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Schenectady, New York

CORROSION OF REACTOR STRUCTURAL MATERIALS  
IN HIGH-TEMPERATURE WATER

II. STATIC CORROSION BEHAVIOR AT 600 TO 680°F

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ABSTRACT

Possible structural materials for use in the primary coolant system and for fuel element fabrication in a water-cooled and -moderated reactor were extensively corrosion-tested in static water and various aqueous media at 600 and 680°F. Both AISI Type 304 and AISI Type 347 stainless steels were found to react with water at these temperatures at a rate of 3 to 5 mg of metal/dm<sup>2</sup>/500 hr. Carbon steel (ASTM A201) corroded at a rate approximately 10 times greater (40 mg/dm<sup>2</sup>/mo). Steels containing 2 1/4 and 5% chromium corroded at about 80 mg/dm<sup>2</sup>/mo.

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INTRODUCTION

In support of advanced water-cooled reactor designs, work was undertaken in the Chemistry and Chemical Engineering Section of KAPL to investigate the corrosion behavior of certain structural materials in water at temperatures in the range between 500°F, where much work has been done by other investigators, and approximately 700°F. Accordingly static corrosion tests have been run at 600 and 680°F, all for relatively short periods of times (500 to 1000 hr). In light of other experience, it seems certain that these tests are sufficiently long to ensure reliable comparative corrosion rates. However, these rates cannot be considered as final engineering proof that a structural material is satisfactory for a particular reactor application.

As mentioned above, the corrosion behavior of many reactor structural materials had been thoroughly investigated in water at about 500°F. There was only a limited amount of work done at higher temperatures. Most of this work was done at Argonne National Laboratory, Westinghouse Atomic Power Division, and Babcock & Wilcox Company Research Center. This early work is covered by several reports.\*

Since many types of materials are of interest in reactor operation, the program included studies of the corrosion behavior of the following general classes:

Ceramics. These are useful as electrical insulators or bearing materials in high-temperature water.

Primary coolant systems materials. In this work specimens of likely materials were tested at the lower temperature, 600°F.

Fuel element construction materials. Since this service is the most severe in the reactor from the standpoint of temperature, the more corrosion resistant materials were tested at 680°F. Clearly fuel element materials cannot be chosen without regard for the nuclear properties. Our selection was limited, thus, to those deemed promising by the nuclear design engineers.

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\*ANL-4519, "Corrosion of Some Structural Materials," by C. S. Bredin, et al. October 1948 - June 1951.

Babcock & Wilcox Research Center Report No. 5312, "Corrosion of Some Structural Materials in High-Temperature Water with Controlled Concentrations of Oxygen or Hydrogen," by S. C. Datsco, September 25, 1953.

WAPD-92, "Corrosion Behavior of Zircaloy-2," by K. M. Golden and D. E. Thomas, February 19, 1954.

EXPERIMENTAL TECHNIQUESEquipmentTypes of Autoclaves

The following commercial autoclaves were used for all of the tests:

1. A 250-ml Reactor Assembly as per Drawing 20-1711, Autoclave Engineers, Inc., Erie, Pa. [Figure 1 (KS-774)].
2. Micro Reaction Vessel, Cat. No. 406-356X4, American Instrument Co., Silver Springs, Md.

A limited experience was gained with a third type:

3. A 250-ml Reactor Assembly Cat. No. 6025-AS-347 with the Autoclave Engineers Self Sealing Closure, Autoclave Engineers, Inc., Erie, Pa.

All of the above autoclaves are fabricated of AISI Type 347 stainless steel. The first two use a confined gasket-type closure, while the third utilizes a modified Bridgeman closure. A disassembled view of the Autoclave Engineers Reactor Assembly Autoclave (Type 1) is given in Figure 1 (KS-774). This design has proved very easy to use, principally because of the Acme-type threads by which the cap is fitted to the autoclave body.

In practice the hole at the top of all of the autoclaves is sealed with a plug and gland nut. The confined gasket autoclaves required the use of a torque wrench to obtain a leakproof closure. Once the closure technique was mastered the autoclave gave excellent service in the region tested, the upper limits being a maximum temperature of 680°F and maximum pressure of about 3000 psi. Limited experience with the modified Bridgeman seal (Type 3) indicates that it is difficult to seal and consequently unreliable for these tests. This is logical since the Bridgeman seal is designed for use with pressures considerably higher than any encountered here.

Furnaces

Autoclaves were heated in two ways:

1. Vertical Heat Treating Furnaces, Model PT 2222, manufactured by the K. H. Huppert Co., Chicago, Ill.
2. Autoclave Heating Jackets, Cat. No. 406-35H 1, manufactured by the American Instrument Company.

The pit-type furnaces accommodate 8 to 10 autoclaves at one time. Heating coils are open around the sides and a powerful circulating fan ensures a remarkably uniform distribution of heat. A temperature "map" was made using an autoclave with a thermocouple well. Except for the corners, where the temperature was 10 to 15°F low, a maximum variation of less than 2°F was found. Altogether the performance of the Huppert furnaces has been very satisfactory.

//

The heating jackets are designed to fit the American Instrument Company autoclaves and generally gave very good service. However, no study of the temperature distribution within them was made.

### Temperature Control

Proper temperature in the furnaces is maintained by a Pyrovane Controller (Minneapolis-Honeywell Regulator Co., Philadelphia, Pa.). Overtemperature safety limits consist of a second Pyrovane Controller set about 10°F higher and a pressure gauge with electrical contacts. The gauge (Ashcroft Durogage Model 1077 T manufactured by Manning, Marwell, and Moore, Stratford, Conn.) is connected to a partially filled (water) autoclave located in the center of the furnace. Since pressure is a very sensitive function of temperature and the error in the gauges is relatively small in terms of temperature, the controller is adjusted to give a pressure corresponding to the desired temperature. The control and safety limit circuits have given reliable service for more than a year. A schematic circuit diagram is shown in Figure 2. A photograph of a furnace and control panel is presented in Figure 3 (1121756).

Experience has shown that the main temperature fluctuation, amounting to a few °F, occurs on opening the furnace for changing autoclaves. These are of short duration, of course. Results of the afore-mentioned temperature map convinced us that temperature variations amount to less than 2°F and that our test temperatures can be conservatively placed at  $600 \pm 5^\circ\text{F}$  and  $680 \pm 5^\circ\text{F}$ .

Temperature control of the heating jackets is achieved in one case by a Pyrovane and in the other case by a Micromax (Leeds and Northrup Co., Philadelphia, Pa.). Overtemperature safety limit for each jacket is provided by a Sensitrol (Model 705, Type 2, manufactured by the Weston Electrical Instrument Company). As an additional safety measure against overpressure, blowout disks are used. Bulk temperature was checked on one or two occasions by attaching a pressure gauge to the autoclave. Temperature fluctuations amounted to less than 2°F and the average temperature was within 5°F of the nominal temperature on any given run.

### Autoclave Filling Procedure

Details of the procedures used in filling the autoclaves in the glove box are given in Appendix A. It is sufficient to say here that the upper limit on the total amount of oxygen trapped in the autoclave is 0.1 mg. Thus any corrosion or scale formation was a result of reaction with water and not oxygen.

RESULTS AND DISCUSSION

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General

It was decided early that in the case of metals reliable measurements of corrosion and/or scale formation could be made only if the specimens could be adequately descaled after test. Considerable effort was devoted to studying the various descaling procedures, many of which satisfactorily descale ferrous alloys corroded in high-temperature water. The details of this work as well as details of the descaling methods appear in KAPL-1198.\* Accordingly little or no attention was paid to specimen appearance. The only exceptions to this occurred when a material showed abnormally high rates of corrosion or failed completely. Here specimen appearance is good indication of what took place.

Thus the data on metallic specimens include three numbers:

$W_0$   $\equiv$  original weight of the specimen

$W_s$   $\equiv$  weight of the specimen after being corroded in the test solution.

This is equal to the specimen weight plus the weight of the oxygen, water of hydration, etc. taken up by the specimen surface less the scale which has been removed, one way or another.

$W_D$   $\equiv$  weight of the specimen after removal of the scale. A correction for the amount of unreacted metal removed in the descaling process is applied to this.

Clearly these three quantities may be combined in three subtractive pairs, thus:

$\Delta W_1 = W_s - W_0$   $\equiv$  over-all weight change of the specimen and may be positive or negative.

$\Delta W_2 = W_0 - W_D$   $\equiv$  weight of metal reacted.

$\Delta W_3 = W_s - W_D$   $\equiv$  amount of scale removed by the descaling process.

Since if two of the W's are given the third may be readily calculated, the following are tabulated for all materials which can be descaled successfully:

$\Delta W_2/A \times t$ , the "corrosion rate by descaling", which is considered to be much the best index of amount of corrosion.

$\Delta W_3/A \times t$ , the "scale formation rate."

t, the "time of test."

\*KAPL-1198, "Corrosion of Reactor Structural Materials in High-Temperature Water. I. Descaling Method," by R. Fowler, Jr., D. L. Douglas, and F. C. Zydes, August 27, 1954.

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The area of the specimen A is expressed in square decimeters. In the case of the 1/2-in. x 4-in. specimens,  $A = 0.25 \text{ dm}^2$ . For zirconium, the one material which cannot be descaled, only the rate of scale formation is reported. All the evidence points to the fact that the  $\text{ZrO}_2$  film is retained on the specimen, thus its weight is a good measure of extent of corrosion.

In the paragraphs which follow, the results for each material or class of materials are discussed briefly. The detailed results are included in the tables following the discussion. Analysis of materials is given in Appendix B. This work was undertaken in the light of the knowledge of previous extensive work at lower temperatures; thus, it is not surprising that most of the metals tested were quite resistant to corrosion, at least in pure water. Certain of the ceramics and also plated metals proved to corrode excessively.

#### Ceramic Materials (Table 1)

Various ceramic materials were tested in pure water and dilute aqueous solutions at  $600^\circ\text{F}$ . Aluminum oxide (either pressed and fired in the KAPL Ceramic Laboratory or supplied by Linde Air Products as synthetic sapphire) and pressed thorium oxide, showed little corrosion in pure water, dilute base (0.001 M NaOH), and dilute acid (0.001 N  $\text{H}_2\text{SO}_4$ ).

The extent of corrosion was determined by weighing the specimens before and after test and by analyzing the final "solution" for the major constituent of the ceramic. There is some error involved in the weighing since no great precautions were taken to ensure that the materials were dried to exactly the same degree before each weighing. Hence the corrosion rates determined by analyzing the solution (tabulated as Analyzed Loss) in Table 1 are more reliable.

#### Zirconium and Zircaloy-2 (Tables 2, 3, and 4)

The rates of weight increase observed for crystal-bar zirconium, Bureau of Mines zirconium, and Zircaloy-2 in pure water\* at  $600^\circ\text{F}$  were equal to approximately  $+10 \text{ mg/cm}^2/500 \text{ hr}$ . Increase in temperature to  $680^\circ\text{F}$  roughly doubled the rate of weight increase in the same time interval. These results are in good agreement with those of other investigators.\*\* Changing the water conditions by adding hydrogen or  $1 \times 10^{-3} \text{ M LiOH}$  (at temperature) did not change the rate of weight increase at either temperature. Specimens of Bureau of Mines zirconium failed in  $6 \times 10^{-3} \text{ M LiOH}$  while the other two materials suffered only a slight increase in corrosion rate.

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\*Pure water in the case of all tests in this report is water initially having a resistivity greater than  $10^6 \text{ ohm cm}$  and an oxygen content of less than 0.1 ppm. Since the gas space in the autoclave is filled with helium, the water contains considerable dissolved helium at temperature.

\*\*WAPD-92, "Corrosion of Zircaloy-2," by D. E. Thomas, February 19, 1954.

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AISI Type 347 Stainless Steel (Tables 5, 6, 7, 8, and 9)

This material was extensively tested at 600 and at 680°F. Not only were many different water conditions tried, but also different surface treatments and different lots of steel were investigated. A general conclusion is that Type 347 stainless steel effectively resists attack by water at these temperatures. After long exposure, specimens emerge with a dull black film which can be rubbed off only partially. Corrosion rates in pure water, water containing hydrogen in any amount, 0.02 M  $H_3BO_3$  (Figure 4), and LiOH up to  $10^{-3}$  M and in 0.02 M ammonium borate (Figure 5) average 3 to 5 mg/dm<sup>2</sup>/500 hr. Noteworthy is the fact that hydrogen in the water in amounts up to 3500 cc/liter does not affect the rate of corrosion. This is in contrast to a protective effect of hydrogen at concentrations of 50 cc/liter and greater at 500°F reported by other observers.\* Surface finish in the range tested had little effect, although in some cases the electropolished specimens showed lower corrosion rates. The method is not sensitive enough to detect the small differences between various surface finishes. Higher corrosion rates (10 to 20 mg/dm<sup>2</sup>/500 hr) were observed in  $5 \times 10^{-3}$  M LiOH and in 0.02 M  $H_3BO_3$  adjusted to pH 9.5 with LiOH.

AISI Type 304 Stainless Steel (Tables 10, 11, and 12)

No significant difference regarding the resistance to attack by the water conditions tested was noted between the annealed AISI type 304 and the various lots of AISI Type 347 stainless steels. The best demonstration of the equivalence of Types 304 and 347 stainless steel will be found in Figures 4 and 5, where the corrosion of both materials in 0.02 M boric acid and in 0.02 M ammonium borate are plotted together.

Miscellaneous High Nickel Alloys (A-Nickel, Table 13; Illium, Table 14; Inconel, Table 15; and Carpenter 20, Table 16)

In general, the other high nickel materials react very slowly with pure water at 600°F, showing corrosion rates by descaling of about 5 mg/dm<sup>2</sup>/500 hr. Hydrogen in small amounts markedly reduces the extent of reaction, doubtless because the hydrogen, water, nickel, and nickel oxide equilibrium lies on the water side at these temperatures.

Iron-Chromium Alloys (AISI Type 410, Table 17; Croloy 9, Table 18; Croloy 5, Table 19; AISI Type 502, Table 20; ASTM Type A357 P5, Table 21; ASTM A335 P22, Table 22)

The iron-chromium alloys tested (ranging from 12 to 2 1/4% chromium) all showed about the same corrosion rates by descaling in pure water at 600°F, approximately 50 mg/dm<sup>2</sup>/500 hr. This corrosion rate is roughly a factor of 10 greater than the AISI Type 347 stainless steel, and a factor of 2 greater than ASTM A201 carbon steel.

\*Babcock & Wilcox Co. Research Center Report No. 5312, op.cit.

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Long term corrosion rates of  $40 \text{ mg/dm}^2/500 \text{ hr}$  are obtained for both the 5% chrome and 2 1/4% chrome alloys from Figures 6 and 7, respectively.

Carbon Steel (ASTM Type A201, Table 23, 24, and 25; ASTM A283, Table 26)

ASTM Type A201 carbon steel in pure water at  $600^\circ\text{F}$  showed a corrosion rate of approximately  $35 \text{ mg/dm}^2/500 \text{ hr}$ . The addition of hydrogen did not affect this rate; however, the additions of  $\text{LiOH}$  or  $\text{H}_3\text{BO}_3$  decreased the rate somewhat. Figures 8, 9, 10, and 11 present most of the corrosion data on carbon steel in graphical form. Long term corrosion rates estimated from the slopes of the curves in Figures 8, 9, 10, and 11 average about  $20 \text{ mg/dm}^2/500 \text{ hr}$  for carbon steel in static water at  $600^\circ\text{F}$ .

Table 1. Corrosion of Ceramic Materials in Water and Dilute Aqueous Solutions at 600°F

Material	Corrosion Medium	Wt Change, mg/cm <sup>2</sup>	Analyzed Loss, (1) mg/cm <sup>2</sup>	Time of Test, hr	Body Density	Firing Temp, °C
ThO <sub>2</sub>						
Test 1	H <sub>2</sub> O	-0.00	1x10 <sup>-3</sup>	403	9.23	1750
2	NaOH, pH 11	-0.055	1.7x10 <sup>-3</sup>	472		
3(2)	0.001 N H <sub>2</sub> SO <sub>4</sub>	-0.048	2.8x10 <sup>-4</sup>	234 1/2		
90% Al <sub>2</sub> O <sub>3</sub> + 10% Cr <sub>2</sub> O <sub>3</sub>	H <sub>2</sub> O	+0.06	0.30	259	3.62	1900
90% Al <sub>2</sub> O <sub>3</sub> + 10% Cr <sub>2</sub> O <sub>3</sub>	H <sub>2</sub> O	-0.36	1.6x10 <sup>-3</sup> (Cr+Al)	744		
Al <sub>2</sub> O <sub>3</sub>						
Test 1	H <sub>2</sub> O	-0.01	9x10 <sup>-3</sup>	283	3.91	1900
2	H <sub>2</sub> O	-0.02	4x10 <sup>-3</sup>	282		
3	H <sub>2</sub> O	-0.03	6x10 <sup>-3</sup>	594		
4	NaOH, pH 9	+0.02	1.1x10 <sup>-2</sup>	262		
5	NaOH, pH 11	-0.21	3.0x10 <sup>-3</sup>	454		
6(2)	0.001 N H <sub>2</sub> SO <sub>4</sub>	2.7x10 <sup>-3</sup>	2.8x10 <sup>-3</sup>			
Sapphire (3)						
Test 1	H <sub>2</sub> O	-0.13	6x10 <sup>-3</sup>	282		
2	H <sub>2</sub> O	-0.03	3x10 <sup>-3</sup>	594		
3	NaOH, pH 9	-0.02	5x10 <sup>-3</sup>	262		
4	NaOH, pH 11	-0.37	4x10 <sup>-4</sup>	454		
5(2)	0.001 N H <sub>2</sub> SO <sub>4</sub>	-0.025	2.8x10 <sup>-3</sup>	234 1/2		
Carboloy 608 (Cr <sub>3</sub> C <sub>2</sub> )	H <sub>2</sub> O	+0.05	2.1x10 <sup>-4</sup>	232 1/2	7.55	
	H <sub>2</sub> O	+0.04	1x10 <sup>-4</sup>	281 1/2		
Al <sub>2</sub> O <sub>3</sub> crucible	H <sub>2</sub> O	-0.049	0.031	744	3.91	1900
B <sub>4</sub> C (4)	H <sub>2</sub> O	-0.10	--	576		
BeO	H <sub>2</sub> O	-0.01	--	504		1950
Teflon	H <sub>2</sub> O	-0.88	--	235		

(1) Analysis of the water or solution remaining in the autoclave.

(2) Run simultaneously in platinum liner in the autoclave.

(3) Linde Aire Product Co.

(4) Norton Company "Norbide."

Table 2. Static Corrosion of Crystal-Bar Zirconium<sup>(1)</sup>

<u>Test Condition</u>	<u>Temp, °F</u>	<u>Time of Test, hr</u>	<u>Increase, mg/dm<sup>2</sup>/500 hr</u>	<u>Appearance</u>
200 cc/l He	600	498	+7.5 +8	Tight black film Tight black film
400 cc/l He	680	528	+16 +15	Tight black film Blisters
200 cc/l H <sub>2</sub>	600	600	+11 +9	Black film with blisters Black film with blisters
400 cc/l H <sub>2</sub>	680	520	+13 +13	Tight black film Tight black film
Initial, LiOH 1.1x10 <sup>-3</sup> M Final, OH <sup>-</sup> 1.4x10 <sup>-3</sup> M 200 cc/l He	600	570	+11 +10	Tight black film Tight black film
Initial, LiOH 2.1x10 <sup>-4</sup> M Final, OH <sup>-</sup> 1.0x10 <sup>-4</sup> M 400 cc/l He	680	495	+19 +17	Tight black film Tight black film
Initial, LiOH 1x10 <sup>-3</sup> M Final, OH <sup>-</sup> 1.7x10 <sup>-3</sup> M 400 cc/l He	680	498	+15.5 +12	Tight black film Tight black film
Initial, LiOH 5.8x10 <sup>-3</sup> M Final, OH <sup>-</sup> 4.9x10 <sup>-3</sup> M 400 cc/l He	680	473	+25.5 +47	Tight black film One blister
Initial, LiOH 5.7x10 <sup>-3</sup> M Final, OH <sup>-</sup> 9.3x10 <sup>-3</sup> M 400 cc/l He	680	473	+24 +42	Tight black film Tight black film

(1) All zirconium materials pickled in 10% HNO<sub>3</sub> - 2% HF as per Appendix A.

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Table 3. Static Corrosion of Bureau of Mines Zirconium<sup>(1)</sup>

<u>Test Condition</u>	<u>Temp, °F</u>	<u>Time of Test, hr</u>	<u>Rate of Wt Increase, mg/dm<sup>2</sup>/500 hr</u>	<u>Appearance</u>
200 cc/1 He	600	498	+10 +11	Tight black film Tight black film
400 cc/1 He	680	528	+18 +19	Tight black film Tight black film
200 cc/1 H <sub>2</sub>	600	600	+13 +9	Tight black film Tight black film
400 cc/1 H <sub>2</sub>	680	520	+20.5 +19	Tight black film Tight black film
Initial, LiOH 1.1x10 <sup>-3</sup> M Final, OH <sup>-</sup> 1.4x10 <sup>-3</sup> M 200 cc/1 He	600	570	+12 <sup>(2)</sup> +11	Tight black film Tight black film
Initial LiOH 2.1x10 <sup>-4</sup> Final, OH <sup>-</sup> 1.0x10 <sup>-4</sup> 400 cc/1 He	680	495	+22 +21	Tight black film Tight black film
Initial LiOH 1x10 <sup>-3</sup> M Final, OH <sup>-</sup> 1.7x10 <sup>-3</sup> M 400 cc/1 He	680	498	+19 +18	Tight black film Tight black film
Initial, LiOH 5.8x10 <sup>-3</sup> M Final, OH <sup>-</sup> 4.9x10 <sup>-3</sup> M 400 cc/1 He	680	473	+2007 +1980	White oxide coat White oxide coat
Initial, LiOH 5.7x10 <sup>-3</sup> M Final, OH <sup>-</sup> 9.3x10 <sup>-3</sup> M 400 cc/1 He	680	473	+1970 +1880	White oxide coat White oxide coat

(1) All zirconium materials pickled in 10% HNO<sub>3</sub> - 2% HF as per Appendix A.

(2) Figures refer to individual specimens. This information applies throughout all of the tables.

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Table 4. Static Corrosion of Zircaloy-2<sup>(1)</sup>

<u>Test Condition</u>	<u>Temp, °F</u>	<u>Time of Test, hrs</u>	<u>Rate of Weight Increase mg/dm<sup>2</sup>/500 hrs</u>	<u>Appearance</u>
200 cc/l He	600	498	+13 +11	Tight black film Tight black film
400 cc/l He	680	528	+20 +20	Tight black film Tight black film
200 cc/l H <sub>2</sub>	600	600	+12 +12	One large white patch Tight black film
400 cc/l H <sub>2</sub>	680	520	+19 +20	Tight black film Tight black film
Initial, 2500 cc/l H <sub>2</sub> Final, 1400 cc/l H <sub>2</sub>	680	595	+20 +20 +19 +19 +18 +18	Tight black film Tight black film Tight black film Tight black film Tight black film Tight black film
Initial, LiOH 1.1x10 <sup>-3</sup> M Final, OH <sup>-</sup> 1.4x10 <sup>-3</sup> M 200 cc/l He	600	570	+13 +13	Tight black film Tight black film
Initial, LiOH 2.1x10 <sup>-4</sup> M Final, OH <sup>-</sup> 1.0x10 <sup>-4</sup> M 400 cc/l He	680	495	+21 +23	Tight black film Tight black film
Initial, LiOH 1x10 <sup>-3</sup> M Final, OH <sup>-</sup> 1.7x10 <sup>-3</sup> M 400 cc/l He	680	498	+23 +24	One small white area Tight black film
Initial, LiOH 5.8x10 <sup>-3</sup> M Final, OH <sup>-</sup> 4.9x10 <sup>-3</sup> M 400 cc/l	680	473	+27.5 +29.5	Tight black film Tight black film
Initial, LiOH 5.7x10 <sup>-3</sup> M Final, OH <sup>-</sup> 9.3x10 <sup>-3</sup> M 400 cc/l He	680	473	+26 +28	Tight black film Tight black film
Initial, 0.25 M H <sub>3</sub> BO <sub>3</sub> ad- justed at room temperature with NH <sub>4</sub> OH to pH 10. Final, pH 10.15 Final borate = 0.12 M 400 cc/l He	680	217	+16 <sup>(2)</sup> +22 <sup>(2)</sup> +18 <sup>(2)</sup> +18 <sup>(2)</sup> +19 <sup>(2)</sup> +20 <sup>(2)</sup>	Tight black film Tight black film Tight black film Tight black film Tight black film Tight black film

(1) All zirconium materials pickled in 10% HNO<sub>3</sub> - 2% HF as per Appendix A.

(2) Expressed in mg/dm<sup>2</sup>/217 hr.

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Table 5. Static Corrosion of AISI Type 347 Stainless Steel

<u>Test Condition</u>	<u>Temp, °F</u>	<u>Surface Treatment</u>	<u>Time of Test, hr</u>	<u>Corrosion Rate by Descaling, mg/dm<sup>2</sup>/500 hr</u>	<u>Scale Formation Rate, mg/dm<sup>2</sup>/500 hr</u>
200 cc/1 He	600	As-rolled, pickled	566	-1	+2
			481	-1	+2
			441	-1	+10(1)
			441	0	+8(1)
			481	-2*	+4*
200 cc/1 He	600	Emery-polished, pickled	566	-2	+4.5
			481	-2	+3
			481	-5*	+6*
200 cc/1 He	600	Electropolished	481	0	+1
				0	0
400 cc/1 He	680	As-rolled, pickled	504	-2	+3.5
				-3	+5
				-3	+5
		As-rolled, pickled	496	-4*	+7*
			496	0	+5
400 cc/1 He	680	Emery-polished, pickled	566	-5	+7.5
			496	-2	+5
			566	-5**	+9**
400 cc/1 He	680	Electropolished	504	-1.5	+2
				-1.5	+3
			496	0	+3
200 cc/1 H <sub>2</sub>	600	As-rolled, pickled	566	-1	+1
			553	-4	+7
			553	-2*	+4*
200 cc/1 H <sub>2</sub>	600	Emery-polished, pickled	553	-2	+5
			566	-1	+5.5
			553	-5*	+7*
200 cc/1 H <sub>2</sub>	600	Electropolished	553	0	0
			553	-1	+2
400 cc/1 H <sub>2</sub>	600	As-rolled, pickled	549	-3	+6
Initial, 400 cc/1 H <sub>2</sub>	600	As-rolled, pickled	502	0	0
Final, 100 cc/1 H <sub>2</sub>	600	Emery-polished, pickled	549	-4	+6
400 cc/1 H <sub>2</sub>			549	-4	+6
			548	-5	+6

\*Throughout the remaining tables, the asterisk indicates that the analysis of this material is given in the appendix under AISI Type 347, Lot B. All other material is Lot A unless otherwise stated.

\*\*Throughout the remaining tables, two asterisks indicate that analysis of this material is given in the appendix under AISI Type 347 M-1.

(1) Value high because of residue other than scale on sample at end of test.

Table 6. Static Corrosion of AISI Type 347 Stainless Steel

Test Condition	Temp, °F	Surface Treatment	Time of Test, hr	Corrosion Rate by Descaling, mg/dm <sup>2</sup> /500 hr	Scale Formation Rate, mg/dm <sup>2</sup> /500 hr
Initial, 400 cc/l H <sub>2</sub> Final 100 cc/l H <sub>2</sub>	600	Emery-polished, pickled	502	-1	+2
400 cc/l H <sub>2</sub>	600	Electropolished	549	-1 0	+3 +2
Initial, 400 cc/l H <sub>2</sub> Final, 100 cc/l H <sub>2</sub>	600	Electropolished	502	-1 -1	+2 +2
400 cc/l H <sub>2</sub>	680	Emery-polished, pickled	616	-2 -5**	+2 +5**
870 cc/l H <sub>2</sub>	680	As-rolled, pickled	571	-1	+2
		Emery-polished, pickled	571 571	-2** -3	+4** +4
1300 cc/l H <sub>2</sub>	680	As-rolled, pickled	499	-2	+2
		Emery-polished, pickled	499	-2	+3
		Electropolished	499 499	-2 -3	+3 +4
Initial, 3500 cc/l H <sub>2</sub> Final, 3000 cc/l H <sub>2</sub>	680	As-rolled, pickled	532	-1	+1.5
		Emery-polished, pickled	532	-2	+3
		Electropolished	532 532	-1 -2	+2 +3
Initial, LiOH 2x10 <sup>-4</sup> M Final, OH <sup>-</sup> 3.9x10 <sup>-4</sup> M 200 cc/l He	600	As-rolled, pickled	425	0	0
		As-rolled, pickled	425	-9*	+9*
		Emery-polished, pickled	425	-6	+9
			425	-7*	+9*
		Electropolished	425	-6	+8
			425	-4	+5
Initial, LiOH 2.2x10 <sup>-4</sup> M Final, OH <sup>-</sup> 5.5x10 <sup>-4</sup> M 400 cc/l He	680	As-rolled, pickled	473	-3	+6
			473	-2*	+5*
		Emery-polished, pickled	473	-4	+10
			473	-2*	+5*
		Electropolished	473	-2	+7
			473	-4	+9
Initial, LiOH 1x10 <sup>-3</sup> M Final, OH <sup>-</sup> 1.2x10 <sup>-3</sup> M 200 cc/l He	600	As-rolled, pickled	576	-2	+4
			576	-8	+9
		Emery-polished, pickled	576	-8	+9
		Electropolished	576	-1	+1
			576	-2	+2
Initial, NaOH 1x10 <sup>-3</sup> M Final, OH <sup>-</sup> 1x10 <sup>-3</sup> M 400 cc/l He	680	As-rolled, pickled	751	-3	+2
		Emery-polished, pickled	751	-3	+2

Table 7. Static Corrosion of AISI Type 347 Stainless Steel

Test Condition	Temp, °F	Surface Treatment	Time of Test, hr	Corrosion Rate by Descaling, mg/dm <sup>2</sup> /500 hr	Scale Formation Rate, mg/dm <sup>2</sup> /500 hr
Initial, LiOH 1x10 <sup>-3</sup> M Final, OH <sup>-</sup> 1.2x10 <sup>-3</sup> M 400 cc/l He	680	As-rolled, pickled	526	-4	+7
		Emery-polished, pickled	526	-8*	+11*
		Electropolished	526	-4	+5
			526	-3	+5
Initial, LiOH 5.7x10 <sup>-3</sup> M Final, OH <sup>-</sup> 4.6x10 <sup>-3</sup> M 200 cc/l He	600	As-rolled, pickled	481	-1	+3
			481	-3*	+3*
Initial, LiOH 5.7x10 <sup>-3</sup> M Final, OH <sup>-</sup> 3.7x10 <sup>-3</sup> M 200 cc/l He	600	As-rolled, pickled	671	-3	+6
			671	-5*	+5*
Initial, LiOH 5.7x10 <sup>-3</sup> M Final, OH <sup>-</sup> 4.6x10 <sup>-3</sup> M 200 cc/l He	600	Emery-polished, pickled	481	-3	+6
			481	-5*	+5*
Initial, LiOH 5.7x10 <sup>-3</sup> M Final, OH <sup>-</sup> 3.7x10 <sup>-3</sup> M 200 cc/l He	600	Emery-polished, pickled	671	-3	+5
			671	-4*	+5*
Initial, LiOH 5.7x10 <sup>-3</sup> M Final, OH <sup>-</sup> 4.6x10 <sup>-3</sup> M 200 cc/l He	600	Electropolished	481	0	+1
			481	-1	+2
Initial, LiOH 5.7x10 <sup>-3</sup> M Final, OH <sup>-</sup> 3.7x10 <sup>-3</sup> M 200 cc/l He	600	Electropolished	671	-1	+1
			671	-1	+1
Initial, LiOH 4.8x10 <sup>-3</sup> M Final, OH <sup>-</sup> 4.4x10 <sup>-3</sup> M 400 cc/l He	680	As-rolled, pickled	498	-17	+19
Initial, LiOH 5.7x10 <sup>-3</sup> M Final, LiOH 5.8x10 <sup>-3</sup> M 400 cc/l He	680	As-rolled, pickled	495	-7	+21
				-13*	+24*
Initial LiOH 4.8x10 <sup>-3</sup> M Final, OH <sup>-</sup> 4.4x10 <sup>-3</sup> M 400 cc/l He	680	Emery-polished, pickled	498	-10	+19
Initial, 5.7x10 <sup>-3</sup> M Final, 5.8x10 <sup>-3</sup> M 400 cc/l He	680	Emery-polished, pickled	495	-19	+35
				-12*	+21*
Initial, LiOH 4.8x10 <sup>-3</sup> M Final, OH <sup>-</sup> 4.4x10 <sup>-3</sup> M 400 cc/l He	680	Electropolished	498	-7	+19
			498	-5	+15

Table 8. Static Corrosion of AISI Type 347 Stainless Steel

Test Condition	Temp, °F	Surface Treatment	Time of Test, hr	Corrosion Rate by Descaling, mg/dm <sup>2</sup> /500 hr	Scale Formation Rate, mg/dm <sup>2</sup> /500 hr
Initial, $5.7 \times 10^{-3}$ M Final, $5.8 \times 10^{-3}$ M 400 cc/l	680	Electropolished	495	-7	+16
			495	-8	+16
Initial, 0.25 M H <sub>3</sub> BO <sub>3</sub> adjusted at room temperature with NH <sub>4</sub> OH to pH 10 Final, pH 9.2 Final, 1.3 M 400 cc/l He	680	Emery-polished, pickled	72	4 <sup>(1)</sup> 2 <sup>(1)</sup>	10 <sup>(1)</sup> 2 <sup>(1)</sup>
		Electropolished	72	0 <sup>(1)</sup>	0 <sup>(1)</sup>
Initial, 0.25 M H <sub>3</sub> BO <sub>3</sub> adjusted at room temperature with NH <sub>4</sub> OH to pH 10 Final, pH 9.75 Final borate 0.31 400 cc/l He	680	As-rolled, pickled	210	6 <sup>(1)</sup>	9 <sup>(1)</sup>
			210	8 <sup>(1)</sup>	12 <sup>(1)</sup>
		Emery-polished, pickled	210	1 <sup>(1)</sup>	2 <sup>(1)</sup>
			210	3 <sup>(1)</sup>	4 <sup>(1)</sup>
Electropolished	210	4 <sup>(1)</sup>	4 <sup>(1)</sup>		
	210	2 <sup>(1)</sup>	4 <sup>(1)</sup>		
Initial, 0.02 M H <sub>3</sub> BO <sub>3</sub> adjusted at room temperature with LiOH to pH 9.5 Final, pH 11.7 Final borate 0.016 400 cc/l He	680	As-rolled, pickled	603	17*	33*
			603	14	34
		Emery-polished, pickled	603	16*	30*
			603	14	33
Electropolished	603	19	38		
	603	19	38		
See Graph of the Following Results in Figure 5					
Initial, 0.02 M H <sub>3</sub> BO <sub>3</sub> Final borate 0.023 400 cc/l He	680	As-rolled, pickled	191	-2	+4*
			191	0	+3
		Emery-polished, pickled	191	-4*	+4*
			191	0	+8
		Electropolished	191	0	+5
191	0	+3			
Initial, 0.02 M H <sub>3</sub> BO <sub>3</sub> Final borate 0.010 400 cc/l He	680	As-rolled, pickled	599	-3	+7
			599	-3*	+6*
		Emery-polished, pickled	599	-3*	+6*
			599	-2	+4
		Electropolished	599	-1	+4
599	-1	+4			
Initial, 0.02 M H <sub>3</sub> BO <sub>3</sub> Final borate 0.019 400 cc/l He	680	As-rolled, pickled	813	-2	+2
			813	-1*	+2*
		Emery-polished, pickled	813	-1*	+1*
			813	-1	+1
		Electropolished	813	-2	+2
813	-2	+2			

(1) Expressed in mg/dm<sup>2</sup> because of the short test time.

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Table 9. Static Corrosion of AISI Type 347 Stainless Steel

Test Condition	Temp, °F	Surface Treatment	Time of Test, hr	Corrosion Rate by Descaling, mg/dm <sup>2</sup> /500 hr	Scale Formation Rate, mg/dm <sup>2</sup> /500 hr
Initial, 0.02 M H <sub>3</sub> BO <sub>3</sub> Final borate 0.022 400 cc/l He	680	As-rolled, pickled	1027	-1*	+1*
			1027	-2	+2
		Emery-polished, pickled	1027	-1	+1
			1027	-1	+2
See Graph of the Following Results in Figure 6					
Initial, 0.02 M H <sub>3</sub> BO <sub>3</sub> adjusted at room temperature with NH <sub>4</sub> OH to pH 9.25 Final borate 0.010 M 400 cc/l He	680	As-rolled, pickled	384	-5*	+4*
			384	-3*	+4*
		Emery-polished, pickled	384	-3	+9
			384	-2	+4
Initial, 0.02 M H <sub>3</sub> BO <sub>3</sub> adjusted at room temperature with NH <sub>4</sub> OH to pH 9.5 Final, pH 8.10 Final borate 0.011 M 400 cc/l He	680	As-rolled, pickled	594	-3	+7
			594	-2*	+3*
		Emery-polished, pickled	594	-3	+5
			594	-3*	+4*
Initial, 0.02 M H <sub>3</sub> BO <sub>3</sub> adjusted at room temperature with NH <sub>4</sub> OH to pH 9.5 Final, pH 9.25 Final borate 0.014 M 400 cc/l He	680	As-rolled, pickled	813	-2	+4
			813	-2*	+1*
		Emery-polished, pickled	813	-3	+7
			813	-3*	+2*
Initial, 0.02 M H <sub>3</sub> BO <sub>3</sub> adjusted at room temperature with NH <sub>4</sub> OH to pH 9.5 Final, pH 9.15 Final borate 0.021 M	680	As-rolled, pickled	1082	-2*	+2*
		Emery-polished, pickled	1082	-2*	+3*
			1082	-2	+4
		Electropolished	1082	-2	+2
			1082	-2	+3

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Table 10. Static Corrosion of AISI Type 304 Stainless Steel

Test Condition	Temp, °F	Surface Treatment	Time of Test, hr	Corrosion Rate by Descaling, mg/dm <sup>2</sup> /500 hr	Scale Formation Rate, mg/dm <sup>2</sup> /500 hr
200 cc/l He	600	As-rolled, pickled	566	-2	+5
		Emery-polished, pickled	481	-2	+5
		Emery-polished, pickled	566	-3	+6
			481	-6	+10
400 cc/l He	680	As-rolled, pickled	566	-2	+5
			496	-2	+6
		Emery-polished, pickled	566	-4	+8.5
			496	-3	+7
200 cc/l H <sub>2</sub>	600	As-rolled, pickled	566	-1	+2
			553	-2	+4
		Emery-polished, pickled	566	-2	+5
			566	-5	+7
400 cc/l H <sub>2</sub> Final, 400 cc/l H <sub>2</sub>	600	As-rolled, pickled	548	-4	+6
Initial, 400 cc/l H <sub>2</sub> Final, 100 cc/l H <sub>2</sub>	600	As-rolled, pickled	502	-5	+11
		Emery-polished, pickled	502	-2	+7
Initial, 400 cc/l H <sub>2</sub> Final, 400 cc/l H <sub>2</sub>	600	Emery-polished, pickled	548	-4	+5
400 cc/l H <sub>2</sub>	680	As-rolled, pickled	616	-3	+4
			616	-3	+6
870 cc/l H <sub>2</sub>	680	As-rolled, pickled	571	-3	+5
		Emery-polished, pickled	571	-3	+5
1300 cc/l H <sub>2</sub>	680	As-rolled, pickled	499	-3	+5
		Emery-polished, pickled	499	-1	+2
Initial, 3500 cc/l H <sub>2</sub> Final, 3000 cc/l H <sub>2</sub>	680	As-rolled, pickled	532	-3	+4
		Emery-polished, pickled	532	-1.5	+3
Initial, LiOH 2x10 <sup>-4</sup> M Final, OH <sup>-</sup> 3.9x10 <sup>-4</sup> M 200 cc/l He	600	As-rolled, pickled	425	-6	+8
		Emery-polished, pickled	425	-13	+18
Initial, LiOH 2.2x10 <sup>-4</sup> M Final, OH <sup>-</sup> 5.5x10 <sup>-4</sup> M 400 cc/l He	680	As-rolled, pickled	473	-4	+8
		Emery-polished, pickled	473	-4	+9
Initial, LiOH 1x10 <sup>-3</sup> M Final, OH <sup>-</sup> 1.3x10 <sup>-3</sup> M 200 cc/l He	600	As-rolled, pickled	576	-8	+9
		Emery-polished, pickled	576	-9	+14

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Table 11. Static Corrosion of AISI Type 304 Stainless Steel

<u>Test Condition</u>	<u>Temp, °F</u>	<u>Surface Treatment</u>	<u>Time of Test, hr</u>	<u>Corrosion Rate by Descaling, mg/dm<sup>2</sup>/500 hr</u>	<u>Scale Formation Rate, mg/dm<sup>2</sup>/500 hr</u>
Initial, NaOH $1 \times 10^{-3}$ M Final, OH <sup>-</sup> $1 \times 10^{-3}$ M 400 cc/l He	680	As-rolled, pickled Emery-polished, pickled	751 751	-3 -4	+3 +3
Initial, LiOH $1 \times 10^{-3}$ M Final, OH <sup>-</sup> $1.2 \times 10^{-3}$ M 400 cc/l He	680	As-rolled, pickled Emery-polished, pickled	526 526	-9 -12	+12 +18
Initial, LiOH $5.7 \times 10^{-3}$ M Final, OH <sup>-</sup> $4.6 \times 10^{-3}$ M 200 cc/l He	600	As-rolled, pickled Emery-polished, pickled Emery-polished, pickled	481 481	-3 -9	+6 +10
Initial, LiOH $5.7 \times 10^{-3}$ M Final, OH <sup>-</sup> $3.7 \times 10^{-3}$ M 200 cc/l He	600	As-rolled, pickled Emery-polished, pickled	671 671	-6 -12	+17 +19
Initial, LiOH $4.8 \times 10^{-3}$ M Final, OH <sup>-</sup> $4.4 \times 10^{-3}$ M 400 cc/l He	680	Emery-polished, pickled	498	-20 -16	+33 +30
Initial, LiOH $5.7 \times 10^{-3}$ M Final, OH <sup>-</sup> $5.8 \times 10^{-3}$ M 400 cc/l He	680	As-rolled, pickled	495 495	-12 -23	+23 +41
Initial, 0.25 M H <sub>3</sub> BO <sub>3</sub> ad- justed at room temper- ature with NH <sub>4</sub> OH to pH 10 Final, pH 9.75 Final borate = 0.31 M 400 cc/l He	680	As-rolled, pickled Emery-polished, pickled Emery-polished, pickled	210 210 210	-1(1) -1(1) -1(1)	+1(1) +2(1) +1(1)
Initial, 0.02 M H <sub>3</sub> BO <sub>3</sub> adjusted at room temperature with LiOH to pH 9.5 Final, pH 11.7 Final borate = 0.016 M 400 cc/l He	680	As-rolled, pickled Emery-polished, pickled	603 603	-16 -19	+38 +38
See Graph of the Following Results in Figure 5					
Initial, 0.02 M H <sub>3</sub> BO <sub>3</sub> Final borate = 0.023 M 400 cc/l He	680	As-rolled, pickled	191	-1	+8
Initial, 0.02 M H <sub>3</sub> BO <sub>3</sub> Final borate = 0.010 M 400 cc/l He	680	As-rolled, pickled Emery-polished, pickled	599 599	-4 -3	+8 +7

(1) Expressed in mg/dm<sup>2</sup> because of the short time of test.

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Table 12. Static Corrosion of AISI Type 304 Stainless Steel

<u>Test Condition</u>	<u>Temp, °F</u>	<u>Surface Treatment</u>	<u>Time of Test, hr</u>	<u>Corrosion Rate by Descaling, mg/dm<sup>2</sup>/500 hr</u>	<u>Scale Formation Rate, mg/dm<sup>2</sup>/500 hr</u>
Initial, 0.02 M H <sub>3</sub> BO <sub>3</sub>	680	As-rolled, pickled	813	-1	+2
Final borate 0.019	680	Emery-polished, pickled	813	-1	+4
400 cc/l He					
Initial, 0.02 M H <sub>3</sub> BO <sub>3</sub>	680	As-rolled, pickled	1027	-2	+5
Final borate 0.022	680	As-rolled, pickled	1027	-2	+4
400 cc/l He					
See Graph of the Following Results in Figure 6					
Initial, 0.02 M H <sub>3</sub> BO <sub>3</sub>	680	As-rolled, pickled	384	-4	+9
adjusted at room		Emery-polished, pickled	384	-3	+9
temperature with NH <sub>4</sub> OH					
to pH 9.5					
Final, pH 9.25					
Final borate 0.010					
400 cc/l He					
Initial, 0.02 M H <sub>3</sub> BO <sub>3</sub>	680	As-rolled, pickled	594	-2	+6
adjusted at room		Emery-polished, pickled	594	-1	+4
temperature with NH <sub>4</sub> OH					
to pH 9.5					
Final, pH 8.10					
Final borate 0.011 M					
400 cc/l He					
Initial, 0.02 M H <sub>3</sub> BO <sub>3</sub>	680	As-rolled, pickled	813	-3	+7
adjusted at room		Emery-polished, pickled	813	-5	+9
temperature with NH <sub>4</sub> OH					
to pH 9.5					
Final, pH 9.25					
Final borate 0.014					
400 cc/l He					
Initial, 0.02 M H <sub>3</sub> BO <sub>3</sub>	680	As-rolled, pickled	1082	-3	+5
adjusted at room		Emery-polished, pickled	1082	-4	+6.5
temperature with NH <sub>4</sub> OH					
to pH 9.5					
Final, pH 9.15					
Final borate 0.021 M					
400 cc/l He					

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Table 13. Static Corrosion of Nickel

<u>Test Condition</u>	<u>Temp, °F</u>	<u>Surface Treatment</u>	<u>Time of Test, hr</u>	<u>Corrosion Rate by Descaling, mg/dm<sup>2</sup>/500 hr</u>	<u>Scale Formation Rate, mg/dm<sup>2</sup>/500 hr</u>
200 cc/l He	600	As-rolled, pickled	568	-4	+8
			568	-2	+4
			441	-5	+41(1)
			441	-8	+40
			441	-9	+44
			441	-9	+50
200 cc/l H <sub>2</sub>	600	As-rolled, pickled	568	-1	+2
			568	-1	+3
Initial, LiOH $1 \times 10^{-3}$ M	600	As-rolled pickled	498	-1	+1
Final, OH <sup>-</sup> $0.48 \times 10^{-3}$ M				0	0

(1) Carbonaceous deposits on specimens from water contaminated with oil.

29Table 14. Static Corrosion of ILLIUM

<u>Test Condition</u>	<u>Temp, °F</u>	<u>Surface Treatment</u>	<u>Time of Test, hr</u>	<u>Corrosion Rate by Descaling, mg/dm<sup>2</sup>/500 hr</u>	<u>Scale Formation Rate, mg/dm<sup>2</sup>/500 hr</u>
200 cc/l He	600	Emery-polished, pickled	568	-3	+4
			568	-4	+6
			441	-2	+5
			441	-2	+3
200 cc/l H <sub>2</sub>	600	Emery-polished, pickled	568	0	0
			568	0	+1
Initial, LiOH 1x10 <sup>-3</sup> M Final, 0.68x10 <sup>-3</sup> M 200 cc/l He	600	As-rolled, washed	498	-2	+2
			498	-2	+2

30Table 15. Static Corrosion of Inconel

<u>Test Condition</u>	<u>Temp, °F</u>	<u>Surface Treatment</u>	<u>Time of Test, hr</u>	<u>Corrosion Rate by Descaling mg/dm<sup>2</sup></u>	<u>Scale Formation Rate, mg/dm<sup>2</sup></u>
200 cc/l He	600	Emery-polished, pickled	568	-7	+10
			568	-8	+13
200 cc/l H <sub>2</sub>	600	Emery-polished, pickled	568	0	0
			568	0	+1
Initial, LiOH 1x10 <sup>-3</sup> M Final, 0.6 x 10 <sup>-3</sup> M 400 cc/l He	600	As-rolled, washed	498	-7	+3
			498	-1	0
Initial, 0.25 H <sub>3</sub> BO <sub>3</sub> adjusted at room temper- ature with NH <sub>4</sub> OH to pH 10 Final, pH 9.75 Final borate 0.31 400 cc/l He	680	As-rolled, washed	72	0(1)	2(1)
				0(1)	1(1)

(1) Expressed in mg/dm<sup>2</sup> because of the short time.

31

Table 16. Static Corrosion of Carpenter 20

<u>Test Condition</u>	<u>Temp, °F</u>	<u>Time of test, hr</u>	<u>Corrosion Rate by Descaling, mg/dm<sup>2</sup>/500 hr</u>	<u>Scale Formation Rate, mg/dm<sup>2</sup>/500 hr</u>
200 cc/l He	600	501	-6	+11
			-7	+10
			-4	+8
			-6	+14
			-1	+6
			-6	+10
			441	+12
			441	+12
			441	+8
			441	-2

32

Table 17. Static Corrosion of AISI Type 410 Steel

<u>Test Condition</u>	<u>Temp, °F</u>	<u>Surface Treatment</u>	<u>Time of Test, hr</u>	<u>Corrosion Rate by Descaling, (1) mg/dm<sup>2</sup></u>	<u>Scale Formation Rate, (1) mg/dm<sup>2</sup></u>
200 cc/1 He	600	Emery-polished, pickled	280	-64	+95
			280	-64	+96
			441	-44	+58
			441	-49	+64
			441	-41	+57
			376	-44	+70
			376	-34	+51
400 cc/1 He	680	Emery-polished, pickled	439	-82	+120
				-83	+127
200 cc/1 H <sub>2</sub>	600	Emery-polished, pickled	280	-22	+39
			280	-22	+48
985 cc/1 H <sub>2</sub>	600	Emery-polished, pickled	258	-77	+113
			258	-75	+110
Initial, LiOH 1x10 <sup>-3</sup> M Final, OH <sup>-</sup> 0.2x10 <sup>-3</sup> M	600	Emery-polished, pickled	282	-66	+112
			282	-66	+112

(1) Expressed in terms of time of test since times were considerably less than 500 hr.

Table 18. Static Corrosion of Croloy 9

<u>Test Condition</u>	<u>Temp, °F</u>	<u>Surface Treatment</u>	<u>Time of Test, hr</u>	<u>Corrosion Rate by Descaling<sup>(1)</sup> mg/dm<sup>2</sup></u>	<u>Scale Formation Rate,<sup>(1)</sup> mg/dm<sup>2</sup></u>
200 cc/l He	600	Emery-polished, pickled	280	-71	+106
			280	-65	+96
400 cc/l He	680	Emery-polished, pickled	439	-90	+125
				-89	+122
200 cc/l H <sub>2</sub>	600	Emery-polished, pickled	280	-32	+52
			280	-22	+35
985 cc/l H <sub>2</sub>	600	Emery-polished, pickled	258	-71	+98
			258	-69	+94
Initial LiOH $1 \times 10^{-3}$ M Final, OH <sup>-</sup> $0.2 \times 10^{-4}$ M 200 cc/l He	600	Emery-polished, pickled	282	-50	+69
			282	-56	+77

(1) Expressed in terms of time of test.

34  
 Table 19. Static Corrosion of Croloy 5

<u>Test Condition</u>	<u>Temp, °F</u>	<u>Surface Treatment</u>	<u>Time of Test, hr</u>	<u>Corrosion Rate by Descaling mg/dm<sup>2</sup>/500 hr</u>	<u>Scale Formation Rate, mg/dm<sup>2</sup>/500 hr</u>
200 cc/l He	600	Emery-polished, pickled	441	-64	+87
			441	-63	+82
			441	-82	+110
			441	-75	+104
400 cc/l He	680	Emery-polished, pickled	424	-48	+77
				-43	+67
				-43	+70
				-49	+77

Table 20. Static Corrosion of AISI Type 502 Stainless Steel

<u>Test Condition</u>	<u>Temp, °F</u>	<u>Surface Treatment</u>	<u>Time of Test, hr</u>	<u>Corrosion Rate by Descaling, mg/dm<sup>2</sup>/500 hr</u>	<u>Scale Formation Rate, mg/dm<sup>2</sup>/500 hr</u>
200 cc/l He	600	Emery-polished, pickled	501	-100	+133
			501	-99	+133
			498	-117	+152
			498	-113	+148
			498	-165	+212
			376	-61	+85
			376	-66	+91
			376	-61	+85
			376	-44	+69
			376	-45	+69
			376	-48	+72
			441	-48	+71

36

Table 21. Static Corrosion of ASTM Type A357 P5 Steel (5% Cr, 1/2% Mo)

<u>Test Condition</u>	<u>Temp, °F</u>	<u>Surface Treatment</u>	<u>Time of Test, hr</u>	<u>Corrosion Rate by Descaling,<sup>(1)</sup> mg/dm<sup>2</sup></u>	<u>Scale Formation Rate<sup>(1)</sup> mg/dm<sup>2</sup></u>
See Graph of the Following Results in Figure 7					
200 cc/l He	600	Emery-polished, pickled	161	-60	+89
			161	-64	+89
			376	-46	+70
			376	-46	+70
			376	-57	+80
			376	-56	+78
			780	-106	+147
			780	-89	+130
200 cc/l H <sub>2</sub>	600	Emery-polished, pickled	120	-56	+82
			120	-67	+96
			300	-57	+82
			300	-57	+63
			506	-101	+142
			506	-96	+140
			506	-89	+126
			932	-136	+165
932	-115	+164			

(1) Expressed in terms of time of test.

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• Table 22. Static Corrosion of ASTM Type A335 P22 Steel  
(2 1/4% Cr, 1% Mo)

<u>Test Condition</u>	<u>Temp, °F</u>	<u>Surface Treatment</u>	<u>Time of Test, hr</u>	<u>Corrosion Rate by Descaling<sup>(1)</sup> mg/dm<sup>2</sup></u>	<u>Scale Formation Rate,<sup>(1)</sup> mg/dm<sup>2</sup></u>
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See Graph of the Following Results in Figure 8

200 cc/l He	600	Emery-polished pickled	113	-91	+117
			113	-68	+90
			113	-73	+95
			113	-65	+86
			113	-68	+89
			113	-65	+89
			113	-61	+82
			472	-102	+139
			472	-105	+141
			472	-97	+132
			472	-104	+142
			472	-108	+145
			472	-122	+138
			1214	-186	+250
			1214	-131	+186
			1214	-170	+239
			1214	-155	+214
			1214	-155	+210
			1214	-190	+263
			1214	-132	+188

(1) Expressed in terms of time of test.

38  
Table 23. Static Corrosion of ASTM Type A201 Carbon Steel

<u>Test Condition</u>	<u>Temp, °F</u>	<u>Surface Treatment</u>	<u>Time of Test, hr</u>	<u>Corrosion Rate by Descaling,<sup>(1)</sup> mg/dm<sup>2</sup></u>	<u>Scale Formation Rate,<sup>(1)</sup> mg/dm<sup>2</sup></u>
See Graph of the Following Results in Figure 9					
200 cc/l He	600	Emery-polished, pickled	161	-17	+29
			161	-20	+32
			306	-40	+95
			306	-37	+62
			376	-22	+36
			376	-28	+44
			376	-43	+82
			376	-37	+71
			474	-34	+56
			474	-30	+46
			780	-52	+73
780	-45	+66			
200 cc/l H <sub>2</sub>	600	Emery-polished, pickled	104	-16	+26
			104	-25	+34
			322	-21	+32
			322	-21	+32
			505	-38	+56
			931	-48	+70
			931	-51	+75
See Graph of the following Results in Figure 10					
Initial, LiOH 1x10 <sup>-3</sup> M Final, OH <sup>-</sup> 0.8x10 <sup>-3</sup> M 200 cc/l He	600	Emery-polished, pickled	119	-5	+16
			119	-14	+26
Initial, LiOH 1x10 <sup>-3</sup> M Final, OH <sup>-</sup> 0.9x10 <sup>-3</sup> M 200 cc/l He	600	Emery-polished, pickled	520	-17	+43
			520	-29	+50
			520	-21	+38
			520	-21	+38
Initial, LiOH 1x10 <sup>-3</sup> M Final, OH <sup>-</sup> 0.8x10 <sup>-3</sup> M 200 cc/l He	600	Emery-polished, pickled	640	-24	+34
			640	-23	+35
			640	-27	+40
			640	-19	+33
Initial, LiOH 1x10 <sup>-3</sup> M Final, OH <sup>-</sup> 1.4x10 <sup>-3</sup> M 200 cc/l He	600	Emery-polished, pickled	934	-16	+37
			934	-34	+59
			934	-33	+56
			934	-31	+54
			934	-23	+45
			934	-25	+45
Initial LiOH 5.6x10 <sup>-3</sup> M Final OH <sup>-</sup> 5x10 <sup>-3</sup> M 200 cc/l He	600	Emery-polished, pickled	112	-10	+39
			112	-14	+32
			112	-17	+31

(1) Expressed in terms of time of the test.

Table 24. Static Corrosion of ASTM Type A201 Carbon Steel

<u>Test Condition</u>	<u>Temp, °F</u>	<u>Surface Treatment</u>	<u>Time of Test, hr</u>	<u>Corrosion Rate by Descaling mg/dm<sup>2</sup></u>	<u>Scale Formation Rate, mg/dm<sup>2</sup></u>
Initial, LiOH 5.6x10 <sup>-3</sup> M Final, OH <sup>-</sup> 5x10 <sup>-3</sup> M 200 cc/l He	600	Emery-polished, pickled	169	-13	+59
			169	-27	+63
Initial, LiOH 5.6x10 <sup>-3</sup> M Final, OH <sup>-</sup> 5x10 <sup>-3</sup> M 200 cc/l He	600	Emery-polished, pickled	390	-31	+58
			390	-18	+42
			390	-27	+35
Initial, LiOH 5.6x10 <sup>-3</sup> M Final, OH <sup>-</sup> 5x10 <sup>-3</sup> M 200 cc/l He	600	Emery-polished, pickled	447	-17	+70
			447	-16	+38
			447	-46	+82
See Graph of the following Results in Figure 11					
Initial, LiOH 1x10 <sup>-2</sup> M Final, OH <sup>-</sup> 0.7x10 <sup>-2</sup> M 200 cc/l He	600	Emery-polished, pickled	174	-9	+58
			174	-10	+64
			174	-10	+63
			174	-11	+68
			174	-11	+79
			174	-10	+68
Initial, LiOH 1x10 <sup>-2</sup> M Final OH <sup>-</sup> 0.6x10 <sup>-2</sup> M 200 cc/l He	600	Emery-polished, pickled	425	-25	+73
			425	-26	+77
			425	-26	+91
			425	-24	+105
			425	-36	+126
			425	-25	+116
Initial, LiOH 1x10 <sup>-2</sup> M Final, OH <sup>-</sup> 0.9x10 <sup>-2</sup> M 200 cc/l He	600	Emery-polished, pickled	617	-50	+122
			617	-41	+116
			617	-54	+127
			617	-18	+70
			617	-39	+98
			617	-37	+95
Initial, LiOH 1x10 <sup>-2</sup> M Final, OH <sup>-</sup> 0.3x10 <sup>-2</sup> M 200 cc/l He	600	Emery-polished, pickled	1146	-41	+61
			1146	-59	+95
			1146	-41	+69
			1146	-45	+70
			1146	-34	+68
			1146	-47	+84
See Graph of the Following Results in Figure 12					
Initial, H <sub>3</sub> BO <sub>3</sub> 0.02 M 200 cc/l He	600	Emery-polished, pickled	102	-7	+12
			102	-9	+16
			102	-9	+18
			102	-6	+9
			102	-9	+1
			102	-8	

40

Table 25. Static Corrosion of ASTM A201 Carbon Steel

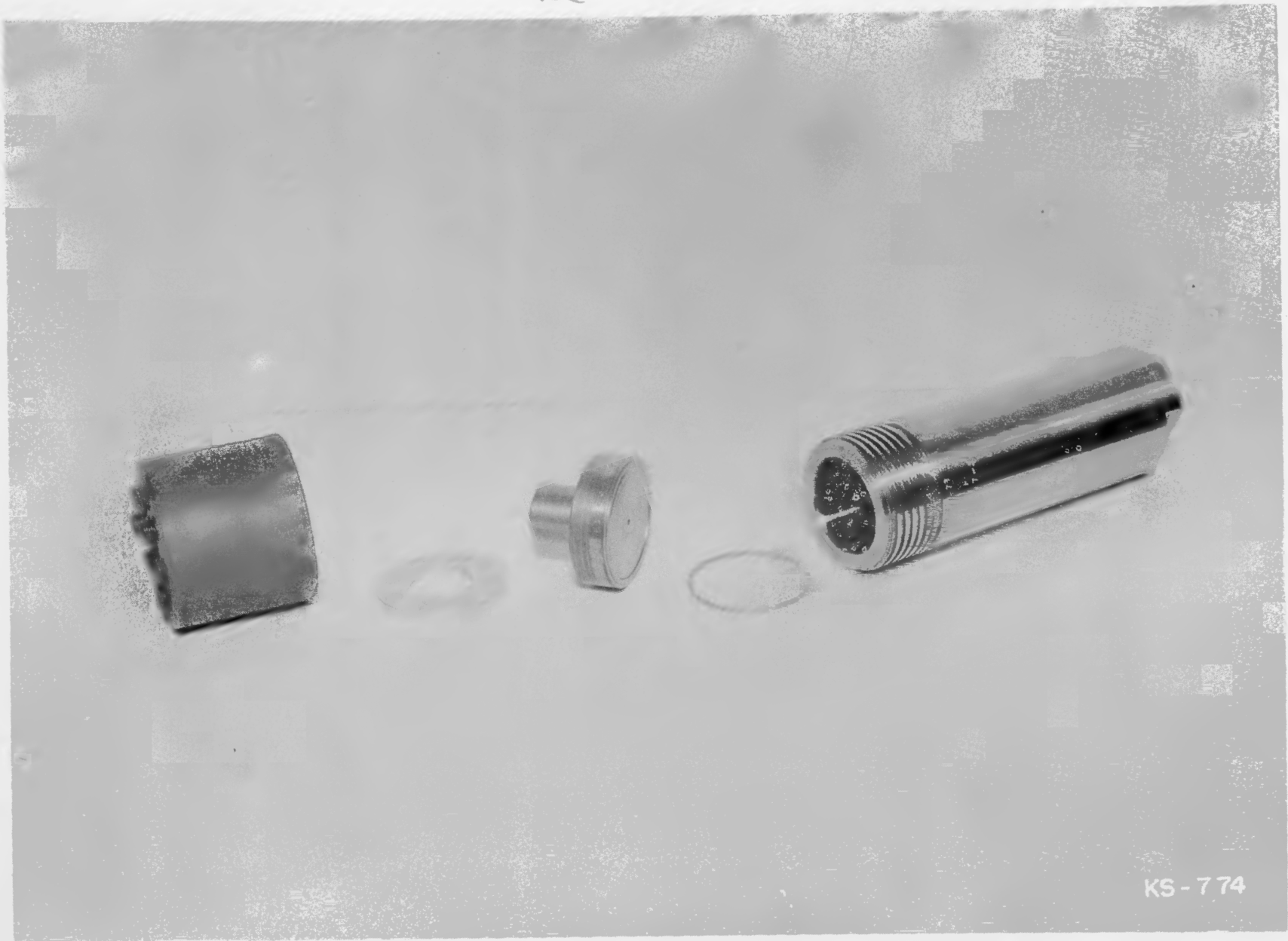
<u>Test Condition</u>	<u>Temp, °F</u>	<u>Surface Treatment</u>	<u>Time of Test, hr</u>	<u>Corrosion Rate by Descaling, mg/dm<sup>2</sup>/500 hr</u>	<u>Scale Formation Rate, mg/dm<sup>2</sup>/500 hr</u>
Initial, H <sub>3</sub> BO <sub>3</sub> 0.02 M Final borate 0.022 200 cc/l He	600	Emery-polished, pickled	198	-7	+18
			198	-7	+18
			198	-9	+22
			198	-8	+23
			198	-8	+19
			198	-9	+24
Initial, H <sub>3</sub> BO <sub>3</sub> 0.02 M 200 cc/l He	600	Emery-polished, pickled	617	-18	+36
			617	-25	+46
			617	-19	+31
			617	-32	+48
			617	-16	+27
			617	-29	+43
Initial, H <sub>3</sub> BO <sub>3</sub> 0.02 M Final borate 0.012 200 cc/l He	600	Emery-polished, pickled	1032	-29	+40
			1032	-27	+43
			1032	-38	+55
			1032	-29	+49
			1032	-33	+53
			1032	-33	+50

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Table 26. Static Corrosion of ASTM A 283 Carbon Steel

<u>Test Condition</u>	<u>Temp, °F</u>	<u>Surface Treatment</u>	<u>Time of Test, hr</u>	<u>Corrosion Rate by Descaling, mg/dm<sup>2</sup></u>	<u>Scale Formation Rate, mg/dm<sup>2</sup></u>
0.25 M H <sub>3</sub> BO <sub>3</sub> adjusted at room temper- ature with NH <sub>4</sub> OH to pH 10 Final, pH 9.75 Final, H <sub>3</sub> BO <sub>3</sub> 0.31 400 cc/l He	680	Emery-polished, pickled	210	-3	15
			210	-3	11
			210	-2	15
			210	-0	11
200 cc/l He	680	Emery-polished, pickled	161	-7	+12
				-7	+16
			306	-43	+63
				-25	+38

42



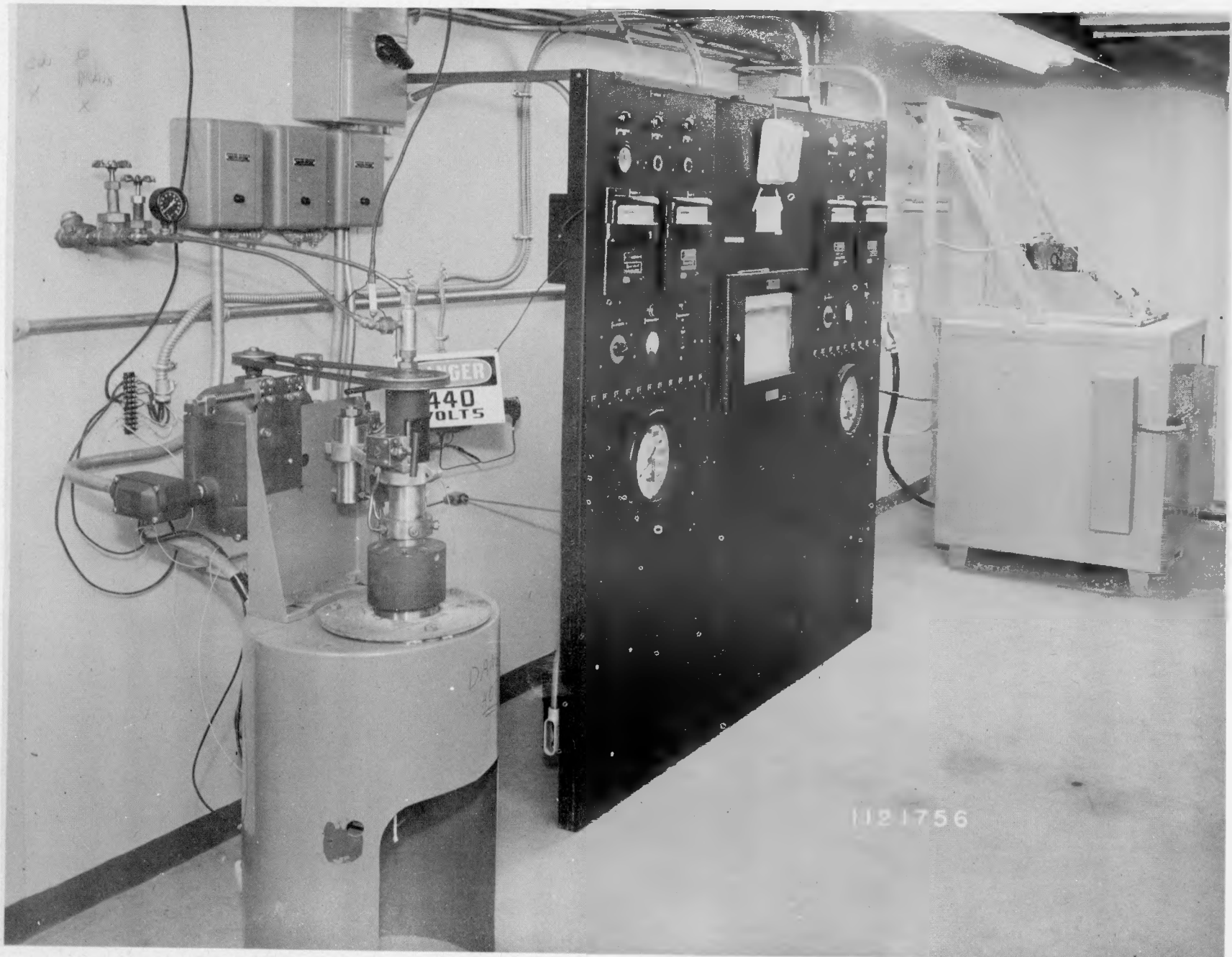
704 039

KS-774



44

721 04A



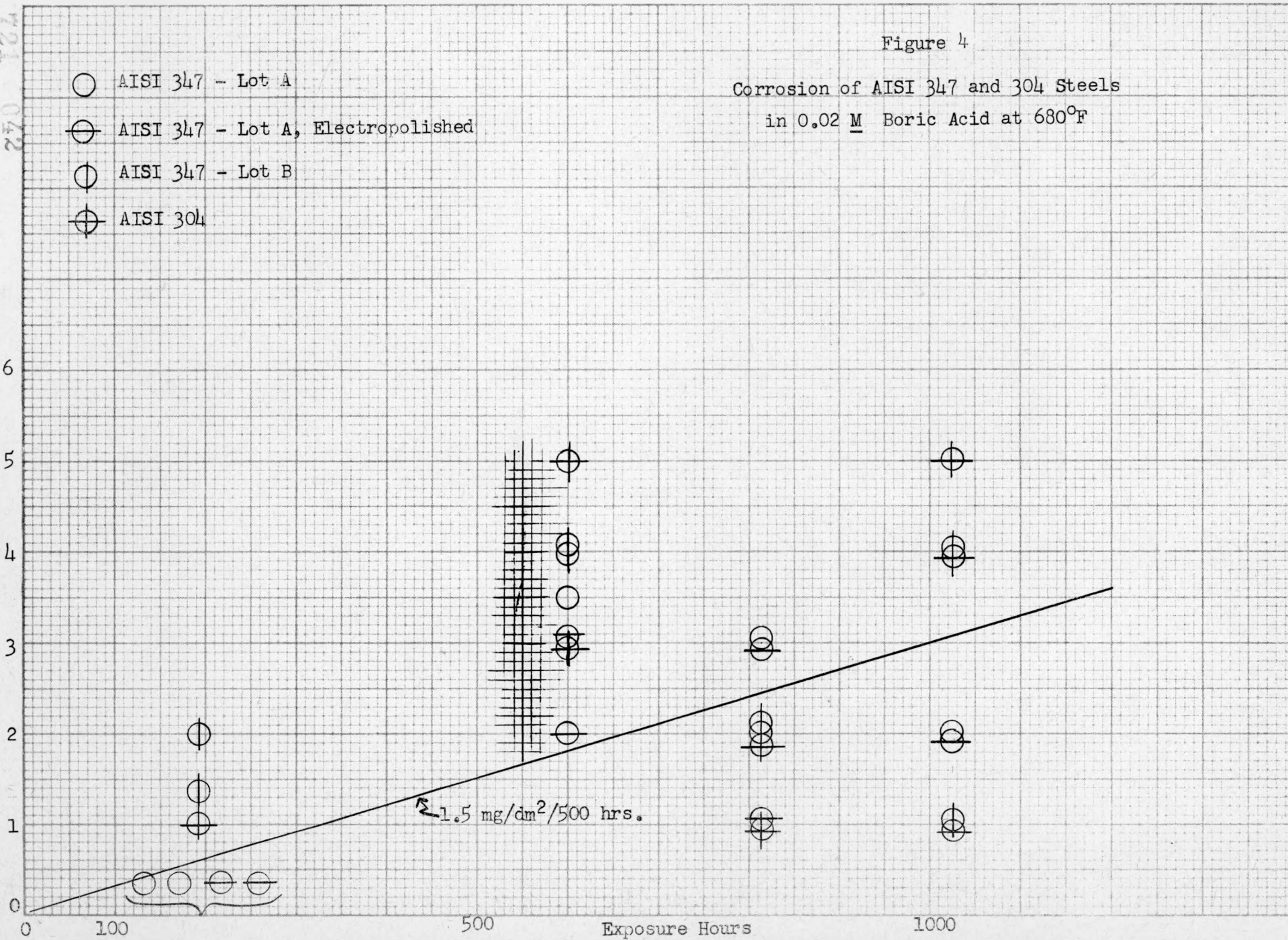
1121756

Figure 4

Corrosion of AISI 347 and 304 Steels  
in 0.02 M Boric Acid at 680°F

- AISI 347 - Lot A
- ⊖ AISI 347 - Lot A, Electropolished
- ⊙ AISI 347 - Lot B
- ⊕ AISI 304

Corrosion by Descaling in mg/dm<sup>2</sup>



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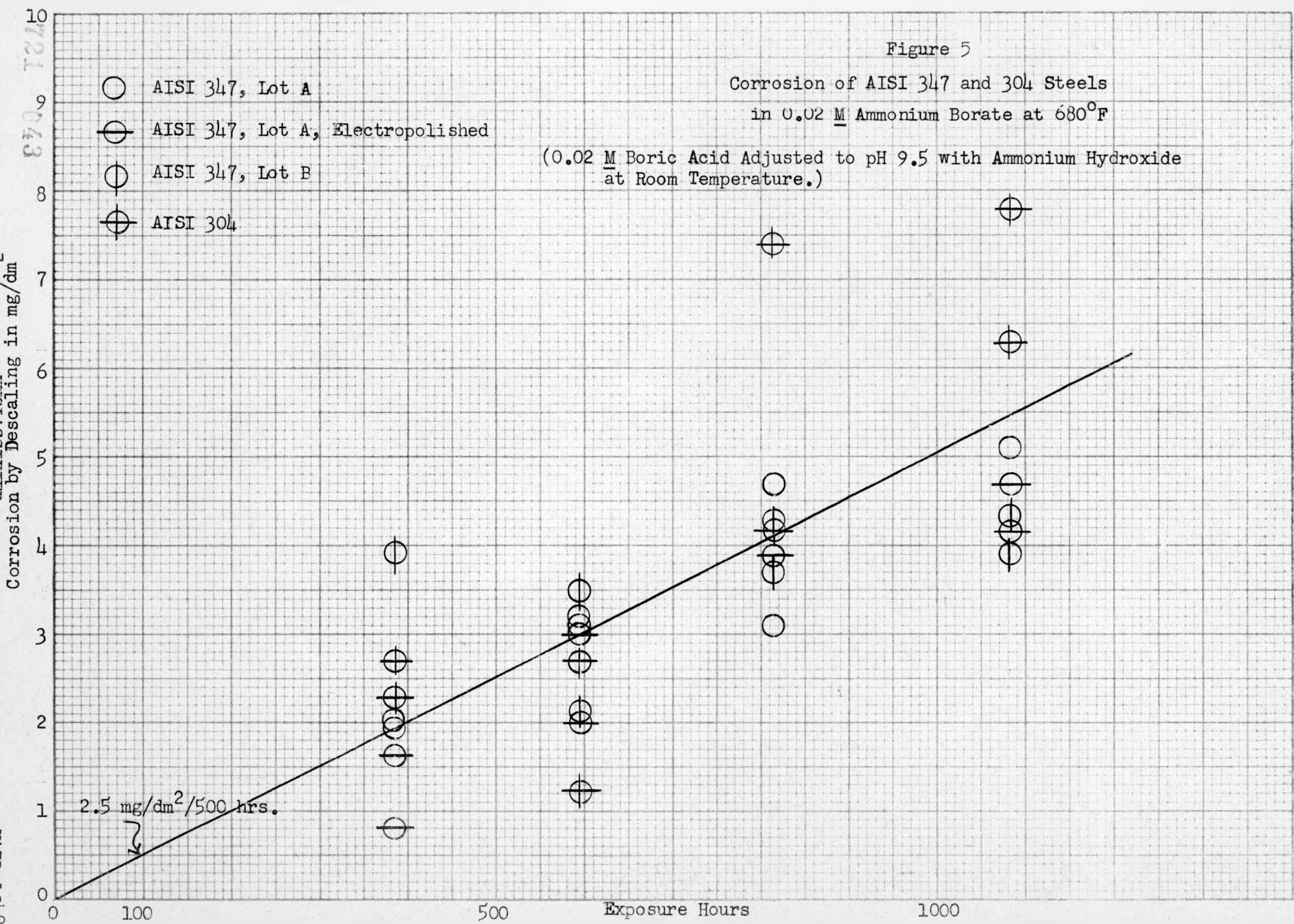
53

Figure 5

Corrosion of AISI 347 and 304 Steels  
in 0.02 M Ammonium Borate at 680°F

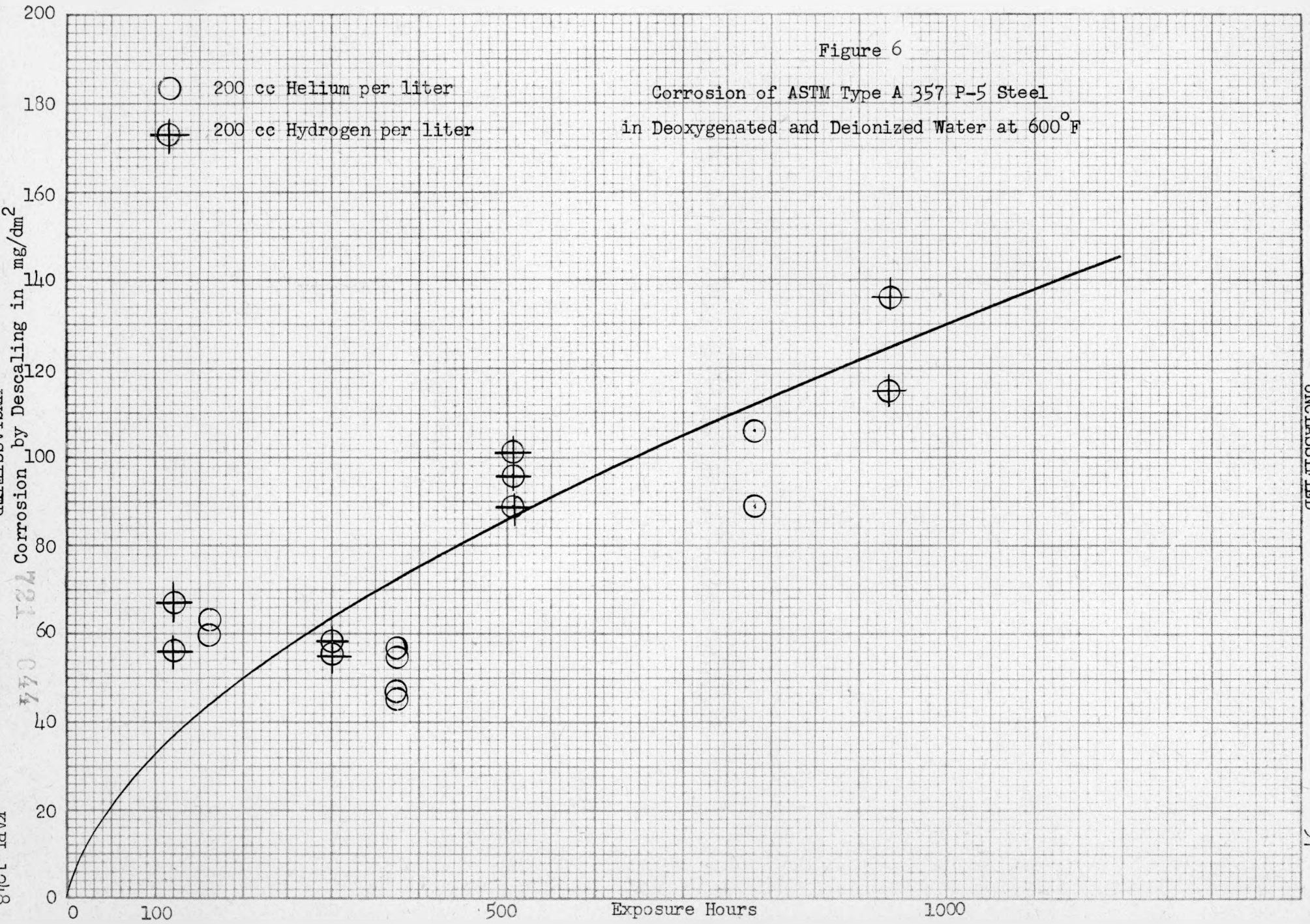
(0.02 M Boric Acid Adjusted to pH 9.5 with Ammonium Hydroxide  
at Room Temperature.)

- AISI 347, Lot A
- ⊖ AISI 347, Lot A, Electropolished
- ⊙ AISI 347, Lot B
- ⊕ AISI 304



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UNCLASSIFIED

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Figure 7

Corrosion of ASTM Type A 335 P-22 Steel  
in Deoxygenated and Deionized Water at 600°F

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Corrosion by Descaling in mg/dm<sup>2</sup>

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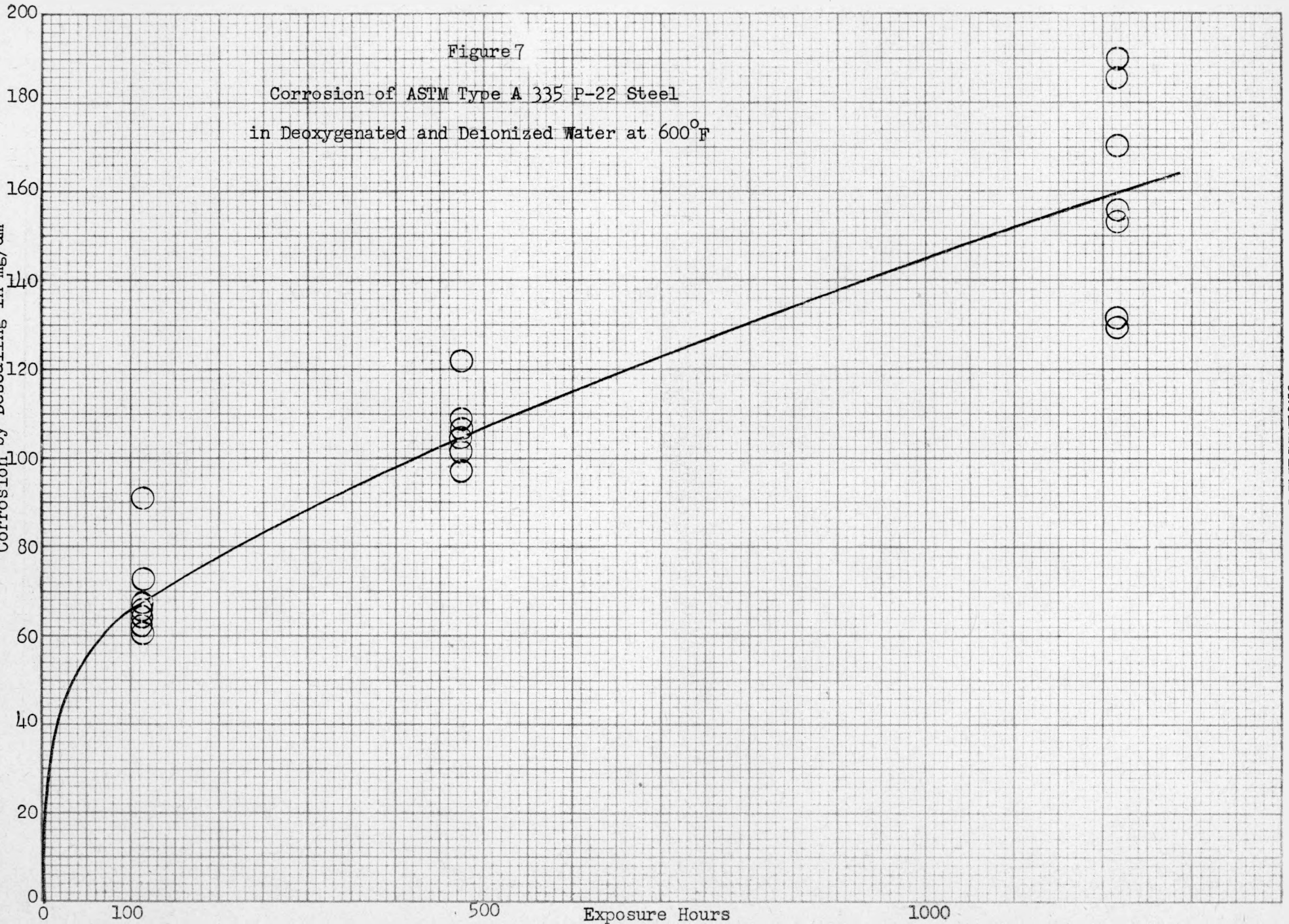


Figure 8

Corrosion of ASTM Type A 201 Carbon Steel  
in Deoxygenated and Deionized Water at 600°F

- 200 cc Helium per liter
- ⊕ 200 cc Hydrogen per liter

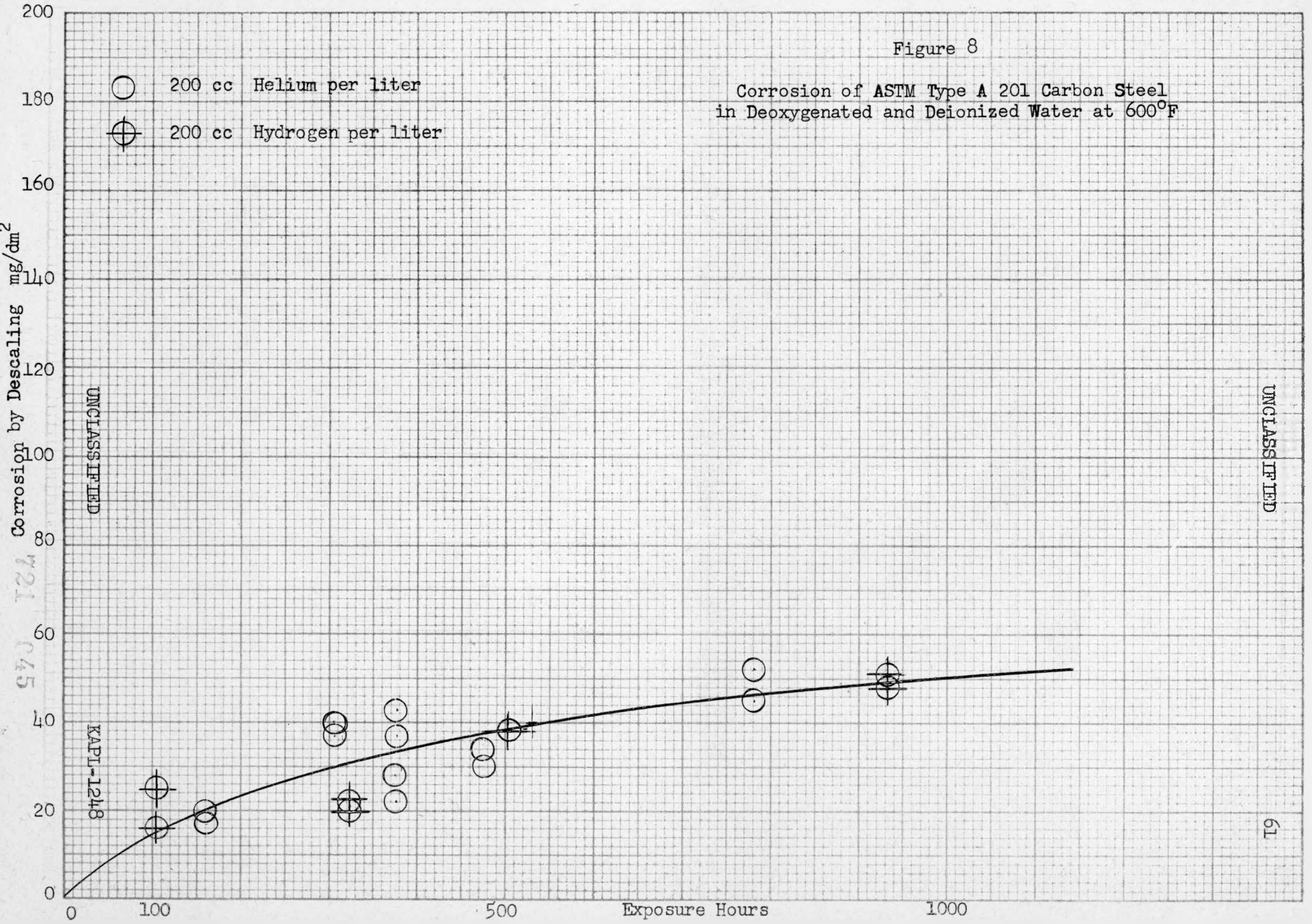


Figure 9

Corrosion of ASTM Type A 20L Carbon Steel  
in  $1 \times 10^{-3}$  M Lithium Hydroxide at  $600^{\circ}\text{F}$

- $1 \times 10^{-3}$  M LiOH
- ⊕  $5 \times 10^{-3}$  M LiOH

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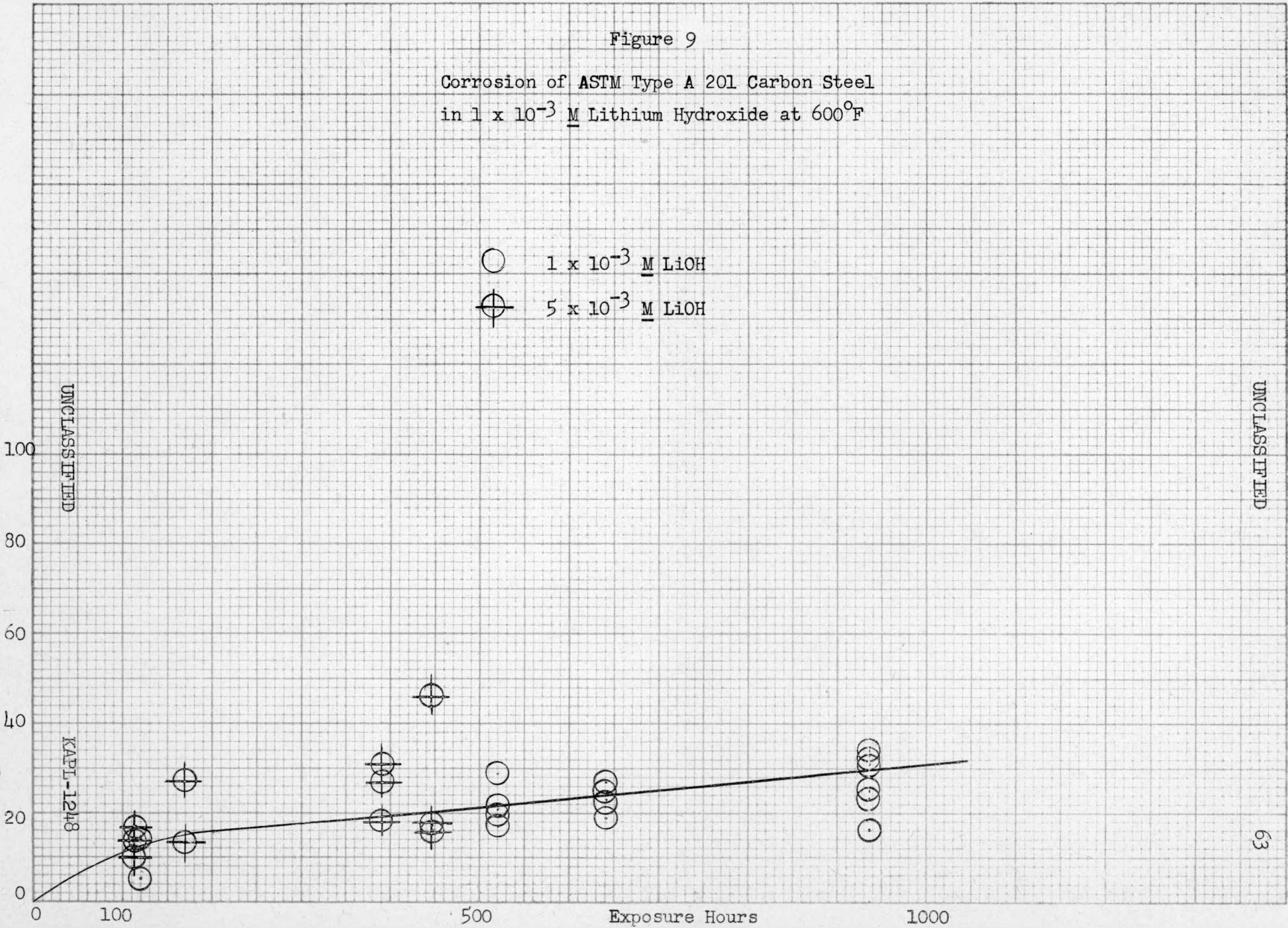


Figure 10

Corrosion of ASTM Type A 201 Carbon Steel  
in  $1 \times 10^{-2} M$  Lithium Hydroxide at  $600^{\circ}F$

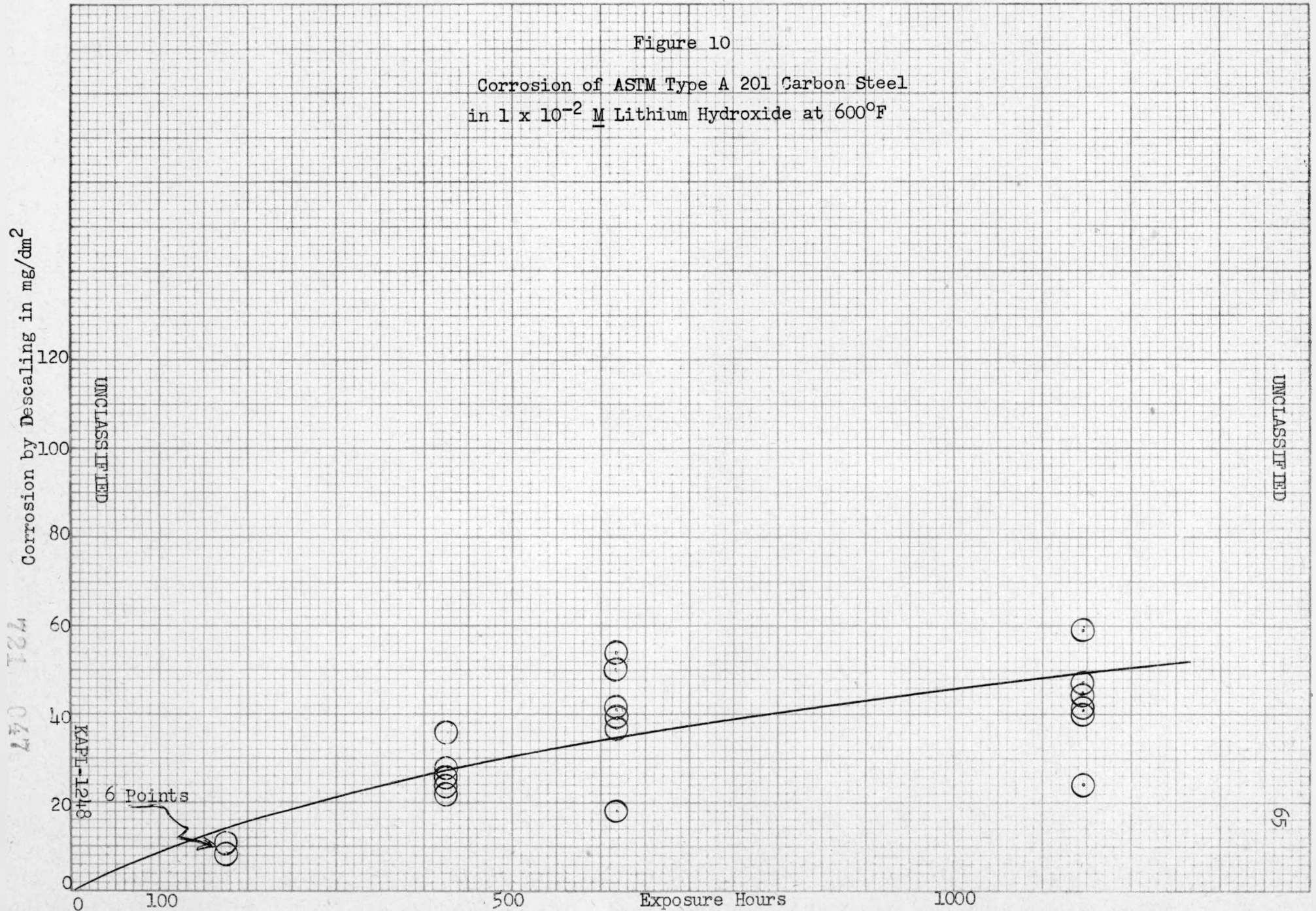


Figure 11

Corrosion of ASTM Type A 201 Carbon Steel  
in 0.02 M Boric Acid at 600°F

Corrosion by Descaling in mg/dm<sup>2</sup>

W21  
048

120

100

80

60

40

20

0

0

100

500

Exposure Hours

1000

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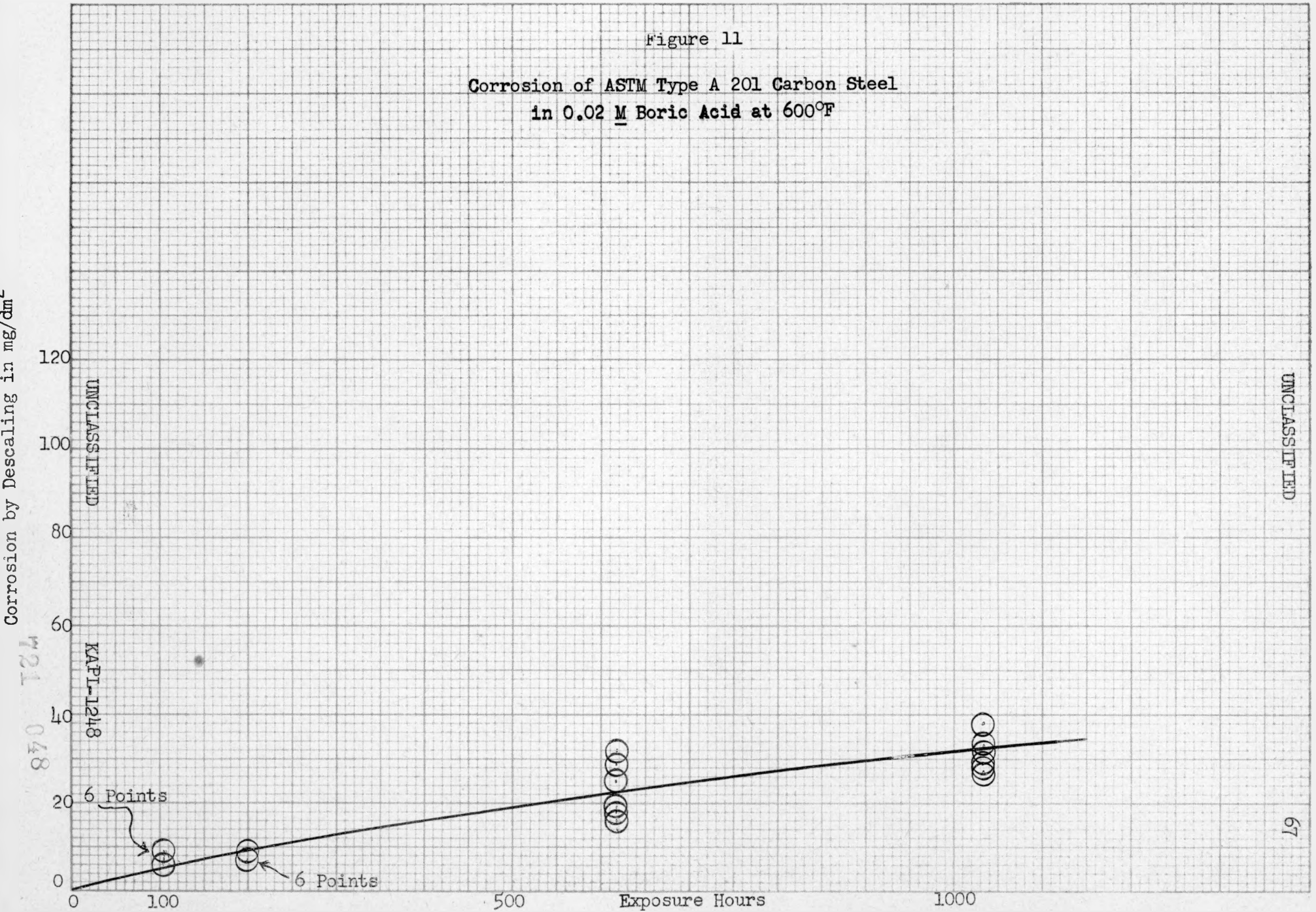
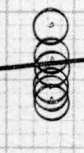
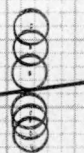
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6 Points

6 Points



## APPENDIX A

SPECIMENS AND PREPARATION

Specimens were cut from sheet stock 0.015 to 0.040 inch thick. Dimensions were 1/2 inch x 4 inch and a 3/16-inch hole was punched close to one end. The total specimen area was thus about 25 cm<sup>2</sup>. After cutting, all specimens except the AISI 347 and AISI 304 materials were annealed to relieve cold working of the specimen. The stock materials have been analyzed by the Metallurgy and Chemical Analysis Activity, and the compositions are given in Appendix B.

The stainless steel specimens in general were treated in one of three ways:

1. As-rolled and pickled, in which case the coupons were immersed in a hot 50% HNO<sub>3</sub> - 5% HF mixture until uniform bubble evolution over the entire surface occurred.
2. Emery-polished and pickled, in which case the coupons were hand-polished with grade 0 emery polishing paper (Behr-Manning, Troy, N. Y.) and then given the above pickling treatment. No great efforts were made to make the polishing absolutely uniform from one specimen to another.
3. Electropolished, in which case the coupons were made the anode at 1/2 amp/in.<sup>2</sup> for 15 minutes in an appropriate bath (42 wt % phosphoric acid, 47 wt % glycerine, 11 wt % water) maintained at about 90°C.

Zirconium specimens are pickled in 20% HNO<sub>3</sub> - 2% HF to uniform bubble evolution.

Mild steel and low alloy steel specimens were emery polished and pickled in 10% HNO<sub>3</sub> to uniform bubble evolution.

In any given test, from four to eight specimens were placed in the autoclave. Four or five of these were fastened together with a stainless steel bolt, stainless steel spacers being used to maintain a minimum separation of 1/4 inch between specimens. The remaining coupons were placed loose in the autoclave. The geometry is such that there was no chance of two coupons having a mutual contact area greater than that corresponding to an edge. Mild steel specimens in conducting solutions were insulated from the stainless steel by Al<sub>2</sub>O<sub>3</sub> spacers.

WEIGHING AND DESCALING

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The coupons were carefully dried and weighed before and after test and the gross weight change recorded. The ferrous alloys were then descaled by one of the following two procedures: (1) electrolytic method, in which the coupons were made cathodes for four minutes at 1 amp/in.<sup>2</sup> in 2.5% sulfuric acid containing 0.2% quinoline ethiodide, or (2) sodium hydride bath, in which the specimens were dipped for a few minutes in a 1 to 2% solution of NaH in molten NaOH at  $370 \pm 10^\circ\text{C}$  and subsequently quenched in water, dipped in 5 to 10% H<sub>2</sub>SO<sub>4</sub> (with inhibitor) and rinsed in water and acetone. Full details of these descaling methods and others, including blank corrections to be used with various materials, have been published in a separate report.\* After descaling the specimens are again carefully dried and weighed. No satisfactory method of descaling zirconium has yet been developed.

AUTOCLAVE FILLING PROCEDURE

As in any well designed experiment, the prime consideration is to control as closely as possible the conditions under which the reactions under study are taking place. In this case the purity of the water, gas, and other additives used in filling the autoclaves must be known accurately. In particular it is necessary to keep oxygen, or any other active substance, out of the autoclave. This is relatively simple in the case of the water: one pass through a 2-ft bed of Duolite S-10 Deoxygenating Resin (Chemical Process Co., Redwood City, Calif.) followed by a 2-ft bed of MB-1 deionizing resin (Rohm & Haas Co., Philadelphia, Pa.) reduces the oxygen content of laboratory deionized water to less than 0.01 ppm. At the same time the conductivity is reduced to less than  $1 \times 10^{-6}$  (ohm<sup>-1</sup>-cm<sup>-1</sup>) indicating a very low dissolved salt content. It is entirely impractical to obtain an atmosphere, say of helium or nitrogen, of comparable purity. However, one can set 0.1 mg as a reasonable upper limit to the oxygen trapped in the autoclave. Since the gas space amounts to about 100 cc, an oxygen content of less than 0.1% by volume will suffice even for helium and hydrogen. This is well within the purity limits of the ordinary commercial gases.

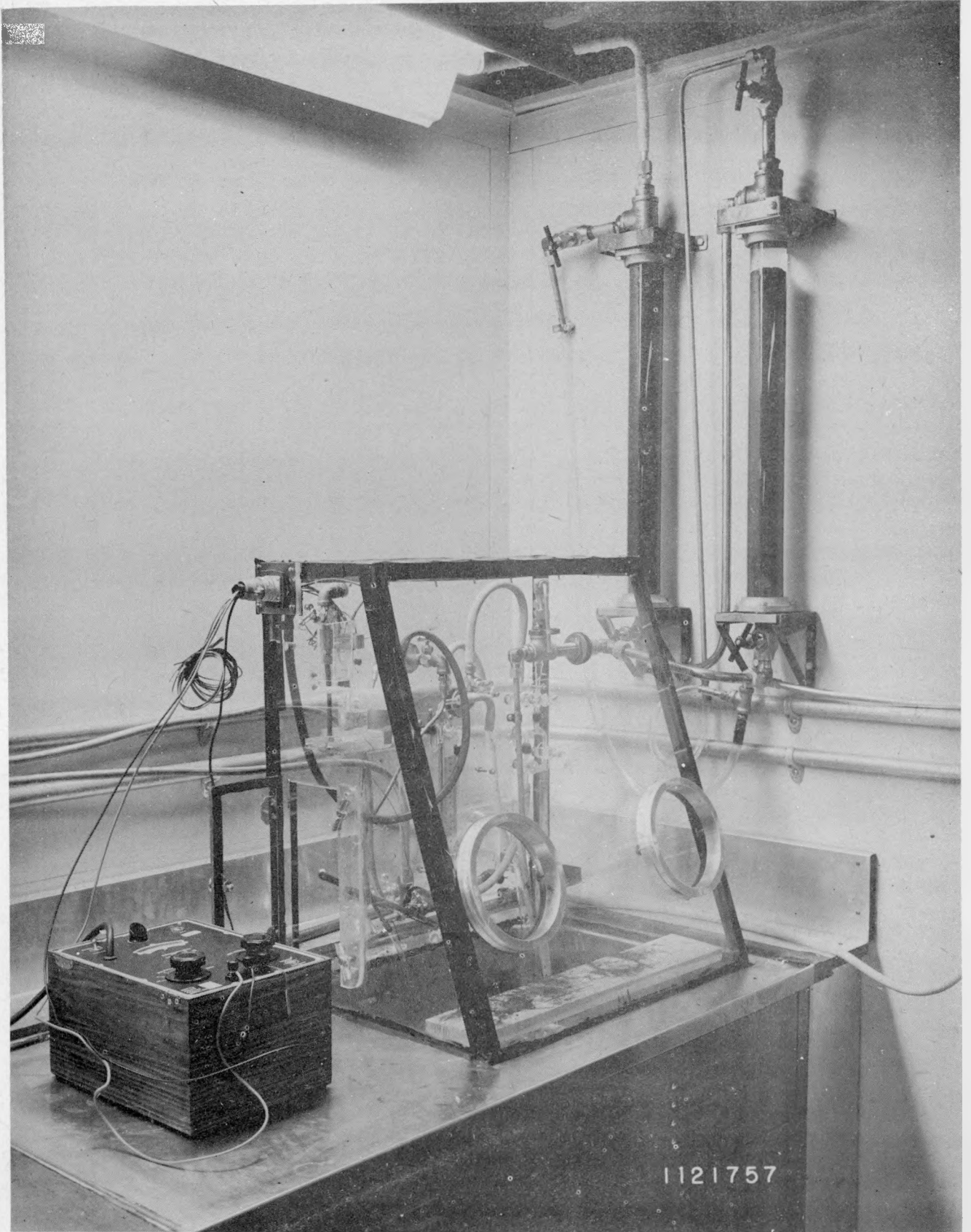
In order to exclude air as much as possible while filling the autoclaves, a gloved box of lucite was built over an ordinary laboratory sink (Figure 12). As can be seen in the photograph the deoxygenated-deionized water is piped into the box. Ordinary line nitrogen, containing about 1% O<sub>2</sub> is used in flushing the air out of the box. The actual filling procedure was as follows:

1. The autoclave was rinsed and filled with the desired amount of water.
2. Specimens and soluble salt additives (by means of lambda pipets) were added next.

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\*KAPL-1198, op.cit.

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3. The cover was placed on the autoclave and the thrust bolts tightened sufficiently to make the vessel vacuum tight.
4. A fitting (connected by rubber tubing to a small manifold) was threaded into the hole in the autoclave cover; the nitrogen in the gas space flushed out and replaced with helium by several cycles of evacuation followed by filling with helium. This results in a filling which has less than the prescribed maximum amount of oxygen. The fitting was quickly removed and replaced by a plug. That the gas remains sufficiently pure after this operation was proved in separate experiments by replacing the fitting and drawing the gas into an evacuated bulb.
5. The autoclave was removed to a bench vise and the thrust bolts carefully tightened with a torque wrench.

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Analysis of Material<sup>(1)</sup>

<u>Material</u>	<u>C</u>	<u>Si</u>	<u>Cr</u>	<u>Ni</u>	<u>Mn</u>	<u>S</u>	<u>P</u>	<u>Cb-Ta</u>	
Bureau of Mines Zirconium	850 ppm	300 ppm	45 ppm	5 ppm	35 ppm	-	-	-	
Crystal-bar Zirconium	890 ppm	120 ppm	6 ppm	20 ppm	0.5 ppm	-	-	-	
Zircaloy-2	1000 ppm	200 ppm	450 ppm	350 ppm	4 ppm	-	-	-	
AISI Type 347	Lot A	0.059%	0.34%	18.5%	11.34%	1.50%	0.005%	0.011%	0.52%
	Lot B	0.062%	0.62%	18.94%	10.93%	1.38%	0.018%	0.024%	0.77%
AISI Type 347 M-1		0.04%	0.31%	17.0%	13.9%	0.40%	0.005%	0.017%	0.38%
AISI Type 304		0.039%	0.42%	18.64%	8.96%	1.12%	0.020%	0.023%	-
Nickel	-	-	-	~99.5%	0.18%	-	-	-	
Inconel	0.056%	0.16%	14.4%	76.4%	0.23%	0.005%	-	0.07%	
Illium-R	0.058%	0.44%	22.1%	63.6%	0.50%	0.005%	0.0006%	-	
Carpenter 20	0.057%	0.82%	19.8%	28.3%	0.87%	0.007%	0.011%	-	
AISI Type 410	0.127%	0.31%	12.16%	0.41%	0.44%	0.025%	0.014%	-	
Croloy 9	0.095%	0.69%	8.19%	-	0.41%	0.021%	0.027%	-	
Croloy 5	0.083%	0.36%	5.08%	0.20%	0.43%	-	-	-	
AISI Type 502	0.061%	0.25%	4.88%	-	0.35%	0.024%	0.021%	-	
ASTM Type A357 P-5	0.138%	0.27%	6.13%	-	0.33%	0.026%	0.009%	-	
ASTM Type 335 P 22	0.166%	0.36%	2.21%	-	0.35%	0.017%	0.007%	-	
ASTM Type A201	0.185%	0.18%	-	-	0.73%	0.033%	0.010%	-	
ASTM Type A283	0.163%	0.004%	-	-	0.35%	0.023%	0.007%	-	

(1) Per cent by weight.

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Analysis of Material (continued)

<u>Material</u>	<u>Mo</u>	<u>Zr</u>	<u>Cu</u>	<u>Co</u>	<u>Fe</u>	<u>Other</u>			
Bureau of Mines Zirconium	Not found	Balance	35 ppm	Not found	850 ppm	4 ppm N <sub>2</sub> ,	< 3 ppm Sn,	16 ppm H <sub>2</sub> ,	29 ppm O <sub>2</sub>
Crystal-bar Zirconium	Not found	Balance	35 ppm	Not found	180 ppm	4 ppm N <sub>2</sub> ,	< 3 ppm Sn,	39 ppm H <sub>2</sub> ,	17 ppm O <sub>2</sub>
Zircaloy-2	Not found	Balance		Not found	0.18%	4 ppm N <sub>2</sub> ,	1.49% Sn,	17 ppm H <sub>2</sub> ,	8 ppm O <sub>2</sub>
AISI Type 347	Lot A	-	0.15%	-	Balance				-
	Lot B	-	-	-	Balance				-
AISI Type 347 M-1	0.011%	-	0.127%	0.03%	Balance				-
AISI Type 304	-	-	-	-	Balance				-
Nickel	-	-	-	-	-				-
Inconel	-	-	0.31%	-	7.14%	0.08% Al,	0.22% Ti		
Illium-R	3.6%	-	2.42%	-	6.63%				-
Carpenter 20	1.9%	-	3.59%	-	43.1%				-
AISI Type 410	-	-	-	-	Balance				-
Croloy 9	0.52%	-	-	-	Balance				-
Croloy 9	0.45%	-	-	-	Balance				-
AISI Type 502	-	-	-	-	Balance				-
ASTM Type A357 P-5	0.56%	-	-	-	Balance				-
ASTM Type 335 P 22	0.88%	-	-	-	Balance				-
ASTM Type A201	-	-	0.31%	-	Balance				-
ASTM Type A283	-	-	0.005%	-	Balance				-

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