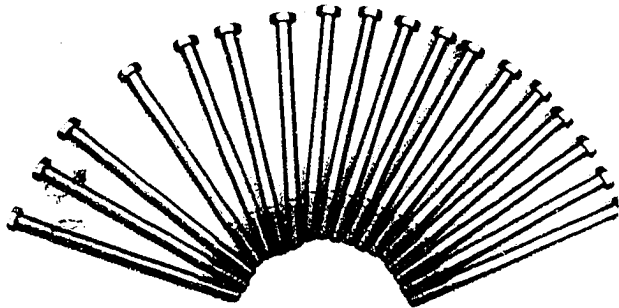


Figure 13. Eighteen medium carbon steel bolts plated electrolytically with thick clear chromate zinc and tested with low salt concentration solution in the main tank of the salt spray machine: Appearance after test.

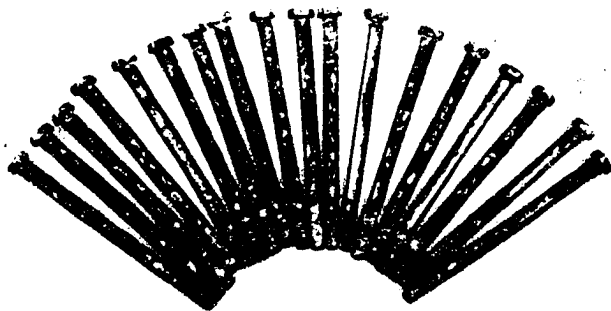


Figure 14. Eighteen medium carbon steel bolts plated electrolytically with thin yellow dichromate zinc:  
Appearance before test.

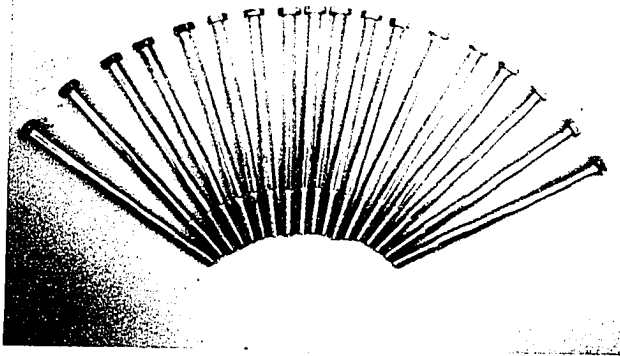


Figure 15. Eighteen medium carbon steel bolts plated electrolytically with thin yellow dichromate zinc and tested with low salt concentration solution in the main tank of the salt spray machine: Appearance after test.

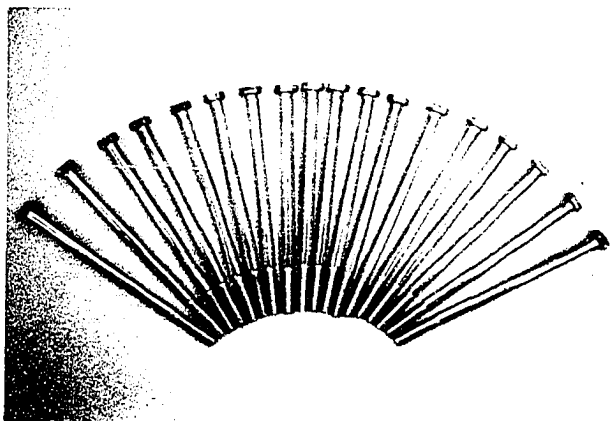


Figure 16. Eighteen medium carbon steel bolts plated electrolytically with thick yellow dichromate zinc:  
Appearance before test.

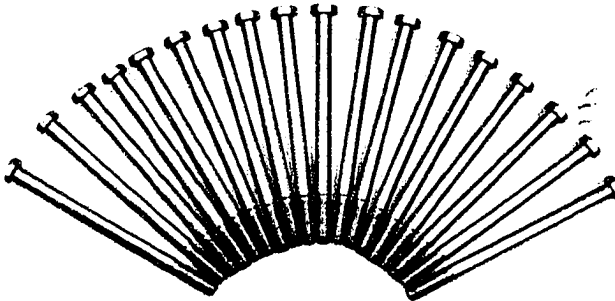


Figure 17. Eighteen medium carbon steel bolts plated electrolytically with thick yellow dichromate zinc and tested with low salt concentration solution in the main tank of the salt spray machine: Appearance after test.

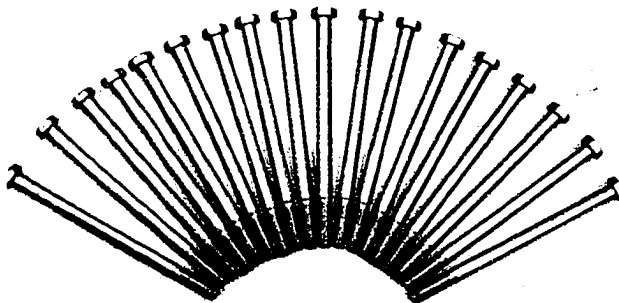


Figure 18. Plain medium carbon steel bolts: Appearance before test.

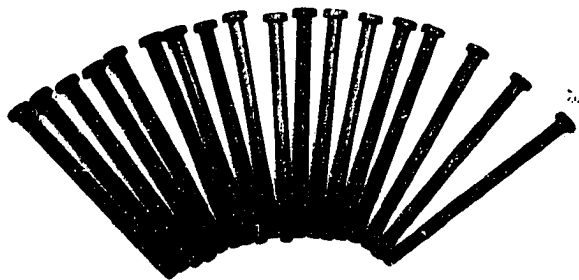


Figure 19. Plain medium carbon steel bolts tested with low salt concentration solution in the main tank of the salt spray machine: Appearance after test.

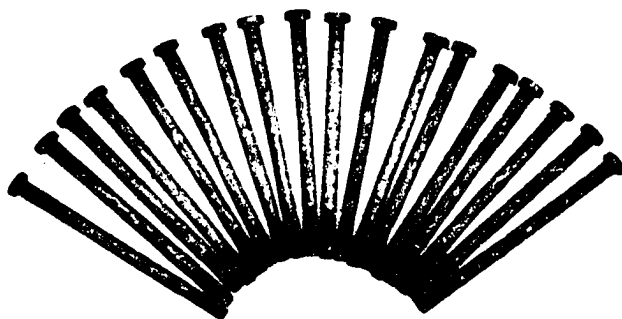


Figure 20. Eighteen medium carbon steel bolts plated with thin clear chromate zinc: Appearance before test.

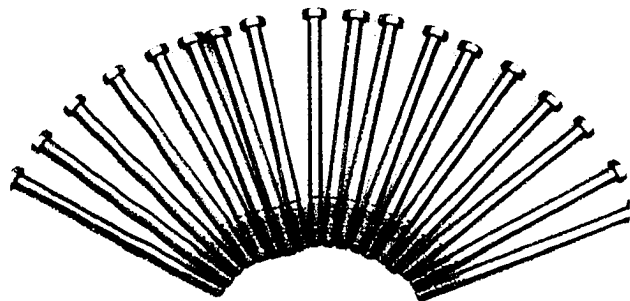


Figure 21. Eighteen medium carbon steel bolts plated with thin clear chromate zinc and tested with high salt concentration solution in the main tank of the salt spray machine: Appearance after test.

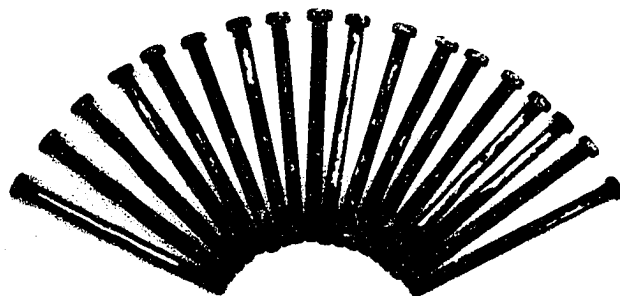


Figure 22. Eighteen medium carbon steel bolts plated electrolytically with thick clear chromate zinc: Appearance before test.

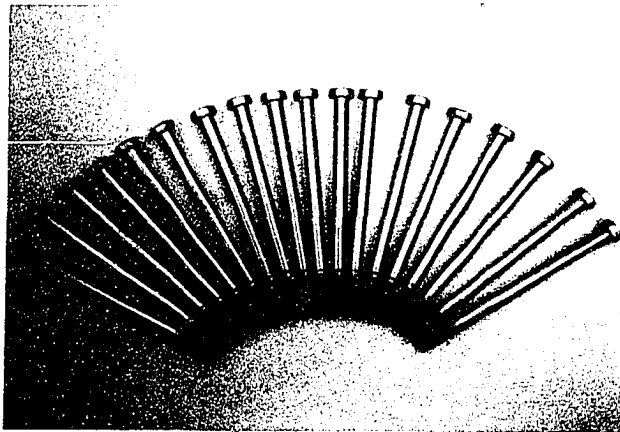


Figure 23. Eighteen medium carbon steel bolts plated electrolytically with thick clear chromate and tested with high salt concentration solution in the main tank of the salt spray machine: Appearance after test.

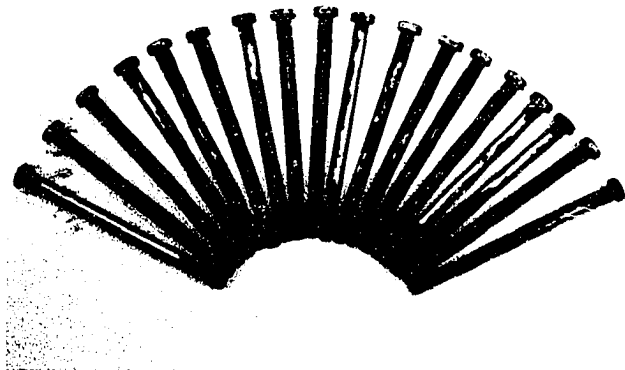


Figure 24. Eighteen medium carbon steel bolts plated electrolytically with thin yellow dichromate zinc:  
Appearance before test.

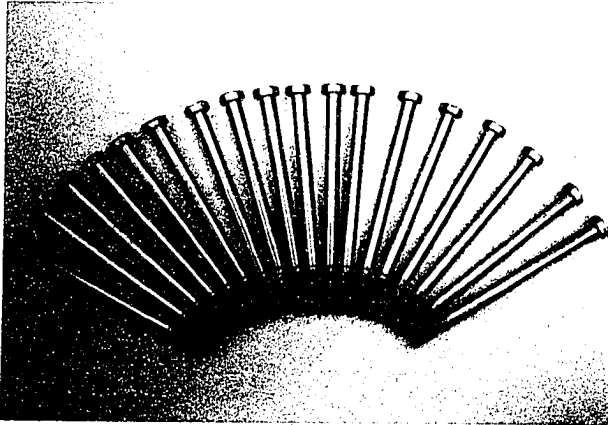


Figure 25. Eighteen medium carbon steel bolts plated electrolytically with thin yellow dichromate zinc and tested with high salt concentration solution in the main tank of the salt spray machine: Appearance after test.

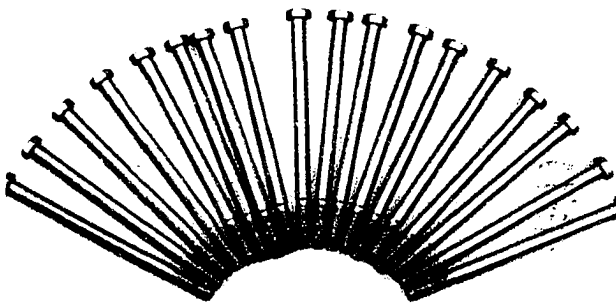




Figure 26. Eighteen medium carbon steel bolts plated electrolytically with thick yellow dichromate zinc: Appearance before test.

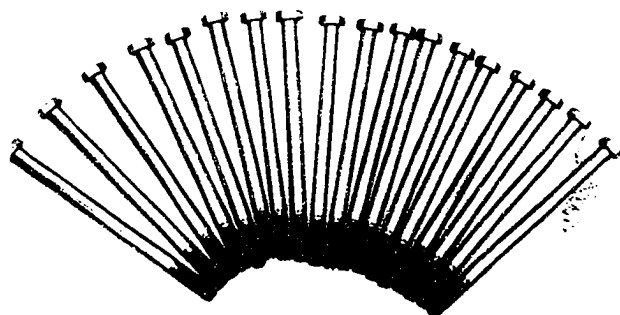


Figure 27. Eighteen medium carbon steel bolts plated electrolytically with thick yellow dichromate and tested with high salt concentration solution in the main tank of the salt spray machine: Appearance after test.

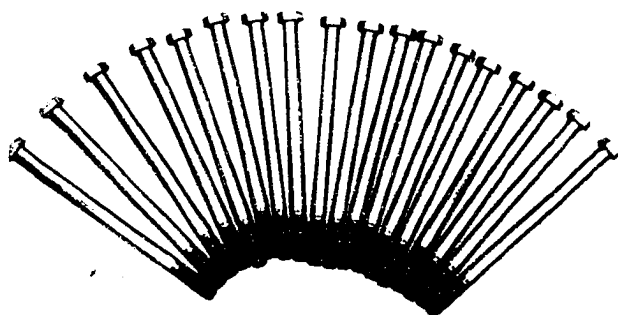


Figure 28. Plain medium carbon steel bolts: Appearance before test.

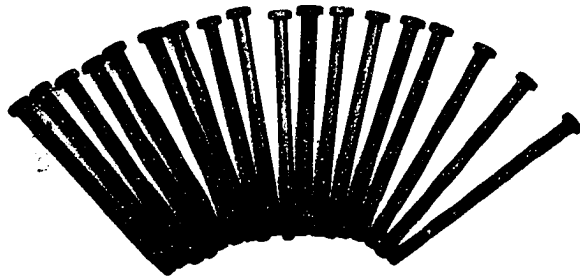
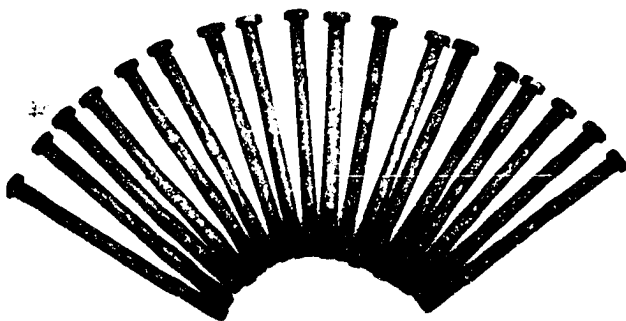


Figure 29. Plain medium carbon steel bolts tested with high salt concentration solution in the main tank of the salt spray machine: Appearance after test.



### The Research Hypothesis

The statistics used in this research were that of the Analysis of Variance, commonly referred to as ANOVA. It is a statistical technique that seeks to determine whether differences in the values of a variable can be explained by categorization of the observations (Mirer, 1988). According to Witte (1989 p. 345), ANOVA tests the null hypothesis for two, three, or more population means by classifying total variability into two dependent components: variability between groups and variability within groups. He noted that F reflects the ratio of the observed differences between all sample means known as the variability between groups in the numerator and the estimated error terms known as variability within groups in the denominator term, that is,

$$F = \frac{\text{Variability between groups}}{\text{Variability within groups.}}$$

Wiersma (1991, p. 330) explained that the sampling distribution for the ratio of two variances is the F distribution. He added that in nature the numerical values of the distribution are all positive. The researcher made use of this information to test the research hypothesis according to the result of the computer generated output of the study.

The research question stated was as follows: Is coating thickness correlated with the corrosion resistance

of medium carbon steel plated bolts? This question can be answered when considering the general linear model test result in Figure 30. The null hypothesis for this research question was as follows:  $H_0$  = There is no correlation between plating thickness and corrosion resistance of medium carbon steel bolts.

The alternative hypothesis was  $H_A$ : There is a correlation between plating thickness and corrosion resistance of medium carbon steel plated bolts. Considering the percentage loss of thickness called Loss 2, the result of the test was that the P-value is 0.0001 which is a very small number. Neter, Wasserman, and Kutner (1993) noted that the P-value for a sample outcome is the probability that the sample outcome could have been more extreme than the observed one when  $\mu = \mu_0$ . They added that large P-values support  $H_0$  while small P-values support the  $H_A$  (p. 12). For a P-value of 0.0001 the researcher supported the alternative hypothesis which stated that there is a correlation between the plating thickness and the corrosion resistance of plated bolts. Harnett and Murphy (1983) commenting on the P-value noted: "If  $\alpha$  exceeds the P-value: reject  $H_0$  and if the P-value exceeds  $\alpha$ : accept  $H_0$ " (p. 320). Again since the P-value of the experiment Loss 2 was 0.0001 and the nominal alpha level for this research was 0.05, it is evident that  $\alpha = 0.05$  exceeded the P-value (0.0001). The researcher rejected the null hypothesis ( $H_0$ ). This means that the

Figure 30. General linear models procedure for loss 2.

The SAS System

General Linear Models Procedure  
Class Level Information

Class	Levels	Values
TRT	3	1 2 3
BALT	2	1 2
THICKNS	3	0 1 2

Number of observations in data set = 180

General Linear Models Procedure

Dependent Variable: LOSS2

Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	9	26.10683389	2.90073932	1392.87	0.0001
Error	170	0.35403889	0.00208258		
Corrected Total	179	26.46087278			

	R-Square	C. V.	Root MSE	LOSS2 Mean
	0.986430	11.63012	0.0456353	0.3923889

Source	DF	Type I SS	Mean Square	F Value	Pr > F
TRT	2	23.77144083	12.88572042	6187.38	0.0001
BALT	1	0.22401389	0.22401389	107.87	0.0001
THICKNS	1	0.00241736	0.00241736	1.16	0.2828
TRT*BALT	2	0.08350417	0.04175208	20.65	0.0001
TRT*THICKNS	1	0.01625625	0.01625625	7.81	0.0058
BALT*THICKNS	1	0.00918403	0.00918403	4.41	0.0372
TRT*BALT*THICKNS	1	0.00001736	0.00001736	0.01	0.9274

Source	DF	Type III SS	Mean Square	F Value	Pr > F
TRT	1	18.84283403	18.84283403	9047.82	0.0001
BALT	1	0.16323615	0.16323615	79.35	0.0001
THICKNS	1	0.00241736	0.00241736	1.16	0.2828
TRT*BALT	1	0.02780069	0.02780069	13.21	0.0004
TRT*THICKNS	1	0.01625625	0.01625625	7.81	0.0058
BALT*THICKNS	1	0.00918403	0.00918403	4.41	0.0372
TRT*BALT*THICKNS	1	0.00001736	0.00001736	0.01	0.9274

alternative hypothesis was accepted. There indeed is a correlation between the plating thickness and the corrosion resistance of medium carbon steel bolts plated electrolytically with clear chromate zinc or yellow dichromate zinc.

The F-value for the overall model of Loss 2 is 1392.87 which is a very high value. Borg and Gall (1989) noted that the statistical tool used to test for significance of differences between variances is the analysis of variance or the F test. The value of F is the ratio of the variances. Borg and Gall also noted that the larger the F ratio, the less likely it is that the variances of the populations from which the samples were drawn are equal. They noted that if the F ratio exceeds the significance level set by the researcher, the null hypothesis should be rejected. In this particular general linear model, an F-value of 1392.87 shows that the overall model is significant and also that there is a reason to support the alternative hypothesis ( $H_A$ ). There is a correlation between the plating thickness and the corrosion resistance of medium carbon plated bolts.

Harnett and Murphy (1983) stated that in the general linear model, the total variation or the sum of squares total (SST) is equal to the unexplained variation or the sum of squares errors (SSE) plus the explained variation or the sum of squares regression (p. 469).

$$SST = SSE + SSR$$

Considering Table 12, the SST = 50.4309644, the SSE = 4.93041111 and the sum of square regression is equal to 45.50055333.

$$\begin{array}{rcc} SST & & SSE & & SSR \\ 50.4309644 & = & 4.93041111 & + & 45.50055333 \end{array}$$

Harnett and Murphy also noted that the mean square regression (MSR) is equal to the sum of squares regression divided by the degree of freedom between groups in Loss 1.

$$MSR = \frac{SSR}{DF \text{ (between)}} = \frac{45.50055333}{9} = 5.05561704$$

They also noted that the mean square error (MSE) is equal to the sum of squares error divided by the degree of freedom within groups (p. 513); for Loss 1 the following equation applies:

$$MSE = \frac{SSE}{DF \text{ (within)}} = \frac{4.9304111}{170} = 0.02900242$$

Applying the previous formula for F, Harnett and Murphy

$$\text{noted that } F = \frac{MSR}{MSE} = \frac{5.05561704}{0.02900242} = 174.32.$$

Looking at the dependent variable Loss 2, an analogue reasoning was used to figure out the meaning of the computer output. The sum of squares total (SST) = 26.46087278 and the sum of squares regression (SSR) was 26.10683389 and finally the sum of squares error was (SSE) was 0.35403889.

$$\begin{aligned} \text{SST} &= \text{SSE} + \text{SSR} \\ 26.46087278 &= 0.35403889 + 26.10683389 \end{aligned}$$

The mean square regression equal the sum of squares regression (SSR) divided by the degree of freedom between groups ( $DF_{\text{between}}$ ) = 9.

$$\text{MSR} = \frac{\text{SSR}}{\text{DF (between)}} = \frac{26.10683389}{9} = 2.90075932.$$

The Mean Square Error equal the sum of squares error (SSE) divided by the degree of freedom within ( $DF_{\text{within}}$ ) = 170.

$$\text{MSE} = \frac{\text{SSE}}{\text{DF (within)}} = \frac{0.35403889}{170} = 0.00208258.$$

The F-value was also computed using Harnett and Murphy (1983) notation

$$F = \frac{\text{MSR}}{\text{MSE}} = \frac{2.90075932}{0.00208258} = 1399.87.$$

The researcher also looked at the second dependent variable: weight loss called (Loss 1). The general linear model test result of the interaction between treatment, salt, and thickness in Figure 31 yielded the following results: F-value = 1392.87 and the P-value = 0.0001 for the overall model. It means that this model is very significant.

As stated earlier, Borg and Gall (1989) noted that the larger the F-value, the less likely the variance of the population from which the samples were drawn are equal and



if the F-value exceeds the significance level set by the researcher, the null hypothesis should be rejected. Here again, the researcher concludes that there is a correlation between the plating thickness and the corrosion resistance of medium carbon plated fasteners.

In both Figures 30 and 31, looking at the individual variables and their F-value and P-value, one sees that the independent variable, thickness, by itself is not significant at all. In both cases its P-value was high and its F-value was low. This phenomenon is due to the fact that thickness doesn't really matter as a single entity. In order for thickness to matter it has to be involved with other factors such as treatment and salt interaction. There are many factors that can determine the significance of this variable. Also in both Tables, the three-way interaction between treatment, salt and thickness appeared not to be significant. In both cases the P-value is high and the F-value is low respectively. Here again, the researcher concluded that thickness, treatment and salt interaction may not be significant if certain conditions are not present. These conditions are humidity, contaminations, salinity in the atmosphere, and possibly temperature also.

The researcher wondered what would be the effect of the other interaction if thickness was not a factor. What would be the result if two-way interaction rather than three-way interaction was considered? In Figure 32, the researcher

generated a new general linear model with two-way interactions. The results were as follows: the F-value for the overall model was 192.63 and the overall P-value was 0.0001. As stated earlier, a low P-value and a high F-value denotes that the overall experiment was significant. Looking at individual two-way interactions, all 3 two-way interactions were significant. (TrT \* salt) had P-value = 0.0001 and F-value 169.59; interaction between treatment and thickness (TrT \* Thickns) was also significant. The P-value for their interaction was 0.0001 and their F-value was 25.30. Finally, the interaction between salt and thickness (salt \* thickns) was also significant. Their P-value was 0.0003 and their F-value was 13.83.

**Figure 31.** General linear models procedure for loss 1.

The SAS System

General Linear Models Procedure  
Class Level Information

Class	Levels	Values
TRT	3	1 2 3
SALT	2	1 2
THICKNS	3	0 1 2

Number of observations in data set = 180

General Linear Models Procedure

Dependent Variable: LOSS1

Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	9	48.50053333	5.383392592	174.32	0.0001
Error	170	4.93041111	0.02900242		
Corrected Total	179	50.43094444			
	R-Square	C.V.	Root MSE	LOSS1 Mean	
	0.902234	19.14430	0.1703010	0.8993336	

Source	DF	Type I SS	Mean Square	F Value	Pr > F
TRT	2	23.77010036	11.88505028	409.80	0.0001
SALT	1	13.93633536	13.93633536	481.19	0.0001
THICKNS	1	0.00022300	0.00022300	0.01	0.9299
TRT*SALT	2	6.81304722	3.25733361	112.32	0.0001
TRT*THICKNS	1	0.74334444	0.74334444	25.70	0.0001
SALT*THICKNS	1	0.40744944	0.40744944	14.05	0.0002
TRT*SALT*THICKNS	1	0.10671111	0.10671111	3.68	0.0568

Source	DF	Type III SS	Mean Square	F Value	Pr > F
TRT	1	21.02222300	21.02222300	724.84	0.0001
SALT	1	15.35683319	15.35683319	529.80	0.0001
THICKNS	1	0.00022300	0.00022300	0.01	0.9299
TRT*SALT	1	4.99322300	4.99322300	172.23	0.0001
TRT*THICKNS	1	0.74334444	0.74334444	25.70	0.0001
SALT*THICKNS	1	0.40744944	0.40744944	14.05	0.0002
TRT*SALT*THICKNS	1	0.10671111	0.10671111	3.68	0.0568

**Figure 32. General linear models procedure for two way interaction between samples: Loss 1.**

**The SAS System**

**General Linear Models Procedure**  
**Class Level Information**

Class	Levels	Values
TRT	3	1 2 3
BALT	2	1 2
THICKNS	3	0 1 2

**General Linear Models Procedure**

Dependent Variable: LOSS1

Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	8	45.37384222	5.67423028	192.63	0.0001
Error	171	5.03712222	0.02945686		
Corrected Total	179	50.40964444			
	R-Square	C.V.	Root MSE	LOSS1 Mean	
	0.900118	19.29390	0.1716300	0.8893556	

Source	DF	Type I SS	Mean Square	F Value	Pr > F
TRT	2	23.77010056	11.88505028	403.47	0.0001
BALT	1	13.93563556	13.93563556	473.77	0.0001
THICKNS	1	0.00022500	0.00022500	0.01	0.9305
TRT*BALT	2	6.51506722	3.25753361	110.59	0.0001
TRT*THICKNS	1	0.74534444	0.74534444	25.30	0.0001
BALT*THICKNS	1	0.40746944	0.40746944	13.83	0.0003

Source	DF	Type III SS	Mean Square	F Value	Pr > F
TRT	1	21.02222500	21.02222500	713.66	0.0001
BALT	1	15.47533889	15.47533889	523.36	0.0001
THICKNS	1	0.00022500	0.00022500	0.01	0.9305
TRT*BALT	1	4.99522500	4.99522500	169.58	0.0001
TRT*THICKNS	1	0.74534444	0.74534444	25.30	0.0001
BALT*THICKNS	1	0.40746944	0.40746944	13.83	0.0003

### Results of Research Questions

#### Question One

The first research question that this study attempted to answer was: Is coating correlated to corrosion resistance of fasteners?

In order to answer this question, the researcher considered the data processed by the VAX 6000-410 computer using the statistical software called SAS System. Looking at the first independent variable and the results of the computer output, the researcher computed the percentage weight loss. The procedure developed by Moniz (1992) for computing the percentage reduction of the area of a bolt was used here as follows:

$$\text{Percentage Reduction of Area} = \frac{A_o - A_r}{A_o} \times 100$$

where  $A_o$  = original area

$A_r$  = final area

100 = constant.

Area =  $\pi r^2$  let that be A

Following the same procedure, the researcher computed the percentage thickness loss.

$$\% \text{ Thickness Loss} = \frac{\text{Thickness Before} - \text{Thickness After}}{\text{Thickness Before}} \times 100$$

The percentage thickness loss for bolts plated with thin yellow zinc and tested with low salt concentration solution in the salt spray tank was computed as follows:

$$\frac{1.005 - 0.820}{1.005} \times 100 = 18.4\% \text{ thickness loss.}$$

The percentage thickness loss for bolts plated with thick yellow zinc and tested with low salt concentration solution in the salt spray tank was computed as follows:

$$\frac{1.021 - 0.833}{1.021} \times 100 = 18.4\% \text{ thickness loss.}$$

The percentage thickness loss for bolts plated with thin clear chromate zinc and tested with low salt concentration solution in the main tank of the salt spray machine was computed as follows:

$$\frac{0.984 - 0.125}{0.984} \times 100 = 87.2\% \text{ thickness loss.}$$

The percentage thickness loss for bolts plated with thick clear chromate zinc and tested with low salt concentration solution in the main tank of the salt spray machine was also computed:

$$\frac{1.051 - 0.146}{1.051} \times 100 = 86.1\% \text{ thickness loss.}$$

Using these results, Table 4 was constructed.

Table 4

Nominal Mean Loss of Thickness (Loss 2) and Percentage Thickness Loss. Test Done with Low Salt (Salt = 1).

Treatment	THICKNESS		
	Plain (0)	Thin (1)	Thick (2)
Plain 1	0	--	--
Yellow 2	--	0.186 18.4%	0.188 18.4%
Clear 3	--	0.860 87.2%	0.906 86.1%

Again, following the same procedure, the percentage thickness loss for bolts tested with high salt concentration solution in the main tank of the salt spray machine was also computed. Percentage thickness loss for bolts plated with thin yellow dichromate zinc and tested with high salt concentration solution in the main tank of the salt spray machine was computed as follows:

$$\frac{0.978 - 0.893}{0.978} \times 100 = 8.5\% \text{ thickness loss.}$$

Percentage thickness loss for bolts plated with thick yellow dichromate zinc and tested with high salt concentration solution in the main tank of the salt spray machine was also computed as follows:

$$\frac{1.028 - 0.972}{1.028} \times 100 = 5.6\% \text{ thickness loss.}$$

Percentage thickness loss for bolts plated with thin clear zinc chromate and tested with high salt concentrated solution in the main tank of the salt spray machine was also computed as follows:

$$\frac{1.002 - 0.186}{1.002} \times 100 = 81.4\% \text{ thickness loss.}$$

Percentage thickness loss for bolts plated with thick clear zinc chromate and tested with high salt concentrated solution in the main tank of the salt spray machine was computed and the result was as follows:

$$\frac{1.023 - 0.195}{1.023} \times 100 = 80.9\% \text{ thickness loss.}$$

Using the result of bolts tested with high salt concentration (Salt 2) Table 5 was constructed for comparison of both the nominal thickness loss and the percentage thickness loss.

Table 5

Nominal Mean Loss of Thickness (Loss 2) and Percentage Thickness Loss. Test Done with High Salt (Salt = 2).

Treatment	THICKNESS		
	Plain (0)	Thin (1)	Thick (2)
Plain 1	0	--	--
Yellow 2	--	0.085 8.5%	0.057 5.6%
Clear 3	--	0.816 81.4%	0.828 80.9%



The data in Table 4 show that bolts electrolytically plated with yellow zinc dichromate are more corrosion resistant than the ones coated electrolytically with clear zinc chromate. Also, the data shown in this table indicate that thickness does not matter as long as the bolts are coated with yellow zinc dichromate. That is, the specimen will endure the same percentage loss.

The data in Table 5 reveal for the groups of bolts subjected to a high salt concentration in this test that the bolts plated electrolytically with yellow zinc dichromate are more corrosion resistant than the ones coated with clear zinc chromate. Bolts plated with yellow zinc dichromate performed similarly in a salted, humid, oceanlike climate.

These results are consistent with previous research findings (e.g., Zaki, 1988). Zaki stated that thick coating would be anything above 0.00025" and thin coating would be any coating in the range of 0.0002" or less. These findings hold true provided that there is no "slough off" problem. "Slough off" is known as the problem that occurs when the chromate films during drying time of the plating process would not adhere to the surface being coated. Furthermore, these results are also supported by Cook et al. (1992) findings that thickness and uniformity of the coating are important because they determine bolt/nut corrosion resistance; this ensures the protection and the economical use of coated parts.

The researcher also examined the first dependent variable nominal weight loss (Loss 1) using the computer output to compute the percentage weight loss.

The percentage weight loss for bolts with no coating (plain) tested with low salt concentrated solution in the main tank of the salt spray machine (TRT = 1, Salt = 1, Thickns = 0) was computed as follows:

$$\frac{83.609 - 82.010}{83.609} \times 100 = 1.91\% \text{ weight loss.}$$

The percentage weight loss for bolts plated with thin yellow dichromate zinc and tested with low salt concentrated solution in the main tank of the salt spray machine (TRT = 2, Salt = 1, Thickns = 1) was computed as follows:

$$\frac{83.730 - 82.714}{83.730} \times 100 = 1.21\% \text{ weight loss.}$$

The percentage loss for bolts plated with thick yellow dichromate zinc and tested with low salt concentrated solution in the main tank of the salt spray machine (TRT = 2, Salt = 1, Thickns = 2) was also computed as follows:

$$\frac{84.210 - 83.496}{84.210} \times 100 = 0.84\% \text{ weight loss.}$$

The percentage weight loss for bolts plated with thin clear zinc chromate and tested with low salt concentrated solution in the main tank of the salt spray machine (TRT = 3, Salt = 1, Thickns = 1) was also computed as follows:

$$\frac{83.824 - 82.615}{83.824} \times 100 = 1.44\% \text{ weight loss.}$$

The percentage weight loss for bolts plated with thick clear zinc chromate and tested with low salt concentrated solution in the main tank of the salt spray machine (TRT = 3, Salt = 1, Thickns = 2) was computed as follows:

$$\frac{84.377 - 83.074}{84.377} \times 100 = 1.54\% \text{ weight loss.}$$

The data in Table 6 show the results of the computed percentage weight loss of bolts tested with low salt-concentrated solution in the main tank of the salt spray machine. It contains both the nominal weight loss and the percentage weight loss of these bolts. These data show that bolts electrolytically plated with yellow zinc dichromate provide better corrosion resistance than the ones coated electrolytically with clear zinc chromate. It can also be said that the results of this study revealed that a thick coating provides an even better corrosion resistance.

The same test was also conducted with high salt concentration in the salt spray tank. The researcher also computed the percentage weight loss for bolts coated with thin and thick yellow dichromate zinc and clear zinc chromate. This test was conducted with high concentrated salt solution in the main tank of the salt spray machine. The percentage weight loss for bolts with no coatings (Plain) and tested with high concentrated salt solution in

Table 6

Nominal Mean Weight Loss (Loss 1) and Percentage Weight Loss. Test Done with Low Salt (Salt = 1).

Treatment	WEIGHT		
	Plain (0)	Thin (1)	Thick (2)
Plain 1	1.60 1.91%	--	--
Yellow 2	--	1.016 1.21%	0.714 0.84%
Clear 3	--	1.209 1.44%	1.303 1.54%

the main tank of the salt spray machine (TRT = 1, Salt = 2, Thickns = 0) was computed as follows:

$$\frac{83.622 - 82.948}{83.622} \times 100 = 0.80\% \text{ weight loss.}$$

The percentage weight loss for bolts plated with thin yellow dichromate zinc and tested with high concentrated salt solution in the main tank of the salt spray machine (TRT = 2, Salt = 2, Thickns = 1) was computed as follows:

$$\frac{83.767 - 83.749}{83.767} \times 100 = 0.02\% \text{ weight loss.}$$

The percentage weight loss for bolts plated with thick yellow dichromate zinc and tested with high concentrated salt solution in the main tank of the salt spray machine

(TRT = 2, Salt = 2, Thickns = 2) was also computed as follows:

$$\frac{84.218 - 84.181}{84.218} \times 100 = .04\% \text{ weight loss.}$$

The percentage weight loss for bolts plated with thin clear zinc chromate and tested with high concentrated salt solution in the main tank of the salt spray machine (TRT = 3, Salt = 2, Thickns = 1) was computed as follows:

$$\frac{83.767 - 82.702}{83.767} \times 100 = 1.27\% \text{ weight loss.}$$

The percentage weight loss for bolts plated with thick clear zinc chromate and tested with high concentrated salt solution in the main tank of the salt spray machine (TRT = 3, Salt = 2, Thickns = 2) was finally computed as follows:

$$\frac{84.399 - 83.136}{84.399} \times 100 = 1.49\% \text{ weight loss.}$$

Using the above results, Table 7 was constructed in the same manner as Tables 4, 5, and 6.

The data in Table 7 revealed that bolts plated electrolytically with yellow zinc dichromate provide better corrosion resistance than the ones coated electrolytically with clear zinc chromate since they provided the least mean weight loss and the least percentage weight loss. The data in Table 7 can also be used to answer Question One.

Table 7

Nominal Mean Weight Loss (Loss 1) and Percentage Weight Loss. Test Done with High Salt (Salt = 2).

Treatment	WEIGHT		
	Plain (0)	Thin (1)	Thick (2)
Plain 1	0.674 1.21%	--	--
Yellow 2	--	0.017 0.02%	0.037 0.04%
Clear 3	--	1.064 1.27%	1.263 1.49%

#### Question Two

The second research question was: Is clear chromate coating more protective than the yellow dichromate coating on fasteners? Based on the mean weight loss from the data in Tables 6 and 7 and the mean thickness from data in Tables 4 and 5, it is evident that coating bolt electrolytically with clear zinc chromate is not enough to provide good corrosion resistance. In most industrial settings, the choice of coating bolts with clear zinc chromate or yellow zinc dichromate is an economical one. Based on these findings, it seems worthwhile to plate bolts with yellow zinc dichromate as it has better corrosion resistance. As noted by Uhlig and Revie (1985) the direct costs of replacing damaged equipment due to failure of corroded bolts can be eliminated by protecting them against corrosion. The

researcher suggests that yellow zinc dichromate could be a solution to the problem.

### Question Three

Third research question was: In a salt spray test, does higher concentration of salt cause more corrosion on parts? In order to answer this question, data from Tables 4, 5, 6, and 7 need to be examined. Also data in Table 4 and 5 were constructed in order to provide the researcher with the lowest mean thickness loss. These tests were conducted with low and high salt concentration respectively. When comparing data in both tables, it can be concluded that:

1. In salt spray test, higher salt concentration does cause more corrosion.
2. With low salt concentration, the least thickness loss was 0.186 for thin bolts plated electrolytically with yellow dichromate zinc and 0.188 for thick bolts plated electrolytically with yellow zinc dichromate.
3. With high salt concentration, the least thickness loss was 0.057 for thick and 0.085 for thin bolt plated with yellow zinc dichromate. Comparing these figures, the researcher could not claim that high concentration of salt in a salt spray test does cause more corrosion. May be there were other factors in the interaction between the treatment, such as the thickness and the salt concentration that caused this inconclusiveness.

The data in Tables 6 and 7 were also examined in order to support this claim. These data were derived from tests conducted in order to evaluate the mean weight loss (Loss 2). These tests were conducted with low and high salt concentration respectively. Data from Table 6 were generated using the results of a test conducted with low salt concentration; the results were: 1.016 and 0.714 as the least weight losses. The 1.21 was the percentage mean weight loss for bolts coated with a thin layer of yellow dichromate zinc and 0.84 was the percentage mean weight loss for bolts coated with a thick layer of yellow dichromate zinc. Data from Table 7 were also generated with the results of a test conducted with high salt concentration. The results given were: 0.017 and 0.037 for the least weight losses. The result 0.02 was the percentage mean weight loss for fasteners coated with a thin layer of yellow dichromate zinc and 0.04 was the percentage mean weight loss for fasteners coated with a thick layer of yellow dichromate zinc. Here again, a comparison of both tables reveals that higher salt concentration in the salt spray test caused parts to corrode more. However, it can also be argued that, based on the data shown in Tables 4 and 5, higher salt concentration in a salt spray does cause parts to lose their thickness thereby losing their protection against corrosion.



#### Question Four

The fourth research question was: What is the effect of coating thickness on the corrosion resistance of zinc coated medium carbon steel bolts? In order to answer this question, data from Tables 4, 5, 6 and 7 were examined.

1. Considering the data in Table 4, the least thickness loss in a test run with low salt concentration with the yellow dichromate-coated bolts with both the thin and thick coating, the question cannot be answered for the least thickness loss was equal to 0.186 for both.

2. Considering the data in Table 5, the least thickness loss in a test run with high salt concentration solution was 0.057, for the yellow dichromate with thick coating.

3. The data in Table 6 illustrate that the least amount of weight loss in a test run with low salt concentration solution was 0.714, for the yellow dichromate with a thick coating.

4. Finally, considering the data in Table 7, the least amount of weight loss in a test run with high salt concentration solution was 0.017; the yellow dichromate with a thin coating. Based on these categorical results, it was concluded that a thick coating of yellow dichromate would provide a good corrosion resistance to fasteners than a thin one. Again, this also depends on the interaction effects between the salt, the treatment, and the thickness.

Question Five

The fifth research question was: What is the appropriate coating thickness for medium carbon steel bolts? The data in Tables 4, 5, 6, and 7 reveal the following:

1. The least minimum thickness loss in Table 4 for a test run with low salt concentration was 0.186 and 0.188 (the yellow dichromate) with both thin and thick coatings respectively. Based on those results, one cannot determine for sure the appropriate coating thickness for a bolt; but can say at least a 0.0002" coating thickness with the yellow dichromate would ensure an adequate protection because the minimum required thickness of this coating was 0.0002".

Considering the data in Table 5, the least thickness loss for test run with high salt concentration in the main tank was 0.057 or 5.6% thickness loss (the yellow dichromate) with a thick coating. The least weight loss from data in Table 6 for a test run with low salt concentration in the main tank is 0.714 or 0.84% weight loss (the yellow dichromate) with a thick coating.

Finally, the least amount of weight loss from data in Table 7 for a test run with high salt concentration in the main tank was 0.017 or 0.02% weight loss. Here again the thin coating passed the test. This result was sufficient enough to conclude that a minimum coating thickness would provide adequate protection for a bolt if it was coated with yellow dichromate zinc.

CHAPTER 5  
CONCLUSIONS AND RECOMMENDATIONS

Conclusions

This study was undertaken with two stated problems:

1. To investigate the correlation between plating thickness and corrosion resistance of medium carbon fasteners under salt spray conditions on two types of zinc.
2. To compare the level of protection of clear chromate coating and the yellow dichromate zinc on medium carbon steel bolts under salt spray moisture conditions.

Upon reviewing the available literature, the researcher concluded that by testing the various coated bolts according to the ASTM B117 standard data would be obtained to determine if a correlation existed. Furthermore, a correlation could be established between the corrosion resistance and the types of coating. The various tests run during this investigation proved that one type of coating is better than the other in terms of longer periods of protection and a weather condition could be a determinant factor in the longevity of a plated fastener. The two dependent variables, loss in thickness (Loss 2) and loss in weight (Loss 1), appeared to be of limited value. The independent variables with their subcategories also were limited in their choice. Data in Tables (4, 5, 6 and 7) proved that some predictions could be made concerning the yellow zinc coating. The data in Table 4 clearly

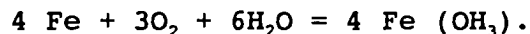
supported Zaki (1988) who reported, as noted in the review of literature, that the single-dip clear chromate films contain a lower content of chrome and offer less corrosion protection than the colored types. Let us recall that the yellow dichromate plating on the medium carbon steel bolts experience less percentage weight and thickness loss in the salt spray testing of this experiment. In other words, data in Table 4 revealed that yellow zinc is a coating that can provide the maximum time of protection for medium carbon steel bolts in both high and low salt concentration atmosphere.

As far as the data in Table 5 were concerned, these findings were also in agreement with Zaki's (1988) conclusions regarding color and appearance. The yellow dichromate plated bolts lost their color after being subjected to the salt spray test in the fog chamber. This loss of color could be translated to the loss of thickness which in return meant less protection against corrosion. Zaki stated that failure to meet corrosion resistance for the particular coating applied could be due to insufficient thickness for the required type of chromate, contamination of chromate film with metallic impurities. Some findings related to Nelson (1963) were of great importance during the experiment. The problem of zinc peeling of the base material did not occur. The platers were aware of it and they took Nelson's recommendations to avoid it. The data in

Tables 4 and 5 also illustrated a degree of consistency in the findings of Cook et al. (1992). They investigated various types of coatings on steel bolts. They found that the thickness and uniformity of the coating are important because they determine bolt/nut thread fit. This also determined the longevity of the bolt/nut when they are tightened. Considering the data found in Tables 4 and 5, the clear chromate loss of thickness and loss of weight was a concrete example of the failure that would result due to non-uniformity of the coating because most of it was gone during the test. The data in Table 5 also rated yellow zinc as a coating that can provide a maximum time of protection for medium carbon steel bolts.

As noted in the IFI Reference Book that corrosion occurs by direct chemical attack when the base material is soluble in corrosion medium. Talking about corrosion reactions, Moniz (1992) noted that corrosive environments are electrolytes that contain positive and negative ions. The anode and cathode reactions are the two most important components of corrosion reactions. Moniz stated that in an anode reaction, metal atoms are converted into positively charged ions and electrons, leading to dissolution of the metal. He also noted that in the cathode reaction, specific positively charged ions in the electrolyte consume the electrons. It was previously noted that uniform corrosion occurs on iron or steel items. In this research the items

tested are medium carbon steel bolts, therefore, the type of corrosion that occurred during the experiment would be denoted as uniform corrosion which was supported by Moniz. In the electrochemical reaction of corrosion, the positive charge passing from the metal into the electrolyte at anode locations on the metal surface is balanced by an equal amount of negative charge passing from electrolyte to the metal at cathode locations. In electrical terms, when corrosion occurs, a current flows from the anode to the cathode. Since the anode and cathode currents are equal and opposite, no electric shock is experienced from a corroding system. When the corrosion is general, as was the case with the medium carbon bolts with no coating and tested with both high and low salt concentrated solution, the corrosion is also uniform. In this case, the anode and cathode sites are switching continuously, causing relatively uniform metal loss over the surface. Groshart (1986) noted the chemistry of iron corrosion in the following equation:



This was the case where ferric hydroxide dehydrated to form ferric oxide which is nothing but corrosion. According to the data in Table 6, the control group of bolts corroded during the testing and lost nearly 2% of their weight. This result supported the IFI Reference book notes pertaining to corrosion. Moreover, from the data in Table 6 the bolts

coated with thin and thick clear chromate zinc seem to have lost substantially in terms of percentage weight loss. This result was to be consistent with Zaki (1988) which stated that bolts coated with clear chromate zinc will tend to perform less than the ones coated with yellow dichromate zinc. According to the data in Table 5, bolts coated electrolytically with thin clear chromate zinc and the ones coated electrolytically with thick clear chromate zinc lost 81.4% and 80.9% of their thickness respectively. The bolts coated with thin yellow dichromate zinc and those coated with thick yellow dichromate zinc performed relatively better than the others. Their results were 1.21% and 0.84% respectively. It was concluded that the yellow dichromate zinc had a good protection in salt spray testing. The data in Table 6 also revealed yellow zinc as the best coating capable of resisting corrosion on medium carbon steel bolts.

According to the data in Table 7, the bolts coated electrolytically with thin clear chromate zinc seemed to have lost more percentage weight than those in the control group. This could be explained by two phenomenons:

1. The salt concentration solution was high.
2. The coating on these groups of samples was completely gone and part of the base metal was also attacked by corrosion. These made more loss in terms of weight. These findings proved that bolts plated electrolytically with clear zinc are far less protective than those coated

with yellow dichromate zinc. The data in Table 7 also revealed that bolts coated with thin and thick clear chromate lost 1.27% and 1.49% of their weight respectively. In this group even the ones coated with thick clear chromate lost more than the ones with a thin coating. Bolts coated with thin and thick yellow dichromate lost 0.2% and 0.4% of their weight respectively. Finally, the data in Table 7 also rated yellow dichromate zinc as the best coating capable of resisting corrosion for a long period of time.

Altogether, the data from these four tables enabled the researcher to draw the conclusion that yellow dichromate zinc coating provides better coating on medium carbon steel bolts than the clear chromate zinc. Furthermore, the data from these four tables substantiated Zaki's (1988) findings that was granting 100-300 hours of salt spray resistance to white corrosion for the yellow dichromate zinc.

#### Suggestions and Summary

##### Suggestions

The results of this research demonstrated that life of bolts coated with various types of zinc could be short or long. Should manufacturers consider the geographical location with high moisture content and high pollution in the atmosphere in order to distribute their final products such as cars, tractors, refrigerators, motorcycles, etc.? The answer here is yes.



If considering a dry area such as Arizona, manufacturers may need not worry about plating parts with yellow dichromate zinc. However, they may need to plate their parts with thick clear chromate (0.0003" or above). However, it would be worth spending extra money to coat medium carbon steel bolts with the yellow dichromate zinc.

If considering a coastal area with high humidity and high pollution in the atmosphere such as New York or Los Angeles, the researcher suggests that manufacturers plate their products with yellow dichromate zinc.

As stated before, electrolytes occur naturally in our everyday environments; they are: humidity, fog, dew, condensation, and rain. These electrolytes have a broad variation in their electrical conductivity. It was also noted that the corrosion survival expectancy of steel is enhanced considerably when exposed to the conditions in the high humidity of southern states such as Florida and Texas as opposed to the ultra-dry desert climates of Nevada and Arizona (Moniz, 1992). The fog chamber of the salt spray apparatus was certainly humid; this humidity considered as an electrolyte caused the corrosion on the bolts subjected to various tests. This simulation of various humid regions of the United States proved that parts manufactured for these types of regions should be protected. The results of this study revealed that the yellow dichromate zinc would be the appropriate type of coating. Once again, this research

proved that it is worth spending the extra dollars to plate parts with yellow dichromate zinc.

The plating of parts with yellow dichromate zinc not only provides protection against corrosion but it also eliminates unnecessary expenditures associated with corroded parts. For example, a corroded part can fail and break a piece of equipment can break down. May be that was the reason why Uhlig and Revie (1985) noted that a better protection of metals, especially fasteners, would bring about improvements in two significant areas. The first area of improvement would be economic: that which is caused by the prevention of direct and indirect losses. As stated before in Chapter One, the direct losses include the costs involved in replacing corroded parts in most industrial settings. Moreover, there is also a direct cost associated with tooling losses due to corrosion. Uhlig and Revie estimated the approximate losses of tooling costs alone for electrical utilities in the United States of America to be tens of millions of dollars each year. Though the indirect cost of corrosion losses could not be assessed as easily as the direct costs of losses to corrosion, Donovan (1986) estimated the indirect costs of losses to corrosion to be billions of dollars each year.

A corroded part can also injure employees on the job site or consumers using products with unprotected fasteners. Thus, this leads to the second area of

improvement that protected fasteners such as the one coated with yellow dichromate zinc can bring about. This area of improvement is the one dealing with the safety of operating equipment. As stated in Chapter One, fasteners are used in many industries and they are used to hold together things such as vessels, automobiles, airplanes, tractors, bridges, etc. If well plated, these fasteners would hold as manufacturers expected them to and thereby avoid accidents that would cost not only dollars, but also human lives.

#### Summary

This study was conducted under the ASTM B117 guidelines for salt spray testing. The different tests run by the researcher rated the yellow dichromate zinc as best choice of coating for medium carbon steel bolts for products going to areas with high humidity and high pollution in the atmosphere. The results of the study also proved that thickness doesn't matter as long as the fasteners are plated with a minimum of 0.0002" thickness of the yellow dichromate zinc.

Furthermore, these results also proved that there definitely is a correlation between the plating thickness and the corrosion resistance of medium carbon fasteners plated electrolytically with zinc: clear chromate or yellow dichromate.

The five research questions were answered based on the results of this investigation. The more precise conclusions regarding these questions in this study were as follows:

1. Medium carbon steel bolts plated electrolytically with yellow dichromate zinc are more corrosion resistant than the one plated with clear chromate zinc. These findings were consistent with Zaki (1988) who found the yellow dichromate zinc as more protective than the clear one.

2. The yellow dichromate zinc coating appeared to be more protective than the clear chromate zinc on medium carbon steel bolts. Looking at Figures 10 and 11 (see Appendix D) it can be noticed that the bolts coated with clear chromate lost 85% of their coating during the test. Corrosion resulted and the thread became brittle.

3. A high salt concentration in the atmosphere is not the only factor that make parts corrode; perhaps the humidity and other pollutants can also be factors contributing to the corrosion of fasteners. However, this research reveals that layers of coating can be lost with a high concentration of salt in the atmosphere. As a matter of fact, the bolts plated with yellow dichromate did lose their bright color and became dull (see Appendix D).

4. Though the minimum thickness required for the protection of medium carbon steel fasteners is 0.0002"

(Zaki, 1988), more protection can be provided for fasteners thicker than 0.0002".

5. In an industrial setting where the question of coating parts with either clear chromate zinc or yellow dichromate zinc seems to be the daily question, the choice now will clearly be that of the yellow dichromate zinc because it is proven to be more protective.

6. The clear chromate zinc does not provide a good protection for medium carbon fasteners beyond a certain period of time estimated at 60 hours in the fog chamber of the salt spray apparatus.

#### Recommendations for Further Study

Based on the outcomes of this study, several recommendations for further studies are made.

1. During the electrolytic plating of medium carbon fasteners, phenomenon called hydrogen embrittlement may occur. It was noted in the Industrial Fasteners Institute book (IFI, 1988) that hydrogen has no beneficial effects on steel; hydrogen can cause serious damage to steel. Hydrogen embrittlement causes fastener failures; it causes fractures of a given fastener into two separate pieces. This phenomenon called hydrogen embrittlement occurs during various stages in the making of a medium carbon steel fastener. It also can occur during fasteners plating, particularly during acid pickling and alkaline cleaning. Before and during the plating process, atomic hydrogen is

absorbed into the fastener's surface. The deposited metallic coating then entraps the hydrogen. When the fasteners are tightened, the hydrogen migrates towards points of highest stress concentration. Pressure builds until the strength of the base metal is exceeded and minutes after, surface rupture occurs. Hydrogen known to be exceptionally mobile, penetrates quickly into the newly formed cracks. This pressure-rupture-penetration cycle continues until the fastener fractures, usually within hours of first stress application.

According to Moniz (1992), embrittlement is the loss of ductility in a metal caused by corrosion. Hydrogen assisted cracking is a form of embrittlement that constitutes a potential problem at service temperatures between  $-5^{\circ}$  F and  $250^{\circ}$  F. At low temperatures, the rate of hydrogen diffusion is extremely low. Higher temperatures encourage hydrogen to diffuse out of the metal rather than enter it. Carbon steel fasteners not only go through heat treatment during the time they are manufactured, but also go through the process of plating thus increasing the chance of hydrogen assisted cracking. A study can be conducted to see whether there exists a correlation between hydrogen caused cracks and the corrosion of fasteners.

2. The researcher would also like to recommend a similar study to investigate the correlation between plating thickness of nuts plated electrolytically with clear

chromate zinc or yellow dichromate zinc and their corrosion resistance using a wide range of thickness.

3. Another area of interest would be the correlation between wrenching torque and plating thickness of bolts.

4. Another study similar to this one can be done with temperature incorporated as another independent variable. The reason for this is that engines are put together with plated bolts; during operation, the engine can get as hot as 200 degrees or more.

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**Appendix A**  
**Standard Test Method of Salt Spray (Fog) Testing**



Designation: B 117 - 90

## Standard Test Method of Salt Spray (Fog) Testing<sup>1</sup>

This standard is issued under the fixed designation B 117; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

*This standard has been approved for use by agencies of the Department of Defense to replace Method 811.1 of Federal Test Method Standard No. 131B. Consult the DoD Index of Specifications and Standards for the specific year of issue that has been adopted by the Department of Defense.*

### 1. Scope

1.1 This test method sets forth the conditions required in salt spray (fog) testing for specification purposes. Suitable apparatus which may be used to obtain these conditions is described in Appendix X1. The method does *not* prescribe the type of test specimen or exposure periods to be used for a specific product, nor the interpretation to be given to the results. It should be noted that there is seldom a direct relation between salt spray (fog) resistance and resistance to corrosion in other media, because the chemistry of the reactions, including the formation of films and their protective value, frequently varies greatly with the precise conditions encountered. Comments on the use of the test in research will be found in Appendix X2. For evaluation of corrosive conditions, see Appendix X3.

NOTE 1—This method is applicable to salt spray (fog) testing of ferrous and nonferrous metals, and is also used to test inorganic and organic coatings, etc., especially where such tests are the basis for material or product specifications.

1.2 The values stated in SI units are to be regarded as standard. The inch-pound units in parentheses are provided for information.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

### 2. Referenced Documents

#### 2.1 ASTM Standards:

- B 287 Method of Acetic Acid-Salt Spray (Fog) Testing<sup>2</sup>
- B 368 Method for Copper-Accelerated Acetic Acid-Salt Spray (Fog) Testing (CASS Test)<sup>3</sup>

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee G-1 on Corrosion of Metals, and is the direct responsibility of Subcommittee G01.05 on Laboratory Corrosion Tests.

Current edition approved March 30, 1990. Published May 1990. Originally published as B 117 - 39 T. Last previous edition B 117 - 85<sup>11</sup>.

<sup>2</sup> Discontinued—See 1987 Annual Book of ASTM Standards, Vols 02.05 and 03.02.

<sup>3</sup> Annual Book of ASTM Standards, Vol 02.05.

- D 609 Methods for Preparation of Steel Panels for Testing Paint, Varnish, Lacquer, and Related Products<sup>4</sup>
- D 1193 Specification for Reagent Water<sup>5</sup>
- D 1654 Method for Evaluation of Painted or Coated Specimens Subjected to Corrosive Environments<sup>4</sup>
- E 70 Test Method for pH of Aqueous Solutions with the Glass Electrode<sup>6</sup>

### 3. Apparatus

3.1 The apparatus required for salt spray (fog) testing consists of a fog chamber, a salt solution reservoir, a supply of suitably conditioned compressed air, one or more atomizing nozzles, specimen supports, provision for heating the chamber, and necessary means of control. The size and detailed construction of the apparatus are optional, provided the conditions obtained meet the requirements of this method.

3.2 Drops of solution which accumulate on the ceiling or cover of the chamber shall not be permitted to fall on the specimens being tested.

3.3 Drops of solution which fall from the specimens shall not be returned to the solution reservoir for respraying.

3.4 Material of construction shall be such that it will not affect the corrosiveness of the fog.

### 4. Test Specimens

4.1 The type and number of test specimens to be used, as well as the criteria for the evaluation of the test results, shall be defined in the specifications covering the material or product being tested or shall be mutually agreed upon by the purchaser and the seller.

### 5. Preparation of Test Specimens

5.1 Metallic and metallic-coated specimens shall be suitably cleaned. The cleaning method shall be optional depending on the nature of the surface and the contaminants, except that it shall not include the use of abrasives other than a paste of pure magnesium oxide nor of solvents which are corrosive or will deposit either corrosive or protective films. The use of a nitric acid solution for the chemical cleaning, or passivation, of stainless steel specimens is permissible when agreed upon by the purchaser and the seller. Care shall be taken that specimens are not recontaminated after cleaning by excessive or careless handling.

<sup>4</sup> Annual Book of ASTM Standards, Vol 06.01.

<sup>5</sup> Annual Book of ASTM Standards, Vol 11.01.

<sup>6</sup> Annual Book of ASTM Standards, Vol 15.05.



5.2 Specimens for evaluation of paints and other organic coatings shall be prepared in accordance with applicable specification(s) for the material(s) being tested, or as agreed upon by the purchaser and supplier. Otherwise, the test specimens shall consist of steel meeting the requirements of Methods D 609 and shall be cleaned and prepared for coating in accordance with applicable procedure of Methods D 609.

5.3 Specimens coated with paints or nonmetallic coatings shall not be cleaned or handled excessively prior to test.

5.4 Whenever it is desired to determine the development of corrosion from an abraded area in the paint or organic coating, a scratch or scribed line shall be made through the coating with a sharp instrument so as to expose the underlying metal before testing. The conditions of making the scratch shall be as defined in Method D 1654, unless otherwise agreed upon between the purchaser and seller.

5.5 Unless otherwise specified, the cut edges of plated, coated, or duplex materials and areas containing identification marks or in contact with the racks or supports shall be protected with a suitable coating stable under the conditions of the test, such as ceresin wax.

NOTE 2—Should it be desirable to cut test specimens from parts or from preplated, painted, or otherwise coated steel sheet, the cut edges shall be protected by coating them with paint, wax, tape, or other effective media so that the development of a galvanic effect between such edges and the adjacent plated or otherwise coated metal surfaces, is prevented.

## 6. Position of Specimens During Test

6.1 The position of the specimens in the salt spray chamber during the test shall be such that the following conditions are met:

6.1.1 Unless otherwise specified, the specimens shall be supported or suspended between 15 and 30° from the vertical and preferably parallel to the principal direction of horizontal flow of fog through the chamber, based upon the dominant surface being tested.

6.1.2 The specimens shall not contact each other or any metallic material or any material capable of acting as a wick.

6.1.3 Each specimen shall be so placed as to permit free settling of fog on all specimens.

6.1.4 Salt solution from one specimen shall not drip on any other specimen.

NOTE 3—Suitable materials for the construction or coating of racks and supports are glass, rubber, plastic, or suitably coated wood. Bare metal shall not be used. Specimens shall preferably be supported from the bottom or the side. Slotted wooden strips are suitable for the support of flat panels. Suspension from glass hooks or waxed string may be used as long as the specified position of the specimens is obtained, if necessary by means of secondary support at the bottom of the specimens.

## 7. Salt Solution

7.1 The salt solution shall be prepared by dissolving  $5 \pm 1$  parts by weight of sodium chloride in 95 parts of water conforming to Type IV water in Specification D 1193. The salt used shall be sodium chloride substantially free of nickel and copper and containing on the dry basis not more than 0.1% of sodium iodide and not more than 0.3% of total

impurities. Some salts contain additives that may act as corrosion inhibitors; careful attention should be given to the chemical content of the salt. Upon agreement between purchaser and seller, analysis may be required and limits established for elements or compounds not specified in the chemical composition given above.

7.2 The pH of the salt solution shall be such that when atomized at 35°C (95°F) the collected solution will be in the pH range of 6.5 to 7.2 (Note 4). Before the solution is atomized it shall be free of suspended solids (Note 5). The pH measurement shall be made electrometrically at 25°C (77°F) using a glass electrode with a saturated potassium chloride bridge in accordance with Method E 70, or colorimetrically using bromothymol blue as indicator, or short range pH paper which reads in 0.2 or 0.3 of a pH unit (Note 6).

NOTE 4—Temperature affects the pH of a salt solution prepared from water saturated with carbon dioxide at room temperature and pH adjustment may be made by the following three methods:

(1) When the pH of a salt solution is adjusted at room temperature, and atomized at 35°C (95°F), the pH of the collected solution will be higher than the original solution due to the loss of carbon dioxide at the higher temperature. When the pH of the salt solution is adjusted at room temperature, it is therefore necessary to adjust it below 6.5 so the collected solution after atomizing at 35°C (95°F) will meet the pH limits of 6.5 to 7.2. Take about a 50-mL sample of the salt solution as prepared at room temperature, boil gently for 30 s, cool, and determine the pH. When the pH of the salt solution is adjusted to 6.5 to 7.2 by this procedure, the pH of the atomized and collected solution at 35°C (95°F) will come within this range.

(2) Heating the salt solution to boiling and cooling to 95°F for maintaining it at 95°F for approximately 48 h before adjusting the pH produces a solution the pH of which does not materially change when atomized at 35°C (95°F).

(3) Heating the water from which the salt solution is prepared to 35°C (95°F) or above, to expel carbon dioxide, and adjusting the pH of the salt solution within the limits of 6.5 to 7.2 produces a solution the pH of which does not materially change when atomized at 35°C (95°F).

NOTE 5—The freshly prepared salt solution may be filtered or decanted before it is placed in the reservoir, or the end of the tube leading from the solution to the atomizer may be covered with a double layer of cheesecloth to prevent plugging of the nozzle.

NOTE 6—The pH can be adjusted by additions of dilute copper hydrochloric acid or copper sodium hydroxide solutions.

## 8. Air Supply

8.1 The compressed air supply to the nozzle or nozzles for atomizing the salt solution shall be free of oil and dirt (Note 7) and maintained between 69 and 172 kN/m<sup>2</sup> (10 and 25 psi) (Note 8).

NOTE 7—The air supply may be freed from oil and dirt by passing it through a water scrubber or at least 610 mm (2 ft) of suitable cleaning material such as sheep's wool, excelsior, slag wool, or activated alumina.

NOTE 8—Atomizing nozzles may have a "critical pressure" at which an abnormal increase in the corrosiveness of the salt fog occurs. If the "critical pressure" of a nozzle has not been established with certainty, control of fluctuation in the air pressure within plus or minus 0.7 kN/m<sup>2</sup> (0.1 psi), by installation of a suitable pressure regulator valve<sup>7</sup> mini-

<sup>7</sup> Registered U. S. Patent Office.

<sup>8</sup> The Nullimatic pressure regulator (or equivalent) manufactured by Moore Products Co., H and Lycoming Sts., Philadelphia, PA 19124, is suitable for this purpose.

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mizes the possibility that the nozzle will be operated at its "critical pressure."<sup>9</sup>

### 9. Conditions in the Salt Spray Chamber

**9.1 Temperature**—The exposure zone of the salt spray chamber shall be maintained at  $35 \pm 1.1 - 1.7^\circ\text{C}$  ( $95 \pm 2 - 3^\circ\text{F}$ ). The temperature within the exposure zone of the closed cabinet shall be recorded at least twice a day at least 7 h apart (except on Saturdays, Sundays, and holidays when the salt spray test is not interrupted for exposing, rearranging, or removing test specimens or to check and replenish the solution in the reservoir).

**NOTE 9**—A suitable method to record the temperature is by a continuous recording device or by a thermometer which can be read from outside the closed cabinet. The recorded temperature must be obtained with the salt spray chamber to avoid a false low reading because of wet-bulb effect when the chamber is open.

**9.2 Atomization and Quantity of Fog**—At least two clean fog collectors shall be so placed within the exposure zone that no drops of solution from the test specimens or any other source shall be collected. The collectors shall be placed in the proximity of the test specimens, one nearest to any nozzle and the other farthest from all nozzles. The fog shall be such that for each  $80\text{ cm}^2$  of horizontal collecting area there will be collected in each collector from 1.0 to 2.0 mL of solution per hour based on an average run of at least 16 h (Note 10). The sodium chloride concentration of the collected solution shall be  $5 \pm 1$  weight % (Note 11). The pH of the collected solution shall be 6.5 to 7.2. The pH measurement shall be made electrometrically or colorimetrically using bromothymol blue as the indicator.

**NOTE 10**—Suitable collecting devices are glass funnels with the stems inserted through stoppers into graduated cylinders or crystallizing dishes. Funnels and dishes with a diameter of 10 cm have an area of about  $80\text{ cm}^2$ .

**NOTE 11**—A solution having a specific gravity of 1.0255 to 1.0400 at  $25^\circ\text{C}$  ( $77^\circ\text{F}$ ) will meet the concentration requirement. The concentration may also be determined as follows: Dilute 5 mL of the collected solution to 100 mL with distilled water and mix thoroughly; pipet a 10-mL aliquot into an evaporating dish or casserole; add 40 mL of distilled water and 1 mL of 1 % potassium chromate solution (chloride-free) and titrate with 0.1 N silver nitrate solution to the first appearance of a permanent red coloration. A solution that requires between 3.4 and 5.1 mL of 0.1 N silver nitrate solution will meet the concentration requirements.

**9.3** The nozzle or nozzles shall be so directed or baffled that none of the spray can impinge directly on the test specimens.

### 10. Continuity of Test

**10.1** Unless otherwise specified in the specifications covering the material or product being tested, the test shall be continuous for the duration of the entire test period. Continuous operation implies that the chamber be closed and the spray operating continuously except for the short daily

interruptions necessary to inspect, rearrange, or remove test specimens, to check and replenish the solution in the reservoir, and to make necessary recordings as described in Section 9. Operations shall be so scheduled that these interruptions are held to a minimum.

### 11. Period of Test

**11.1** The period of test shall be as designated by the specifications covering the material or product being tested or as mutually agreed upon between the purchaser and the seller.

**NOTE 12**—Recommended exposure periods are to be as agreed upon by the purchaser and seller, but exposure periods of multiples of 24 h are suggested.

### 12. Cleaning of Tested Specimens

**12.1** Unless otherwise specified in the specifications covering the material or product being tested, specimens shall be treated as follows at the end of the test:

**12.1.1** The specimens shall be carefully removed.

**12.2** Specimens may be gently washed or dipped in clean running water not warmer than  $38^\circ\text{C}$  ( $100^\circ\text{F}$ ) to remove salt deposits from their surface, and then immediately dried. Drying shall be accomplished with a stream of clean, compressed air.

### 13. Evaluation of Results

**13.1** A careful and immediate examination shall be made for the extent of corrosion of the dry test specimens or for other failure as required by the specifications covering the material or product being tested or by agreement between the purchaser and the seller.

### 14. Records and Reports

**14.1** The following information shall be recorded, unless otherwise prescribed in the specifications covering the material or product being tested:

**14.1.1** Type of salt and water used in preparing the salt solution,

**14.1.2** All readings of temperature within the exposure zone of the chamber,

**14.1.3** Daily records of data obtained from each fog-collecting device including the following:

**14.1.3.1** Volume of salt solution collected in millimetres per hour per  $80\text{ cm}^2$ ,

**14.1.3.2** Concentration or specific gravity at  $35^\circ\text{C}$  ( $95^\circ\text{F}$ ) of solution collected, and

**14.1.3.3** pH of collected solution.

**14.4** Type of specimen and its dimensions, or number or description of part,

**14.5** Method of cleaning specimens before and after testing,

**14.6** Method of supporting or suspending article in the salt spray chamber,

**14.7** Description of protection used as required in 5.5,

**14.8** Exposure period,

**14.9** Interruptions in test, cause and length of time, and

**14.10** Results of all inspections.

**NOTE 13**—If any of the atomized salt solution which has not contacted the test specimens is returned to the reservoir, it is advisable to record the concentration or specific gravity of this solution also.

<sup>9</sup> It has been observed that periodic fluctuations in air pressure of  $\pm 3.4\text{ kN/m}^2$  (0.5 psi) resulted in about a twofold increase in the consistency of the fog from a nozzle which was being operated at an average pressure of  $110\text{ kN/m}^2$  (16 psi). Controlling the fluctuations within  $\pm 0.7\text{ kN/m}^2$  (0.1 psi), however, avoided any increase in the consistency of the salt fog. See Durney, V. M. and Cavanagh, W. R., "Apparatus and Factors in Salt Fog Testing," *Proceedings, ASTM*, Vol 48, 1948, p. 153.

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15. Precision and Bias<sup>10</sup>

15.1 The reproducibility of results in the salt spray test is highly dependent on the type of specimens tested and the evaluation criteria selected as well as the control of the operating variables. In any testing program, sufficient replicates should be included to establish the variability of the results. Substantial variability is often observed when similar specimens are tested in different fog chambers even though

the testing conditions are nominally similar and within the ranges specified in this method.

15.2 The salt spray (fog) test is intended to reproduce the corrosion that occurs in atmospheres containing salt spray or splash. It has been widely observed, however, that rankings of different alloys or coating systems, or both, do not necessarily fall in the same order as atmospheric tests in marine or road salt splash environments. This test has been more useful in rating the relative resistance of a specific type of protective coating, for example, hot-dip zinc coatings on steel. Interpretation of the results of this method beyond this purpose must be verified by actual exposure tests.

<sup>10</sup> Supporting data are available on loan from ASTM Headquarters. Request RR: G01-1003.

## APPENDICES

## (Nonmandatory Information)

## X1. CONSTRUCTION OF APPARATUS

## X1.1 Cabinets

X1.1.1 Standard salt spray cabinets are available from several suppliers, but certain pertinent accessories are required before they will function according to this method and provide consistent control for duplication of results.

X1.1.2 The salt spray cabinet consists of the basic chamber, an air-saturator tower, a salt solution reservoir, atomizing nozzles, specimen supports, provisions for heating the chamber, and suitable controls for maintaining the desired temperature.

X1.1.3 Accessories such as a suitable adjustable baffle or central fog tower, automatic level control for the salt reservoir, and automatic level control for the air-saturator tower are pertinent parts of the apparatus.

X1.1.4 The cabinet should be of sufficient size to test adequately the desired number of parts without overcrowding. Small cabinets have been found difficult to control

and those of less than 0.43-m<sup>3</sup> (15-ft<sup>3</sup>) capacity should be avoided.

X1.1.5 The chamber may be made of inert materials such as plastic, glass, or stone, but most preferably is constructed of metal and lined with impervious plastics, rubber, or epoxy-type materials or equivalent.

## X1.2 Temperature Control

X1.2.1 The maintenance of temperature within the salt chamber can be accomplished by several methods. It is generally desirable to control the temperature of the surroundings of the salt spray chamber and to maintain it as stable as possible. This may be accomplished by placing the apparatus in a constant-temperature room, but may also be achieved by surrounding the basic chamber of a jacket containing water or air at a controlled temperature.

X1.2.2 The use of immersion heaters in an internal salt solution reservoir or of heaters within the chamber is detrimental where heat losses are appreciable, because of solution evaporation and radiant heat on the specimens.

X1.2.3 All piping which contacts the salt solution or spray should be of inert materials such as plastic. Vent piping should be of sufficient size so that a minimum of back pressure exists and should be installed so that no solution is trapped. The exposed end of the vent pipe should be shielded from extreme air currents that may cause fluctuation of pressure or vacuum in the cabinet.

## X1.3 Spray Nozzles

X1.3.1 Satisfactory nozzles may be made of hard rubber, plastic, or other inert materials. The most commonly used type is made of plastic. Nozzles calibrated for air consumption and solution-atomized are available. The operating characteristics of a typical nozzle are given in Table X1.1.

X1.3.2 It can readily be seen that air consumption is relatively stable at the pressures normally used, but a marked reduction in solution sprayed occurs if the level of the solution is allowed to drop appreciably during the test. Thus,

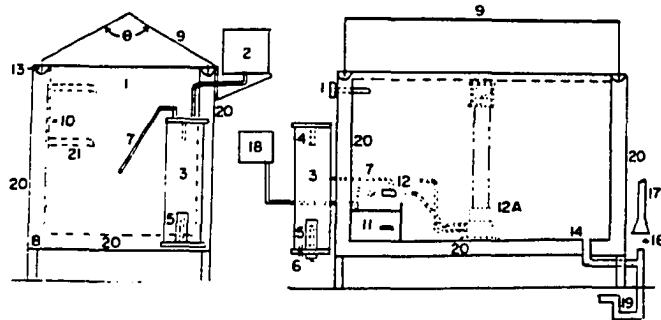
TABLE X1.1 Operating Characteristics of Typical Spray Nozzle

Siphon Height, in.	Air Flow, L/min				Solution Consumption, mL/h			
	Air Pressure, psi				Air Pressure, psi			
	5	10	15	20	5	10	15	20
4	19	26.5	31.5	36	2100	3840	4584	5256
8	19	26.5	31.5	36	636	2760	3720	4320
12	19	26.5	31.5	36	0	1380	3000	3710
16	19	26.6	31.5	36	0	780	2124	2904

Siphon Height, cm	Air Flow, dm <sup>3</sup> /min				Solution Consumption, cm <sup>3</sup> /h			
	Air Pressure, kPa				Air Pressure, kPa			
	34	69	103	138	34	69	103	138
10	19	26.5	31.5	36	2100	3840	4584	5256
20	19	26.5	31.5	36	636	2760	3720	4320
30	19	26.5	31.5	36	0	1380	3000	3710
40	19	26.6	31.5	36	0	780	2124	2904

B 117



- 4—Angle of lid, 90 to 125°
- 1—Thermometer and thermostat for controlling heater (item No. 8) in base
- 2—Automatic water leveling device
- 3—Humidifying tower
- 4—Automatic temperature regulator for controlling heater (item No. 5)
- 5—Immersion heater, nonrusting
- 6—Air inlet, multiple openings
- 7—Air tube to spray nozzle
- 8—Strip heater in base
- 9—Hinged top, hydraulically operated, or counterbalanced
- 10—Brackets for rods supporting specimens, or test table
- 11—Internal reservoir
- 12—Spray nozzle above reservoir, suitably designed, located, and baffled
- 12A—Spray nozzle housed in dispersion tower located preferably in center of cabinet
- 13—Water seal
- 14—Combination drain and exhaust. Exhaust at opposite side of test space from spray nozzle (item 12), but preferably in combination with drain, waste trap, and forced draft waste pipe (items 16, 17, and 19).
- 15—Complete separation between forced draft waste pipe (item 17) and combination drain and exhaust (items 14 and 19) to avoid undesirable suction or back pressure
- 16—Forced draft waste pipe
- 17—Automatic leveling device for reservoir
- 18—Waste trap
- 19—Waste trap
- 20—Air space or water jacket
- 21—Test table or rack, well below roof area

FIG. X1.1 Typical Salt Spray Cabinet

the level of the solution in the salt reservoir must be maintained automatically to ensure uniform fog delivery during the test.<sup>11</sup>

X1.3.3 If the nozzle selected does not atomize the salt solution into uniform droplets, it will be necessary to direct the spray at a baffle or wall to pick up the larger drops and prevent them from impinging on the test specimens. Pending a complete understanding of air-pressure effects, etc., it is important that the nozzle selected shall produce the desired condition when operated at the air pressure selected. Nozzles are not necessarily located at one end, but may be placed in the center and can also be directed vertically up through a suitable tower.

**X1.4 Air for Atomization**

X1.4.1 The air used for atomization must be free of grease, oil, and dirt before use by passing through well-maintained filters. Room air may be compressed, heated, humidified, and washed in a water-sealed rotary pump, if the temperature of the water is suitably controlled. Otherwise

cleaned air may be introduced into the bottom of a tower filled with water, through a porous stone or multiple nozzles. The level of the water must be maintained automatically to ensure adequate humidification. A chamber operated according to this method and Appendix will have a relative humidity between 95 and 98 %. Since salt solutions from 2 to 6 % will give the same results (though for uniformity the limits are set at 4 to 6 %), it is preferable to saturate the air at temperatures well above the chamber temperature as insurance of a wet fog. Table X1.2 shows the temperatures, at different pressures, that are required to offset the cooling effect of expansion to atmospheric pressure.

X1.4.2 Experience has shown that most uniform spray chamber atmospheres are obtained by increasing the atom-

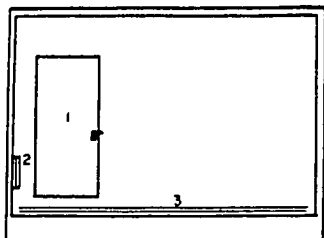
TABLE X1.2 Temperature and Pressure Requirements for Operation of Test at 95°F

	Air Pressure, psi			
	12	14	16	18
Temperature, °F	114	117	119	121
	Air Pressure, kPa			
	83	96	110	124
Temperature, °C	46	47	48	49

<sup>11</sup> A suitable device for maintaining the level of liquid in either the saturator tower or reservoir of test solution may be designed by a local engineering group, or may be purchased from manufacturers of test cabinets as an accessory.



## B 117



NOTE—The controls are the same, in general as for the laboratory cabinet (Fig. X1.1), but are sized to care for the larger cube. The chamber has the following features:

- (1) Heavy insulation.
- (2) Refrigeration door with drip rail, or pressure door with drip rail, inward-swinging sill.
- (3) Low-temperature auxiliary heater, and
- (4) Duck boards on floor, with floor sloped to combination drain and air exhaust.

FIG. X1.2 Walk-in Chamber, 1.5 by 2.4 m (5 by 8 ft) and Upward in Overall Size

izing air temperature sufficiently to offset heat losses, except those that can be replaced otherwise at very low-temperature gradients.

### X1.5 Types of Construction

X1.5.1 A modern laboratory cabinet is shown in Fig.

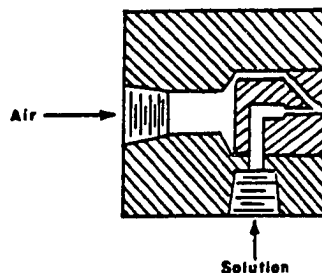


FIG. X1.3 Typical Spray Nozzle

X1.1. Walk-in chambers are not usually constructed with a sloping ceiling due to their size and location. Suitably located and directed spray nozzles avoid ceiling accumulation and drip. Nozzles may be located at the ceiling, or 0.91 m (3 ft) from the floor directed upward at 30 to 60° over a passageway. The number of nozzles depends on type and capacity and is related to the area of the test space. An 11- to 19-dm<sup>3</sup> (3- to 5-gal) reservoir is required within the chamber, with the level controlled. The major features of a walk-in type cabinet, which differs significantly from the laboratory type, are illustrated in Fig. X1.2. Construction of a plastic nozzle, such as is furnished by several suppliers, is shown in Fig. X1.3.

## X2. USE OF THE SALT SPRAY (FOG) TEST IN RESEARCH

X2.1 The detailed requirements of this method are primarily for quality acceptance and should not be construed as the optimum conditions for research studies. The test has been used to a considerable extent for the purpose of comparing different materials or finishes with an acceptable standard. The recent elimination of many cabinet variables and the improvement in controls have made the three ASTM Salt Spray Tests: Method B 117, B 287, and B 368, into useful tools for many industrial and military production and qualification programs.

X2.2 The test has been used to a considerable extent for the purpose of comparing different materials or finishes. It should be noted that there is seldom a direct relation between salt spray (fog) resistance and resistance to corrosion in other media, because the chemistry of the reactions, including the formation of films and their protective value, frequently varies greatly with the precise conditions encountered. Informed personnel are aware of the erratic composition of basic alloys, the possibility of wide variations in quality and thickness of plated items produced on the same racks at the same time, and the consequent need for a mathematical determination of the number of specimens required to constitute an adequate sample for test purposes. In this connection it is well to point out that Method B 117 is not applicable to the study or testing of decorative chromium

plate (nickel-chromium or copper-nickel-chromium) on steel or on zinc-base die castings or of cadmium plate on steel. For this purpose Methods B 287 and B 368 are available, which are also considered by some to be superior for comparison of chemically-treated aluminum (chromated, phosphated, or anodized), although final conclusions regarding the validity of test results related to service experience have not been reached. Method B 117 is considered to be most useful in estimating the relative behavior of closely related materials in marine atmospheres, since it simulates the basic conditions with some acceleration due to either wetness or temperature or both.

X2.3 When a test is used for research, it may prove advantageous to operate with a different solution composition or concentration or at a different temperature. In all cases, however, it is desirable to control the temperature and humidity in the manner specified, and to make certain that the composition of the settled fog and that of the solution in the reservoir are substantially the same. Where differences develop, it is necessary to control conditions so that the characteristics of the settled fog meet the specified requirements for the atmosphere.

X2.4 Material specifications should always be written in terms of the standard requirements of the appropriate salt spray method, thereby making it possible to test a variety of materials from different sources in the same equipment.



### X3. EVALUATION OF CORROSIVE CONDITIONS

**X3.1 General**—This appendix covers test panels and procedures for evaluating the corrosive conditions within a salt spray cabinet. The procedure involves the exposure of steel test panels and the determination of their mass losses in a specified period of time. This may be done monthly or more frequently to insure consistent operation over time. It is also useful for correlating the corrosive conditions among different cabinets.

**X3.2 Test Panels**—The required test panels, 76 mm by 127 mm by 0.8 mm, are made from SAE 1010 commercial grade cold-rolled carbon steel (UNS G10080).

**X3.3 Preparation of Panels Before Testing**—Clean panels before testing by degreasing only, so that the surfaces are free of dirt, oil, or other foreign matter that could influence the test results. After cleaning, weigh each panel on an analytical balance to the nearest 1.0 mg and record the mass.

**X3.4 Positioning of Test Panels**—Place a minimum of two weighed panels in the cabinet, with the 127-mm length supported 30° from vertical. Place the panels in the prox-

imity of the condensate collectors. (See Section 6.)

**X3.5 Duration of Test**—Expose panels to the salt fog for 48 to 168 h.

**X3.6 Cleaning of Test Panels After Exposure**—After removal of the panels from the cabinet, rinse each panel immediately with running tap water to remove salt, and rinse in reagent grade water (see Specification D 11 3 Type IV). Chemically clean each panel for 10 min at 20–25°C in a fresh solution prepared as follows:

Mix 1000 mL of hydrochloric acid (Sp. Gr. 1.19) with 1000 mL reagent grade water (D 1193, Type IV) and add 10 grams of hexamethylene tetramine. After cleaning, rinse each panel with reagent grade water (Type IV) and dry (see 12.2).

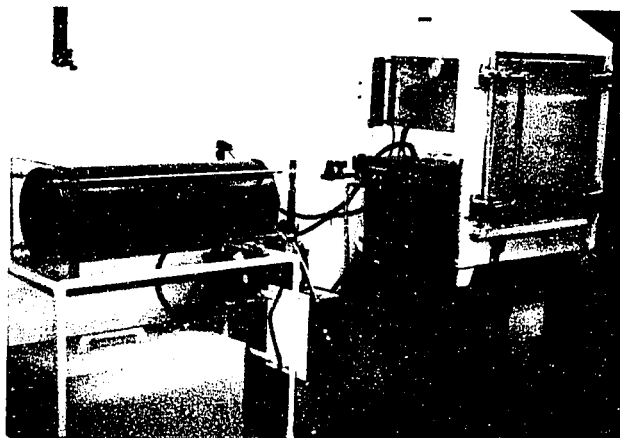
**X3.7 Determining Mass Loss**—Immediately after drying, determine the mass loss by reweighing and subtracting panel mass after exposure from its original mass. The mass should not vary 20 % from test to test.

**X3.7.1** Data generated in the Interlaboratory Study using this method are available from ASTM as Research Report.

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of the standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.

**Appendix B**  
**Salt Spray Testing Apparatus**



**Appendix C**  
**Computer Generated Output for the Study**

The SAS System

Obs	ID	TET	SALT	WGTBEF	WGTAFY	THICKBEF	THICKAFY	THICKNS	LOSS1	LOSS2
1	1	1	1	83.62	81.94	0.11	0.11	0	1.68	0.00
2	1	1	1	85.67	81.95	0.12	0.12	0	1.72	0.00
3	1	1	1	83.62	81.93	0.11	0.11	0	1.69	0.00
4	1	1	1	83.61	81.98	0.10	0.10	0	1.63	0.00
5	1	1	1	83.64	81.96	0.11	0.11	0	1.68	0.00
6	1	1	1	83.63	82.02	0.10	0.10	0	1.61	0.00
7	1	1	1	83.66	82.04	0.10	0.10	0	1.62	0.00
8	1	1	1	83.62	81.89	0.10	0.10	0	1.73	0.00
9	1	1	1	83.67	82.02	0.09	0.09	0	1.65	0.00
10	10	1	1	83.57	81.93	0.12	0.12	0	1.64	0.00
11	11	1	1	83.63	81.97	0.11	0.11	0	1.64	0.00
12	12	1	1	83.70	82.04	0.13	0.13	0	1.62	0.00
13	13	1	1	83.58	81.96	0.12	0.12	0	1.62	0.00
14	14	1	1	83.25	82.60	0.11	0.11	0	1.64	0.00
15	15	1	1	83.69	82.05	0.13	0.13	0	1.66	0.00
16	16	1	1	83.52	81.86	0.10	0.10	0	1.62	0.00
17	17	1	1	83.67	82.03	0.12	0.12	0	1.62	0.00
18	18	1	1	83.62	82.00	0.09	0.09	0	1.60	0.00
19	19	1	1	83.80	82.70	0.99	0.99	1	1.10	0.16
20	20	2	2	83.73	82.72	1.04	0.81	1	1.03	0.23
21	21	2	2	83.68	82.63	0.01	0.72	1	1.05	0.29
22	22	2	2	83.77	82.65	0.99	0.78	1	1.06	0.20
23	23	2	2	83.77	82.61	1.04	0.74	1	0.66	0.28
24	24	2	2	83.66	82.61	1.04	0.78	1	1.03	0.28
25	25	2	2	83.14	82.14	0.92	0.81	1	2.00	0.17
26	26	2	2	83.68	83.08	0.97	0.82	1	0.60	0.15
27	27	2	2	83.77	83.05	1.02	0.84	1	0.72	0.18
28	28	2	2	83.73	82.60	0.98	0.81	1	1.17	0.17
29	29	2	2	83.78	82.70	1.01	0.80	1	1.05	0.21
30	30	2	2	83.77	82.50	1.02	0.84	1	1.27	0.18
31	31	2	2	83.63	82.90	0.99	0.88	1	0.73	0.19
32	32	2	2	83.77	82.71	0.97	0.78	1	1.06	0.19
33	33	2	2	83.82	82.72	1.03	0.88	1	1.18	0.19
34	34	2	2	83.76	82.70	1.03	0.87	1	1.06	0.16
35	35	2	2	83.72	82.68	0.97	0.86	1	1.04	0.11
36	36	2	2	83.68	82.64	1.01	0.89	1	1.04	0.12
37	37	2	2	84.25	83.14	1.00	0.80	1	1.05	0.17
38	38	2	2	84.27	83.24	1.03	0.81	1	1.03	0.15
39	39	2	2	84.34	83.31	1.05	0.78	1	1.00	0.14
40	40	2	2	84.16	83.16	1.03	0.84	1	0.64	0.14
41	41	2	2	84.18	83.50	1.05	0.71	1	0.40	0.14
42	42	2	2	84.21	83.61	1.06	0.90	1	1.12	0.27
43	43	2	2	84.28	83.16	1.03	0.81	1	0.64	0.16
44	44	2	2	84.27	83.63	1.01	0.83	1	0.64	0.09
45	45	2	2	84.15	83.16	1.00	0.92	1	0.58	0.18
46	46	2	2	84.17	83.59	0.99	0.85	1	0.53	0.19
47	47	2	2	84.31	83.78	1.04	0.87	1	0.51	0.16
48	48	2	2	84.21	83.70	1.03	0.85	1	0.53	0.17
49	49	2	2	84.12	83.59	0.99	0.82	1	0.53	0.17
50	50	2	2	84.14	83.71	0.99	0.74	1	0.43	0.23
51	51	2	2	84.31	83.64	1.00	0.90	1	0.37	0.10
52	52	2	2	84.24	83.70	1.03	0.92	1	0.34	0.11
53	53	2	2	84.21	83.64	1.01	0.96	1	0.53	0.05
54	54	2	2	84.24	83.64	1.05	0.91	1	0.42	0.14
55	55	2	2	83.83	83.68	0.98	0.74	1	1.25	0.06
56	56	2	2	83.98	82.67	0.97	0.15	1	1.23	0.82

The SAS System

OBS	ID	TRT	SALT	WGTBEF	WGTAFY	THICKBEF	THICKAFY	THICKENS	LOSS1	LOSS2
57	3	J	1	83.87	82.59	0.98	0.17	1	1.28	0.81
58	4	J	1	83.78	82.54	0.97	0.13	1	1.24	0.84
59	5	J	1	83.82	82.74	1.00	0.10	1	1.10	0.90
60	6	J	1	83.80	82.59	1.01	0.10	1	1.21	0.91
61	7	J	1	83.47	82.47	0.97	0.11	1	1.00	0.86
62	8	J	1	83.83	82.61	0.98	0.14	1	1.22	0.84
63	9	J	1	83.77	82.56	1.03	0.13	1	1.21	0.90
64	10	J	1	83.75	82.57	1.00	0.13	1	1.18	0.83
65	11	J	1	83.86	82.64	0.98	0.12	1	1.22	0.87
66	12	J	1	83.81	82.40	0.97	0.10	1	1.21	0.87
67	13	J	1	83.79	82.59	0.99	0.11	1	1.21	0.88
68	14	J	1	83.78	82.66	0.96	0.13	1	1.12	0.85
69	15	J	1	83.87	82.68	0.99	0.14	1	1.27	0.87
70	16	J	1	83.85	82.58	0.98	0.11	1	1.16	0.83
71	17	J	1	83.90	82.54	0.98	0.13	1	1.19	0.88
72	18	J	1	83.86	82.67	0.98	0.10	1	1.19	0.85
73	1	J	1	84.64	83.23	1.03	0.10	1	1.29	0.81
74	2	J	1	84.39	83.10	1.01	0.10	1	1.24	0.89
75	3	J	1	84.27	83.01	1.08	0.13	1	1.41	0.95
76	4	J	1	84.36	82.95	1.08	0.14	1	1.28	0.85
77	5	J	1	84.60	83.12	1.01	0.10	1	1.28	0.85
78	6	J	1	84.43	83.13	1.03	0.10	1	1.31	0.99
79	7	J	1	84.45	83.14	1.09	0.10	1	1.24	0.88
80	8	J	1	84.41	83.13	1.01	0.10	1	1.33	0.89
81	9	J	1	84.11	82.78	1.01	0.13	1	1.33	0.98
82	10	J	1	84.43	83.10	1.11	0.11	1	1.29	0.92
83	11	J	1	84.45	83.16	1.03	0.13	1	1.31	0.91
84	12	J	1	84.34	82.83	1.04	0.13	1	1.29	0.91
85	13	J	1	84.40	83.18	1.03	0.13	1	1.24	0.92
86	14	J	1	84.32	83.17	1.08	0.14	1	1.13	0.92
87	15	J	1	84.43	83.11	1.13	0.15	1	1.12	0.98
88	16	J	1	84.44	83.02	1.04	0.11	1	1.42	0.93
89	17	J	1	84.44	83.20	1.04	0.14	1	1.25	0.90
90	18	J	1	84.27	82.98	1.04	0.14	1	1.29	0.88
91	1	J	1	83.47	82.99	0.13	0.13	0	0.80	0.80
92	2	J	1	83.44	82.94	0.12	0.12	0	0.80	0.80
93	3	J	1	83.88	82.98	0.14	0.14	0	0.80	0.80
94	4	J	1	83.96	82.87	0.13	0.13	0	0.80	0.80
95	5	J	1	83.88	82.84	0.10	0.10	0	0.80	0.80
96	6	J	1	83.86	82.98	0.10	0.10	0	0.80	0.80
97	7	J	1	83.94	82.74	0.13	0.13	0	0.80	0.80
98	8	J	1	83.99	82.97	0.10	0.10	0	0.80	0.80
99	9	J	1	83.68	83.02	0.11	0.11	0	0.80	0.80
100	10	J	1	83.84	82.85	0.10	0.10	0	0.47	0.80
101	11	J	1	83.88	82.91	0.11	0.11	0	0.84	0.80
102	12	J	1	83.88	83.09	0.10	0.10	0	0.74	0.80
103	13	J	1	83.72	82.98	0.11	0.11	0	0.80	0.80
104	14	J	1	83.60	82.95	0.11	0.11	0	0.82	0.80
105	15	J	1	83.63	82.83	0.11	0.11	0	0.82	0.80
106	16	J	1	83.63	83.00	0.14	0.14	0	0.84	0.80
107	17	J	1	83.47	83.10	0.14	0.14	0	0.89	0.80
108	18	J	1	83.47	82.98	0.13	0.13	0	0.81	0.80
109	1	N	N	83.89	83.88	0.93	0.93	0	-0.81	0.89
110	2	N	N	83.67	83.68	0.91	0.91	0	0.83	0.85
111	3	N	N	83.83	83.80	0.98	0.98	1	0.83	0.89
112	4	N	N	83.74	83.71	0.98	0.99	1	0.83	0.89

The SAS System

DBS	ID	TRY	SALT	MCTBEF	MCTAFT	THICKBEF	THICKAFT	THICKRS	LOSS1	LOSS2
123	5			83.63	83.62	0.96	0.81	1	0.01	0.15
124	6			83.75	83.73	0.92	0.83	1	0.02	0.09
125	7			83.79	83.78	0.96	0.87	1	0.01	0.09
126	8			83.77	83.73	1.00	0.94	1	0.04	0.06
127	9			83.73	83.72	1.00	0.94	1	0.01	0.07
128	10			83.79	83.78	0.96	0.94	1	0.01	0.07
129	11			83.71	83.69	0.97	0.88	1	0.02	0.09
130	12			83.83	83.83	0.97	0.84	1	0.02	0.13
131	13			83.61	83.80	1.01	0.84	1	0.01	0.15
132	14			83.81	83.80	1.03	0.93	1	0.02	0.08
133	15			83.74	83.73	1.00	0.97	1	0.01	0.03
134	16			83.87	83.84	0.99	0.94	1	0.03	0.05
135	17			83.82	83.80	1.01	0.93	1	0.02	0.08
136	18			84.12	84.27	1.05	0.89	1	0.05	0.16
137	19			84.23	84.20	1.04	0.93	1	0.03	0.11
138	20			84.23	84.19	1.00	0.91	1	0.04	0.09
139	21			84.16	84.10	1.07	1.01	1	0.06	0.06
140	22			84.21	84.20	1.03	0.98	1	0.01	0.05
141	23			84.23	84.20	1.00	0.97	1	0.03	0.03
142	24			84.38	84.34	1.07	1.00	1	0.04	0.07
143	25			84.29	84.26	1.04	1.01	1	0.03	0.03
144	26			84.23	84.19	1.02	1.00	1	0.04	0.02
145	27			84.30	84.27	1.06	1.00	1	0.03	0.06
146	28			84.09	84.05	1.03	1.01	1	0.03	0.04
147	29			84.19	84.17	1.07	1.02	1	0.04	0.04
148	30			84.19	84.15	1.05	0.99	1	0.04	0.04
149	31			84.19	84.15	1.09	0.99	1	0.04	0.05
150	32			84.21	84.19	1.09	0.99	1	0.04	0.04
151	33			84.24	84.22	1.01	0.98	1	0.02	0.03
152	34			84.27	84.24	1.01	0.97	1	0.01	0.04
153	35			84.11	84.09	0.99	0.94	1	0.01	0.03
154	36			83.84	83.73	1.00	0.94	1	1.13	0.82
155	37			83.79	83.71	0.94	0.86	1	1.08	0.78
156	38			83.87	83.78	0.97	0.88	1	1.09	0.79
157	39			83.82	83.67	0.94	0.81	1	1.15	0.73
158	40			83.74	83.63	0.99	0.83	1	1.15	0.74
159	41			83.68	83.72	1.02	0.86	1	0.94	0.86
160	42			83.84	83.69	0.99	0.88	1	1.15	0.81
161	43			83.84	83.86	1.01	0.97	1	0.98	0.84
162	44			83.36	83.36	1.03	0.91	1	1.00	0.79
163	45			83.74	83.69	1.03	0.86	1	1.07	0.87
164	46			83.77	83.82	1.00	0.88	1	0.95	0.82
165	47			83.84	83.78	1.00	0.87	1	1.06	0.87
166	48			83.84	83.69	1.05	0.89	1	1.05	0.87
167	49			83.74	83.68	1.00	0.89	1	1.04	0.87
168	50			83.76	83.71	0.99	0.88	1	1.05	0.85
169	51			83.80	83.74	1.03	0.89	1	0.99	0.81
170	52			83.68	83.69	1.00	0.88	1	1.00	0.82
171	53			84.17	83.08	1.00	0.88	1	1.29	0.82
172	54			84.01	83.17	1.02	0.86	1	1.04	0.86
173	55			84.42	83.10	1.03	0.81	1	1.32	0.82
174	56			84.38	83.25	1.01	0.82	1	1.23	0.79
175	57			84.33	83.18	1.03	0.82	1	1.15	0.80
176	58			84.43	83.15	1.02	0.83	1	1.28	0.79
177	59			84.33	83.20	1.03	0.88	1	1.13	0.87
178	60			84.38	83.14	1.04	0.89	1	1.24	0.88
179	61			84.46	83.01	1.05	0.81	1	1.67	0.84
180	62			84.27	83.84	1.03	0.86	1	1.23	0.79
181	63			84.37	83.14	1.05	0.88	1	1.23	0.82
182	64			84.24	83.84	1.00	0.87	1	1.22	0.83
183	65			84.41	83.08	1.00	0.86	1	1.22	0.83
184	66			84.35	83.18	1.03	0.82	1	1.21	0.83
185	67			84.34	83.15	1.03	0.84	1	1.21	0.83
186	68			84.41	83.16	1.01	0.88	1	1.25	0.83
187	69			84.04	83.17	1.00	0.87	1	1.29	0.83
188	70			84.34	83.15	1.02	0.88	1	1.19	0.84

Variable	n	Mean	Std Dev	Minimum	Maximum
ID	188	9.50000000	1.2025993	1.00000000	18.00000000
TRY	188	2.20000000	0.7584189	1.00000000	3.00000000
SALT	188	1.50000000	0.5013947	1.00000000	2.00000000
MCTBEF	188	83.7250000	0.3118119	83.25000000	84.25000000
MCTAFT	188	83.8279442	0.3049773	83.26000000	84.33000000
THICKBEF	188	0.9326467	0.3611753	0.80000000	1.20000000
THICKAFT	188	0.9442778	0.3651164	0.80000000	1.20000000
THICKRS	188	1.20000000	0.7584189	1.00000000	2.00000000
LOSS1	188	0.8895554	0.5307893	-0.01000000	2.00000000
LOSS2	188	0.9923889	0.3844816	0.99000000	0.99000000



## The SAS System

TRT=1 SALT=1 THICKNS=0

Variable	N	Mean	Std Dev	Minimum	Maximum
ID	18	9.5000000	5.3385391	1.0000000	18.0000000
WGTBEF	18	83.4094444	0.1000278	83.2500000	83.7000000
WGTAFY	18	82.0105556	0.1570115	81.8600000	82.4000000
THICKBEF	18	0.1088889	0.0132349	0.0800000	0.1300000
THICKAFY	18	0.1088889	0.0132349	0.0800000	0.1300000
LOSS1	18	1.5988889	0.2393344	0.4500000	1.7300000
LOSS2	18	0	0	0	0

TRT=1 SALT=2 THICKNS=0

Variable	N	Mean	Std Dev	Minimum	Maximum
ID	18	9.5000000	5.3385391	1.0000000	18.0000000
WGTBEF	18	83.4222222	0.0580671	83.4800000	83.7200000
WGTAFY	18	82.9477778	0.0921990	82.7400000	83.1000000
THICKBEF	18	0.1216667	0.0188648	0.1000000	0.1600000
THICKAFY	18	0.1216667	0.0188648	0.1000000	0.1600000
LOSS1	18	0.6744444	0.0826205	0.5600000	0.9000000
LOSS2	18	0	0	0	0

TRT=2 SALT=1 THICKNS=1

Variable	N	Mean	Std Dev	Minimum	Maximum
ID	18	9.5000000	5.3385391	1.0000000	18.0000000
WGTBEF	18	83.7300000	0.1610626	83.2700000	84.1600000
WGTAFY	18	82.7144444	0.2213919	82.1600000	83.1100000
THICKBEF	18	1.0055556	0.0279121	0.9700000	1.0400000
THICKAFY	18	0.8200000	0.0441455	0.7200000	0.8900000
LOSS1	18	1.0155556	0.3305160	0.2200000	2.0000000
LOSS2	18	0.1855556	0.0551172	0.1100000	0.2900000

TRT=2 SALT=1 THICKNS=2

Variable	N	Mean	Std Dev	Minimum	Maximum
ID	18	9.5000000	5.3385391	1.0000000	18.0000000
WGTBEF	18	84.2100000	0.0788147	84.0100000	84.3400000
WGTAFY	18	83.4966667	0.2299872	83.1400000	83.7800000
THICKBEF	18	1.0216667	0.0233263	0.9900000	1.0600000
THICKAFY	18	0.8338889	0.0905429	0.6100000	0.9600000
LOSS1	18	0.7133333	0.2541453	0.3700000	1.1200000
LOSS2	18	0.1677778	0.0974109	0.0500000	0.4200000

## The SAS System

TRT=2 SALT=2 THICKNS=1

Variable	N	Mean	Std Dev	Minimum	Maximum
ID	18	9.5000000	5.3385391	1.0000000	18.0000000
MGTBEF	18	83.7666667	0.0772315	83.6100000	83.9900000
MGTAFT	18	83.7494444	0.0747916	83.5900000	83.9800000
THICKBEF	18	0.9783333	0.0336505	0.9100000	1.0300000
THICKAFT	18	0.8933333	0.0517914	0.8100000	0.9700000
LOSS1	18	0.0172222	0.0112749	-0.0100000	0.0400000
LOSS2	18	0.0850000	0.0486202	0.0200000	0.1600000

TRT=2 SALT=2 THICKNS=2

Variable	N	Mean	Std Dev	Minimum	Maximum
ID	18	9.5000000	5.3385391	1.0000000	18.0000000
MGTBEF	18	84.2183333	0.0907667	83.9900000	84.3800000
MGTAFT	18	84.1816667	0.0967866	83.9500000	84.3400000
THICKBEF	18	1.0288889	0.0282612	0.9900000	1.0700000
THICKAFT	18	0.9722222	0.0375038	0.8900000	1.0200000
LOSS1	18	0.0366667	0.0197037	0.0100000	0.0900000
LOSS2	18	0.0566667	0.0342997	0.0200000	0.1600000

TRT=3 SALT=1 THICKNS=1

Variable	N	Mean	Std Dev	Minimum	Maximum
ID	18	9.5000000	5.3385391	1.0000000	18.0000000
MGTBEF	18	83.8244444	0.0624238	83.6700000	83.9200000
MGTAFT	18	82.6155556	0.0555425	82.5400000	82.7400000
THICKBEF	18	0.9844444	0.0178952	0.9600000	1.0300000
THICKAFT	18	0.1250000	0.0203643	0.1000000	0.1700000
LOSS1	18	1.2088889	0.0723643	1.0000000	1.3400000
LOSS2	18	0.8594444	0.0283823	0.8100000	0.9100000

TRT=3 SALT=1 THICKNS=2

Variable	N	Mean	Std Dev	Minimum	Maximum
ID	18	9.5000000	5.3385391	1.0000000	18.0000000
MGTBEF	18	84.3777778	0.0898728	84.1100000	84.4800000
MGTAFT	18	83.0744444	0.1244859	82.7800000	83.2500000
THICKBEF	18	1.0514444	0.0358510	1.0100000	1.1300000
THICKAFT	18	0.1461111	0.0276828	0.1000000	0.2000000
LOSS1	18	1.3033333	0.0820330	1.1500000	1.5100000
LOSS2	18	0.9055556	0.0494942	0.8100000	0.9900000

## The SAS System

TRT=3 SALT=2 THICKNS=1

Variable	N	Mean	Std Dev	Minimum	Maximum
ID	18	9.5000000	5.3385391	1.0000000	18.0000000
MCTBEF	18	83.7672222	0.0822239	83.5600000	83.8700000
MCTAFT	18	82.7027778	0.0868945	82.4900000	82.8600000
THICKBEF	18	1.0022222	0.0309754	0.9400000	1.0500000
THICKAFT	18	0.1866667	0.0222288	0.1400000	0.2300000
LOSS1	18	0.0466667	0.0998343	0.0000000	1.3500000
LOSS2	18	0.8155556	0.0398833	0.7300000	0.8700000

TRT=3 SALT=2 THICKNS=2

Variable	N	Mean	Std Dev	Minimum	Maximum
ID	18	9.5000000	5.3385391	1.0000000	18.0000000
MCTBEF	18	84.3988889	0.1219879	84.2600000	84.8300000
MCTAFT	18	83.1361111	0.0651820	83.0100000	83.2900000
THICKBEF	18	1.0233333	0.0211438	0.9900000	1.0500000
THICKAFT	18	0.1950000	0.0309173	0.1600000	0.2600000
LOSS1	18	1.2627778	0.1265286	1.1200000	1.6600000
LOSS2	18	0.8283333	0.0250294	0.7900000	0.8700000

TRT=1 SALT=1

Variable	N	Mean	Std Dev	Minimum	Maximum
ID	18	9.5000000	5.3385391	1.0000000	18.0000000
MCTBEF	18	83.6094444	0.1600278	83.2500000	83.7000000
MCTAFT	18	82.0105556	0.1570115	81.8600000	82.6000000
THICKBEF	18	0.1088889	0.0132349	0.0800000	0.1300000
THICKAFT	18	0.1088889	0.0132349	0.0800000	0.1300000
THICKNS	18	0	0	0	0
LOSS1	18	1.9988889	0.2393346	0.6500000	1.7300000
LOSS2	18	0	0	0	0

TRT=1 SALT=2

Variable	N	Mean	Std Dev	Minimum	Maximum
ID	18	9.5000000	5.3385391	1.0000000	18.0000000
MCTBEF	18	83.6222222	0.0586671	83.4800000	83.7200000
MCTAFT	18	82.9477778	0.0921990	82.7400000	83.1000000
THICKBEF	18	0.1216667	0.0188648	0.1000000	0.1400000
THICKAFT	18	0.1216667	0.0188648	0.1000000	0.1400000
THICKNS	18	0	0	0	0
LOSS1	18	0.6766667	0.0826205	0.5600000	0.9000000
LOSS2	18	0	0	0	0

TRT=2 SALT=1

Variable	N	Mean	Std Dev	Minimum	Maximum
ID	36	9.5000000	5.2617216	1.0000000	18.0000000
MCTBEF	36	83.9700000	0.2736108	83.2700000	84.3400000
MCTAFT	36	83.1055556	0.4547932	82.1600000	83.7800000
THICKBEF	36	1.0136111	0.0266354	0.9700000	1.0600000
THICKAFT	36	0.8269444	0.0711867	0.6100000	0.9600000
THICKNS	36	1.5000000	0.5078926	1.0000000	2.0000000
LOSS1	36	0.8666667	0.3384371	0.2200000	2.0000000
LOSS2	36	0.1866667	0.0780110	0.0500000	0.4200000

## The SAS System

----- TRT=3 SALT=2 THICKNS=1 -----

Variable	N	Mean	Std Dev	Minimum	Maximum
ID	18	9.5000000	5.3385391	1.0000000	18.0000000
MGTBEF	18	83.7672222	0.0822339	83.5600000	83.8700000
MGTAFY	18	82.7027778	0.0868945	82.4900000	82.8600000
THICKBEF	18	1.0022222	0.0309754	0.9400000	1.0500000
THICKAFY	18	0.1866667	0.0222288	0.1600000	0.2300000
LOSS1	18	1.0644444	0.0998365	0.9400000	1.3500000
LOSS2	18	0.8155556	0.0398855	0.7300000	0.8700000

----- TRT=3 SALT=2 THICKNS=2 -----

Variable	N	Mean	Std Dev	Minimum	Maximum
ID	18	9.5000000	5.3385391	1.0000000	18.0000000
MGTBEF	18	84.3988889	0.1219879	84.2600000	84.8300000
MGTAFY	18	83.1361111	0.0651820	83.0100000	83.2900000
THICKBEF	18	1.0233333	0.0211438	0.9900000	1.0500000
THICKAFY	18	0.1950000	0.0309173	0.1600000	0.2600000
LOSS1	18	1.2627778	0.1265286	1.1200000	1.6600000
LOSS2	18	0.8283333	0.0250294	0.7900000	0.8700000

----- TRT=1 SALT=1 -----

Variable	N	Mean	Std Dev	Minimum	Maximum
ID	18	9.5000000	5.3385391	1.0000000	18.0000000
MGTBEF	18	83.6094444	0.1600278	83.2500000	83.7000000
MGTAFY	18	82.0105556	0.1570115	81.8600000	82.6000000
THICKBEF	18	0.1088889	0.0132349	0.0800000	0.1300000
THICKAFY	18	0.1088889	0.0132349	0.0800000	0.1300000
THICKNS	18	0	0	0	0
LOSS1	18	1.5988889	0.2393346	0.6500000	1.7300000
LOSS2	18	0	0	0	0

----- TRT=1 SALT=2 -----

Variable	N	Mean	Std Dev	Minimum	Maximum
ID	18	9.5000000	5.3385391	1.0000000	18.0000000
MGTBEF	18	83.6222222	0.0586671	83.4800000	83.7200000
MGTAFY	18	82.9477778	0.0921990	82.7400000	83.1000000
THICKBEF	18	0.1216667	0.0188648	0.1000000	0.1600000
THICKAFY	18	0.1216667	0.0188648	0.1000000	0.1600000
THICKNS	18	0	0	0	0
LOSS1	18	0.6744444	0.0826205	0.5600000	0.9000000
LOSS2	18	0	0	0	0

----- TRT=2 SALT=1 -----

Variable	N	Mean	Std Dev	Minimum	Maximum
ID	36	9.5000000	5.2617216	1.0000000	18.0000000
MGTBEF	36	83.9700000	0.2736108	83.2700000	84.3400000
MGTAFY	36	83.1055556	0.4547932	82.1600000	83.7800000
THICKBEF	36	1.0136111	0.0266354	0.9700000	1.0600000
THICKAFY	36	0.8269444	0.0711867	0.6100000	0.9600000
THICKNS	36	1.5000000	0.5070926	1.0000000	2.0000000
LOSS1	36	0.8644444	0.3384371	0.2200000	2.0000000
LOSS2	36	0.1866667	0.0780110	0.0500000	0.4200000

The SAS System

General Linear Models Procedure  
Class Level Information

Class	Levels	Values
TRT	3	1 2 3
SALT	2	1 2
THICKNS	3	0 1 2

Number of observations in data set = 180

General Linear Models Procedure

Dependent Variable: LOSS1

Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	9	45.80053333	5.09341704	174.32	0.0001
Error	170	4.93041111	0.02900242		
Corrected Total	179	50.43094444			

R-Square	C. V.	Root MSE	LOSS1 Mean
0.902234	19.14490	0.1703010	0.8873336

Source	DF	Type I SS	Mean Square	F Value	Pr > F
TRT	2	23.77010036	11.88505028	409.80	0.0001
SALT	1	13.93543336	13.93543336	481.19	0.0001
THICKNS	1	0.00022900	0.00022900	0.01	0.9299
TRT*SALT	2	6.81804722	3.25733261	112.32	0.0001
TRT*THICKNS	1	0.74334444	0.74334444	25.70	0.0001
SALT*THICKNS	1	0.40744944	0.40744944	14.05	0.0002
TRT*SALT*THICKNS	1	0.10671111	0.10671111	3.68	0.0568

Source	DF	Type III SS	Mean Square	F Value	Pr > F
TRT	1	21.02222900	21.02222900	724.84	0.0001
SALT	1	15.35483319	15.35483319	529.80	0.0001
THICKNS	1	0.00022900	0.00022900	0.01	0.9299
TRT*SALT	1	4.99322900	4.99322900	172.23	0.0001
TRT*THICKNS	1	0.74334444	0.74334444	25.70	0.0001
SALT*THICKNS	1	0.40744944	0.40744944	14.05	0.0002
TRT*SALT*THICKNS	1	0.10671111	0.10671111	3.68	0.0568

Parameter	Estimate	T for H0: Parameter=0	Pr >  T	Std Error of Estimate
INTERCEPT	1.263777778	31.46	0.0001	0.04014032
TRT				
1	-0.883333333	-10.36	0.0001	0.08474499
2	-1.226111111	-21.60	0.0001	0.08474499
3	0.000000000	.	.	.
SALT				
1	0.040333334	0.71	0.4789	0.05674499
2	0.000000000	.	.	.
THICKNS				
0	0.000000000	.	.	.
1	-0.198333333	-3.49	0.0006	0.05674499
2	0.000000000	.	.	.
TRT*SALT				
1 1	0.823888889	11.01	0.0001	0.08028064
1 2	0.000000000	.	.	.
2 1	0.636111111	7.92	0.0001	0.08028064
2 2	0.000000000	.	.	.
3 1	0.000000000	.	.	.

## The SAS System

TRT=1 SALT=1

Variable	N	Mean	Std Dev	Minimum	Maximum
ID	18	9.5000000	5.3385391	1.0000000	18.0000000
WGTBEF	18	83.6094444	0.1000278	83.2500000	83.7000000
WGTAFI	18	82.0105556	0.1570115	81.8600000	82.6000000
THICKBEF	18	0.1088889	0.0132349	0.0800000	0.1300000
THICKAFI	18	0.1088889	0.0132349	0.0800000	0.1300000
THICKNS	18	0	0	0	0
LOSS1	18	1.5988889	0.2303346	0.6500000	1.7300000
LOSS2	18	0	0	0	0

TRT=1 SALT=2

Variable	N	Mean	Std Dev	Minimum	Maximum
ID	18	9.5000000	5.3385391	1.0000000	18.0000000
WGTBEF	18	83.6222222	0.0586671	83.4800000	83.7200000
WGTAFI	18	82.9477778	0.0921990	82.7400000	83.1000000
THICKBEF	18	0.1216667	0.0188648	0.1000000	0.1600000
THICKAFI	18	0.1216667	0.0188648	0.1000000	0.1600000
THICKNS	18	0	0	0	0
LOSS1	18	0.6744444	0.0826205	0.5600000	0.9000000
LOSS2	18	0	0	0	0

TRT=2 SALT=1

Variable	N	Mean	Std Dev	Minimum	Maximum
ID	36	9.5000000	5.2617216	1.0000000	18.0000000
WGTBEF	36	83.9700000	0.2736108	83.2700000	84.3400000
WGTAFI	36	83.1055556	0.4547932	82.1600000	83.7800000
THICKBEF	36	1.0136111	0.0266354	0.9700000	1.0600000
THICKAFI	36	0.8269444	0.0711867	0.6100000	0.9600000
THICKNS	36	1.5000000	0.5070926	1.0000000	2.0000000
LOSS1	36	0.8644444	0.3384371	0.2200000	2.0000000
LOSS2	36	0.1866667	0.0780110	0.0500000	0.4200000

TRT=2 SALT=2

Variable	N	Mean	Std Dev	Minimum	Maximum
ID	36	9.5000000	5.2617216	1.0000000	18.0000000
WGTBEF	36	83.9925000	0.2436317	83.6100000	84.3800000
WGTAFI	36	83.9655556	0.2391710	83.5900000	84.3400000
THICKBEF	36	1.0036111	0.0399394	0.9100000	1.0700000
THICKAFI	36	0.9327778	0.0598861	0.8100000	1.0200000
THICKNS	36	1.5000000	0.5070926	1.0000000	2.0000000
LOSS1	36	0.0269444	0.0186424	-0.0100000	0.0900000
LOSS2	36	0.0708333	0.0377402	0.0200000	0.1600000

The SAS System

General Linear Models Procedure  
Class Level Information

Class	Levels	Values
TRT	3	1 2 3
BALT	2	1 2
THICKNS	3	0 1 2

General Linear Models Procedure

Dependent Variable: LOSS1

Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	9	45.39384222	5.044315802	192.63	0.0001
Error	171	0.03712222	0.000217089		
Corrected Total	179	50.43096444			

R-Square	C. V.	Root MSE	LOSS1 Mean
0.900118	19.29390	0.1716300	0.8895556

Source	DF	Type I SS	Mean Square	F Value	Pr > F
TRT	2	23.77010056	11.88505028	403.47	0.0001
BALT	1	13.95363356	13.95363356	473.77	0.0001
THICKNS	1	0.00022300	0.00022300	0.01	0.9305
TRT*BALT	2	4.81806722	2.40903361	110.99	0.0001
TRT*THICKNS	1	0.74534444	0.74534444	25.30	0.0001
BALT*THICKNS	1	0.40746944	0.40746944	13.83	0.0003

Source	DF	Type III SS	Mean Square	F Value	Pr > F
TRT	1	21.02222300	21.02222300	713.46	0.0001
BALT	1	13.47333889	13.47333889	523.36	0.0001
THICKNS	1	0.00022300	0.00022300	0.01	0.9305
TRT*BALT	1	4.99322300	4.99322300	169.98	0.0001
TRT*THICKNS	1	0.74534444	0.74534444	25.30	0.0001
BALT*THICKNS	1	0.40746944	0.40746944	13.83	0.0003

Parameter	Estimate	T for H0: Parameter=0	Pr >  T	Std Error of Estimate
INTERCEPT	1.290000000	34.09	0.0001	0.03784086
TRT				
1	-0.618333333	-11.11	0.0001	0.05539334
2	-1.280333333	-23.83	0.0001	0.04994831
3	0.000000000	.	.	.
BALT				
1	-0.013888889	-0.83	0.7796	0.04994831
2	0.000000000	.	.	.
THICKNS				
0	0.000000000	.	.	.
1	-0.252777778	-8.10	0.0001	0.04994831
2	0.000000000	.	.	.
TRT*BALT				
1 1	0.938333333	12.40	0.0001	0.07648171
1 2	0.000000000	.	.	.
2 1	0.748000000	13.02	0.0001	0.05721000
2 2	0.000000000	.	.	.
3 1	0.000000000	.	.	.
3 2	0.000000000	.	.	.
TRT*THICKNS				
1 0	0.000000000	.	.	.
2 1	0.257777778	5.03	0.0001	0.05721000

**Appendix D**

**Color Copies of Before and After Appearance of Bolts Tested**



Figure 12. Eighteen medium carbon steel bolts plated electrolytically with thick clear chromate zinc: Appearance before test.

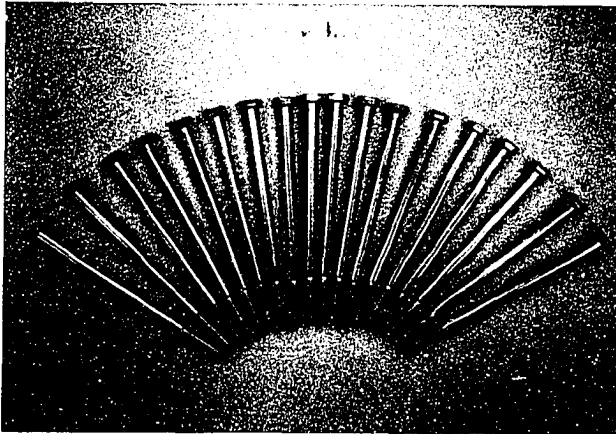


Figure 13. Eighteen medium carbon steel bolts plated electrolytically with thick clear chromate zinc and tested with low salt concentration solution in the main tank of the salt spray machine: Appearance after test.

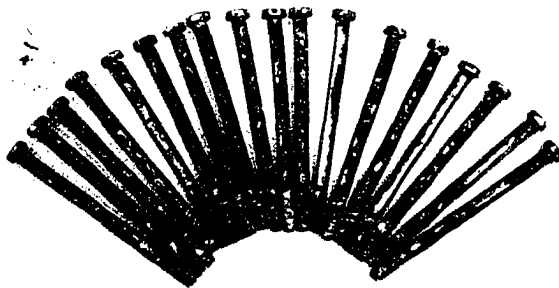


Figure 14. Eighteen medium carbon steel bolts plated electrolytically with thin yellow dichromate zinc:  
Appearance before test.

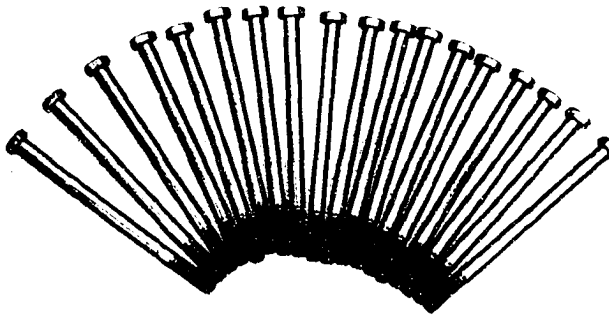


Figure 15. Eighteen medium carbon steel bolts plated electrolytically with thin yellow dichromate zinc and tested with low salt concentration solution in the main tank of the salt spray machine: Appearance after test.

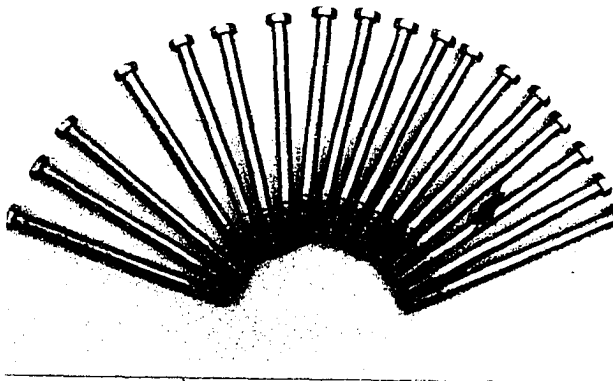


Figure 16. Eighteen medium carbon steel bolts plated electrolytically with thick yellow dichromate zinc:  
Appearance before test.

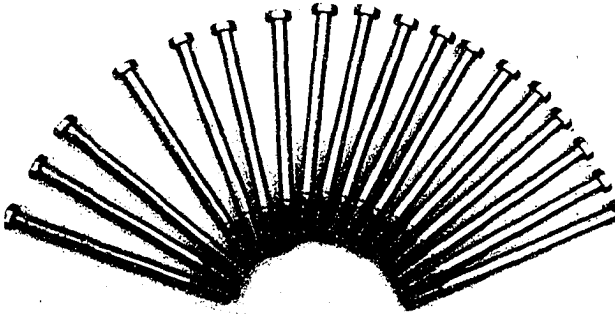


Figure 17. Eighteen medium carbon steel bolts plated electrolytically with thick yellow dichromate zinc and tested with low salt concentration solution in the main tank of the salt spray machine: Appearance after test.

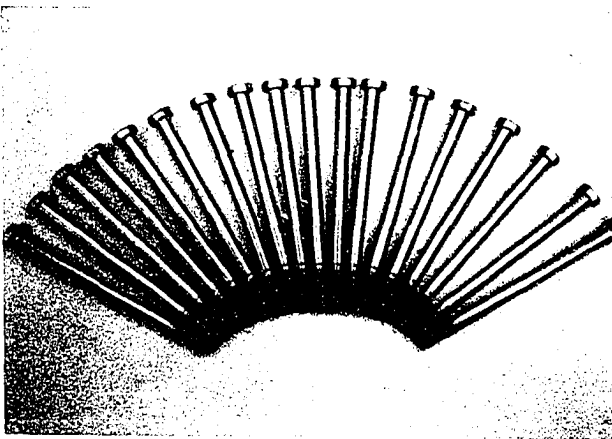


Figure 18. Plain medium carbon steel bolts: Appearance before salt spray test.

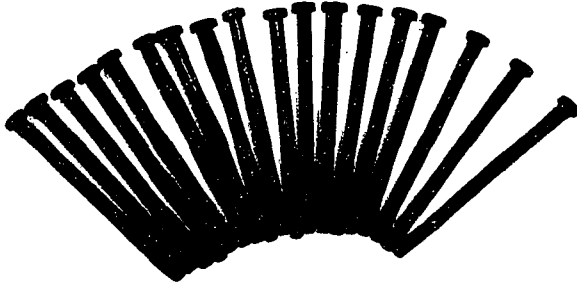


Figure 19. Plain medium carbon steel bolts tested with low salt concentration solution in the main tank of the salt spray machine: Appearance after test.

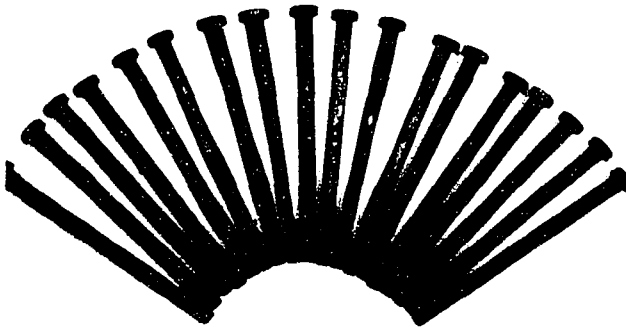


Figure 20. Eighteen medium carbon steel bolts plated with thin clear chromate zinc: Appearance before test.

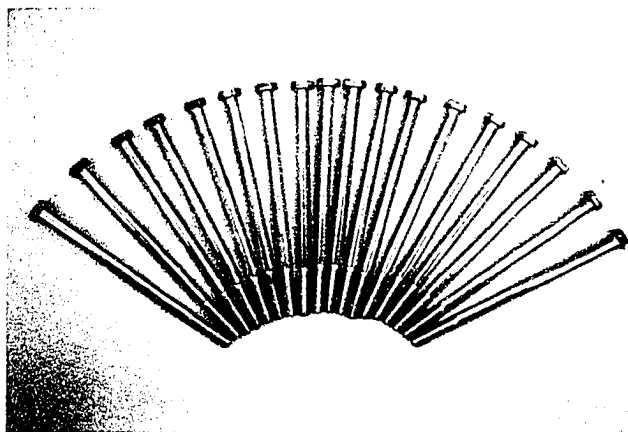


Figure 21. Eighteen medium carbon steel bolts plated with thin clear chromate zinc and tested with high salt concentration solution in the main tank of the salt spray machine: Appearance after test.

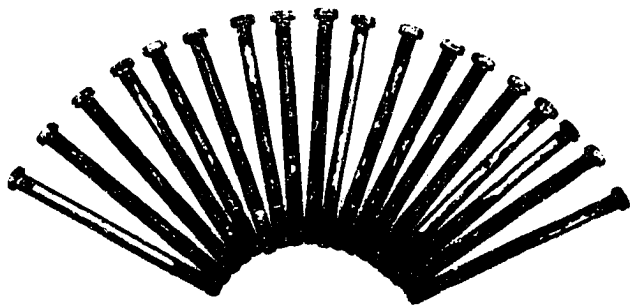


Figure 22. Eighteen medium carbon steel bolts plated electrolytically with thick clear chromate zinc: Appearance before test.

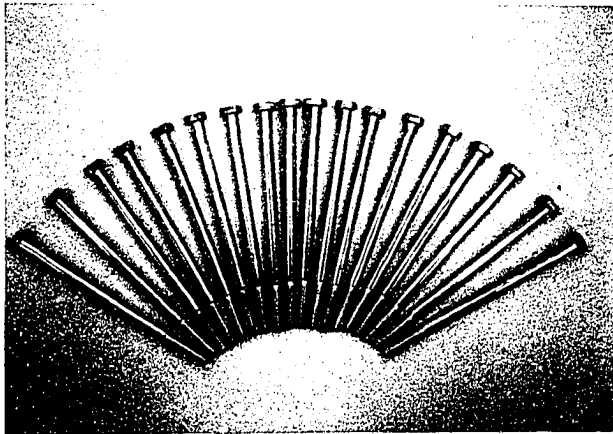


Figure 23. Eighteen medium carbon steel bolts plated electrolytically with thick clear chromate and tested with high salt concentration solution in the main tank of the salt spray machine: Appearance after test.

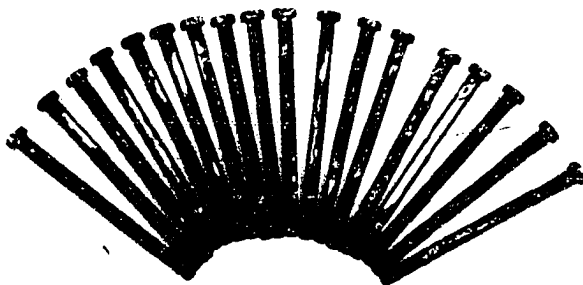


Figure 24. Eighteen Medium carbon steel bolts plated electrolytically with thin yellow dichromate zinc:  
Appearance before test.

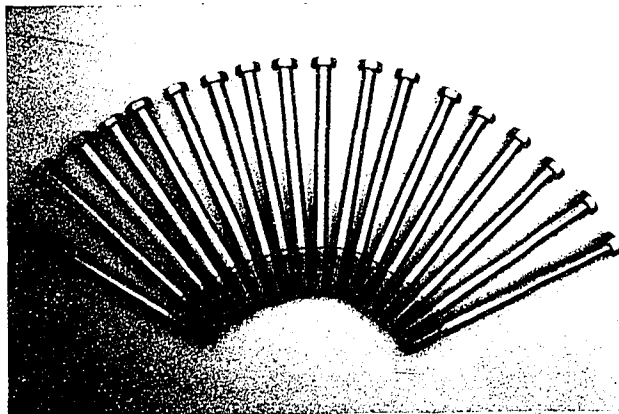


Figure 25. Eighteen medium carbon steel bolts plated electrolytically with thin yellow dichromate zinc and tested with high salt concentration solution in the main tank of the salt spray machine: Appearance after test.

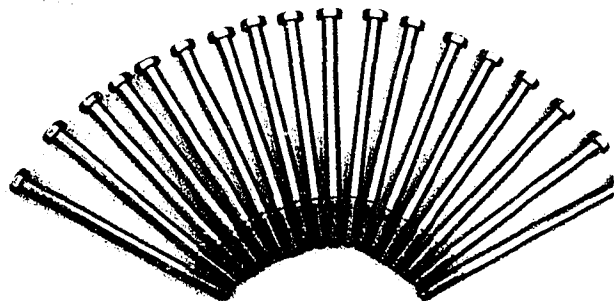


Figure 26. Eighteen Medium carbon steel bolts plated electrolytically with thick yellow dichromate zinc:  
Appearance before test.

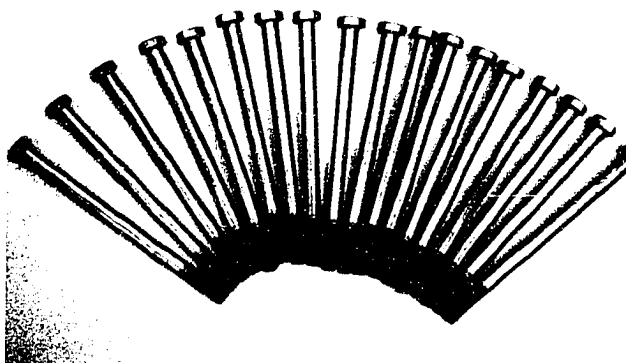


Figure 27 Eighteen medium carbon steel bolts plated electrolytically with thick yellow dichromate and tested with high salt concentration solution in the main tank of the salt spray machine: Appearance after test.

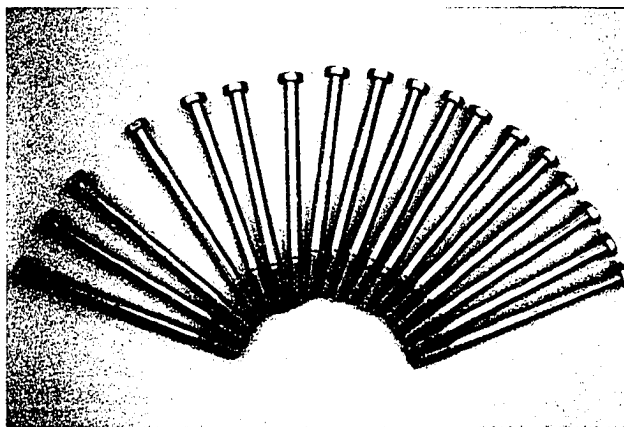




Figure 28. Plain medium carbon steel bolts: Appearance before test.

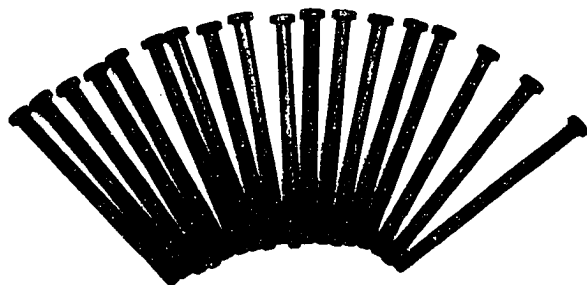
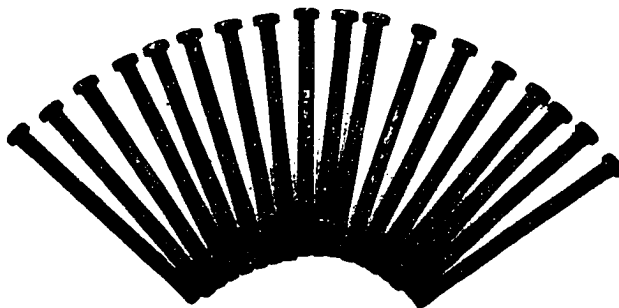


Figure 29. Plain medium carbon steel bolts tested with high salt concentration solution in the main tank of the salt spray machine: Appearance after test.



Appendix E  
Daily Log of Salt Spray Test

**IOWA INDUSTRIAL PRODUCTS, INC.  
SALT SPRAY DAILY INSPECTION**

Date/ Time	p.H. Bal.	Drop rate	Temperature (92-97 F)	Tower heat	Pressure 12-18psi	Interrup. cause	Specific Gravity	By
7/13/93	7.0	---	97.0	119	15.00	---	1.24	Souley B.
7/14/93	7.0	1.5	96.6	119	15.00	---	1.23	Souley B.
7/15/93	7.0	1.6	96.7	119	15.00	---	1.24	Souley B.
7/16/93	7.0	1.6	96.6	119	15.00	---	1.24	Souley B.
7/17/93	7.0	1.5	98.4	119	15.00	---	1.24	Souley B.
7/18/93	7.0	1.5	97.3	119	15.00	---	1.24	Souley B.
7/19/93	7.0	1.5	98.3	119	15.00	---	1.24	Souley B.
7/20/93	7.0	1.5	97.2	119	15.00	---	1.24	Souley B.
7/21/93	7.1	---	96.6	119	15.00	---	1.24	Souley B.
7/22/93	7.0	1.5	96.4	119	15.00	---	1.24	Souley B.
7/23/93	7.0	1.5	97.9	119	15.00	---	1.24	Souley B.
7/24/93	7.0	1.5	97.4	119	15.00	---	1.24	Souley B.
7/25/93	7.0	1.5	96.5	119	15.00	---	1.24	Souley B.
7/26/93	7.0	1.5	98.3	119	15.00	---	1.24	Souley B.
7/27/93	7.0	1.5	101.1	119	15.00	---	1.24	Souley B.
7/28/93	7.0	1.5	100.5	119	15.00	---	1.24	Souley B.

**Daily Log of the Salt Spray Test Run from July 13, 1993 to  
July 28, 1993. (Courtesy of Iowa Industrial Products, Inc.)**