CCL REPORT NO. 268

FINAL REPORT

STUDY OF LONG TERM PACKAGING OF ETHYLFNE GLYCOL ANTIFREEZE

BY

JAMES H. CONLEY

JULY 1969

U. S. ARMY ABERDEEN RESEARCH & DEVELOPMENT CENTEF
COATING & CHEMICAL LABORATORY

Aberdeen Proving Ground
Maryland

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STUDY OF LONG TERM PACKAGING OF ETHYLENE GLYCOL ANTIFREEZE

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ABSTRACT

The object of this study was to investigate conditions which would effect long term packaging of ethylene glycol antifreeze. Factors investigated were pH, dilution, different inhibitor systems and different metals.

Aluminum and tin-coated steel panels were partially immersed in ethylene glycol solutions contained in test jars at room temperatures for a period of ten years. Inhibitors used in this study included borax, borax/glycol condensate, mercaptobenzothiazole, and the sodium salt of mercaptobenzothiazole. pH values ranging from 6 to 10.5 were used. Test solutions contained 0%, 3% and 66-2/3% water.

No system tested was adequate for 10 years storage. All systems exhibited corrosion in the liquid phase and/or the vapor phase.

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I. INTRODUCTION

Coating & Chemical Laboratory was directed by Army Materiel Command to investigate improved antifreeze compounds.

Federal Specification 0-A-548, Type I antifreeze which is specified for Army use in TB-750-651 is composed of undiluted ethylene glycol with borax as an inhibitor. As packaged it has an acidic pH (below 7.0) and tends to cause corrosion of tin containers in long term storage. This material is used in conjunction with 0-I-490a, Inhibitor, Corrosion, Liquid Cooling System. One of the constituents of 0-I-490a is mercaptobenzothiazole (MBT) which greatly enhances the inhibitive properties of borax in ethylene glycol mixtures which have been diluted for vehicle use. However, chemically, MBT is an acid, and was found to rapidly corrode the tin containers when incorporated directly into undiluted 0-A-548 antifreeze packages. The Army, therefore, uses a two package antifreeze system, which is logistically undesirable.

This study was initiated primarily to study conditions which might permit the incorporation of 0-A-548 and 0-I-490a into a one package antifreeze system. Preliminary data on this study were presented in CCL Report Nos. 86 and 100. These data showed that under specified conditions ethylene glycol could be packaged with MBT and stored for short periods in both aluminum and tin coated steel containers.

This present report contains data derived from the study since the two year inspection.

II. DETAILS OF TEST

A. Test Panels.

Aluminum (alloy 2024) panels 2-1/2" by 3", were prepared by abrading with 320A carborundum paper and steel wool (00 Grade), rinsing with hot ethyl alcohol and drying in an oven at 210°F.

Tin coated steel panels, 2-1/2" x 3" were prepared by lightly abrading with steel wool (00 Grade), rinsing in hot ethyl alcohol and drying in an oven at 210°F. The edges of these panels were then dipped in an non-crystalline hydrocarbon wax so that steel edges would not be exposed.

B. Test Jars.

The glass jars used in the test were 14-1/2 ounce screw cap type, approximately 3" in diameter and 4" deep. The jars were closed with screw caps containing lacquered paper pulp liners.

C. Ethylene Glycol.

Ethylene glycol used in the tests was "purified" grade, Fisher Scientific Company. This glycol contained less than 0.5% water.

D. Inhibitors.

1. Borax - The borax used was sodium tetraborate, decahydrate, C.P. It analyzed 101%, calculated as borax, the excess percentage being due to the loss of water of hydration.

2. Borax-glycol condensate - This material was prepared in accordance with the procedure outlined in CCL Report No. 81. It analyzed 14.0 ± 1.0% sodium tetraborate (by weight).

3. Mercaptobenzothiazole - Eastman Kodak 2-mercaptobenzothiazole "practical" grade, was used.

4. Sodium salt of mercaptobenzothiazole - R. T. Vanderbilt's "NACAP", which is a 50% water solution of sodium mercaptobenzothiazole, was used in the tests in amounts corresponding to the quantity of MBT used.

E. Tests Conducted.

1. Test solutions - Thirty-five test solutions used in this study are listed in Tables I and II. A pH of 10.5 was attained by the addition of 5.0N aqueous sodium hydroxide.

2. Test procedures - Approximately 200 ml. of solution was placed in each jar. The metal test panel was approximately one half immersed. This permitted inspection of panels exposed to both the liquid phase and the vapor phase. The screw caps were firmly tightened and the jars stored on a shelf at room temperature.

3. Inspection - One set of duplicate panels were removed in two month intervals up to 8 months for inspections. The duplicate set of panels were left undisturbed for a total of 24 months storage and inspected. The panels were then placed back in the test jars and left undisturbed for a total of 10 years. The final inspection included in this study was made after 10 years storage.

III. RESULTS OF TEST

A. Effect of dilution on tin-coated steel.

Results of inspections up to 8 months and 2 years are included in CCL Report Nos. 86 and 100, respectively. Results in Table I and the photographs show all undiluted samples exhibit heavy corrosion both in the vapor phase and liquid phase. Tests with 97% glycol and 3% water

(Test Nos. 6 and 11) were better than the undiluted samples but were not as good as the tests with 66-2/3% water (Test Nos. 12 to 23A). This shows that dilution decrea es corrosion on tin-coated steel.

B. Effect of dilution on aluminum.

Results of the effect of dilution on aluminum in Table II and the photographs show that dilution increased corrosion in nearly every case. One exception was noted with 2-1/4% borax-glycol condensate, 1/2% NACAP at pH 10.5.

C. Effect of pH.

Increasing the pH of undiluted glycol to 10.5 increased the vapor phase corrosion of aluminum in every case with only a slight difference in the liquid phase corrosion. Increasing the pH to 10.5 at 3% dilution decreased the corrosion of tin-coated steel but at 66-2/3% dilution the increased pH increased corrosion.

D. Comparison of borax-glycol condensate.

Borax-glycol condensate was slightly better than borax in 66-2/3% dilution low pH tests in the liquid phase and in the high pH tests with tin-coated steel. Borax was better than the condensate in the high pH test with 1/2% NACAP at 3% dilution (Table II, Test Nos. 27 and 28).

E. Comparison of MBT and its sodium salt.

The sodium salt of MBT (NACAP) was better than MBT in almost every case. In Test Nos. 8 and 11 with 97% glycol, 2-1/4% borax and 3% water at pH 10.5 the MBT was better than the sodium salt.

F. Best over-all inhibitor combination.

In choosing a single inhibitor combination from this group which would be best on both metals, borax, MBT, and 3% water at pH 10.5 would be the best. At 66-2/3% dilution the condensate with NACAP at pH 10.5 is the best. Even these would not be considered satisfactory for long term storage due to the presence of some corrosion either in the vapor phase or the liquid phase. All corrosion is more severe after 10 years storage and in the same order as reported after 2 years storage.

IV. REFERENCES

1. Authority, AMC Program Directive.

2. Federal Specification O-I-490, Inhibitor, Corrosion, Liquid Cooling System.

3. Federal Specification O-A-548a, Antifreeze, Ethylene Glycol, Inhibited.

4. CCL Report No. 81, Development of an Operational Preservative Hydraulic Brake Fluid, August 1959.

5. CCL Report No. 86, Study of a Newly Developed Inhibitor for Ethylene Glycol in Storage, October 1959.

6. CCL Report No. 100, Storage of Ethylene Glycol with Various Inhibitors, 14 March 1961.

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APPENDIX A

TABLE I

Results of 10-Year Inspection Tin-Coated Steel Panels

Photo No.	Inhibitor Combination	Dilution, % water	Results	Solution Appearance
1	None pH 4.90	Undiluted	Very heavy vapor phase corrosion. Liquid phase full of holes.	Very rusty
2	2-1/2% Borax, pH 5.68	Undiluted	Mod-heavy vapor phase corrosion Tin completely removed in liquid phase. Steel shows intergranular corrosion.	Very rusty
3	2-1/2% Borax-Glycol Condensate pH 5.55	Undiluted	Pitting in vapor phase. Heavy rusting at interface. Tin partially removed in liquid phase. Some intergranular corrosion.	Very rusty
4	2-1/4% Borax, 1/4% MBT, pH 5.65	Undiluted	Heavy vapor phase corrosion. Liquid phase black and pitted.	Very rusty
5	2-1/4% Borax-Glycol Condensate 1/4% MBT, pH 5.55	Undiluted	Heavy vapor phase corrosion. Liquid phase black and pitted.	Very rusty
6	None pH 5.35	3	Heavy vapor phase corrosion. Tin removed from panels in liquid phase. Steel perforated with holes.	Very rusty
7	2-1/4% Borax, 1/2% NACAP, pH 6.00	3	Very heavy vapor phase corrosion. Very heavy liquid phase corrosion.	Very rusty
8	2-1/4% Borax, 1/4% MBT, pH 10.5	3	Slight vapor phase corrosion. Liquid phase okay.	Trace of sediment
9	2-1/4% Borax-Glycol Condensate 1/4% MBT, pH 5.75	3	Very heavy vapor phase corrosion. Excessive pitting in liquid phase.	Very rusty (black)

TABLE I - (Continued)

Photo No.	Inhibitor Combination	Dilution, % water	Results	Solution Appearance
10	2-1/4% Borax-Glycol Condensate, 1/4% MBT, pH 10.5	3	Vapor phase stained. Tin completely removed in liquid phase. Steel shows intergranular corrosion.	Very slight yellow sediment.
11	2-1/2% Borax Condensate 1/2% NACAP, pH 10.5	3	Moderate vapor phase corrosion. Liquid phase coated with crystals but no evidence of pits.	Very slight sediment.
12	None pH 4.55	66-2/3	Very heavy vapor phase corrosion. Panel completely dissolved except under the wax in the liquid phase.	Very rusty
13	2-1/2% Borax pH 7.82	66-2/3	Slight vapor phase corrosion. Liquid phase okay.	Clear
14	2-1/2% Borax-Glycol Condensate, pH 7.85	66-2/3	Light scattered pitting. Liquid phase stained.	Clear
15	2-1/4% Borax, 1/4% MBT pH 7.72	66-2/3	Heavy vapor phase corrosion. Liquid phase stained and coated.	Yellow sediment
16	2-1/2% Borax pH 10.5	66-2/3	Scattered pitting. Filiform corrosion in liquid phase.	Clear
17	2-1/4% Borax, 1/4% MBT, pH 10.5	66-2/3	Very heavy vapor phase corrosion. Heavy pitting and rusting in liquid phase.	Rusty
17A	2-1/4% Borax 1/4% MBT pH 10.5	66-2/3	Very slight vapor phase corrosion. Tin completely removed in liquid phase. Steel show intergranular corrosion.	Slight sediment

TABLE I - (Continued)

Photo No.	Inhibitor Combination	Dilution, % water	Results	Solution Appearance
18	2-1/4% Borax-Glycol Condensate 1/4% MBT, pH 7.55	66-2/3	Very slight vapor phase corrosion. Tin completely removed in liquid phase. Steel shows intergranular corrosion.	Slight yellow sediment
19	2-1/4% Borax-Glycol Condensate 1/4% MBT	66-2/3	Vapor phase okay. Tin partly removed in liquid phase. Some intergranular corrosion.	Slight sediment (white)
20	2-1/4% Borax, 1/2% NACAP pH 7.88	66-2/3	Slight vapor phase corrosion. Slight corrosion and stains in liquid phase.	Heavy white sediment
21	2-1/4% Borax, 1/2% NACAP, pH 10.5	66-2/3	Slight vapor phase corrosion. Light etching with tin removed in one spot.	Very slight sediment
22	2-1/4% Borax-Glycol Condensate, 1/2% NACAP, pH 7.88	66-2/3	Very slight vapor phase corrosion. Moderate-heavy stain in liquid phase.	Heavy light yellow sediment
22A	2-1/4% Borax-Glycol, Condensate, 1/2% NACAP, pH 7.88	66-2/3	Slight vapor phase corrosion. Heavy corrosion at interface on one side. Liquid phase okay.	Moderate-Heavy yellow sediment
23	2-1/4% Borax-Glycol, Condensate, 1/2% NACAP, pH 10.5	66-2/3	Slight vapor phase corrosion. Light yellow stain in liquid phase. No pitting.	Very slight sediment
23A	2-1/4% Borax-Glycol, Condensate, 1/2% NACAP, pH 10.5	66-2/3	Very slight vapor phase corrosion pitting in liquid phase	Slight sediment

TABLE II

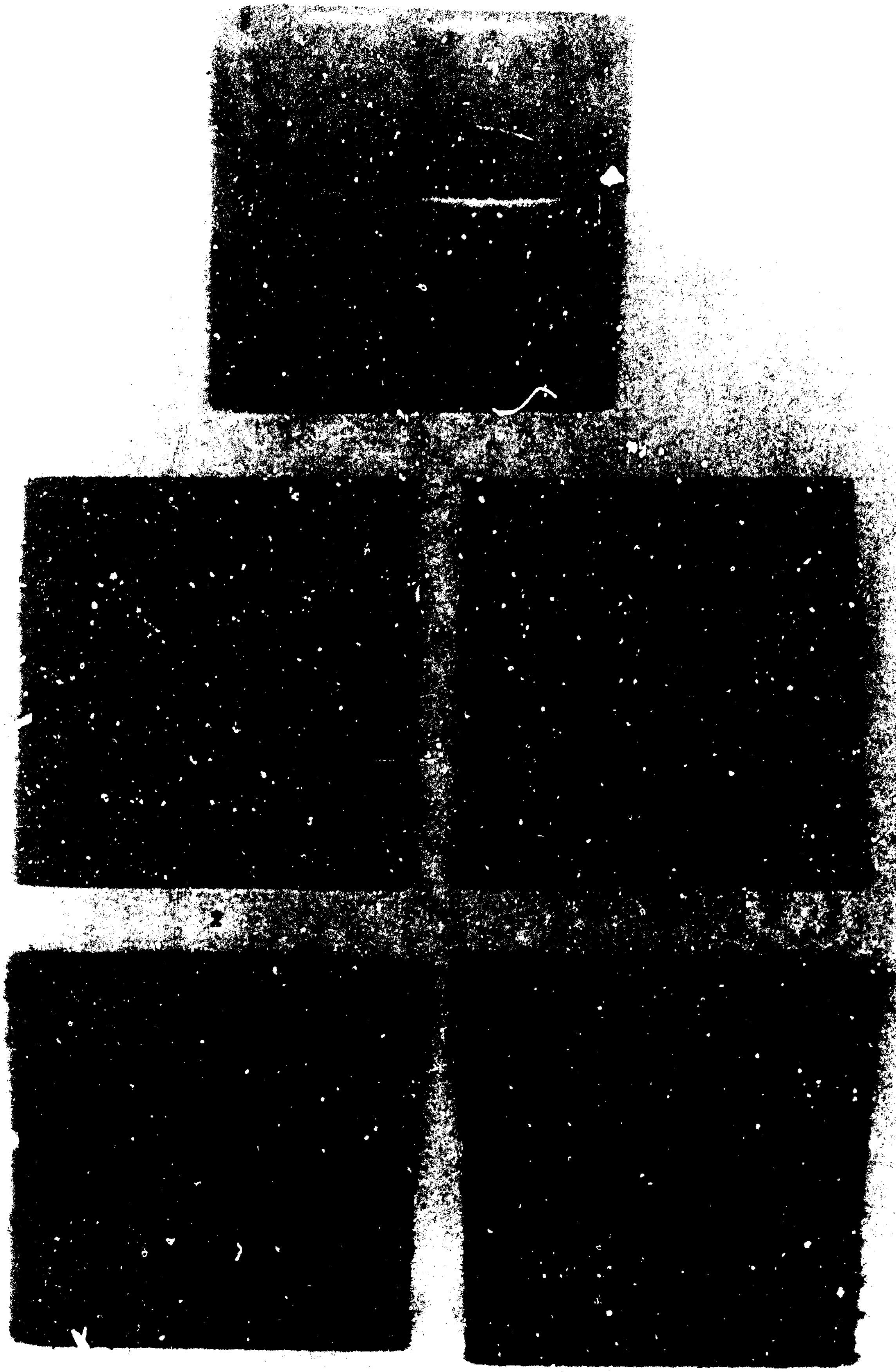
Results of 10 Year Inspection Aluminum Panels

Photo No.	Inhibitor Combination	Dilution, % water	Results	Solution Appearance
24	2-1/4% Borax, 1/4% MBT, pH 10.5	3	Heavy vapor phase corrosion. Moderate-heavy corrosion in liquid phase.	Moderate white sediment
25	2-1/4% Borax-Glycol, Condensate, 1/4% MBT, pH 10.5	3	Moderate vapor phase corrosion. Moderate corrosion in liquid phase.	Very slight sediment
26	2-1/4% Borax, 1/2% NACAP, pH 6.00	3	Moderate vapor phase corrosion. Light stain in liquid phase.	Moderate-Light yellow sediment
27	2-1/4% Borax, 1/2% NACAP, pH 10.5	3	Moderate vapor phase corrosion. Slight-moderate coating in liquid phase	Moderate sediment
28	2-1/4% Borax-Glycol, Condensate, 1/2% NACAP, pH 10.5	3	Moderate vapor phase corrosion. Heavy coating in liquid phase.	Slight sediment
29	2-1/4% Borax, 1/4% MBT, pH 7.72	66-2/3	Moderate-heavy vapor phase corrosion. Moderate-heavy coating in liquid phase.	Moderate white sediment
30	2-1/4% Borax, 1/4% MBT, pH 10.5	66-2/3	Moderate vapor phase corrosion. Heavy coating in liquid phase.	Slight-moderate white sediment
31	2-1/4% Borax-Glycol Condensate, 1/4% MBT, pH 10.5	66-2/3	Moderate vapor phase corrosion. Heavy liquid phase corrosion.	Slight sediment

TABLE II - (Continued)

Photo No.	Inhibitor Combination	Dilution, % water	Results	Solution Appearance
32	2-1/4% Borax, 1/2% NACAP, pH 7.88	66-2/3	Very slight vapor phase corrosion. Slight-moderate stain and pitting in liquid phase.	Moderate-heavy white sediment
33	2-1/4% Borax, 1/2% NACAP, pH 10.5	66-2/3	Moderate vapor phase corrosion. Light scattered pitting and stain in liquid phase.	Slight-moderate sediment
34	2-1/4% Borax-Glycol, Condensate, 1/2% NACAP, pH 7.60	66-2/3	Vapor phase okay. Light overall coating in liquid phase.	Moderate-light yellow sediment
35	2-1/4% Borax-Glycol, Condensate, 1/2% NACAP, pH 10.5	66-2/3	Slight vapor phase corrosion. Moderate pitting in liquid phase.	Very slight sediment

APPENDIX B



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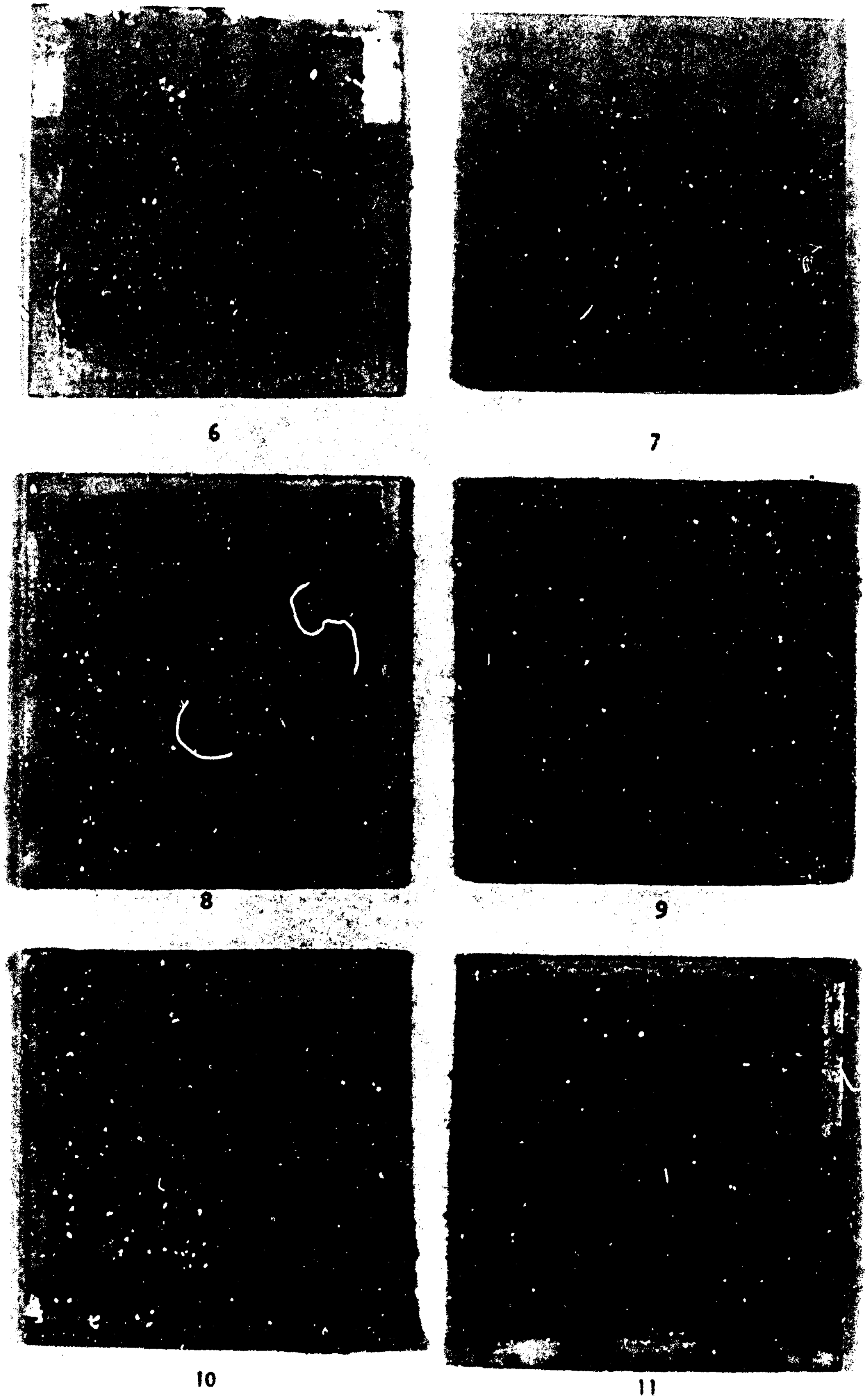
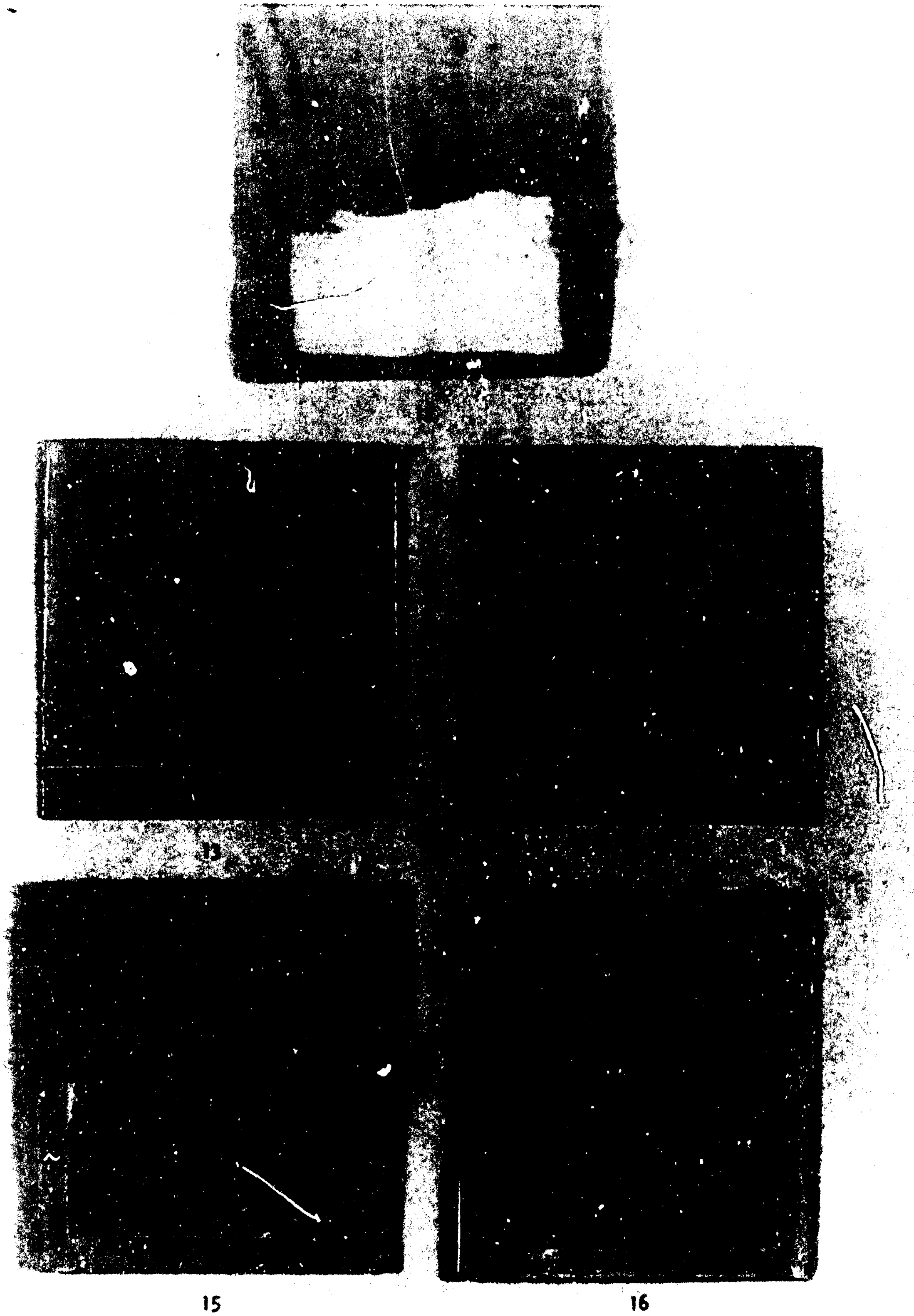
TIN COATED STEEL PANELS

Panels immersed in Undiluted Ethylene Glycol with various Inhibitors



TIN COATED STEEL PANELS

Panels Immersed in 97% Ethylene Glycol, 3% water, with various inhibitors



TIN COATED STEEL PANELS

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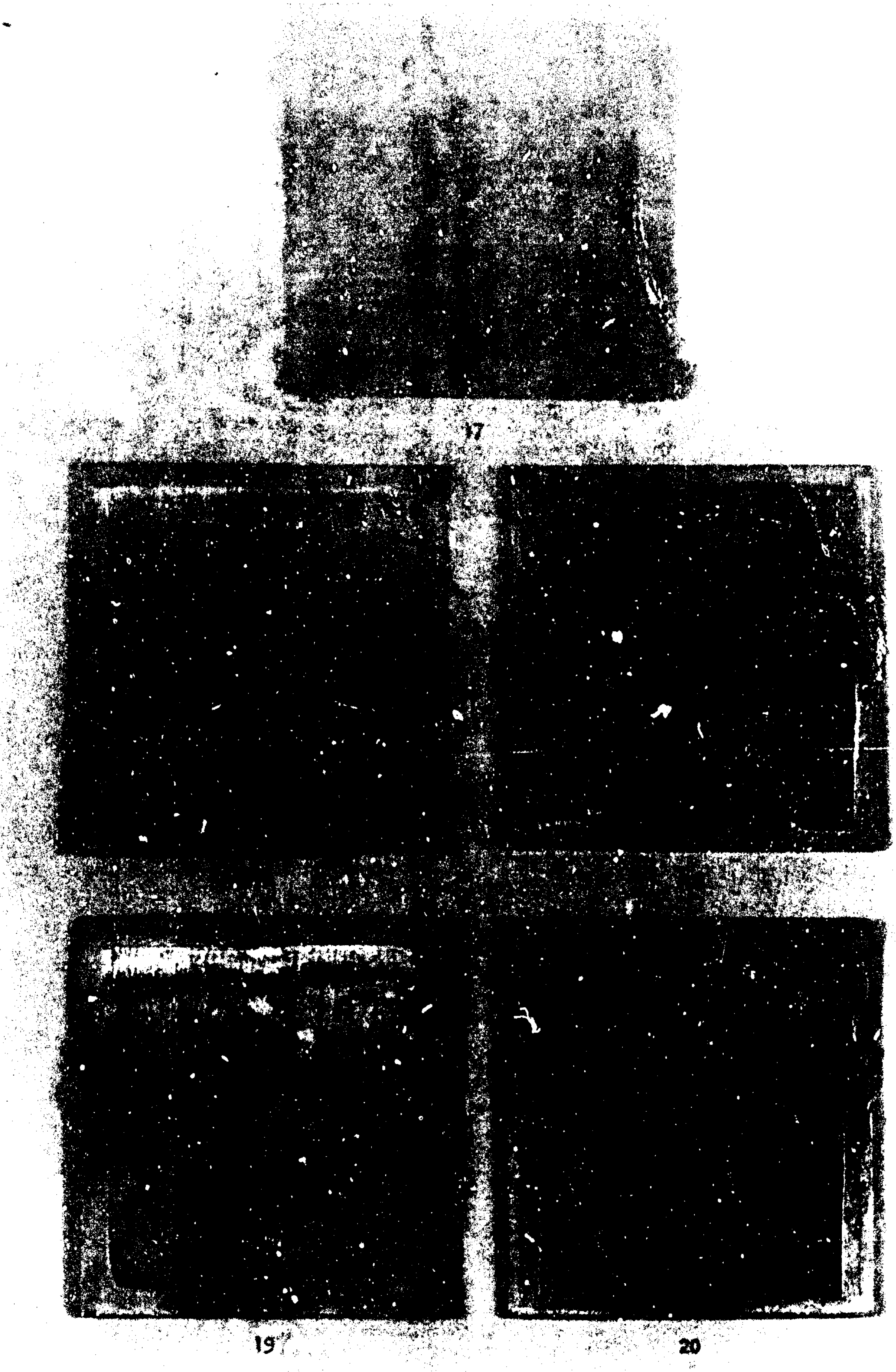


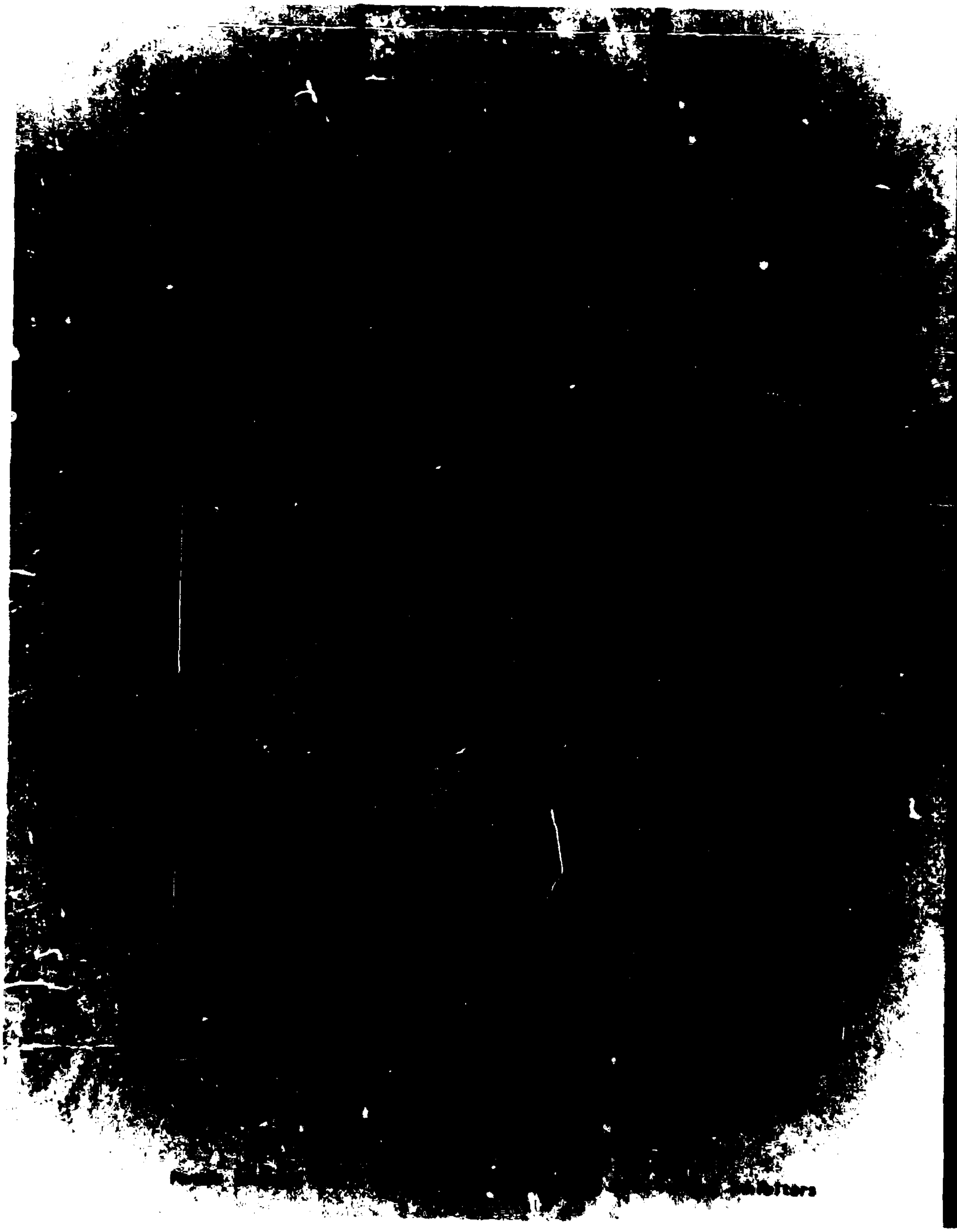
TIN COATED STEEL PANELS

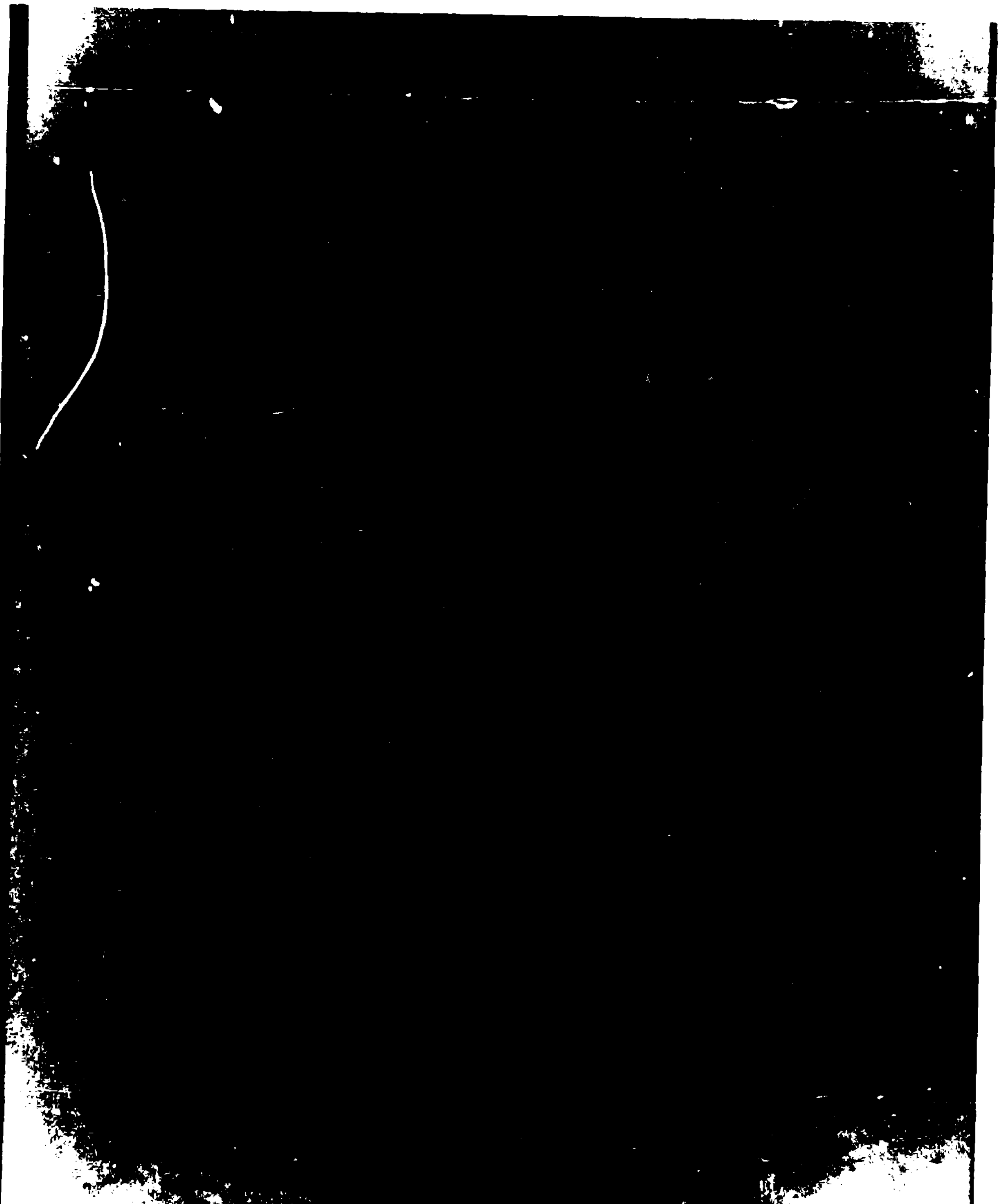
Panels immersed in 33-1/3% Ethylene Glycol, 66-2/3% water, with various inhibitors

23

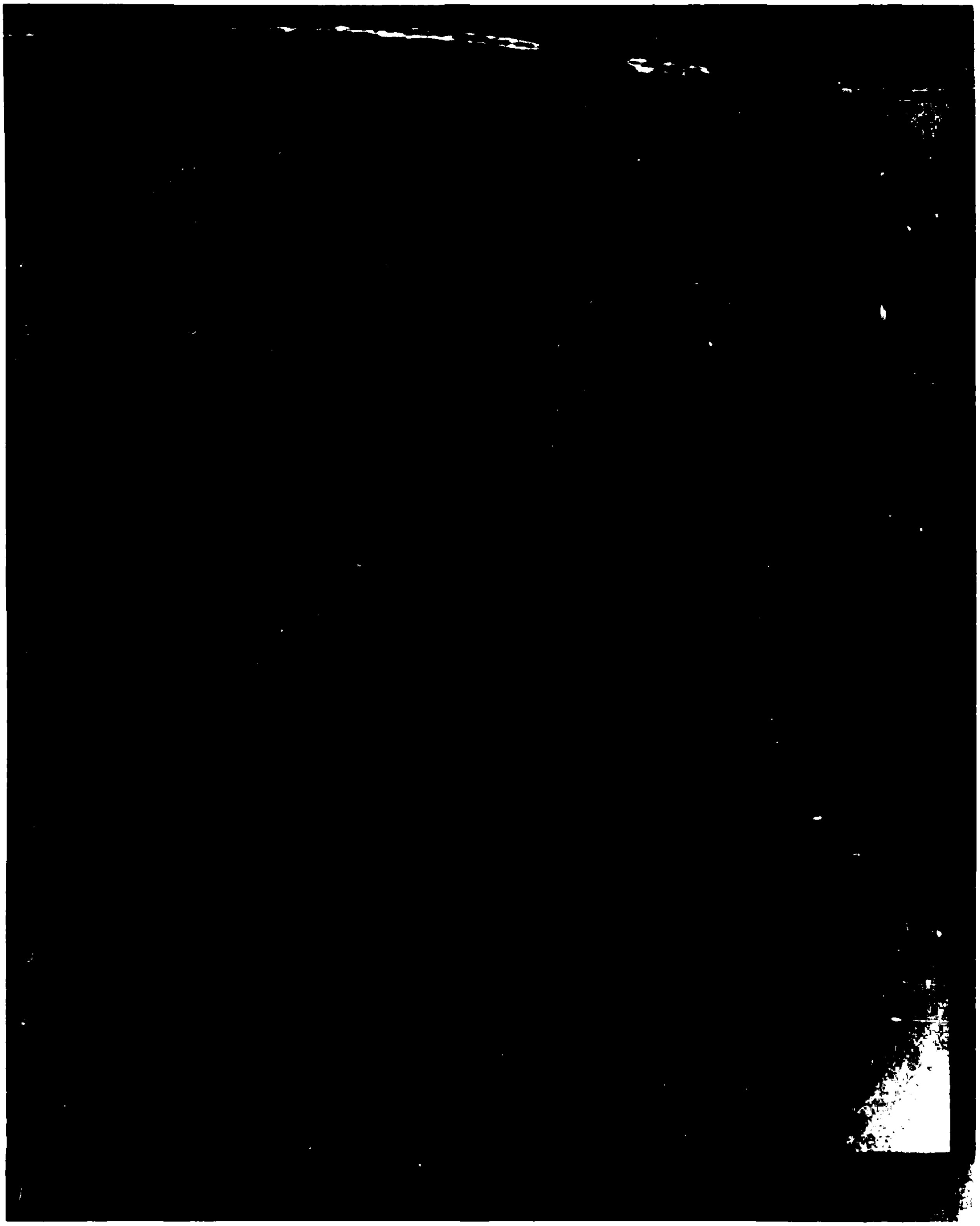
TIN COATED [illegible] PANELS

Panels immersed in 33-1/3% Ethylene Glycol, 66-2/3% water, with various inhibitors





[illegible] Inhibitors



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13. ABSTRACT

The object of this study was to investigate conditions which would effect long term packaging of ethylene glycol antifreeze. Factors investigated were pH, dilution, different inhibitor systems and different metals.

Aluminum and tin-coated steel panels were partially immersed in ethylene glycol solutions contained in test jars at room temperatures for a period of ten years. Inhibitors used in this study included borax, borax-glycol condensate, mercaptobenzothiazole, and the sodium salt of mercaptobenzothiazole. pH values ranging from 6 to 10.6 were used. Test solutions contained 0%, 3% and 66-2/3% water.

No system was adequate for 10 years storage. All systems exhibited corrosion in the liquid phase and/or the vapor phase.

KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
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Corrosion						
Mercaptobenzothiazole						
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