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SOME LOOP EXPERIMENTS IN THE NRX REACTOR TO STUDY THE CORROSION OF MILD STEEL BY FLOWING WATER AT 90°F

CRDC-645

by

G. M. ALLISON

Chalk River, Ontario
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ABSTRACT

This work was undertaken to find the water conditions necessary for minimum corrosion in the mild steel thermal shield recirculating systems in NRX and NRU. This report contains the chemical and corrosion results obtained by operating three mild steel loops in which water at 85-95°F was recirculated through test sections located in J-rod positions in the NRX reactor.

Lowest corrosion rates were found when the water was maintained at pH 10.5 with or without oxygen being present. In both cases the corrosion was general in nature and no pitting occurred. At pH 7 with oxygen present in the water severe pitting took place and the corrosion rate was several times higher than similar conditions without oxygen in the water.

Under oxygen-free conditions the corrosion product was Fe_3O_4. At pH 7 and with 3-5 ppm of O_2 in the water the corrosion product was a mixture of Fe_3O_4 and γ-Fe_2O_3. At high pH with oxygen present Fe_3O_4 predominated with some traces of Fe_2O_3.

The systems tested may be listed in order of increasing corrosiveness: High pH with or without O_2 in the water $<$ water at pH 7 with no O_2 present and continual purification $<$ water with no O_2 present and no purification or pH control $<$ water at pH 7 with 3-5 ppm of O_2 present.

1. Introduction

The loop experiments described in this report were run to determine the least corrosive water conditions for: (a) the NRX mild steel thermal shield cooling system after conversion to a recirculating system, and, (b) the recirculating cooling system for the NRU thermal shields which are also constructed of mild steel.

The thermal shields in the NRX reactor were originally designed to be cooled with Ottawa river water by a once-through system. Sodium hexametaphosphate was added to the river water to reduce corrosion and chlorine was added to kill bacteria. During the renovation of the reactor in 1953, when the upper thermal shields were removed and examined, it was apparent that the phosphate treatment had been far from effective in preventing corrosion. There were large deposits of scale on the inner surface of the shields and associated piping, and serious pitting had occurred under this scale. In some cases the pits had penetrated as far as one-third of the way through some sections of the pipe.

Since this phosphate treatment has been used successfully to prevent corrosion in the steam plants on the project and in the townsite it was felt that the failure in the thermal shields might be due to poor control of the addition of the phosphate compound. This is discussed in a report by R.E. Manson (4). M. Cohen (1) studied sodium hexametaphosphate as a corrosion inhibitor for Ottawa tap water and from a study of his paper it would appear that there could be three reasons for the extensive corrosion in the thermal shields, namely (i) too low a phosphate concentration (ii) variations in the flow velocities within the shields setting up galvanic cells, (iii) low calcium concentration in the water.

After the reactor started up again in March 1954, the phosphate compound used for the water treatment was changed to sodium heptametaphosphate. Chemical analysis indicated that control of the inhibitor addition was still poor. The Reactors Branch decided that better control of water conditions could be achieved if the shields were cooled with recirculated water. Unfortunately these loop experiments were not completed before the recirculation system was started in August, 1955, and for the first few months optimum conditions were not employed.

The NRU thermal shield system is also constructed of mild steel and has been designed for recirculation of the cooling water. Optimum water conditions are also required in this system to keep corrosion to a minimum and to avoid pitting.

The process Engineering Branch undertook to design and operate some mild steel loops to determine the best water conditions for cooling the thermal shields in both reactors. The design was completed and construction had started when the problem was transferred in January 1955 to the Development Chemistry Branch of the newly formed Chemistry and Metallurgy Division. The Radiation Chemistry and Corrosion section of this branch undertook to operate the loops. A few minor changes were made in the design and the loops were finally put into operation in May, 1955.

II. Description of the Loops

Three loops were constructed, in each of which the water could be circulated through a test section in a J-rod position in the NRX reactor. These positions are in the graphite immediately outside the calandria and receive a maximum thermal neutron flux of 1 x 10^{13} n/cm^2/sec. An out-reactor test section is also provided.

The three loops are designated J-1, J-2 and J-3. Complete details of the loops can be found in drawings E-1942-A to S, and in the design manual (5). A flow diagram is included in this report (Figure 1).

Water is circulated at 1-2 igpm by a Chempump through the in-reactor test section which consists of two concentric pipes about 23 feet long, the larger pipe being closed at the bottom. Water flows down the inner pipe at a velocity of about 3 ft/sec and back through the annulus at about 1 ft/sec. Mild steel test specimens are mounted in the bottom of the rod about level with the calandria floor.

From the in-reactor test section, the water flows through an out-reactor test section containing two mild steel specimens and thence through a cooler to the feed tank. The water enters at the top of the feed tank, falls through a vapour phase (He or air) and flows out the bottom of the tank to the pump.

An ion exchange by-pass takes water, at 0.1-0.2 igpm, from the high pressure side of the pump and returns it to the low pressure side of the pump. This by-pass contains a Cuno Filter and an ion exchange column. A conductivity cell is valved in such a manner that the specific conductivity of the water can be measured before and after the resin column. In loop J-2 this by-pass contains two resin columns which are valved so they can be used individually, in series or in parallel. In the other two loops only a single ion exchange column is provided.

Construction has been of mild steel with the exception of the cooler and ion exchange columns which are of stainless steel. The composition of the mild steel used is - 0.1%C; 0.3%Mn; 0.03% Si; 0.04% S.

III. Objectives

Experiments were planned to obtain the following information:

(1) If the recirculating system is under a helium atmosphere, is a deoxygenating resin required to keep the oxygen concentration in the water essentially zero?

(2) Is continuous ion exchange purification of the recirculating water necessary?

(3) Is the corrosion rate of mild steel in high pH water lower than in neutral water under oxygen-free conditions?

(4) What is the effect of high oxygen concentrations in the water on the corrosion of mild steel at pH 7 and at pH 10?

(5) Is the corrosion experienced under the above conditions general in nature, or does localized corrosion occur, i.e., pitting?

(6) Does reactor irradiation affect the corrosion of mild steel in recirculating water?

IV. Operating Conditions

The experiments have been divided into two sections - (1) in which a helium atmosphere was maintained in the feed tanks and the water was essentially oxygen free, and (2) in which the feed tanks were open to the air to provide oxygen to the water. The conditions under which the loops operated are given in Table I below.

Table 1

Operating Conditions of the J-Rod Loops

	J-1		J-2	J-3	
	Exp. 1	Exp. 2		Exp. 1	Exp. 2
Gas over feed tank	He	Air	He	He	Air
pH	not maintained, (see figure 5)	6.5-7.0	6.5-7.0	10.0-10.5	10.0-10.5
Ion exchange resin used	None ★	mixed bed	deoxygenating followed by mixed bed ★★	Mixed bed resin in K^+ and OH^- form in both exp. in J-3 ★★★	
Ion Exchange Flow Rate	0.1-0.2 i.g.p.m. in all loops				
Main Loop Flow Rate	1 - 2 i.g.p.m. in all loops				
Water Temperature (reactor operating)	85-95°F in all loops				
Duration of experiment (months)	7	4	6	9	2(at time of writing)

★ A mixed bed resin column was valved into the circuit for a day or two at the beginning of the experiment to remove any impurities leached from the new piping such as welding flux, etc.

★★ A mixed bed resin column consists of a mixture of a strongly acid cation resin in the H^+ form and a strongly basic anion resin in the OH^- form in a ratio to provide 1 equivalent of H^+ to 1 equivalent of OH^-. The deoxygenating resin is a strongly basic anion resin in the sulphite form. It is followed by a mixed bed resin to remove any sulphite or sulphate which might leak from the sulphite column.

★★★ A mixed bed resin in the K^+ and OH^- form is used to provide and maintain a pH of 10-10.5 and to keep the water free of ions other than K^+ and OH^-.

The loops were operated with the in-reactor test sections mounted out of the reactor for a 2-3 week period to insure that the systems were free of leaks and to obtain some experimental data before in-reactor operation began.

V. Experimental

(a) Analytical Methods

To determine total iron in the loop water, samples were acidified with hydrochloric acid and heated to convert all iron to the ionic form. The o-phenanthroline colorimetric method was then used.

Samples for determining ferrous iron concentrations were taken by flowing loop water through a glass tube (15-20 ml capacity, with a stopcock at each end) connected between the two sampling valves before and after the in-reactor test section as shown on the flow diagram (Figure 1). After flowing water through the tube for 2-3 minutes, the valves and stopcocks were closed and the tube disconnected from the loop. To stabilize the Fe^{++} ion some acid was added through one stopcock by allowing some loop water to flow out the other end. The usual o-phenanthroline colorimetric method was then used omitting the addition of reducing agent.

Samples for dissolved gases were obtained in a similar fashion to those for Fe^{++} using a larger sampling tube (150 ml). The gas was pumped from the sample using the method described in a report by Krenz and Robertson (3) with the one exception that the sample tube was not attached directly to the gas transfer apparatus but to an intermediate flask as shown in Figure 1(a).

The gas was analyzed for H_2, O_2, CO_2, N_2 and He by the method outlined by Robertson and Anderson (6)*. Oxygen concentrations were also determined periodically by a modified Winkler method (7).

(b) pH and Conductivity Measurements

Samples for pH measurements were taken in polythene bottles from the sampling valve at the inlet to the active test section. The pH was determined with a model G Beckman pH meter.

Each loop contained a conductivity cell (cell constant 0.2) in the purification by-pass as shown in Figure 1. Each cell was valved into the system in such a manner that specific conductivities could be determined before and after the ion exchange resin column. An Industrial Instruments conductivity bridge, model RC16, was used.

(c) Radiation Measurements

Measurements of the radiation field at various places on the loops were taken daily with a radiation survey meter, model SU-1B.

Radioactivity in the loop water was determined by evaporating aliquots of loop water on aluminum discs and counting the samples using an end window Geiger counter. Absolute disintegration rates were not determined since only relative results were required.

(d) Ion Exchange Resins

The mixed bed resin used was Rohm and Haas MB-1 resin. In the high pH experiments the resin was converted to the K^+ and OH^- form by washing with 10 resin volumes of 5% KOH solution. This resin is also supplied by Rohm and Haas under the designation XE-149.

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* A detailed report on this gas analysis method is to be published by R. Williams of the Development Chemistry Branch.

The deoxygenating resin was prepared by washing a strongly basic anion resin, 20-50 mesh Dowex 1, with a 3% sodium sulphite solution to convert it to the sulphite form. This resin can also be purchased from the Dow Chemical Co. as X-274 resin.

(e) Measurement of Corrosion Rates

Corrosion rates were determined by measuring the weight losses of the mild steel samples placed in the out-reactor and in-reactor test sections.

The out-reactor test section in each loop was designed to hold two 1/16" mild steel plates, 3 5/8" x 1 3/4". In preparing the specimens for testing they were machined to remove tool marks and were finished with 120 grain emery. One specimen for each loop was cut to the shape shown in the photograph in Figure 7 and the centre square inch was given a special polishing treatment by Dr. M. Cohen at N.R.C., Ottawa, for subsequent electron diffraction measurements on the corrosion film. These specimens were pickled for one minute in 1 M hydrochloric acid, washed with water and acetone and dried. The second specimen for each loop was pickled for one minute in 10% nitric acid, washed with water and acetone and dried. The specimens were weighed before insertion in the out-reactor test section.

Each loop also contained test specimens which were positioned at the bottom of the test section in the radiation field. In each rod there were three rectangular pieces of 1/16" mild steel, 2" x 3/4", and one circular piece of 1/8" mild steel, 1 1/4" in diameter. The circular piece was placed flat on the bottom of the outer pipe while the rectangular pieces were placed on end leaning against the end of the inner pipe.

All specimens, when removed from the loops, were examined with a binocular stereomicroscope before and after descaling. The descaling was done with Clarke's solution (2) which consists of 100 gm hydrochloric acid, 2 gm. antimony trioxide and 5 gm. stannous chloride. After descaling, specimens were weighed to determine the weight loss.

VI. Results and Discussion

(a) Chemical

1. Dissolved Gases:

When the loops were operated with helium in the feed tanks the total gas concentration in the water was usually 10-12 cc/kg and was made up of helium 80-90%, N_2 3-5%, CO_2 1-3% and the remainder H_2 and O_2.

Figures 2 and 3 show the concentrations of H_2 and O_2 in the loop water and also periods of reactor operation for the experiments with He in the feed tanks, i.e. with low O_2 content in the water. No attempt has been made to indicate reactor trips or actual power level and only the longer shut-down periods have been shown. The following points should be noted:

(1) Due to the design of the loops, no data on the rate of formation of gases could be obtained. Any H_2 or O_2 formed from radiation decomposition of the water, or H_2 formed from corrosion, would be distributed between the loop water and the vapour phase in the feed tank. Some vapour phase samples were taken and analyzed on a mass spectrometer and O_2 figures ran between .01 and .03%, while H_2 was between 2 and 6% of the total gas. The H_2 varied with the

concentration in the loop water, but the oxygen did not.

(2) Only in loop 1, exp. 1, (O_2 free, no pH control) was H_2 found in the water during the out-reactor test.

(3) H_2 was found in all the loops when the reactor was operating and the concentration tended to drop during reactor shut-downs. This would indicate that some of the H_2 observed was due to radiation decomposition of water.

(4) O_2 concentrations were normally 0.05 cc/kg, or less, in all three loops, but as can be seen in Figure 2 the concentration was sometimes over 0.1 cc/kg. These higher points did not correspond to peaks in the H_2 curve. In most cases the higher O_2 values were accompanied by higher N_2 values indicating that the O_2 was due to air in the loop water or air contamination in sampling and not to water decomposition. If the H_2 found in the loop water during periods of reactor operation was due to water decomposition the O_2 formed in this reaction must have been disappearing by corrosion of the steel.

Loop J-3 was run for a longer period than J-1 and J-2 and Figure 3 shows a continuation of the H_2, O_2 plot for this loop to include the period at the end of the experiment when radiation induced water decomposition took place. It would appear that something was inhibiting the radiation induced recombination of H_2 and O_2. However, spectrographic analysis showed nothing unusual in the water and only 0.3-0.4 ppm Cl were found (1×10^{-5} M). Renewing the ion exchange resin returned conditions in the loop to near normal for a short period and then once again water decomposition occurred just prior to the end of the experiment. No explanation has been found for this behaviour. During the water decomposition period, the water was found to be 5×10^{-5}M in hydrogen peroxide. Under normal conditions the hydrogen peroxide concentration had been found to be between 0 and 3×10^{-6} M. No H_2O_2 was found in J-1 and J-2.

The H_2 and O_2 data for the second experiment in loop J-1 (3-5 ppm O_2, pH 7) are shown in Figure 4. The lack of O_2 in the water for the first month of operation may be attributed to oxidation of the Fe_3O_4 corrosion film on the loop surfaces to Fe_2O_3 (see corrosion results) plus radiation induced combination of O_2 with corrosion H_2.

When loop J-3 was run with the feed tank open to air, oxygen was found in the water from the beginning (3-5 ppm). This difference in behaviour to J-1 is probably due to the following reasons: Firstly, before this experiment in J-3 was started the loop was used for a test on potassium metaborate solution in which water decomposition took place giving high O_2 concentrations. Then air saturated water was used to flush the loop several times which also helped to oxidize the Fe_3O_4 corrosion film on the loop piping. Secondly, when this second experiment in J-3 was started the reactor was not operating for 2 weeks, which meant there was no removal of O_2 by radiation recombination with H_2 to form water.

2. pH, Conductivity and Iron Concentrations:

Figure 5 shows a plot of these three variables against time for the first experiment in loop J-1 (O_2 free, no pH control). Periods of ion exchange operation are also shown. The high pH and conductivity during the out-reactor test with no ion exchange are thought to be due to the presence of soluble ferrous hydroxide, although the Fe^{++} concentration determined was much lower than expected. The sudden drop in pH at the beginning of the in-reactor test was probably caused by the oxidation of the ferrous hydroxide to insoluble ferric hydroxide. Further evidence for this explanation is that the loop water became cloudy and orange-brown in colour. Unfortunately, ferrous determinations were unreliable at this stage. The water was then cleaned up by the ion exchange circuit. It can be seen that during periods when the ion exchange by-pass was valved off, the total iron concentration and conductivity rose.

In the other two loops, and in experiment 2 in loop 1, the purification by-pass was in use at all times and pH, conductivity, and total iron remained fairly level and with the ranges given in Table II.

Table II

Range of pH, Conductivity and Total Iron Concentration

	pH	Conductivity µmhos	Total Fe ppm
Loop J-2	6.5-7.0	1.0-1.3	0.5-1.0
Loop J-3	10.3-10.5	90-110	0.1-0.5
Loop J-1, Exp.2	6.0-7.0	0.2-0.5	0.2-1.0

The drop in pH with the start-up of the reactor at the beginning of the in-reactor test was noticed in the first experiment in all three loops and the water in each case became cloudy and orange-brown in colour. The actual pH drops were as follows:

J-1 7.9 to 5.4,
J-2 7.1 to 6.4,
J-3 8.1 to 6.6.*

* At the beginning of the test in J-3 some difficulty was experienced in obtaining the desired pH 10.5 and at the time the in-reactor test started the pH was actually only 8.1.

3. Radioactivity of Loop Water

The activity in the water in the three loops was found to be due to Mn^{56} (half life 2.5 hr.) and Fe^{59} (half life 45 days). These radioactive isotopes are produced by neutron bombardment of Mn^{55} and Fe^{58} (0.33% of natural iron). Loop J-1, operating without the purification by-pass, had the highest activity, about 5×10^4 cpm/ml as counted on the top shelf of an end window Geiger counter assembly. This was a factor of 20 higher than activities found in the other two loops and was due to the larger amount of corrosion product in the loop water.

Radiation measurements were made daily at specified points on the loops. Fields at loop 1 were always higher than at the other loops, especially at the feed tank (200-300 mr/hr at contact on J-1 compared to 30-50 mr/hr on the other loops). At no time did the radiation fields create any operating difficulties.

(b) Corrosion

1. Out-reactor Specimens

All the specimens present in the first experiment in each loop, i.e., oxygen-free conditions, were found to be coated with a black oxide which was shown by X-ray diffraction to be magnetite (Fe_3O_4). This black coating was quite non-adherent and much of it could be brushed from the dried specimens. The remainder was removed by descaling in Clarke's solution (2) for 30 seconds. The specimens were weighed and corrosion rates were calculated from the resulting weight loss. The results are shown in Table III.

Table III

Corrosion Rates for Out-Reactor Test Specimens

Loop	pH	Time in Water (days)	Wt.Loss (mg)	Area (dm^2)	Corrosion Rate $mg/dm^2/mo$	Corrosion Rate inches/yr.
A. O_2 absent						
J-1	6.5-7.0	113	211.0	0.676	83	5.0×10^{-4}
J-1	"	55	75.0	"	62	3.7×10^{-4}
J-2	"	116	106.6	0.828	33	2.0×10^{-4}
J-2	"	60	108.8	"	65	3.9×10^{-4}
J-3	10.5	120	37.9	"	11	6.6×10^{-5} ★
J-3	"	150	5.4	"	1.1	6.6×10^{-6}
J-1	"	51	3.2	"	2.3	1.4×10^{-5}
B. O_2 present						
J-1	6.5-7.0	118	951	0.676	351	2.1×10^{-3}
J-3	10.5	51	2.3	0.828	1.6	9.6×10^{-6}

★ This specimen was in loop J-3 during the first experiment when difficulty was experienced in obtaining the proper pH.

Specimens which were exposed to water at pH 7 containing 3-5 ppm O_2(J-1, exp.2) were coated with a heavy corrosion film which consisted of a black oxide covered by a brown oxide. These oxides were shown to be Fe_3O_4 and γ-Fe_2O_3 by X-ray diffraction. When descaled the specimens were found to be very severely pitted, as can be seen in the photograph in Figure 7. Specimen (a) in this photograph is the sample for electron diffraction measurements which has not been descaled. The high corrosion rate found is given in Table III. A section of pipe cut from the loop showed the same mixture of oxides on the surfaces.

Specimens exposed to oxygenated water at pH 10.5 (J-3, exp.2) did not experience this high corrosion rate. The samples appeared very similar to those exposed to oxygen-free water, i.e., mainly black oxide corrosion product, general corrosion with no pitting. The corrosion rate was very similar to that obtained for oxygen-free water at pH 10.5 (Table III). Photographs taken before and after descaling are shown in Figure 9.

The photographs in Figures 6 and 8 show specimens which had been exposed to oxygen-free water at pH 7 and pH 10.5. Microscopic examination of all the specimens exposed to the oxygen-free water systems showed only mild general corrosion with no pitting. Pitting was only observed in water containing oxygen at pH 7.

The results in Table III indicate that the best water conditions of those tested are high pH with or without oxygen present. When no purification of the water was used the average corrosion rate appeared to be somewhat higher than when the water, at similar pH, was kept deionized. The amount of insoluble corrosion product in the loop water was much less in high pH water as indicated by the condition of the ratosights and by the appearance of the water when the loops were drained at the end of each experiment.

Corroded specimens were sent to N.R.C., Ottawa, for electron diffraction measurements, but due to the radioactivity of the corrosion film these measurements have not as yet been made.

2. In-reactor Specimens

No weights were obtained on the in-reactor specimens before they were sealed in the rods which were used for the first experiment in each loop and thus no corrosion rates could be determined. However, microscopic examinations were made after the specimens were descaled. Unlike the specimens in the out-reactor test section, these all showed some pitting which appeared least pronounced on loop J-3 specimens. The black oxide film also appeared to be more compact on these specimens in the high pH water in loop J-3. In all cases one surface appeared to be more severely attacked than the other and the pitting seemed to be concentrated close to the edges. It is thought that the difference in the corrosion of the in-reactor and out-reactor specimens was due to the flow conditions at the bottom of the rods.

In the second experiment in loop J-1 (O_2 present, pH 7) the test specimens in the in-reactor test section had been weighed before being inserted in the rod. After the termination of the experiment two of the rectangular specimens were retrieved, descaled and weighed. They gave corrosion rates of 391 and 368 mg/dm^2/mo compared with 351 mg/dm^2/mo found on the out-reactor specimen and they had the same appearance, i.e., brown oxide over black with extensive pitting.

VII. Conclusions and Summary

The results of these experiments have provided the following information in respect to the objectives listed in Section III of this report:

(1) When the recirculating system is under an inert atmosphere, the oxygen originally present in the water used to fill the system disappears very quickly in the corrosion reaction. Thereafter, the system remains essentially oxygen-free (0.07 ppm or less) even without the use of a deoxygenating resin.

(2) The continuous purification of the recirculating water by ion exchange does not appear necessary. However, it does seem to be advisable in that it reduces the amount of corrosion product in the flowing water and it also reduces the average corrosion rate.

(3) With a very low oxygen concentration in the water (referred to as an essentially oxygen-free condition) the average corrosion rate over a

4 month period at pH 10.5 is considerably lower than at pH 7 - i.e., about 5 mg/dm^2/mo as compared to 50 mg/dm^2/mo.

(4) With a concentration of oxygen in the water of 3-5 ppm, the corrosion rate is very dependent on pH. At pH 7 the average corrosion rate for a 4 month period was about 350 mg/dm^2/mo. At pH 10.5 the average corrosion rate for a 2 month period was only 1.6 mg/dm^2/mo. Therefore, it seems advisable to operate mild steel cooling systems at high pH to avoid the necessity of close control over the O_2 concentration in the water.

(5) Neutral water containing oxygen was found to cause severe pitting in the mild steel specimens in the loop. Actually, a flow pattern was etched into the specimens and is visible in the photographs. The corrosion found in high pH water containing oxygen was only general in nature.

(6) The in-reactor test specimens showed some pitting under water conditions in which the out-reactor specimens showed only general corrosion. This is considered to be due to (a) the turbulent flow at the bottom of the J-rod, (b) the position of the specimens being such that a relatively stagnant condition could exist on one side of them, and (c) the presence of hydrogen peroxide formed by reactor radiation of the water. It would seem therefore that, if conditions are right, reactor radiation can affect the corrosion of mild steel through the water decomposition products.

The results can be summarized by listing the systems tested in order of increasing corrosiveness: high pH with or without O_2 in the water $<$ water at pH 7 with no O_2 present and continual purification $<$ water with no O_2 present and no purification or pH control $<$ water at pH 7 with 3-5 ppm O_2 present.

VIII. Acknowledgements

The author wishes to thank C.J. Scully of the Metallurgy Research Branch for the photographic work. The assistance and co-operation of the NRX staff is greatly appreciated.

IX. References

(1) M. Cohen, Trans. Electrochem. Soc.89, 105(1946), Sodium Hexametaphosphate as a Corrosion Inhibitor for Ottawa Tap Water.

(2) Fowler, Douglas and Zyzes, KAPL 1198, Descaling Methods.

(3) F.H. Krenz and R.F.S. Robertson, CRC-544, Chemical Aspects of the NRU test loop.

(4) R.E. Manson, PDB-94, Review of the Problem of Corrosion Protection for the Reactor Thermal Shield Cooling Water System.

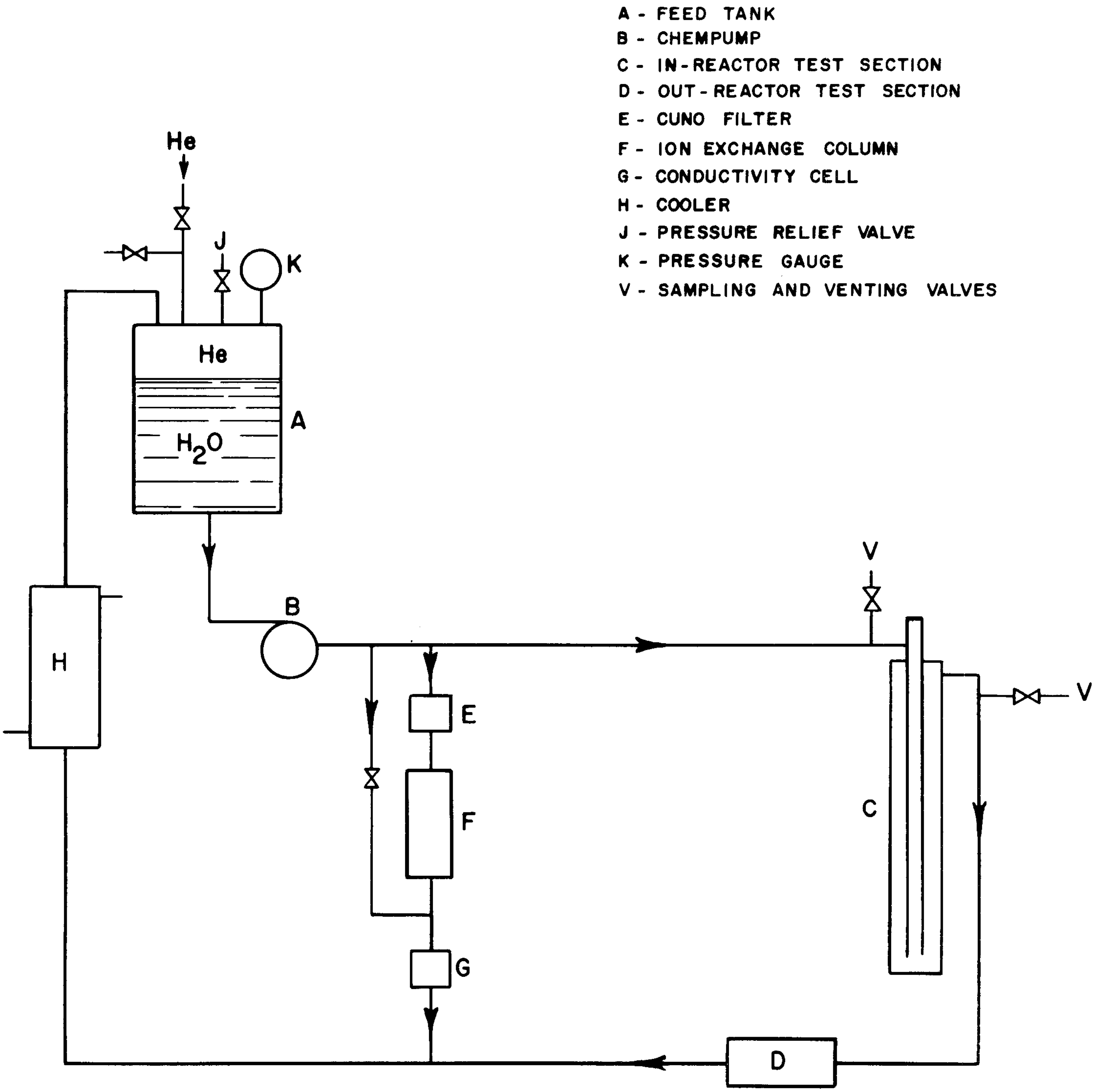
(5) A. Mizzan, DCI-3, Design Manual, J-rod Corrosion Loops.

(6) R.F.S. Robertson and P.G. Anderson, CRDC-596, Out-reactor Tests of the HTP loop.

(7) R. Williams - Modified Winkler Method - to be published.

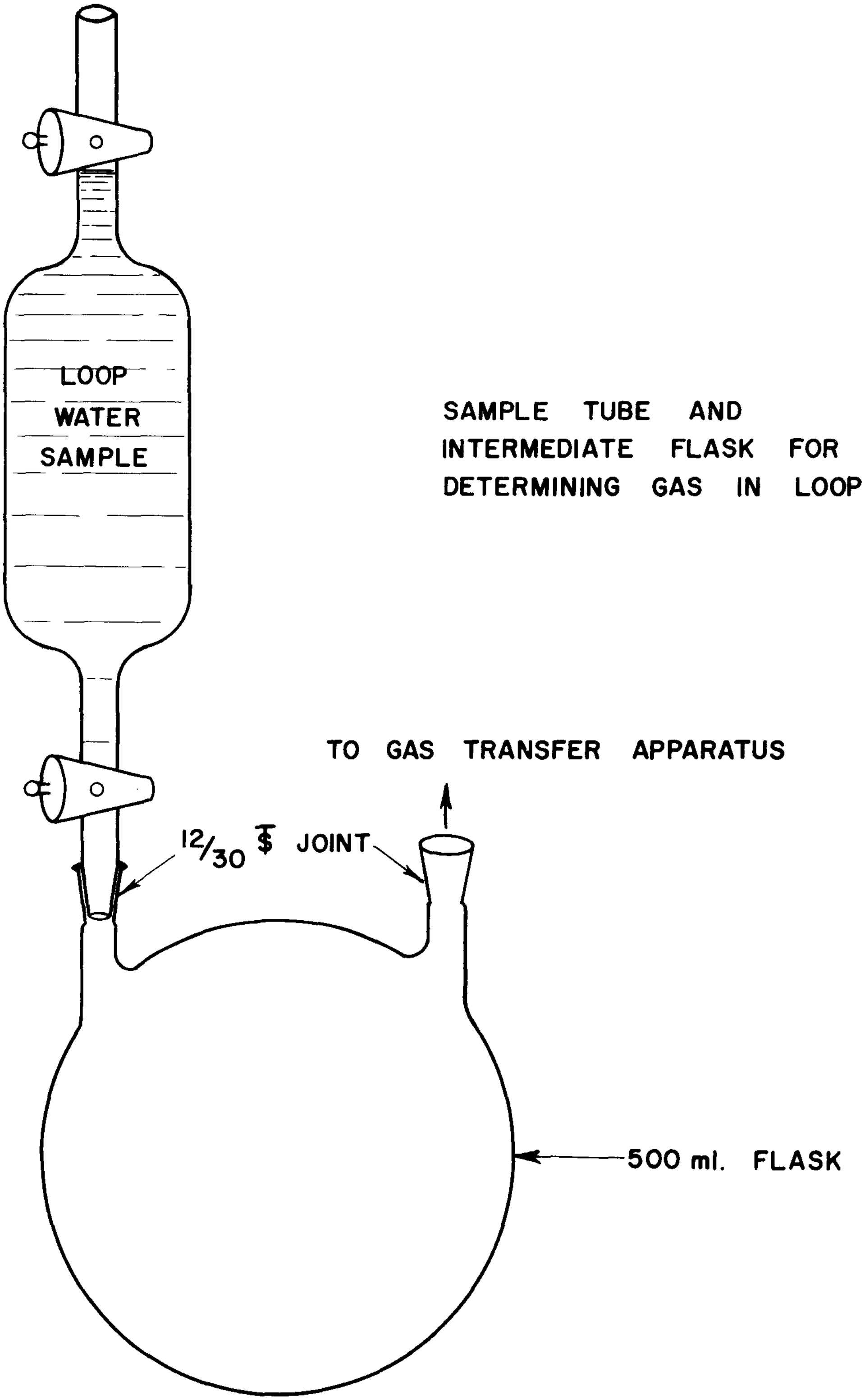
FIG I

FLOW DIAGRAM FOR J-ROD LOOPS



A - FEED TANK
B - CHEMPUMP
C - IN-REACTOR TEST SECTION
D - OUT-REACTOR TEST SECTION
E - CUNO FILTER
F - ION EXCHANGE COLUMN
G - CONDUCTIVITY CELL
H - COOLER
J - PRESSURE RELIEF VALVE
K - PRESSURE GAUGE
V - SAMPLING AND VENTING VALVES

FIG. 1(a)



SAMPLE TUBE AND INTERMEDIATE FLASK FOR DETERMINING GAS IN LOOP WATER

H_2 AND O_2 CONCENTRATIONS IN J-ROD LOOPS

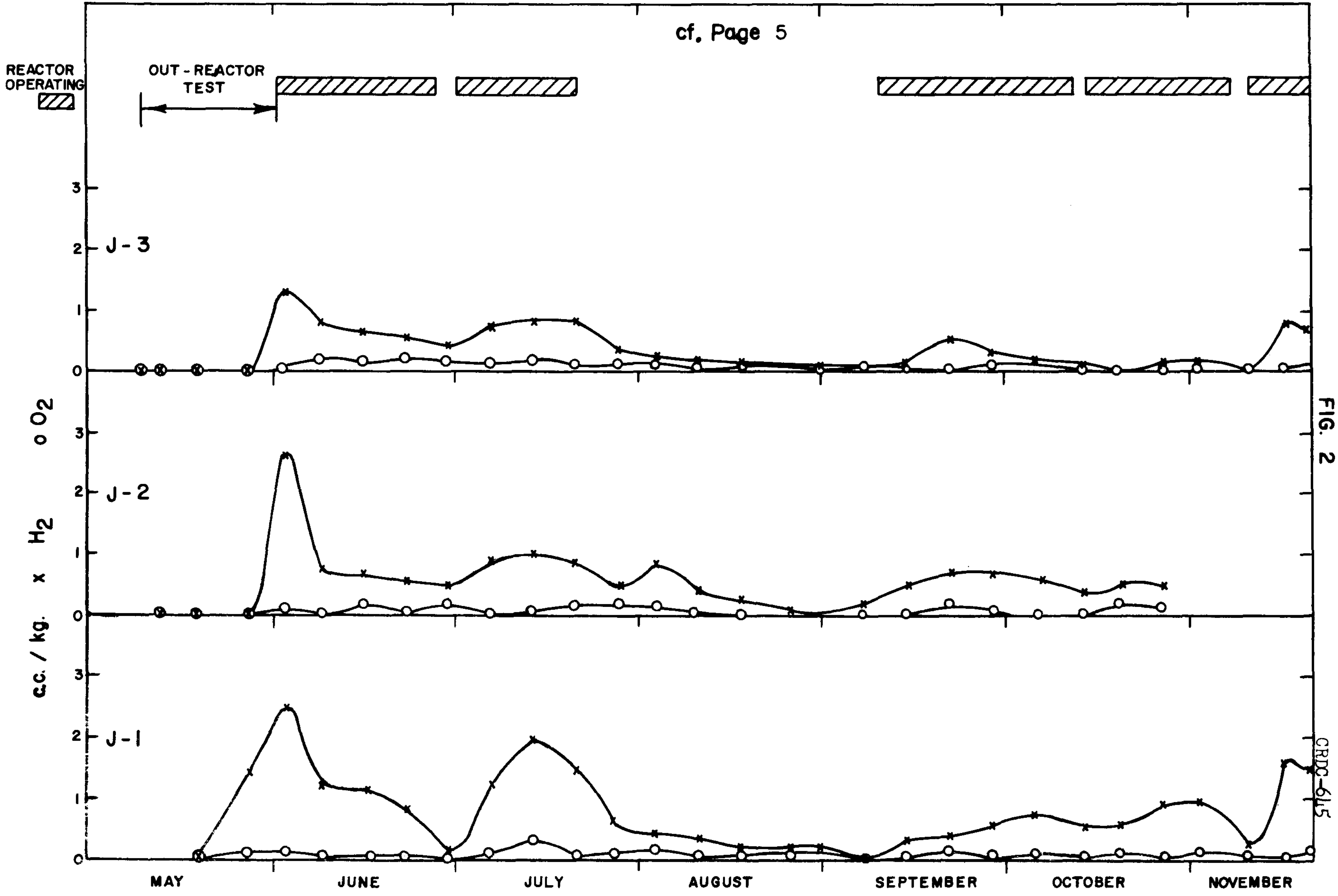


FIG. 2

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FIG. 3

H_2 AND O_2 CONCENTRATIONS IN J-3 (CONT'D. FROM FIG. 2)

cf. Page 6

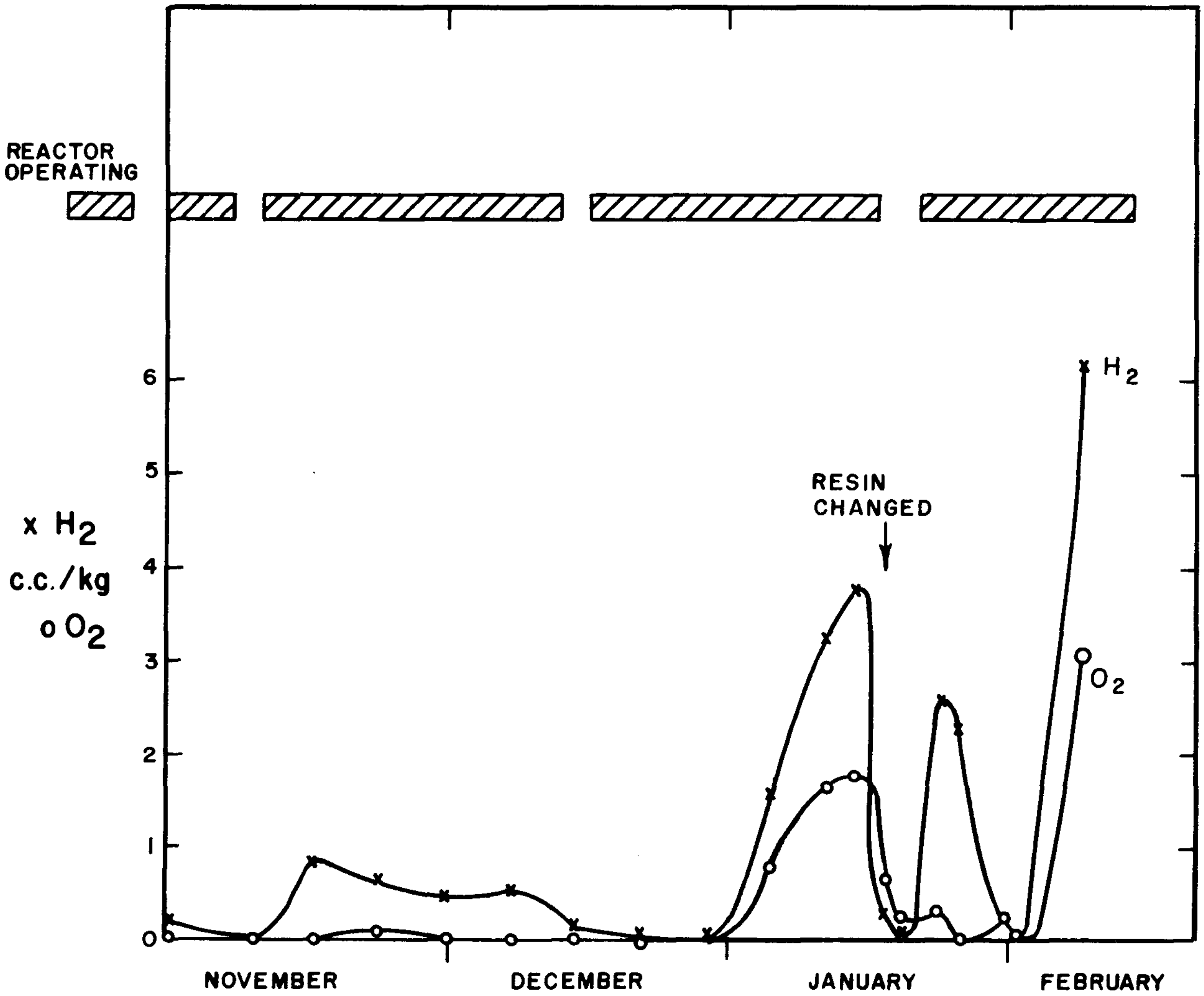
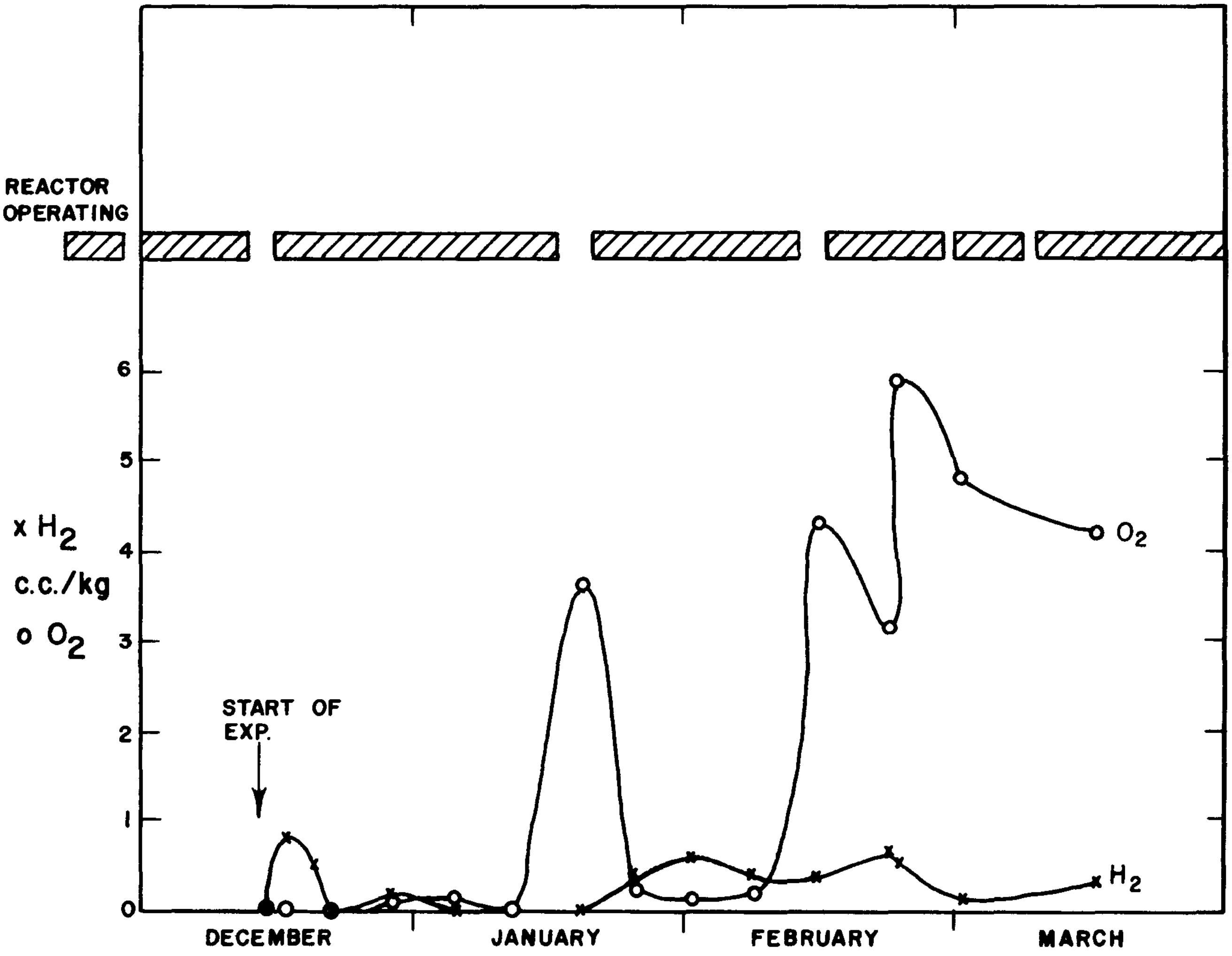


FIG. 4

H_2 AND O_2 CONCENTRATIONS IN J-1
EXPERIMENT 2

cf. Page 6



J-ROD LOOP #1 EXPERIMENT #1

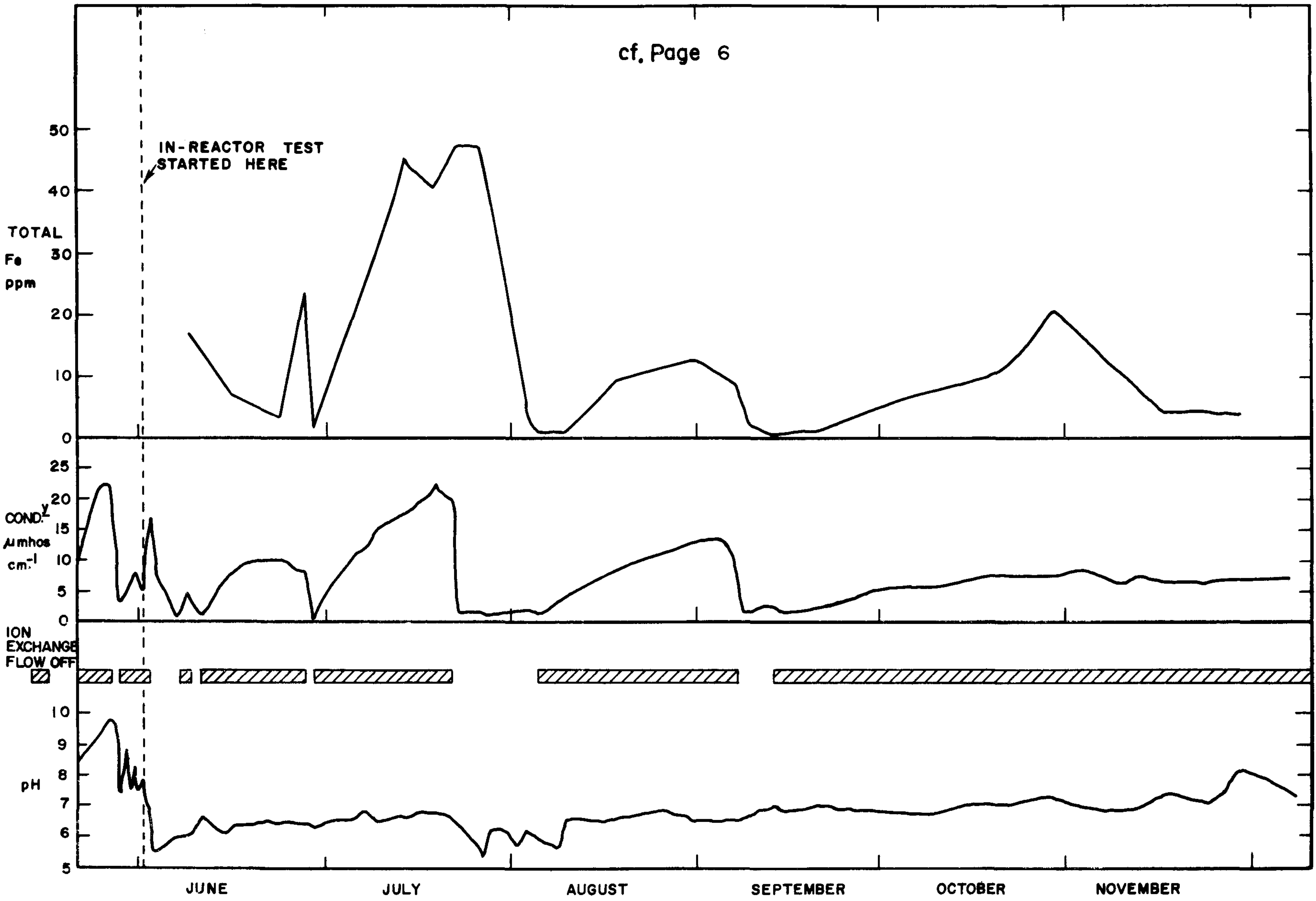
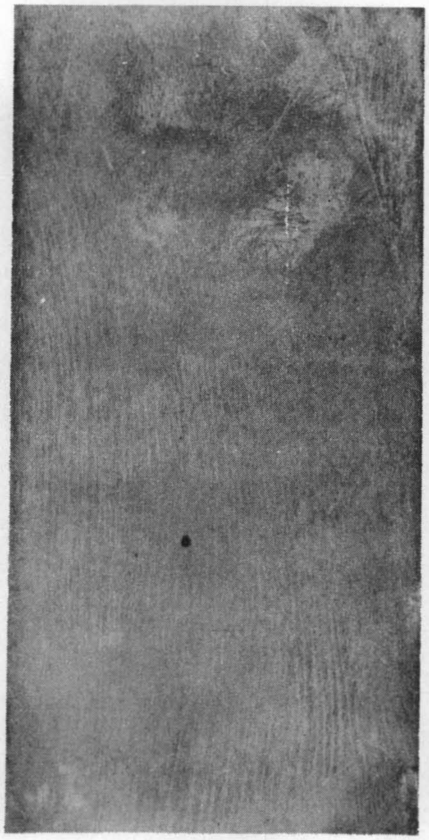


FIG. 5

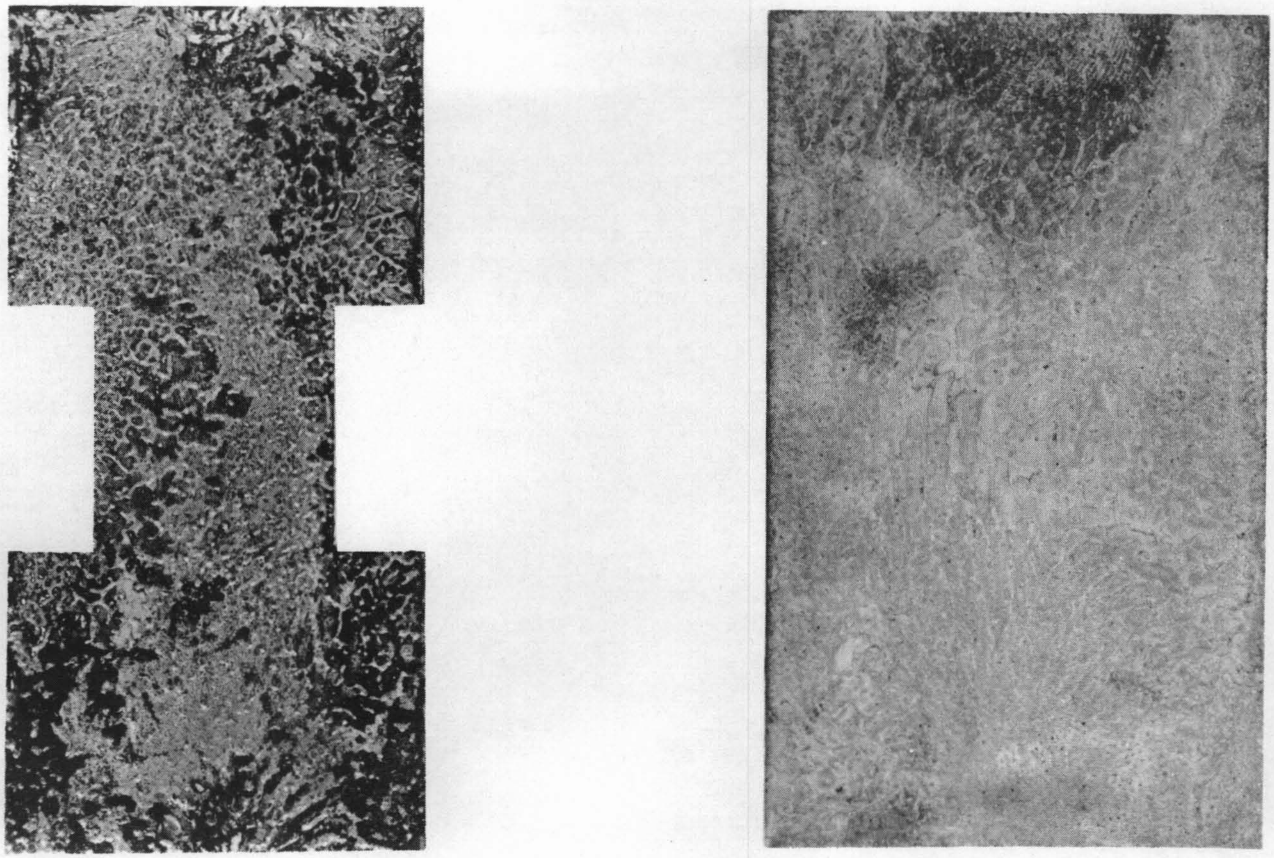
CRDC-645



1.5 X

DESCALED MILD STEEL SPECIMEN FROM OUT-REACTOR TEST SECTION OF LOOP J-2 EXPOSED TO WATER AT pH 6.5 - 7.0, NO O_2 PRESENT FOR 116 DAYS

FIG. 6



1.5 X

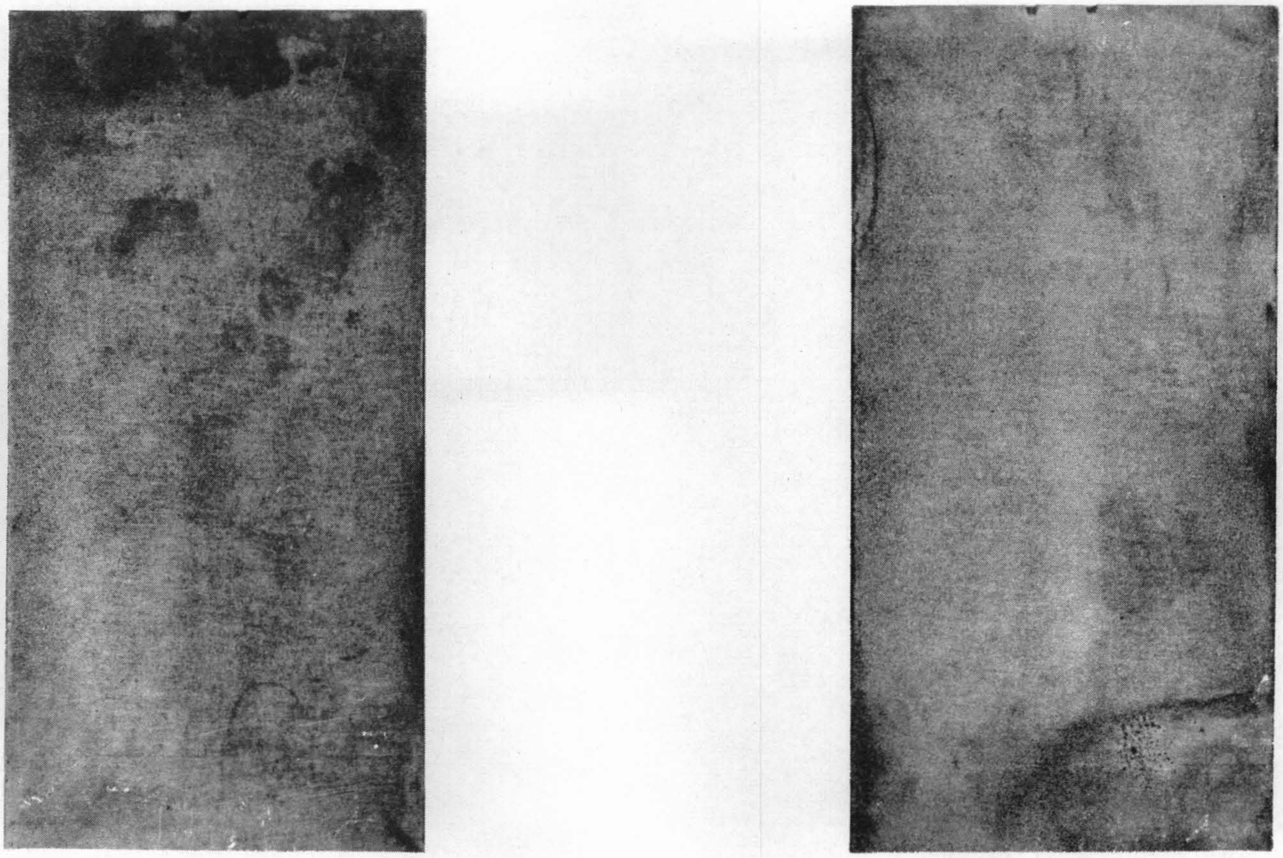
(A) BEFORE DESCALING

1.5 X

(B) AFTER DESCALING

MILD STEEL SPECIMENS FROM OUT-REACTOR TEST SECTION OF LOOP J-1 (EXP. 2) 120 DAYS IN WATER AT pH 6.5 - 7.0 CONTAINING 3-5 ppm O_2

FIG. 7



1.5 X

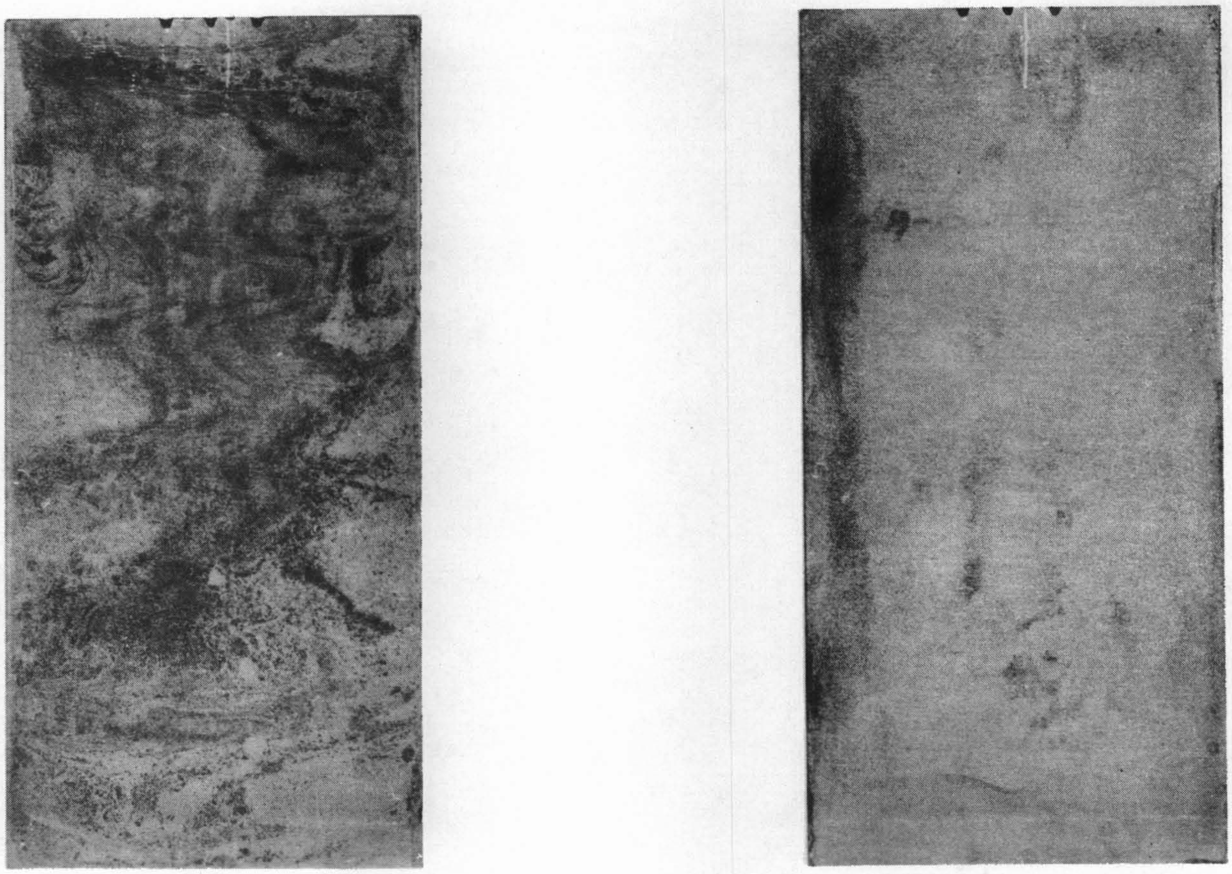
(A) BEFORE DESCALING

1.5 X

(B) AFTER DESCALING

MILD STEEL SPECIMENS WHICH HAD BEEN IN WATER 51 DAYS AT pH 10.5 WITH NO O_2 PRESENT

FIG. 8



1.5 X

(A) BEFORE DESCALING

1.5 X

(B) AFTER DESCALING

MILD STEEL SPECIMENS FROM OUT - REACTOR TEST SECTION OF LOOP J - 3 (EXP. 2) EXPOSED FOR 51 DAYS TO WATER AT pH 10.5 CONTAINING 3-5 ppm O_2

FIG. 9