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ANL/EES-TM-131

CORROSIVITY OF PROPYLENE GLYCOL SOLUTION
IN SOLAR-ENERGY FLUID SYSTEMS

prepared by

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for

Energy and Environmental Systems Division
Argonne National Laboratory
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This report is one of a series of reports prepared by the staff associated with Argonne National Laboratory's Solar Reliability and Materials Program (SRMP). The SRMP, a major activity of the Energy Supply and Utilization Section of the Argonne Energy and Environmental Systems Division, is managed by R.M. Wolosewicz. This report was prepared by a subcontractor, ECA, Inc., and the principal investigators were C.P. Chen and C. Cheng. The program is sponsored by the Department of Energy (DOE) Office of Solar Applications for Buildings, Active Heating and Cooling Division, Field Application Branch; R. Hassett is the DOE Program Manager.

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CONTENTS

ABSTRACT.	1
1 INTRODUCTION	1
2 IN-SITU FLUID MONITORING	3
3 LABORATORY TESTING	8
3.1 Static Autoclave Tests.	8
3.2 Solution-Potential Tests.	8
3.3 Color-Indicator-Strip Tests	8
4 RESULTS AND DISCUSSION	14
5 SUMMARY AND CONCLUSIONS.	21
6 RECOMMENDATIONS.	22
REFERENCES.	23

FIGURES

1 Typical Solar-Energy System with Primary and Secondary Loops.	2
2 Schematic of Argonne National Laboratory Solar-Energy System.	4
3 Schematic of Columbia Gas Solar-Energy System	5
4 Schematic of Perl-Mack Solar-Energy System.	6
5 Fluid-Sensor Schematic.	7
6 Titanium Autoclave System	9
7 Solution-Potential Test Setup	12
8 Solution Potential vs. Time	13
9 Corrosion and Solution Potential vs. Time	15
10 Micrographs of Copper Pitting	16
11 Micrographs of Aluminum Pitting	17
12 Corrosion Rate vs. Solution Potential	19
13 Corrosion Test Loop	20

TABLES

1	Primary-Loop Fluid and Materials at Test Sites.	3
2	Average Solution Potential of Site Fluids	7
3	Descaling Procedures for Corrosion Coupons.	10
4	Static Autoclave Tests of Propylene Glycol Solutions.	11
5	Average Corrosion Rate and Solution Potential	18

CORROSIVITY OF PROPYLENE-GLYCOL SOLUTION IN SOLAR-ENERGY FLUID SYSTEMS

by ECA, Inc.

ABSTRACT

Degradation of inhibited glycol solution leads to corrosion of metals in a solar-energy system. Corrosion was monitored at three sites. To evaluate the extent of corrosion, accelerated laboratory tests were performed. These tests were directed toward controlling fluid corrosion in solar-energy systems.

1 INTRODUCTION

The assessment of corrosion of materials by fluids is a part of the ANL Solar Reliability and Materials Program, sponsored by the Solar Applications Branch at the U.S. Department of Energy (DOE). A typical solar-energy system containing primary and secondary loops is illustrated in Fig. 1. In cold climatic regions, where temperatures can drop below 32°F (0°C), a glycol solution or hydrocarbon oil is often used as the primary fluid to prevent freezing. However, degradation of a glycol solution or the breakdown of its organic inhibitors with time and/or temperature can lead to corrosion at the internal surface of the primary loop. This interim report is a preliminary study of glycol-solution-caused corrosion of the metals in the primary loop.

Inhibited glycol solutions normally degrade, and their inhibitors break down to form acids (e.g., glycolic and formic) that can corrode the metals in the primary loop. During normal operation of the system, the temperature of the primary-loop fluid cycles from 85°F (29°C) to 190°F (88°C). As a result, the corrosivity of the primary-loop fluid increases. When stagnation occurs, temperatures as high as 300-350°F (149-177°C) are possible, and at these high temperatures the solution's corrosivity is increased further. Additional corrosive action can occur if oxygen leaks into or is trapped in the system.

A correlation between in-service data at solar sites and data from accelerated laboratory tests with controlled variables is discussed in this report.

Fig. 1 Typical Solar-Energy System with Primary and Secondary Loops

2 IN-SITU FLUID MONITORING

Texas Instruments, Inc., has developed a voltage corrosivity sensor to detect degradation of antifreeze solutions (inhibited ethylene glycol and water mixture) in automobile engines.¹ When perfected, such a sensor can be mass-produced at a cost of only \$5 to \$10. For in-situ monitoring, a modified version of this fluid sensor (FLS) was installed in the piping section adjacent to the solar collectors, where it continuously monitors changes in solution potential.

Originally, five sites were selected from the DOE Commercial Solar Demonstration Program. These sites are connected by data-acquisition channels to the National Solar Data Network (NSDN) facility in Silver Springs, Md. The transmitted data are subsequently transcribed onto tapes and sent to Argonne National Laboratory (ANL) for analysis. Due to a reduction in funding, the number of sites was reduced from five to three: Argonne National Laboratory Cafeteria (ANL), Argonne, Illinois; Columbia Gas Company (COG), Columbus, Ohio; and Perl-Mack Enterprises (PEM), Denver, Colorado.

A summary description of the fluid and the metals in the primary loop for each of the three sites is given in Table 1, and the schematics of the three sites are shown in Figs. 2-4. Each figure also shows the location of the FLS and its corresponding temperature probe (FC). The FLS is connected to a signal conditioner, which amplifies the solution potential and generates an output signal that is fed to the J-box of the site data-acquisition system. The FLS layout and the signal conditioner are shown in Fig. 5.

The FLS is sensitive to temperature fluctuation, and its voltage reading decreases with increasing temperature.² Since inhibited glycol solutions are stable and not susceptible to degradation at room temperature, 100°F (38°C) was arbitrarily chosen as the minimum temperature ($T_{\min}$) to use in finding the monthly average voltage at each site. The results are summarized in Table 2. In the case of the ANL site, it was necessary to reduce $T_{\min}$ to 75°F (24°C) to assure a reasonable number of hours for averaging the voltage.

Table 1 Primary-Loop Fluid and Materials at Test Sites

Loop Trait	Site and Date Installed		
	ANL, Argonne, Ill. Feb. 9, 1979	COG, Columbus, Ohio May 3, 1979	PEM, Denver, Colo. Feb. 14, 1980
Fluid	50% Citgo inhibited ethylene glycol solution	57% Dowtherm SR-1 inhibited ethylene glycol solution	52% Dowfrost inhibited propylene glycol solution
Materials			
Collector	Carbon steel	Inconel	Galvanized steel
Pipe	Carbon steel	Carbon steel	Copper
Heat exchanger	Carbon steel	Carbon steel	Copper

Fig. 2 Schematic of Argonne National Laboratory Solar-Energy System

- I001 COLLECTOR PLANE TOTAL INSOLATION
- I002 COLLECTOR PLANE DIFFUSED INSOLATION
- I003 NORMAL INCIDENCE PYRHELIOMETER
- T001 OUTDOOR TEMPERATURE

Fig. 3 Schematic of Columbia Gas Solar-Energy System

Fig. 5 Fluid Sensor Schematic

Table 2 Average Solution Potential of Site Fluids

Month	Total hours	Avg. Temp. (°F)	Avg. Voltage (V)
ANL: 50% Citgo inhibited ethylene glycol; $T_{\min} = 75^{\circ}\text{F}$ (24°C)			
Dec. 1979	158	78	-0.004
Feb. 1980	159	85	-0.003
Apr. 1980	148	91	-0.002
COG: 57% Dowtherm SR-1 inhibited ethylene glycol; $T_{\min} = 100^{\circ}\text{F}$ (38°C)			
Dec. 1979	155	129	-0.338
Jan. 1980	37	115	-0.287
Feb. 1980	139	129	-0.287
Mar. 1980	178	130	-0.357
Apr. 1980	208	141	-0.316
PEM: 52% Dowfrost inhibited propylene glycol; $T_{\min} = 100^{\circ}\text{F}$ (38°C)			
Apr. 1980	386	124	-0.034

3 LABORATORY TESTING

Three kinds of laboratory tests were conducted with the glycol solutions:

- 1 Static autoclave tests
- 2 Solution-potential tests
- 3 Color-indicator-strip tests

3.1 STATIC AUTOCLAVE TESTS

A titanium static autoclave system with a stirrer and heat-flux specimen holder was used to study degradation of propylene glycol solutions (see Fig. 6). Titanium was selected for the static autoclave system because it is inert to propylene glycol solutions.

Three types of solution were selected: an uninhibited propylene glycol from Dow Chemical, and two inhibited propylene glycols, Dowfrost* and Sunearth.** Each solution was mixed with ASTM corrosive water (ASTM D-2570-73) in a 1:1 ratio by volume. The water was formulated to contain 100 ppm each of chloride, sulfate, and bicarbonate.

Metal pipe sections were inserted over the Teflon-covered specimen holder, and the metal pipe sections were maintained at a heat flux of 300 Btu/ft². The metals tested include aluminum (6061), copper (CA122), and black iron (1008 carbon steel). The pipe sections were subsequently cleaned for weight-loss measurement according to ASTM standard procedure G1-72 (see Table 3). Similarly, maximum and average pit depths were measured according to ASTM G46-76.

Six test runs were performed. Runs 1 and 2 were thermally cycled between 221°F (105°C) and 329°F (165°C) and runs 3-6 were kept at a constant temperature of 352°F (178°C). The test conditions are summarized in Table 4.

3.2 SOLUTION-POTENTIAL TESTS

The glycol solutions exposed in runs 1-6, as well as sample solutions collected from the three NSDN sites, were measured for solution potential using the test setup shown in Fig. 7. In this test, the solution potential fluctuates for the first 21 hours before a steady voltage is maintained (see Fig. 8). Thus, the potential for each fluid was measured after 24 hours at a solution temperature of 176°F (80°C).

3.3 COLOR-INDICATOR-STRIP TESTS

Three types of commercially available color indicator strips were used. The Nalcool-2000[†] strip is designed for testing Nalcool-2000 inhibited ethylene glycol solutions. The color change when the paper is dipped into the

* Registered trademark of Dow Chemical Company.

** Registered trademark of Sunearth Chemical Company.

† Registered trademark of Nalco Chemical Company.

Fig. 6 Titanium Autoclave System

Table 3 Descaling Procedures for Corrosion Coupons

Coupon Material	Solution			Procedure	
		Ingredients	Amount	Sol. Temp.	Step ^a Time (min)
Aluminum	A	Chromic acid, CrO ₃	20 g	176°F (80°C)	1 Dip in solution A. 10
		Phosphoric acid, H ₃ PO ₄	50 mL		2 If film remains, dip in solution B. 1
		Water	to make 1000 mL		3 If tenacious film remains, use bristle scouring. 1
	B	Nitric acid, HNO ₃		room temp.	
Copper	C	Hydrochloric acid, HCL	25 mL		1 Dip in solution C. 3
					2 If tenacious film remains, use bristle scouring. 0.5-1
		Water	25 mL		
Black iron	D	Sulfuric acid, H ₂ SO ₄	100 mL	122°F (50°C)	1 Scrub with brass scraper and brush.
					2 Use bristle scouring. 5-10
		Rhodine (organic inhibitor)	1.5 mL		3 Dip in solution D. (Foaming may occur; use pan outside.) initial specimen: 1
		Water	to make 1000 mL		4 Use bristle scouring. exposed specimen: 0.5

^aAt the end, rinse specimen in tap water and use ultrasonic cleaning in Freon TF for 3 min.

Table 4 Static Autoclave Tests of Propylene Glycol Solutions

Conditions and Results	50% Dow ^a Run 1 ^c	50% Dowfrost ^b			50% Sunearth ^b	
		Run 2 ^c	Run 3	Run 4	Run 5	Run 6
Test conditions						
Temp. (°F)	221-329	221-329	352	352	352	352
Time (hr)	480-8	700-192	260	500	462	336
Metal loss (mils) ^d						
Iron (1008)	0.21 W	0.14 W	0.04 W	0.12 W	0.12 W	0.10 W
Copper (CA122)	0.01 W	0.11 W	0.01 W	0.03 W	2.8 P _m 1.8 P _a	2.4 P _m 1.4 P _a
Aluminum (6061)	0.01 W	0.35 W	6.6 P _m 5.3 P _a	12.0 P _m 9.8 P _a	8.0 P _m 5.8 P _a	6.4 P _m 5.2 P _a
Solution potential (V) at 176°F (80°C)						
Initial	-0.51	-0.16	-0.16	-0.16	-0.07	-0.07
Final	-0.46	-	-0.2	-0.23	-0.2	-0.17

^aUninhibited propylene glycol solution.

^bInhibited propylene glycol solution.

^cThermally cycled.

^dW = average metal loss; P_m = maximum pit depth; P_a = average pit depth.

solution indicates the freezing-point temperature and inhibitor condition (0-depleted, 600-danger, 200-satisfactory). Similarly, a Dowfrost strip is made for testing Dowfrost (inhibited propylene glycol); the color change relates freezing-point temperature, reserve alkalinity, and inhibitor condition. A Sunearth strip is made for testing Sunearth (inhibited propylene glycol); the color change gives a pH range of 6.4 to 8.4, and inhibitor addition is recommended if the pH is 8 or lower.

All three color indicator strips were used to test the solutions from runs 1-6 and solution samples from the three NSDN sites. The color change in each case was evaluated.

Fig. 7 Solution-Potential Test Setup

Fig. 8 Solution Potential vs. Time

4 RESULTS AND DISCUSSION

Metal loss or pit depth from runs 3-6 (see Table 4) for inhibited propylene glycol solutions (Dowfrost and Sunearth) at the stagnant-full temperature of 352°F (178°C) is plotted in Fig. 9 against exposure times of up to 500 hours. The 500-hour time period is equivalent to 9.6 hours stagnation every week of a one-year period. Black iron (1008 carbon steel) is considered satisfactory, since the metal loss is estimated at about 2.5 mils in 20 years. Under the same conditions, copper is not expected to have any significant metal loss in Dowfrost solution; however, a 20-mil copper absorber plate will pit through within 10 years in Sunearth solution. Both Dowfrost and Sunearth will probably perforate a 20-mil aluminum absorber plate in less than three years.

The pitting behaviors of copper and aluminum, shown in micrographs in Figs. 10 and 11, respectively, are quite different. When copper is exposed to Sunearth solution, the pits follow the extrusion marks in the longitudinal direction of the pipe section. Presumably, surface inclusion may be a factor. With aluminum, scattered pits were observed when the metal was exposed to Dowfrost solution, whereas clusters of pits were noted when it was exposed to Sunearth solution (see Fig. 11).

The relationship between solution potential and metal loss or pit depth is indicated in Table 5 and Fig. 12; the average corrosion rates (in metal loss or maximum pit depth) and the solution potentials are extrapolated by the step method -- i.e., by averaging the values between two incremental test periods. Long-term tests are needed to determine the steady corrosion rates. However Fig. 9, based on limited data from testing the exposed inhibited propylene glycol solutions at 176°F (80°C), shows that 50% Dowfrost solution has one set of solution potentials, while 50% Sunearth solution has another. Thus, the inhibitors and other chemicals added to glycol solution greatly affect the solution potential. In the case of Dowfrost solution, if 1.5 mils/yr, or 30 mils in 20 years, is the limiting factor for the collector system, the critical solution potential is -0.2 V for black iron (1008 carbon steel) and -0.33 V for copper (CA122). For iron in Sunearth solution, the critical solution potential is -0.12 V. There is no critical solution potential for aluminum (6061) in Dowfrost solution or aluminum (6061) and copper (CA122) in Sunearth solution, as shown by the extremely high maximum pitting rate.

To assess the corrosivity of glycol solutions at various sites, it is necessary to conduct laboratory tests to determine the critical solution potential for specific metals under operating and stagnant-full temperature conditions. Results of such a test under stagnant-full conditions are shown in Fig. 12. A specially designed corrosion test loop has just been completed (see Fig. 13). This loop will be able to simulate both operating and stagnant-full in-service conditions.

The results of the indicator-strip tests are as follows:

- Each indicator strip is suitable for a specific metal in a designated solution.

Fig. 9 Corrosion and Solution Potential vs. Time

Run 4
Dowfrost
Control

Run 5
Sunearth
462 hr

Run 6
Sunearth
336 hr

Fig. 10 Micrographs of Copper Pitting

Run 3
Dowfrost
260 hr

Run 4
Dowfrost
500 hr

Run 2
Control

Run 5
Sunearth
462 hr

Run 6
Sunearth
336 hr

Fig. 11 Micrographs of Aluminum Pitting

Table 5 Average Corrosion Rate and Solution Potential

Results	50% Dowfrost		50% Sunearth	
	Run 3, 130 hr	Run 4, 380 hr	Run 5, 399 hr	Run 6, 168 hr
Avg. corrosion or pitting ^a				
Iron (1008)				
(mils)	0.02 W	0.08 W	0.11 W	0.05 W
(mils/yr)	1.34 W	1.84 W	2.42 W	2.61 W
Copper (CA122)				
(mils)	0.005 W	0.02 W	2.6 P _m	1.2 P _m
(mils/yr)	0.34 W	0.46 W	57.1 P _m	62.6 P _m
Aluminum (6061)				
(mils)	3.3 P _m	9.3 P _m	7.2 P _m	3.2 P _m
(mils/yr)	222 P _m	214 P _m	158 P _m	167 P _m
Solution potential (V)				
Initial	-0.16	-0.16	-0.07	-0.07
Final	-0.18	-0.215	-0.185	-0.12

^aW = metal loss; P_m = maximum pit depth

- Sunearth indicator strip is applicable to a Sunearth solution of inhibited propylene glycol and ASTM water used with black iron (1008 carbon steel), but not with aluminum (6061) or copper (CA122).
- Dowfrost indicator strip is applicable to a Dowfrost solution of inhibited propylene glycol and ASTM water used with black iron (1008 carbon steel) or copper (CA122), but not with aluminum (6061).
- Nalcool-2000 indicator strip appears applicable to solutions of Citgo and Dowtherm SR-1 inhibited ethylene glycol and ASTM water used with black iron (1008 carbon steel) in the ANL and COG systems, respectively. But the same indicator strip is not satisfactory for Polar or Jack Frost solution of Dearborn #537 inhibitor added to inhibited ethylene glycol and ASTM water used with black iron (1008 carbon steel). Failure analysis on the Lincoln Housing Authority collector² showed erosion-corrosion and perforation of the outlet carbon-steel tube due to insufficient nitrite content (600 ppm) in Jack Frost solution. On the other hand, the Polar solution containing 3000 ppm nitrite and with Dearborn #537 inhibitor added has been satisfactory in solar hot-water systems of carbon steel.
- Both Nalcool-2000 and Dowfrost indicator strips are satisfactory for obtaining the freezing-point temperature in ethylene or propylene glycol solutions.

Fig. 12 Corrosion Rate vs. Solution Potential

Fig. 13 Corrosion Test Loop

5 SUMMARY AND CONCLUSIONS

Corrosion of metals in inhibited propylene glycol solution is a complex process associated with the degradation of the glycol and the breakdown of the inhibitors. The corrosivity of the fluid and its solution potential are greatly affected by the synergistic effects of various inhibitors and the kind of metal exposed to the fluid.

Commercially available inhibited propylene glycol with Dowfrost appears satisfactory for black iron (1008 carbon steel) and copper (CA122). So far, an inhibitor for solutions used with aluminum has not been developed.

The experimental fluid sensor of Texas Instruments, Inc., is sensitive to temperature and flow fluctuations. Each inhibited propylene glycol solution has a critical potential for a specific metal. Below this voltage, corrosion or pitting becomes significant. Laboratory tests are needed to establish the critical value for specific applications.

Each color indicator strip is designed for evaluating the condition of the inhibitor in a specific inhibited glycol solution. Nalcool-2000 and Dowfrost strips, designed for ethylene and propylene glycol respectively, are interchangeable in detecting freezing-point temperatures.

6 RECOMMENDATIONS

The study of corrosion of metals in inhibited glycol solutions should be continued to establish some simple rules of thumb for solar-energy-system users to follow. It will be necessary to:

- Conduct laboratory tests using the specially designed corrosion loop to simulate the thermal-cycling effects of operating and stagnant-full conditions with respect to time, temperature, and flow rate, as well as metal combinations.
- Calibrate the experimental fluid sensor (FLS) to specific inhibited glycol solutions for mixed metals.
- Consider a side-loop at constant temperature for in-situ monitoring by FLS after laboratory calibration.
- Develop a color indicator strip to detect inhibitor conditions of ethylene or propylene glycol solution in specific metal systems.
- Consider the use of a stable inorganic inhibitor instead of the unstable organic inhibitors presently used in the chemical formulations for glycols. Inorganic inhibitors (e.g., molybdates) are thermally stable at stagnant-full temperatures.

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