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FINAL REPORT

A STUDY ON ELECTRODEPOSITION OF ORGANIC COATINGS FOR POSSIBLE MILITARY USAGE

BY

LT. J. FRAUENHOFFER

&

WILLIAM H. DEAVER

OCTOBER 1966

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### ABSTRACT

A brief discussion on the electrodeposition of organic coatings is presented as the result of an extensive literature survey through recent technical journals and publications. The feasibility of the process for possible military usage was studied using a specific proprietary coating. Laboratory work on processing parameters indicated that electrodeposition would offer advantages for uniformly coating equipment with complex configurations which cannot be adequately coated by other application methods. Before this process can be extensively used more technical data on electrocoating mechanisms, suitable vehicles, pigments, additives, formulation parameters and means for effectively maintaining a uniform composition during electrocoat processing will have to be developed so that the quality of the deposited films can be controlled.

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## 1. INTRODUCTION

Literature in recent years has shown a renewed interest in a subject called "Electrodeposition". The term was first heard back in 1922 when referring to the manufacture of rubber gloves. At that time a patent was issued for the electrodeposition of rubber coatings. Until a few years ago, the idea of electrodeposition of organic coatings lay dormant. Then it was found that a paint coating applied by electrodeposition was comparable to those applied by other existing methods. The big interest developed when the automotive industry found that electrodeposition was a superior method for applying a primer to hard to coat configurations such as recessed seams and channels. (1)

Among the advantages cited for electrodeposition are:

(1) Safety - the film is deposited from a water solution or dispersion, therefore, combustible materials are not present.

(2) Uniform coverage - all parts of the article being protected are uniformly coated. This applies to all seams, sharp corners, edges, recessed areas, and many interior sections that can not be adequately coated by other methods.

(3) Coating appearance - electrodeposition is performed at low solids content which results in an extremely low viscosity solution, which provides rapid run-off and elimination of runs and sags.

(4) Economics - utilization of over 95% of paint has been accomplished since most of excess paint drains back in the coating tank. (2) Solvent cost is reduced to near nothing, since, water being the solvent, ordinary tap water could be used.

(5) Electrodeposition offers the possibility of reducing labor costs by increasing automation. Units which previously had to be coated as individual parts and then assembled can now be coated after assembly.

(6) Process time - although no steps such as pretreatment or baking prior to and after electrodeposition on painting are eliminated, the time consumed in spray or dip coating can be considerably reduced. The article does not have to be dried after cleaning or pretreatment rinses before being electrocoated. The substrate need only be in the coating bath between 0.5 and 2 minutes and after rinsing can be immediately baked since no flash-off time is necessary. The process is also suited to fully conveyORIZED plants.

(7) Elimination of solvent washing - the film after electrodeposition is not readily soluble in water so therefore would not be subject to the refluxing action of the solvent vapors in contained areas.

From the literature available, a comparison of electrodeposition to the two main methods of coating application (spray and dip) is made in Table I, Appendix A. During electrodeposition a dilute

coating is converted from a watersoluble or dispersible form to a dense water-insoluble film on the article being coated. In general the vessel containing the coating is made the cathode and the article being coated the anode. A heat exchanger and agitation are needed to remove heat developed by the passage of current. The agitation also prevents the paint's solids from settling out.

To understand how paint is applied by electrodeposition requires the definition of a few terms that are heard more often in reference to plating than to painting. These terms are electrolysis, electrophoresis, and electro-osmosis and are defined as:

(1) Electrolysis - under the influence of electrical energy, ions move to an electrode and are electrically discharged.

(2) Electrophoresis - small charged solid particles present in a colloidal suspension will move to an electrode under the influence of electrical energy.

(3) Electro-osmosis - under the influence of electrical energy, the liquid phase moves with respect to the solid phase (opposite of electrophoresis.)

According to the literature these mechanisms enable a water soluble (dispersible) paint to be electrodeposited in the following ways. By electrolysis, the resin in solution is carried to the electrode surface (object to be coated) where it is converted to a water-insoluble, dense, uniform layer of coating. Through electrophoresis the insoluble dispersed resins and pigment particles are carried to the electrode surface and simultaneously deposited from the solution. Electroosmosis causes the deposited coating on the electrode to further concentrate by dehydration to yield a coating containing less than 5 percent water prior to curing. (2)

None of these mechanisms however, accounts for the actual deposition of the paint. They effect the transport of the paint to the electrode and compaction after deposition. The actual deposition can be caused by three factors:

(1) The particles coagulate due to a reduction of the negative charge on the particle induced by the primary electrode reaction of discharging the hydroxyl ion with a consequent drop in pH in the vicinity of the anode.

(2) The soluble material concentrates at the anode to a point where the phase volume relationship has exceeded the limit for o/w stability and film formation proceeds in a manner similar to that envisioned for latex film formation.

(3) While both the above take place, the main method of deposition appears to be that of ionic coagulation via reaction with the metallic ions being oxidized at the anode and traveling toward the cathode, e.g.



All investigations report the presence of iron in the films deposited on steel. (3)

Electrodeposition has been used for coating washing machines, (4) for the application of bituminous emulsions to the exterior of oil well casings already placed in the ground, and for the coating of rocket nozzles intended for high temperature service. (5) One automotive user of the electrodeposition process has coated more than 2½ million wheels and not one wheel has been scrapped because of defects caused by the finishing system. (6)

The advantages cited for painting by electrodeposition make this process of considerable interest for the coating of military hardware where configuration makes it difficult to coat by conventional methods. If recessed areas and interior sections that cannot be adequately painted could be uniformly coated the increased corrosion protection obtained would result in the extension of service life for much military equipment. It was therefore considered desirable to study the feasibility of this process for possible military applications.

## II DETAILS OF TEST

For this work a silicon industrial power supply (rectifier), operating on a 440 volt, three phase, sixty cycle AC power source, capable of delivering 20 amperes of 0-500 volts DC with a ripple content of less than 5% was used. The experimental apparatus set-up for electrodeposition is depicted in Figure 1 and consisted of a water jacket cooling system to maintain bath temperature together with a four liter glass beaker as the containing vessel for the paint. A magnetic stirrer provided sufficient agitation to promote proper heat exchange and prevent the settling of paint solids. The panel holder was fabricated from a solid piece of electrically non-conductive poly-vinyl chloride.

The organic coating material used was a water-soluble proprietary primer containing pigments, vehicle, and additives such as solubilizers, dispersants, and thickeners.

Steel panels, 3 by 6 inches, taken from one lot of 1010 cold rolled steel were vapor degreased and then zinc phosphate pre-treated in a bath conforming to Military specification MIL-P-50002, type 2. After immersion in the phosphate bath for 5 minutes, the test panels were thoroughly rinsed and then immersed for 45 seconds in a "chromic acid" final rinse. All panels were dried in a forced hot air oven at 200°F. and placed in a dessicator to cool prior to painting. An untreated steel panel from the same lot was used as the cathode. The temperature of the electrodeposition bath was kept at  $78 \pm 2^\circ\text{F}$ . since most electrodeposition is performed

at room temperature. To establish optimum application conditions, the effect of operating voltage, solids, content, pH and time of deposition were studied with respect to film build and appearance. With these conditions established coated specimens, after curing at 300°F for 30 minutes, were subjected to 20% salt spray, water immersion and a knife test for adhesion. All tests were performed in accordance to MIL-STD-141.

In addition, since the ability of an electrodeposited coating to cover all recesses and edges to a uniform film thickness is dependent upon "throwing power", Triplett and Tube Penetration tests were conducted. The thickness and throwing power for paint is dependent primarily upon the quantity of electricity passed. Films possessing high electrical resistance are desirable especially if regions shielded from the cathode are to be coated. An electrically resistant coating deflects the current away from itself to remote uncoated surfaces. In this way a uniform coating is developed. Therefore, in order to get good throwing power, an electrically resistant coating must be formed.

For the Triplett test, (Figure 2) three 3" X 6" 1010 cold rolled steel panels clamped by means of two spacers to form a wedge-shape were used as the test piece. For the tube penetration test, (Figure 3) a seven inch length of 5/8" steel pipe with a metal strip of the same material inserted inside was used as the test piece.

The Triplett and Tube-Penetration tests were both conducted at 200 volts, 80°F., and 10% solids content. For both tests, the test piece was electrodeposited for 1.5, 3.0, and 5.0 minutes and visually examined. The ratio of the height to which the inside of the pipe was coated to the height to which the outside of the pipe was coated (times 100) is the percentage throwing power for the tube penetration test. The percent throwing power can also be determined by replacing the value determined for the height of the coating on the inside of the pipe with the height the coating attained on the metal strip inserted in the pipe. In the Triplett test the sufficiently coated area is determined and the throwing power is expressed as the percent of this area in relation to the total immersed area.

The ability of the electrodeposition method of painting to apply a uniformly thick film even on edges and recessed areas was also studied by coating various irregular shaped objects. The objects shown in Figure 4 were coated at optimum conditions determined for this paint.

### III DISCUSSION

In varying the operating voltage, solids content, pH and deposition time to establish optimum electrocoating conditions for the coating, it was shown that the amount of material deposited at any given set of conditions is dependent on the amount of current flowing through the paint. Apparently each different coating will have its own set of conditions for optimum deposition. This flow is dependent on the conductivity of the bath, separation of the electrodes, and electrode area. Once the electrode area and separation are set, the current flow is dependent only on the conductivity of the bath. The conductivity of the bath is a function of solids content, viscosity, pH, and temperature. Since electrodeposition builds an electrically resistant film, the current flow will be reduced as the film builds.

If these were the only factors involved, pH, temperature, viscosity, and solids content would be set so that maximum current flow would be obtained; however, from the point of view of electrical efficiency and the evolution of gas or those electrode reactions which interfere with the smooth deposition of paint, the less current passed electrolytically, the better. The point where gas evolution interferes with coating build-up and appearance is called the "rupture point". At the rupture point, excessive gassing occurs at the anode. Therefore the maximum current flow to allow satisfactory film deposition would be that just below the rupture point. Figure 5 shows the effect of varying the voltage on film build when time, pH, solids content, and temperature are held constant. The rupture point for this particular coating using the experimental set-up in Figure 1 occurs slightly under 250 volts. Therefore the optimum operating voltage would be about 225 volts. As indicated by the arrows, above 250V a very uneven film was deposited with the film thickness varying in places as much as 5 mils.

Film build is also a function of time. Figure 6 shows the effect time has on film thickness using a constant voltage of 225 volts, constant solids, and constant temperature. The film appearance is not completely acceptable as it has a roughened look until at least a 1 mil coating is deposited. Therefore the time of electrodeposition would be set at 1 minute at 225 volts.

In general film thickness decreases with pH but the decrease is slight (8) over a small pH range. One explanation for the decrease in film thickness at high pH is that there is a greater tendency for the deposited film to redissolve. Another factor is that many systems are less stable at lower pH values. For this reason, the optimum pH for maximum stability and film thickness must be maintained. The optimum chosen pH for this particular

coating used was 7.2. The problem in relation to pH arises not with choosing the optimum pH but in maintaining that pH. Due to a build-up of cations in the paint bath not deposited with the resin and pigments, the pH increases as the bath is depleted. Figure 7 shows this effect. This problem can be surmounted in four ways:

(1) the bath can be circulated through a cation exchanger resin which will remove the cations.

(2) a cation-deficient replenisher stock may be added to the bath to neutralize the excess cations.

(3) the bath solution can be subjected to dialysis.

(4) the cathode can be surrounded with a semi-permeable membrane and the cations concentrated in an electro dialysis cell. Replacing the water in the cell would remove the cations.

Figure 8 illustrates the depleting effect of electrodeposition on solids content by a large series of panels. As the solids content and pH decreases, the rupture point of the coating also decreases. The solids content, therefore, should be maintained within 1%. Thus if solids were being controlled by bath replenishment the pH would remain at an acceptable level.<sup>(8)</sup> Therefore, for this coating, the optimum operating conditions for electrodeposition to attain a satisfactory film would be 225 volts, 10% solids content, 7.2 pH, and 1 minute deposition time at room temperature.

Corrosion resistance is a function of film thickness as long as the above conditions are kept constant and time alone is used to vary the film thickness. A graph of corrosion resistance versus film thickness is presented in Figure 9. For the electrocoat system used (no topcoat), a 0.6 - 0.7 mil coating thickness would provide 500 hours of protection in a 20% salt spray. The electrocoated object was not susceptible to blistering or loss of adhesion when immersed in water. Film adhesion is rated very good over phosphated steel and is good over unpretreated steel.

Two examples of "throwing power" tests are shown in figures 10 and 11. Figure 10 shows the results obtained using the Triplet test procedure. The outer surface received a complete coat while the inner surface showed partial coating. The panels with the 1/16" separation between were approximately 40% coated while the 1/8" separated panels were approximately 66% coated.

Figure 11 shows the tube penetration test results comparing 4 tubes electrocoated for various periods of time to a similar tube given a conventional dip application. The effectiveness of electrodeposition is clearly evident. Further indication of the effectiveness is illustrated in Table 2 which compares the film thickness obtained by this technique using primer at 10% solids compared to the conventional dip using primer at 40% solids. Note the uniformity of film thickness on the exterior of the electrocoated tubes.

Meaningful film thickness measurements could not be made on the interior surfaces of the conventional dip application due to the non uniformity of coating as evidenced in Figure 11. However, with the electrodeposited film a continual build with time is noted.

Both tests are useful if used properly. The Triplet Test is used for determination of low throwing power materials while the tube penetration test is used for materials of high throwing power. (7)

To achieve high throwing power the film must have high specific resistance to avoid rupturing at the high voltages necessary. Through proper resin selection high specific resistance can be attained although pigmentation, pigment concentration, and method of manufacture are also important factors. (8)

#### IV: CONCLUSION

The use of electrodeposition could be adapted for military material where configuration would make it difficult to coat by other means. However more technology will have to be developed for the control and formulation of coatings to be electrodeposited to provide the quality coatings required for military usage.

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APPENDIX A

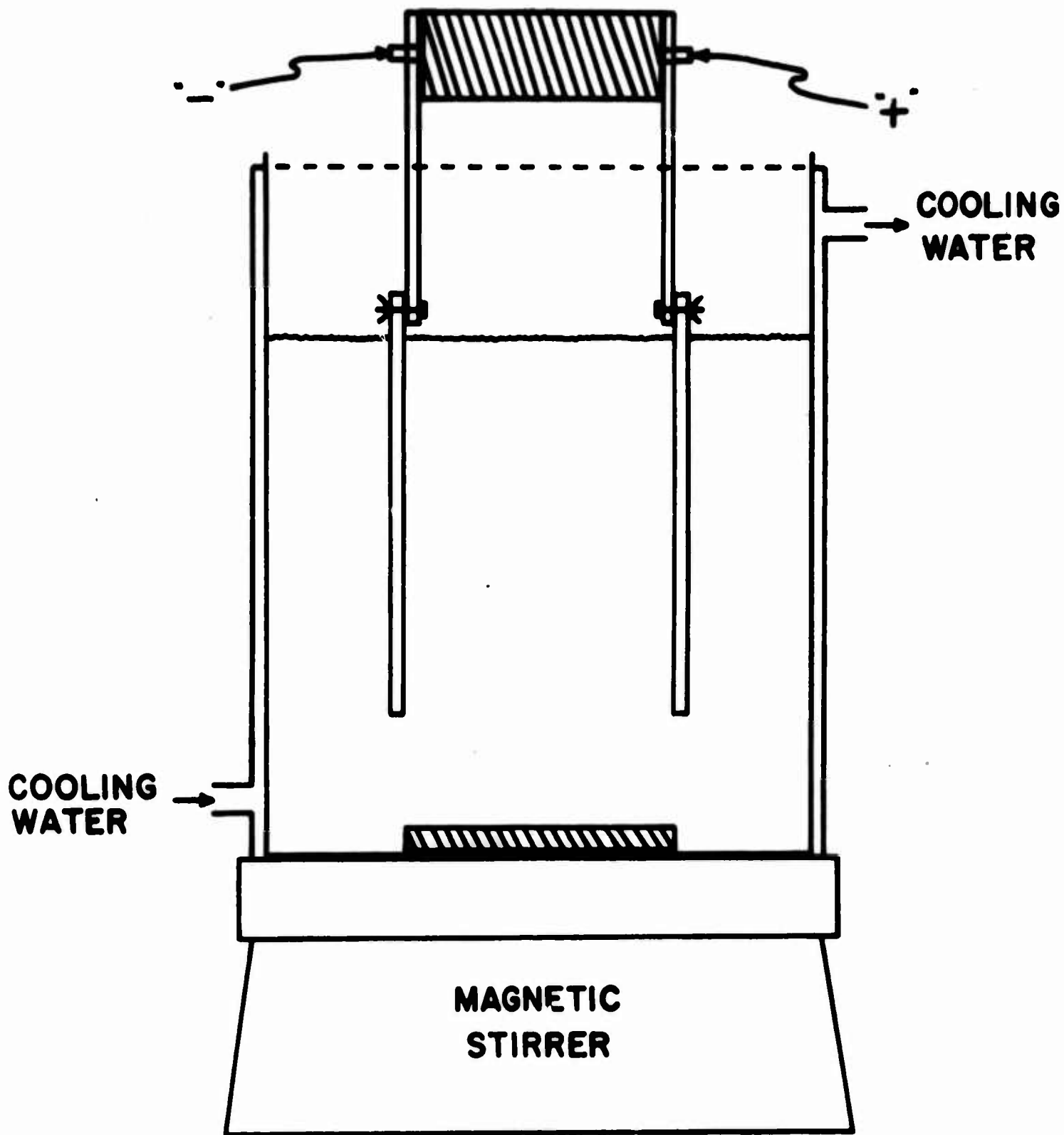
TABLE I

	ELECTROCOATING	SPRAY	DIP
Coating Components	Organic & Pigment	Organic & Pigment	Organic & Pigment
Bath	Multi-phase system (continuous phase + dispersed phase)	Multi-phase system	Multi-phase system
Solvent	Water or organic	Water or organic	Water or organic
Derived Coating	Has to be cured	Can be end product or could be cured	can be end product or could be cured
Deposition	Essentially irreversible	-----	-----
Voltage	About 200 volt	-----	-----
Grams deposited by 96,500 coulombs	2000 g.	-----	-----
Electric Resistance of coating	$10^{+6}$ ohm cm	-----	-----
Electrode Reaction	Discharge of colloidal particles & Chem. reactions	-----	-----
Uniformity of coating	Even all over	Thin in seams, wells, etc.	Thin in seams, well, etc., +wedge effect

TABLE II  
 DRY FILM THICKNESS (MILS)  
 TUBE PENETRATION TEST  
 (MEASURED AT 0.5 INCH INTERVALS)

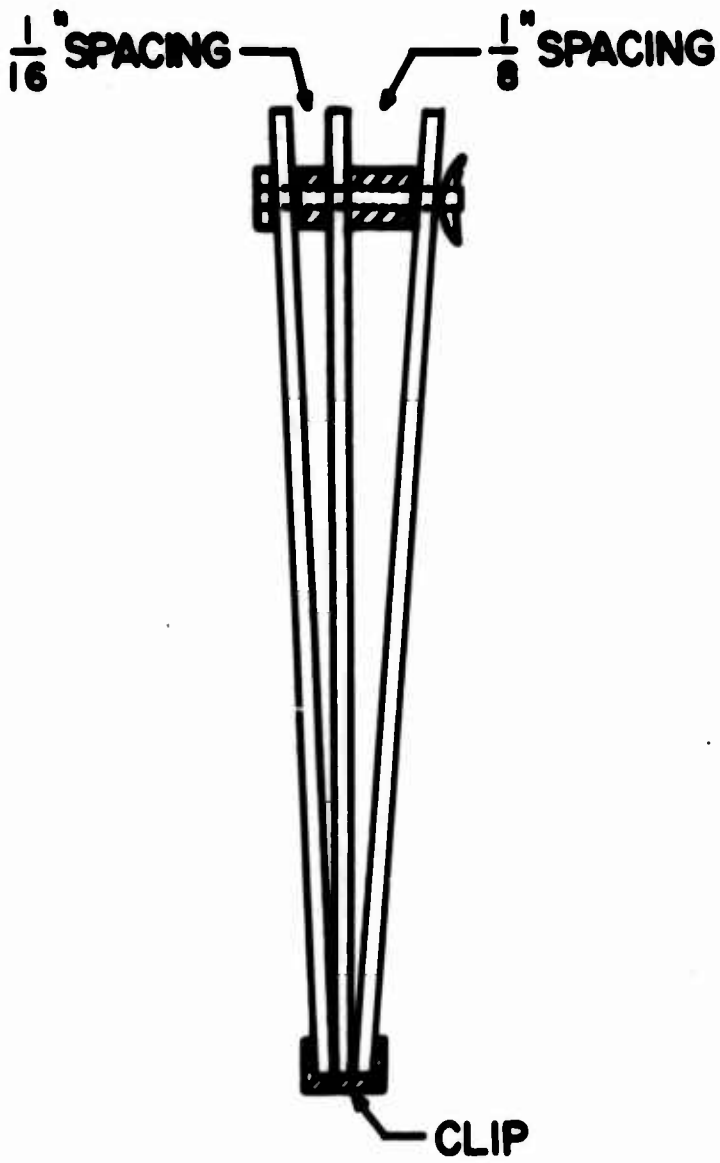
CONVENTIONAL DIP										ELECTROCOAT					
5 Minutes					3 Minutes					1.5 Minutes					
Outside	Inside	Strip	Outside	Inside	Strip	Outside	Inside	Strip	Outside	Inside	Strip	Outside	Inside	Strip	
0.5	0.1	0.2	2.3	0.7	0.9	2.0	0.2	0.3	1.5	-	-	-	-	-	
0.5	0.1	0.4	2.3	0.6	0.8	2.0	0.2	0.4	1.5	-	-	-	-	-	
0.7	0.1	0.1	2.3	0.7	0.9	2.0	0.4	0.5	1.5	-	-	-	-	-	
0.8	0.3	0.1	2.3	0.9	1.0	2.0	0.6	0.6	1.5	-	0.1	-	-	-	
1.0	0.3	0.2	2.3	1.0	1.3	2.0	0.7	0.6	1.5	0.2	0.3	-	-	-	
1.0	0.4	0.2	2.5	1.2	1.3	2.0	1.0	1.0	1.7	0.5	0.3	-	-	-	
1.0	0.4	0.3	2.5	1.4	1.7	2.0	1.2	1.2	1.7	0.6	0.4	-	-	-	
1.1	0.4	0.3	2.5	1.7	2.3	2.0	1.4	1.5	1.7	0.6	0.6	-	-	-	
1.1	0.6	0.3	2.5	1.7	2.7	2.0	1.4	1.8	1.7	0.6	0.9	-	-	-	
1.3	0.8	0.3	2.5	1.7	3.0	2.0	1.8	2.2	1.7	0.8	1.3	-	-	-	
1.3	0.8	0.6	2.5	1.7	3.5	2.0	1.8	2.2	1.7	1.0	1.3	-	-	-	
1.3	1.2	0.6	2.5	2.0	3.5	1.8	1.8	2.2	1.7	1.3	1.3	-	-	-	

APPENDIX B



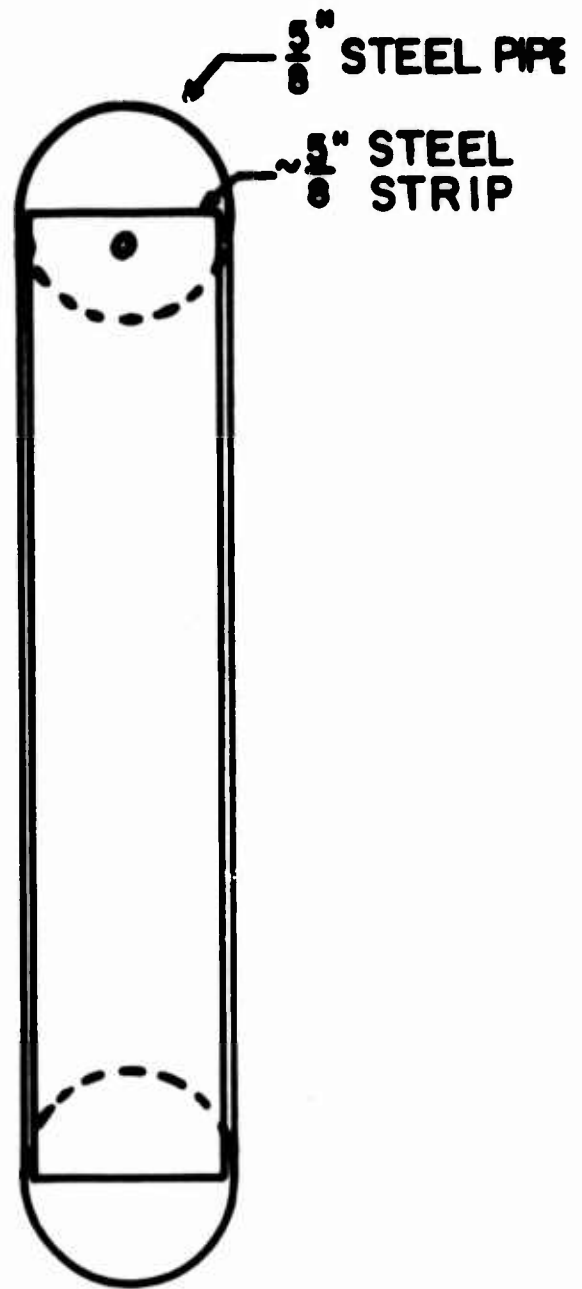
**ELECTRODEPOSITION UNIT**

**FIGURE 1**



**TRIPLET TEST**

**FIGURE 2**



**TUBE PENETRATION TEST**

**FIGURE 3**



ITEMS COATED BY ELECTRODEPOSITION

**FIGURE 4**

### RELATIONSHIP OF FILM BUILD TO VOLTAGE

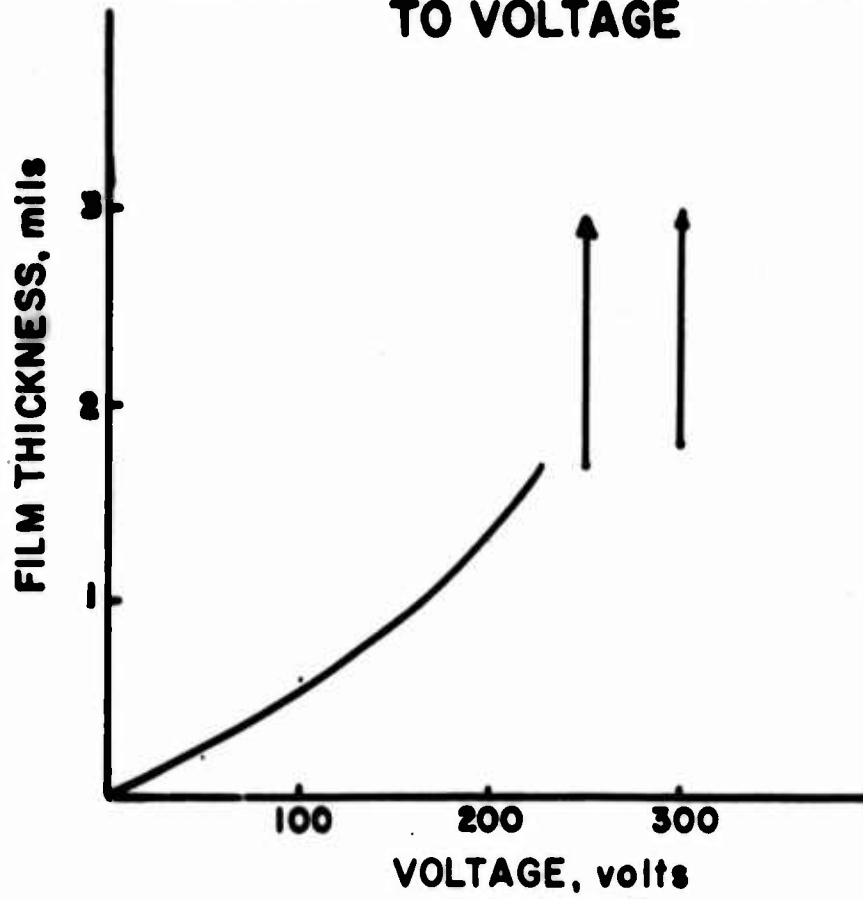


FIGURE 5

### RELATIONSHIP OF FILM BUILD TO TIME OF DEPOSITION

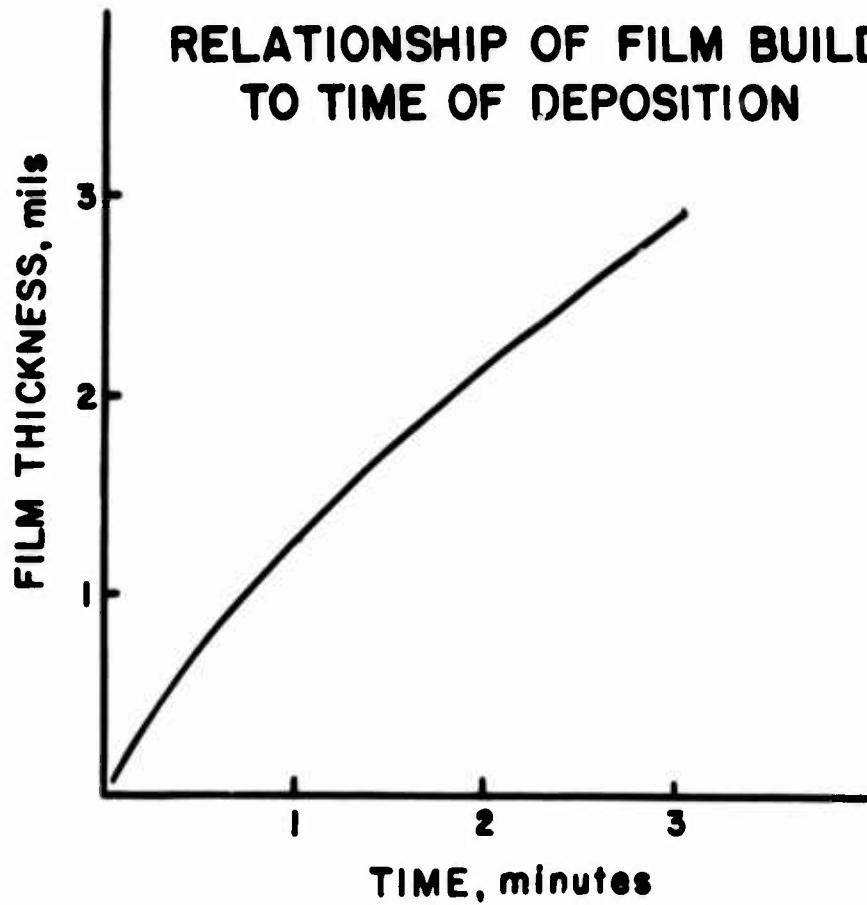
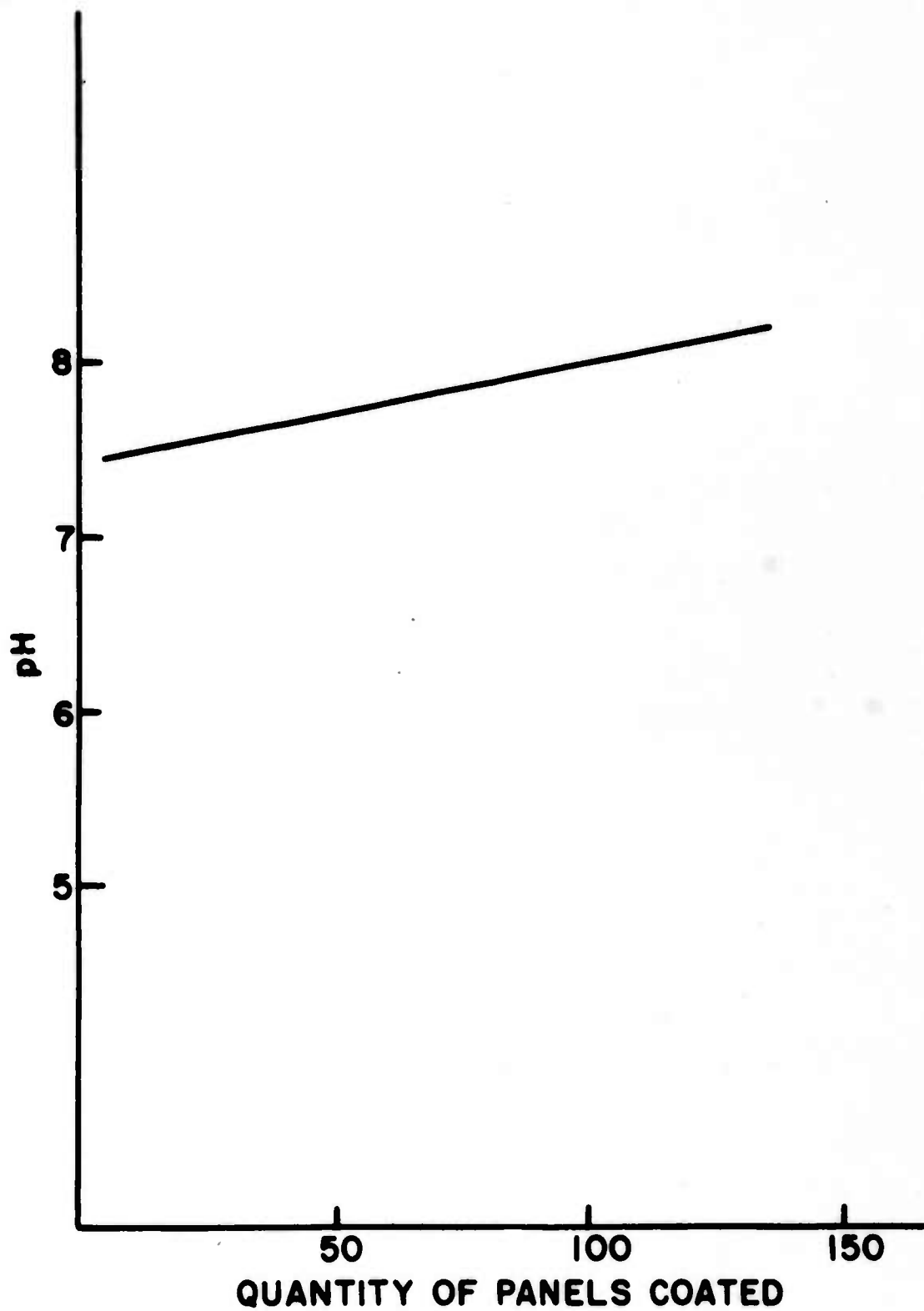


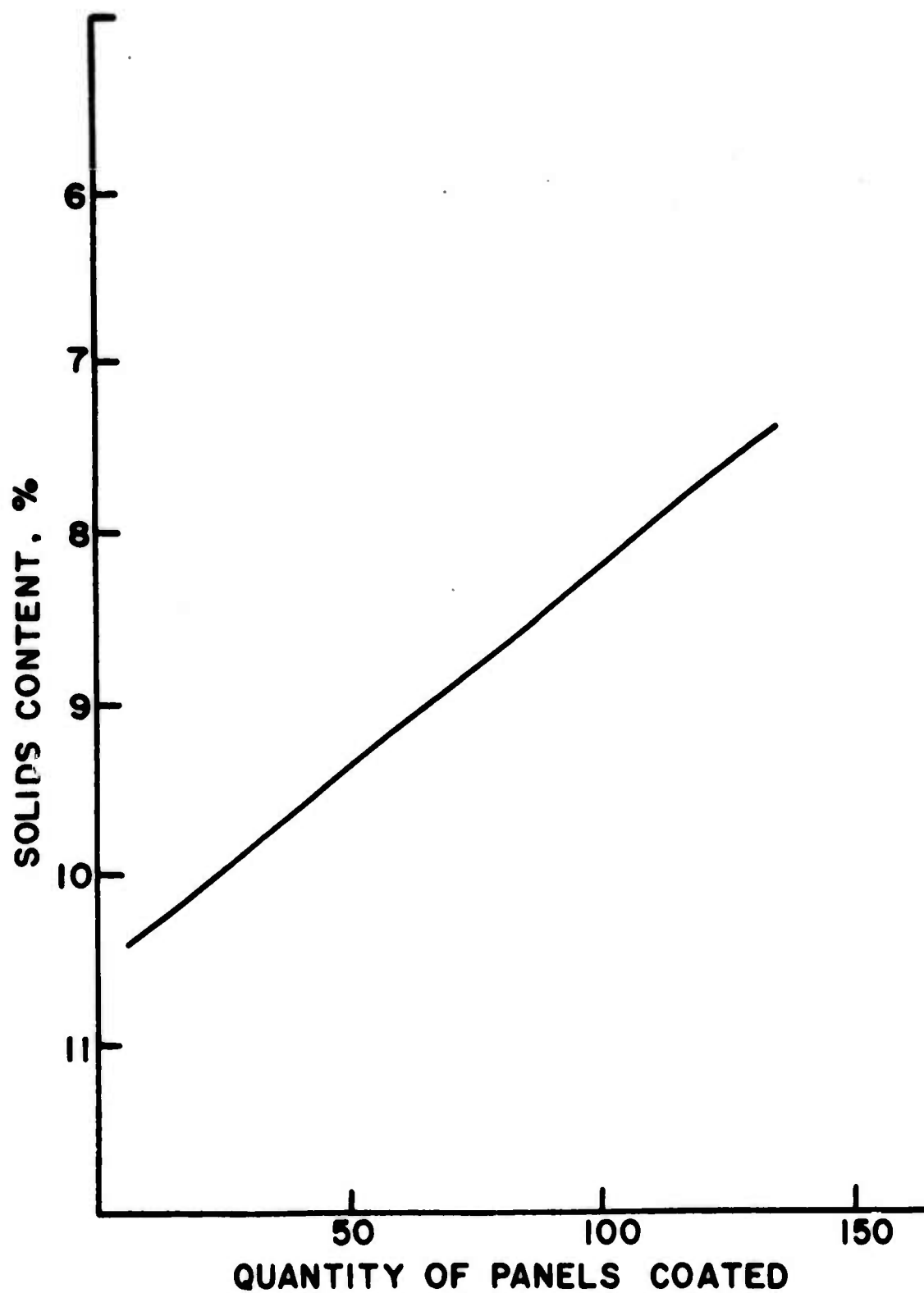
FIGURE 6

# RELATIONSHIP OF pH TO QUANTITY OF PANELS COATED



**FIGURE 7**

# RELATIONSHIP OF SOLIDS CONTENT TO QUANTITY OF PANELS COATED



**FIGURE 8**

## RELATIONSHIP OF CORROSION RESISTANCE TO FILM THICKNESS

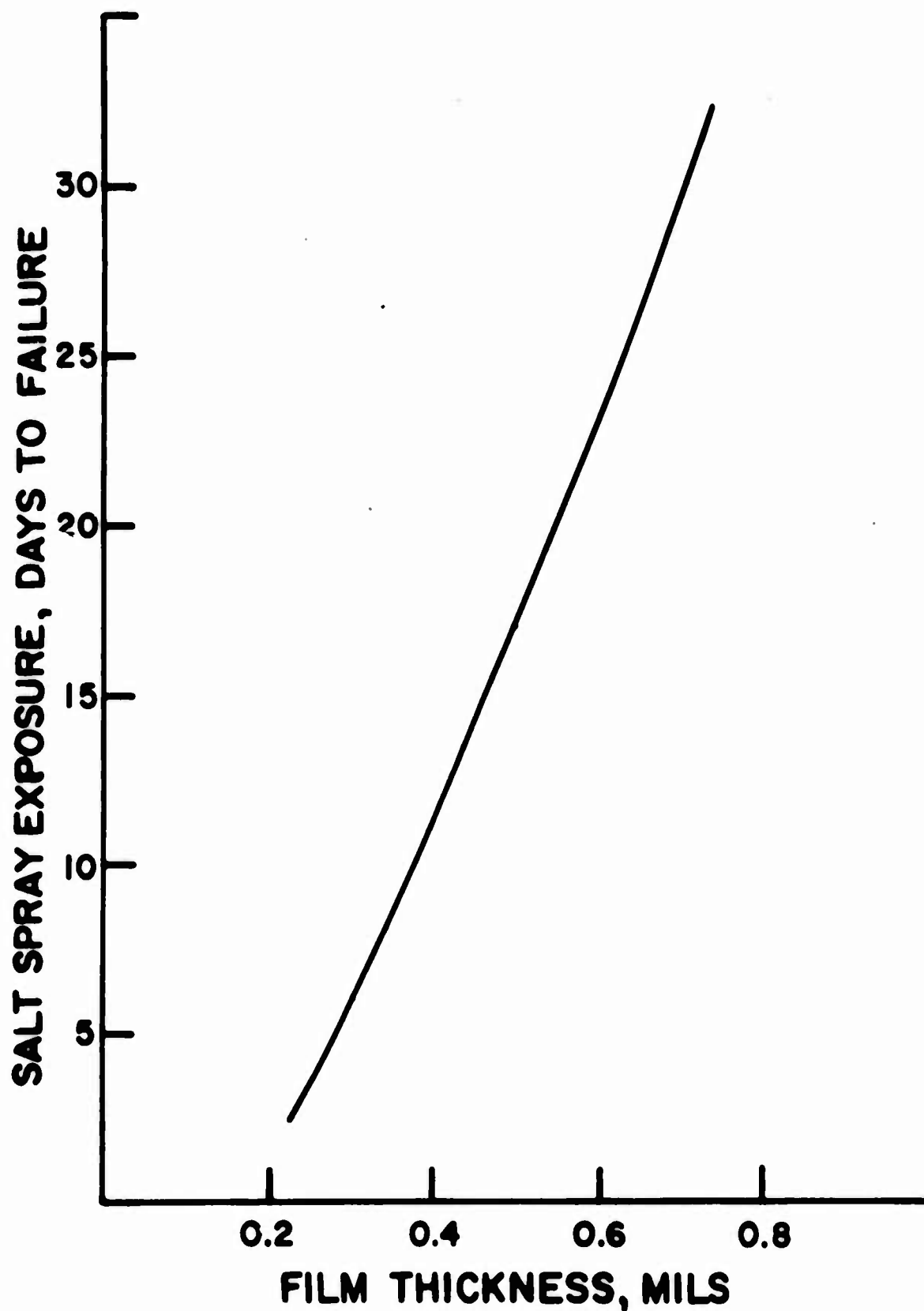
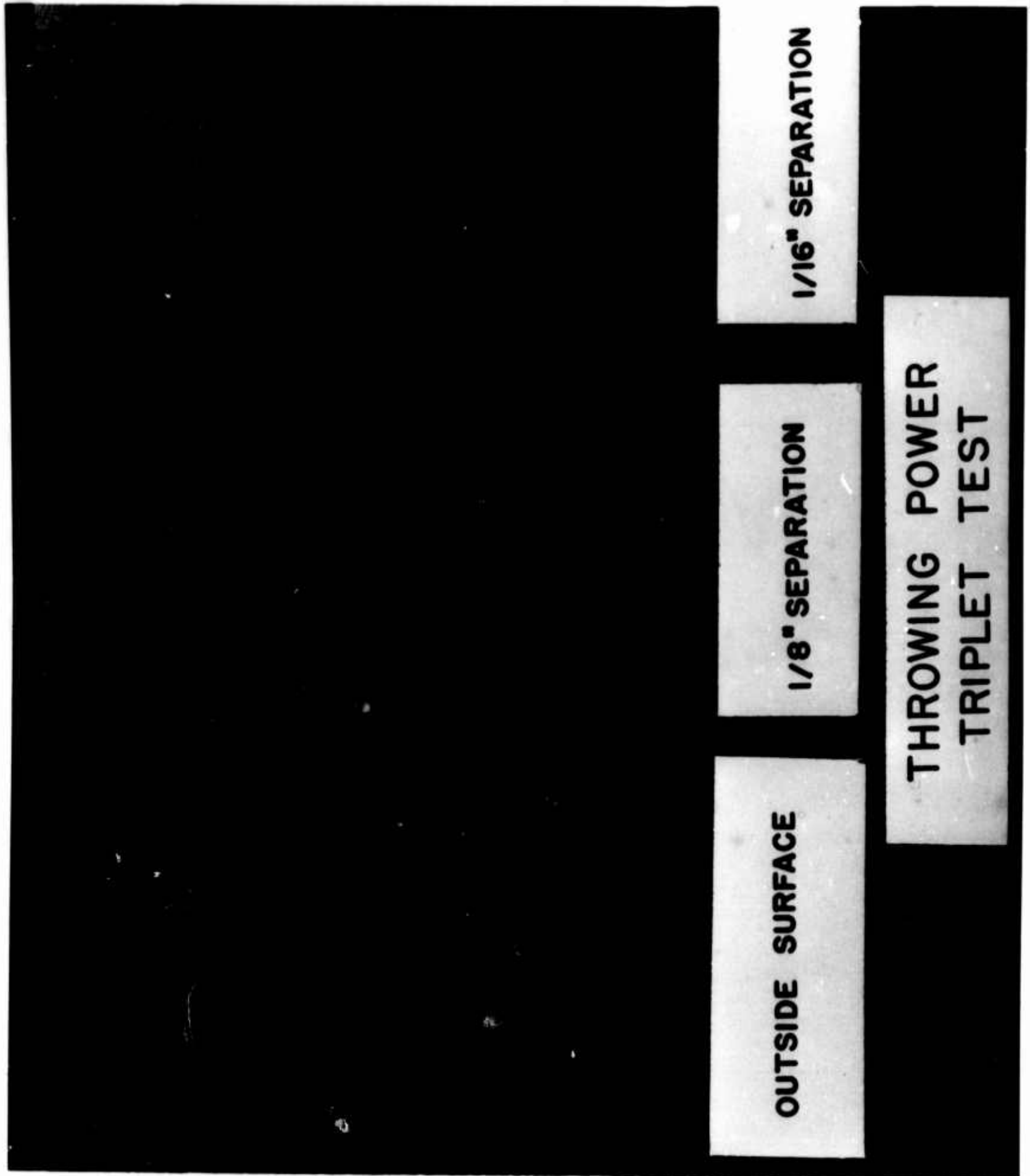


FIGURE 9



OUTSIDE SURFACE

1/8" SEPARATION

1/16" SEPARATION

THROWING POWER  
TRIPLET TEST

**FIGURE 10**

NORMAL  
DIP APPLICATION  
40% SOLIDS

5.0 MINUTES  
200 VOLTS  
10% SOLIDS

3.0 MINUTES  
200 VOLTS  
10% SOLIDS

1.5 MINUTES  
200 VOLTS  
10% SOLIDS

THROWING POWER  
TUBE PENETRATION TEST

FIGURE 11

Unclassified

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11. SUPPLEMENTARY NOTES	12. SPONSORING MILITARY ACTIVITY U. S. Army Materiel Command Washington, D. C. 20315	
13. ABSTRACT <p>A brief discussion on the electrodeposition of organic coatings is presented as the result of an extensive literature survey through recent technical journals and publications. The feasibility of the process for possible military usage was studied using a specific proprietary coating. Laboratory work on processing parameters indicated that electrodeposition would offer advantages for uniformly coating equipment with complex configurations which cannot be adequately coated by other application methods. Before this process can be extensively used more technical data on electrocoating mechanisms suitable vehicles, pigments, additives, formulation parameters and means for effectively maintaining a uniform composition during electrocoat processing will have to be developed so that the quality of the deposited films can be controlled.</p>		

14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
<b>Electrodeposition</b> <b>Organic Coatings</b> <b>Processing parameters</b> <b>Electrophoresis</b> <b>Electro-osmosis</b>						

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