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Section T-1

FILM FORMATION IN "W" ANNULUS

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Secret Notebooks Containing Film Study Data

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213-B	376-B	575-B	904-B	1334
217-B	379-B	610-B	905-B	1335
269-B	380-B	657-B	912-B	

Weekly Memos Covering Results of Film Formation Studies

<u>Memo</u>	<u>For Period Ending</u>
CEK - MUC #26	April 20, 1944
CEK - MUC #46	April 27, 1944
CEK - MUC #47	May 4, 1944
CEK - MUC #48	May 11, 1944
CEK - MUC #60	May 18, 1944
CEK - MUC #61	May 25, 1944
CEK - MUC #122	June 1, 1944
CEK - MUC #124	June 8, 1944
CEK - MUC #125	June 15, 1944
CEK - MUC #126	June 22, 1944
ACM - MUC #12	June 29, 1944
ACM - MUC #13	July 6, 1944
ACM - MUC #24	July 13, 1944
ACM - MUC #25	July 20, 1944
ACM - MUC #26	July 27, 1944
ACM - MUC #27	August 3, 1944
ACM - MUC #31	August 10, 1944
ACM - MUC #32	August 17, 1944
ACM - MUC #57	August 24, 1944
ACM - MUC #80	August 31, 1944
ACM - MUC #81	September 7, 1944
ACM - MUC #82	(September 14, 1944 September 21, 1944)

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FILM FORMATION IN "W" ANNULUS

Summary

Problem Under Investigation:

The studies by the Engineering Development Section on film formation at Site "B" were started when it was discovered in the Hanford corrosion tests that films of iron oxide deposited on the slug and tube surfaces. These films constricted the annular area to such an extent that an increase in pressure beyond that which the pumps could develop was required to maintain steady flow through the tubes. In addition, the heat transfer coefficients were reduced in the test equipment indicating that if a similar condition developed in the pile, its power output would be limited to perhaps as much as 50% of the designed level. Since these conditions are extremely undesirable, it was deemed essential that a condition be found to prevent deposition of these films or a method developed by which they could be removed at reasonable intervals. Because there are no data recorded in the literature that apply specifically to this problem, it was necessary that much basic work be done.

This problem was pursued at CMX and at ML by both the Engineering Development and the Metallurgy Sections. The work of this section was on "W" scale and directed toward the determination of the major factors involved in the deposition, prevention of the formation and removal of the iron oxide film as well as films resulting from other constituents added to or already present in the raw water.

Important Results:

It is hereby stressed, that in interpreting the results presented herein, the water used in these tests differed appreciably from that used at CMX and that there were several other variables involved. For the most part, however, these results did indicate in a broad manner under what conditions the various films might form and how they might be prevented and/or removed. Results from single tests were considered for their value in indicating trend only.

Under conditions of internal heating using demineralized water at pH 6.5 unless otherwise stated, it was found that:

Film formation:

(1) Additions of ferric ion result in increased rates of film formation apparently by precipitation of the iron on the surfaces as the hydrous oxide.

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(2) Ferrous iron appears to require oxidation to ferric before it can deposit as a film.

(3) Single run studies revealed that various anions added before the tubes caused different rates of film formation. Low concentrations of SO_4^{2-} and NO_3^- accelerate film formation while Cl^- , $\text{Fe}(\text{CN})_6^{3-}$ and $\text{Cr}_2\text{O}_7^{2-}$ have very little effect.

(4) Based on single runs at low velocities the rate of iron film formation is faster and the films are thicker than those formed at high velocities.

(5) Preliminary tests at constant velocity indicate that iron film formation is slower under reduced heat flux.

(6) Application of potentials as high as 3 volts across the annulus does not affect film deposition. Electrolytic corrosion is quite severe at this potential.

(7) Aluminum films appeared mainly at the cold end of the annulus and caused increased resistance to water flow with an increase in Al^{+++} concentration.

(8) Sulfate and dichromate had no effect on chromic film formation at pH 6.5. The rate of pressure drop from chromic films deposited from 4.6 pH demineralized water was much less than that obtained at pH 5.2, 6.4 and 7.5.

Film removal:

(9) Oxalic acid removal of iron films should take place with water having a pH below 4.0 to avoid deposition of calcium films.

(10) A pH above 9.0 in the process water should be avoided since calcium or magnesium films will deposit in the absence or presence of silicate.

(11) Neither oxalic acid at pH of 4.5 nor $(\text{NH}_4)_2\text{S}_2\text{O}_8$ at a pH of 7.5 were effective in removal of combined chromic and iron films. Hydrogen peroxide was successful in chromic film removal at pH 7.5.

(12) Silicate was not 100% successful in preventing chromic and iron films at a pH of either 4.6 or 6.5.

(13) As a protective agent disodium dihydrogen pyrophosphate was more successful with iron than with chromic ions.

(14) Calgon proved to be completely successful in preventing deposition of calcium films at pH of 4.2 - 4.7 while removing iron films with oxalic acid. However, iron removal is quite slow at this pH.

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Recommendations:

No recommendations as such are being made since this is a final record report. The results which were obtained were acted upon as seen fit by those responsible for determining treatments and operating procedures for the "W" pile cooling water system.

If, in the future, tests of similar nature are made, it is suggested that, in addition to the precautions noted herein, the following steps be taken:

- (1) Sufficient acid should be added to solutions of cations being tested to insure their addition to the process water in ionic rather than colloidal state (this is open to question as regards our work with chromic).
- (2) Process pipelines should be of non-corrosive materials to eliminate this nearly uncontrollable variable. (Iron was dissolved from our iron process lines).

Prepared by

P. E. Larson

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GENERAL DISCUSSION

Introduction:

The original problem under this program was the corrosion of aluminum under "W" pile conditions. More or less complete studies were to be made by the Metallurgical Section and the most promising of these results were to be evaluated on a semi-works scale by the Engineering Development Section. As a final check on the best conditions for least corrosion as obtained by this section, long time tests were to be conducted at the CMX laboratory at Hanford using the same water that will be used in the pile. However, when corrosion tests were begun at CMX, increases in the pressure required to maintain steady flow were experienced. Examination of the slugs used in these tests revealed that a deposit of what appeared to be iron oxide had formed on the slug and ribbed tubing surfaces to such an extent that the decrease in annular area was sufficient to cause a substantial increase in resistance to flow.

In heat transfer work, a film is usually defined as a stagnant layer of either a liquid or gas along the tube-fluid interface. A modification of this definition occurs in our work in that the term film is specifically intended to apply to a solid scale deposited on the tube from water containing colloidal and/or dissolved solids.

Scale deposits, or hereinafter referred to as films, are extremely undesirable in that the heat transfer coefficients are decreased causing increased metal temperatures, thereby limiting the power output of the pile. The increase in pumping power is also a factor to be considered. It was felt that this problem was of such importance that additional work on corrosion was deferred and all energy was expended on efforts to eliminate or prevent the formation of these films.

A short history of how the principal film forming cations in "W" water entered this study follows: The minimum iron content of the filtered Columbia River water was sufficient to cause a film. Use of sodium silicate had been found to prevent deposition of ferric iron; however, in order to inhibit the abrasive action of the silicate it was necessary to add $\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$. (See MUC - J. P. Howe #222 Meeting Concerning Hanford Cooling Water Problems) When other laboratory workers showed that dichromate underwent a reduction to chromic ion in the presence of pile radiation, film studies on Cr^{+++} assumed a role of major importance. By increasing the pH of the water to 7.5 it was found that pile reduction of dichromate decreased to about zero. But it had formerly been determined that the corrosion rate of 2S aluminum was least at a pH of 6.5. Therefore, pH control became a critical part of the test in order to avoid deposition of Ca^{++} and Mg^{++} films at values somewhat above that which is just sufficient to prevent dichromate reduction. In addition, the formation of an aluminum film would have to be dealt with provided no suitable substitute for alum could be found for use in flocculating the water in the early stages of its treatment. Later in the course of the

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several studies, it was decided that Ferrisul (ferric sulfate) would be a satisfactory flocculating agent, thus eliminating aluminum films from being of major concern.

Although this report covers primarily that part of the film problem investigated by the Engineering Development Section at ML, a very considerable portion of the work was carried on by other groups here and at CMX and Clinton Laboratories. A limited attempt has been made to correlate our work with the results of others. Our objectives may be summarized as (1) Problems dealing with major factors affecting Fe^{+++} , Cr^{+++} , Al^{+++} , Mg^{++} , and Ca^{++} film formation, (2) Problems concerning the removal of the deposited films, and (3) Studies of the prevention of film deposition by use of suitable protectors. At CMX the tests were to be aimed chiefly toward a rapid solution of the film problem in actual "W" water. Detailed laboratory studies of film characteristics were carried out by the Corrosion Group of the Metallurgy Section.

The equipment at ML for the study of film formation consisted of demineralizers for handling raw lake water (chlorine treated but not flocculated or filtered) iron water pipe, equipment for either pre-heating or internally heating the water, 11 ft. lengths of aluminum tubing of slug diameter and 8.3 ft. lengths of ribbed tubing. Tests were begun in January and were ended in September, 1944. A study of the effect of time lag on deposition of films was made in the first 12 tests with water pre-heated in G-fin heat exchangers to temperatures approximating maximum slug surface temperatures expected in the pile. The later experiments were conducted with steam heated specimens. In these tests average heat fluxes of 35 Kw/ft. were used.

Although no such film as had been observed at CMX was obtained in the corrosion studies at ML, subsequent early work in film studies revealed that the film could be made to deposit from demineralized Chicago water and that the phenomena was not peculiar to "W" water. For this reason it was felt that much useful data could be obtained at ML and the tests were continued. In comparing our film work with that at other sites, it must be remembered that all of the ML tests were made in the absence of pile radiation and the majority with internally heated specimens. At Clinton tests were run with pile radiation and at CMX the tests were run without radiation, but with water which was either pre-heated* or externally heated.**

* "Pre-heating" has been defined as that type of heating which takes place in a heat exchanger before the process water enters the equipment where it is desired to study the film effects.

**A system in which steam is passed through the annular space formed by the ribbed tube and a pipe surrounding this tube has been defined as an externally heated system.

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Equipment:

The equipment used by this section in the film studies is pictured schematically in Figure 1.

Water used in tests at Site "B" was Chicago City water (chlorine treated but not flocced or filtered) which was demineralized by substantial removal of dissolved salts by passing in succession through cation and anion exchange resin beds. The demineralizer was purchased from the Illinois Water Treating Company of Rockford, Illinois. The cationic bed consists of ca 50 cu. ft. of Amberlite IR-100, a phenol formaldehyde type resin, which replaces the cations (Ca, Mg, Na, etc.,) in the water with hydrogen ions thus forming mineral acids. These acids are then removed in the anionic unit by passage through ca 21 cu. ft. of Amberlite IR-4 resin, an amine-formaldehyde type adsorbent.

At a water consumption rate of 45 gals./min. sufficient for 2 specimen tubes (about 65,000 gallons per day), the anionic resin required regeneration with a 2-5% soda ash solution after ca 2 days operation, while the cationic unit required regeneration with a 2-5% sulfuric acid after treating 25,000 gallons of water. Continuous operation of the demineralizers during a cationic regeneration was possible since a spare cationic unit was provided.

The purity of the demineralizer effluent was tested continuously by a "Solubridge" conductivity meter placed in the line, and by chemical analyses for chloride made every hour. Units were taken out of service when the conductivity increased rapidly or when the chloride content reached 1 ppm.

From the "Solubridge" the water passed through an integrating volume meter and into the first of two 1800 gallon Lithcote lined storage tanks connected in parallel. Sodium hydroxide solution was injected into the line between the demineralizers and the storage tanks to adjust the pH to the desired value. The water was pumped from the tanks to the test sections in parallel iron lines by means of two 75 gal./min., 231 ft. head, Ingersoll-Rand centrifugal pumps. In the tests in which the water was heated prior to the specimens, heating was done in Griscom-Russell G-fin, concentric tube heat exchangers placed directly after the pumps.

The test sections consisted of two 8 ft. 4-in. lengths of 4 ribbed tubing mounted in suitable water heads as shown in Figure 2. The specimens used were 2S aluminum tubing 1.38 in. O.D. x 1.00 in. I.D. During the last two months of work (runs 99 through 127) the test sections were changed to 9 ft. 2-in. lengths of alclad* 2 ribbed tubing in suitable heads. The new specimens used were 2S aluminum tubing 1.44 in. O.D. x 1.00 in. I.D. The specimen tubes were re-used since in most instances the film could be wiped off. For internal heating, 150 lbs./sq.in. gauge steam was throttled to 50 lbs./sq. in. gauge and passed through the specimen. Steam heating of the hollow specimens caused a relatively large amount of expansion, therefore at the water inlet end of the specimens the heads were mounted on plates that could slide along the

* Alclad tube is 2S aluminum tube with a 72S aluminum cladding on the interior.

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longitudinal axis of the specimen tube. To a great extent the buckling of the ribbed tube was alleviated. There continued to be a noticeable difference in temperature between the upper and lower halves of the outer ribbed tube, thus indicating that warpage of the specimen was still causing a variation in heat transfer. Some of this temperature gradient was attributed to condensate lying on the bottom of the specimen tube. It was intended to conserve steam by use of traps at the ends of the specimens; however, because of operating difficulties the traps were by-passed, and the exit steam lines were allowed to exhaust directly to the sewer.

Four pressure gauges were placed along the ribbed tube as shown in Figure 1. The center two gauges are located $1/3$ and $2/3$ of the distance between the water inlet and outlet. At all these points, piezometer rings were made by drilling 3 - $5/64$ -in. dia. holes 120° apart and through the ribbed tube. This portion of the tube was enclosed (as shown in Figure 2). Temperature readings were taken in the specimen end-fittings with Hg in glass thermometers and control was maintained by varying the amount of steam used. With the aid of Hg manometers attached to taps on either side of stainless steel orifice plates, the operators could manually maintain the desired 20 ft./sec. flow in the annulus. Reagents were metered into the water through the system shown in Figure 3. This type of equipment was found to give accurate control and was very satisfactory in use. All chemicals were added as solutions by positive displacement proportioning pumps. The latter were of 2 models: (1) Hills-McCanna U type and (2) Manzel series 40. The addition points of the added chemicals could be adjusted to give various time lags between the point of addition and that where the process water entered the specimen. Because of the corrosive nature of the reagent solutions which were added, and to prevent contamination or decomposition of the reagents before addition to the test water, the iron pipe lines from the proportioning pumps to the process water were replaced after Run 35 with saran tubing.

In connection with the pressure drop studies an attempt was made to determine the effect of electric charges on film formation. A 30" cataphoresis unit was built in series with one of the long specimens, whereby the hollow tube was electrically insulated from the 2S aluminum de-ribbed tube. By connecting the ribbed tubing and the specimen tubing to a dry cell a potential was impressed across the annulus. Cataphoretic studies were also made by removing the center third of the ribbed tubing of one 8.3 ft. section, and replacing this with a tube from which the ribs had been removed. This center section of de-ribbed tube was then insulated from contact with the specimen by means of the fittings shown in Figure 4.

A small unit for determination of film forming characteristics of the demineralized water at a number of points along the iron pipe line was also constructed. This unit contained an aluminum tube having an inside diameter which was equal to the equivalent diameter of the annulus, and the unit was heated externally with 4 Kw of electrical power in calrod units. Insufficient

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operating personnel prevented us from completing the proposed study with this small calrod unit.

An electrical contact micrometer was developed for the measurement of the thickness of the films. All figures given in this report on film thickness were by measurements made with this instrument. The micrometer is shown in the photograph, Figure 5.

Procedure:

After outlining a series of tests, the desired conditions for a particular run were specified, and detailed starting information was turned over to the operations sub-group. Chemical conditions during the run were maintained by analyses or by stoichiometric additions. The pH was controlled by half hour readings on a Coleman Model 3D or a Beckman Industrial Model pH meter at a temperature of 30°C, and manual control of the proportioned amount of NaOH was based on these determinations. The pH adjustment in the early runs (the first 12) was made at the specimens. The point of adjustment was then changed to the line between the demineralizers and the storage tanks. Peroxide control was obtained by hourly analysis using a calorimetric determination with a starch indicator. Chloride was analyzed hourly by a turbidimetric determination but was not controlled. The chloride content varied from 0 to 1 ppm between demineralizer regenerations. Additions of ferric, ferrous, chromic, sulfate, dichromate and other ions were made stoichiometrically and were not analyzed on a control basis. The average concentration of each added reagent was computed by a meterial balance at the end of a run. Complete analyses on daily composite samples of water for each run were made at the laboratory of the Metallurgy Section. Spot samples were taken throughout the system to follow various changes such as iron pickup from the iron lines leading to the specimens.

In order to avoid having unknown amounts of anions and/or cations in the added solutions, C.P. grade reagents were used to a large extent. Commercial grades of Na_2SO_4 and $\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$ were found satisfactory for our use.

The specimens were 2S aluminum tubing 1.38 in. O.D. x 1.00 in. I.D. and 11 ft. overall length. Surfaces of these specimens were used as received and no attempt was made to remove surface imperfections. These tubes were re-used since in most cases the film could be easily removed by wiping. It was the exception rather than the rule to polish a specimen with emery paper before installing. Sometimes, however, a thin tenacious film which had been baked on during a previous run could be removed only by mechanical abrasion. In all instances the specimens were degreased with acetone before installing.

In order to reduce the effects of unknown and uncontrollable conditions to a minimum so that results could be duplicated and correlated, two specimens were run simultaneously in nearly all tests, using one or the other as a control. Film formation was observed by noting increase in pressure drop at constant flow rate over the length of specimen tube. In some cases the runs were terminated at the point at which the flow rate decreased to such an extent that the Ingersoll-Rand pumps could no longer maintain the desired flow, and in others, at points where it was felt that the data obtained was sufficient for the particular conditions. Runs were usually limited to 24 to 48 hours since this period of operation usually was adequate for the high-spot type of evaluation being made.

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Pertinent data were recorded and composite water samples were taken at half hour intervals during a test.

At the termination of a run the thickness of the film was measured and film samples were taken for any desired tests (film analysis and particle size determination). Pressure drop increase and temperature rise data were plotted as a function of time. The pressure drop was also calculated as percentage increase per hour for each third of the tube and over the whole tube. From the data the water side, steam side and overall heat transfer coefficients were calculated. The decrease in the water film coefficient, h_w , was expressed as percentage decrease per hour. Calculations of h_w are based on the following equations: -

$$(1) \quad h_w = 150 (1 + 0.011 t_a) \frac{V^{0.8}}{D^{0.2}}$$

$$(2) \quad U_1 = \frac{Q_1}{A_1 (\Delta t)_m}$$

$$(3) \quad \frac{1}{U_1} = \frac{1}{h_s} + \frac{L}{k} + \frac{1}{h_w}$$

where:

h_w = film coefficient of water in annulus, Btu/(hr.)(sq.ft.)(°F.).

h_s = film coefficient of steam in inner pipe, Btu/(hr.)(sq.ft.)(°F.).

U_1 = overall heat transfer coefficient based on A_1 , Btu/(hr.)(sq.ft.)(°F.).

A_1 = outer surface area of inner pipe, sq. ft.

k = thermal conductivity of aluminum, Btu/(hr.)(sq.ft.)(°F.)/(ft.).

Q_1 = heat gained by annular fluid, Btu/hr.

V = average water velocity in annulus, ft./sec.

D = $D_2 - D_1$, ordinary equivalent diameter of annulus, inches.

D_2 = inner diameter of outer pipe, inches.

D_1 = outer diameter of inner pipe, inches.

L = thickness of inner pipe, ft.

t_a = average temperature of annular fluid, °F.

$(\Delta t)_m$ = logarithmic mean of terminal temperature differences, °F.

* This formula for startup conditions only.

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The value of h_s is calculated by equation (3) from values of h_w and U_1 obtained in equations (1) and (2) at startup conditions. In calculating the final conditions, U_1 is recalculated and assuming h_s constant throughout the run, h_w is determined by substituting the values in (3), i.e., the final h_w includes the scale coefficient. On the steam side, temperatures were based on the steam saturation pressures. Values of K /linear foot of tube were also calculated for startup and stopping conditions. Calculations of rates as percentage per hour were based on initial values. All pertinent data and calculations for each run were incorporated into a run report.* A sample of this is given in Exhibit A.

Results and Discussion:

Film studies conducted by this section were aimed chiefly toward the determination of the effect of variables controlling film formation. It is believed that the deposition of the films is mainly the precipitation of colloidal hydrous oxides present in, or formed in the water during its passage through the tubes. Thus, conditions affecting the formation or the stability of these colloids would be expected to affect the rate of pressure drop increase. It is stated in the literature that pH, electrolytes, temperature and rate of mixing greatly influence the coagulation rates of the hydrous oxides. In the investigation of these conditions pH is fixed either by that required for minimum corrosion of the aluminum or by that set from balancing corrosion rates and the reduction of dichromate through pile radiation. The rate of mixing or turbulence is set by the geometry of the tubes and may be varied only by virtue of velocity. Unless otherwise specified all tests were made at a pH of either 6.5 or 7.5 and at a velocity of 20 ft./sec.

Effect of time factor on iron film formation:

Early work was done in apparatus equipped with heat exchangers prior to the test sections. Demineralized water of average analysis, as tabulated in Table I (see Appendix), was heated to 85°C at points 5, 25, and 50 seconds time lag before the water entered the specimens, to study the effect of time lag between point of heating and test sections on film deposition. In these time-factor tests pH was adjusted from 4.7 to 6.5 immediately before the specimens. Runs were of 20 to 30 hours duration and were terminated at the point that flow began to decrease or when it appeared definite that the rapid increase in pressure drop would not be experienced.

A possible explanation of the fact that a film was formed at both 5 and 50 second time lags and not at 25 seconds is that the precipitation of a film is dependent upon the time required for the colloidal particles to increase to a size at which they will plate out. The time cycle is such that heating 25 seconds before the specimen precipitates the iron in the intermediate piping thereby reducing its concentration to a value below that at which a film will deposit in the annulus. It would seem that the deposition of a

* See Tables XXIX through XXXV in the Appendix.

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film is a cyclic process depending upon iron pickup, time for particle size growth, point of application of heat and size of particles required for deposition. In the tests where preheating occurred at 5 and 50 sec. before the specimen the cycle was at the film depositing stage when the water passed through the annulus. When the water was preheated 25 seconds before the specimen, the cycle process had already passed through the deposition stage and had reached the stage of pickup and particle growth since no deposit took place in the annulus.

To test this effect of iron pickup, the pH was adjusted at the storage tanks so that the water in the iron pipelines was at pH 6.5 rather than the previous 4.7. By preheating the water 50 seconds before entering the specimens no film was formed during 76 hours operation. This and subsequent tests indicate that increasing the pH to 6.5 reduces the amount of iron pickup.

Conversion to internally heated specimens:

In order to study film formation under conditions more nearly simulating those in a "W" pile it was decided to change the film equipment from a system in which water was preheated to one in which the water was internally heated by steam flowing inside a specimen tube whose outside diameter was equal to that of a slug. The installation of these heated units permitted following more closely the effect of temperature on film deposition and enabled heat transfer studies to be made using heat fluxes of 20 Kw/ft. or greater. All subsequent runs were made with this equipment. In these tests pH was adjusted at the storage tanks and peroxide was added one second before the specimens. Additions of other reagents were made at the points specified. In order to determine the natural film forming properties of demineralized water a preliminary test with no additions other than peroxide was made. It was found that the pressure drop increased at a rate of 0.05%/hr. over a period of 126 hours.

Iron films - ferric sulfate:

It was found that additions of ferric ion added simultaneously with sulfate ion increased rate of film formation. At concentrations of 0.01 to 0.05 ppm Fe^{+++} added, the effect on pressure drop and heat transfer was not markedly different while at 0.2 ppm of added Fe^{+++} very large changes in pressure drop and heat transfer occurred. One run using an added concentration of Fe^{+++} greater than 0.2 ppm gave a rate of increase in pressure drop lower and more nearly equal to those obtained with the low concentration of added Fe^{+++} . It is believed that this phenomenon was caused by the relative supersaturation of the iron in the water under test conditions, resulting in precipitation of the iron in a form that, for the greater part, was carried through rather than deposited on the specimen.

Tests on added ferric ion were made with 2 and 0.2 ppm H_2SO_4 added simultaneously. These concentrations of acid were required to keep the addition solution in ionic form. In tests prior to run 36, ferric additions were made through iron piping. It was found, however, that there was sufficient iron pick-

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up in the addition system to give erroneous results. To eliminate this source of error, saran systems were installed for all reagent pump lines. Only additions made in this manner are reported below unless otherwise specified.

TABLE II

Tests with Added Ferric Ion as $\text{Fe}_2(\text{SO}_4)_3$ and Simultaneous Addition of 2 ppm H_2SO_4

Run No.	Fe^{+++} ppm	Hrs. Oper.	Film Formation Mils/Hr.	Increase in ΔP -%/Hr.**	Decrease in ΔT -%/hr.	Decrease in h_w -%/hr.
36	0.00	41.5	0.02 - 0.06	0.4	0.4	1.1
48	0.05	28.6	-	0.8	0.3	0.8
Ave.*	0.15-0.20	2.5-6.0	0.20 - 0.60	7.0	5.4	11.2

* Average of 5 runs with additions being made through iron piping. Runs are 22-1A, 22-1B, 23-1A, 23-1B, and 23-1C.

** Increase in pressure drop through specimen over that at start of run.

The minimum amount of H_2SO_4 to be added to ferric sulfate solutions in order to insure that the ferric ion was in ionic rather than colloidal form was determined by turbidity tests employing a nephelometer. The minimum sulfuric acid content corresponded to no turbidity.

TABLE III

Tests With Added Ferric Ion as $\text{Fe}_2(\text{SO}_4)_3$ and Simultaneous Addition of Minimum H_2SO_4

Run No.	Fe^{+++} ppm	H_2SO_4 ppm	Hrs. Oper.	Film Formation Mils/hr.	Increase in ΔP -%/hr.	Decrease in ΔT -%/hr.	Decrease in h_w -%/hr.
49	0.00	0.3	13.0	0.00 - 0.04	0.2	0.2	0.3
37	0.01	0.1	36.1	0.01 - 0.06	0.2	0.4	0.9
39	0.05	0.2	30.0	0.01 - 0.04	0.03	0.2	0.6
38	0.2	0.2	4.0	0.00 - 0.54	8.9	2.6	7.5

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Iron films - ferric chloride:

To isolate the effect of sulfate from that of the iron, Fe^{+++} was added from FeCl_3 with added amounts of HCl equivalent to the 2.0 ppm H_2SO_4 used in the sulfate additions. A very rapid increase in pressure drop was again found at 0.2 ppm of added Fe^{+++} ion although the effect was less than that found in runs with equal amounts of SO_4^{--} . Results of these tests are as follows:

TABLE IV

Tests With Added Ferric Ion as FeCl_3 and Simultaneous Addition of 2 ppm Chloride

Run No.	Fe^{+++} ppm	Hrs. Oper.	Film Formation Mils/Hr.	Increase in ΔP -#/hr.	Decrease in ΔT -#/hr.	Decrease in h_w -#/hr.
41	0.00	21.0	0.02 - 0.04	0.1	0.1	0.6
47, 50	0.05	42.8, 34.0	0.01 - 0.07	0.2	0.2	0.7
52	0.2	5.4	0.00 - 0.61	4.3	0.0	0.0

Iron films - various anions:

In order to determine the effect of various anions upon the rate of pressure drop increase the sodium or potassium salt of the anion under test was added 2 seconds before the specimen. With the addition at this point it is believed that the most evident effects produced by the addition are those due to precipitation or stabilization of the colloid by the anion. Single runs, which would give trends rather than conclusive results, were made on these anions at a pH of 6.5 with no other additions except peroxide and iron pickup from the pipes. The SO_4^{--} and NO_3^- anions were found to give rates of pressure drop increase 2 to 3 times greater than those produced by Cl^- , $\text{Fe}(\text{CN})_6^{--}$ and $\text{Cr}_2\text{O}_7^{--}$ when added in concentration of $1.4 \times 10^{-5} \text{N}$ as calculated on the basis of valence charge. The results of these tests are as follows:

TABLE V

Tests With Anions Added as Na or K Salts 2 Seconds before the Specimen

Run No.	Anion	$\frac{5}{\text{Nx}10}$	Hrs. Oper.	Film Formation Mils/hr.	Increase in ΔP -#/hr.	Decrease in ΔT -#/hr.	Decrease in h_w -#/hr.
36	SO_4^{--}	4.4	41.5	0.02 - 0.06	0.4	0.4	1.1
41	Cl^-	5.9	21.0	0.02 - 0.04	0.1	0.1	0.7
44	NO_3^-	3.4	23.8	0.01 - 0.10	0.4	0.4	1.3
45	$\text{Fe}(\text{CN})_6^{--}$	4.4	28.8	0.00 - 0.05	0.03	0.2	0.1
46	$\text{Cr}_2\text{O}_7^{--}$	4.4	28.0	0.01 - 0.04	0.06	0.1	0.8

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Iron films - ferrous sulfate:

Ferrous additions at 2 and 60 seconds before the specimen gave pressure drop increases much lower than those with Fe^{+++} in tests with H_2O_2 adjusted to $2 \times 10^{-5} \text{N}$ at 1 second before the specimen. A comparison of Fe^{++} additions with and without H_2O_2 showed that the pressure drop increase is somewhat lower in the latter case although here again only 1 run was made and the point of Fe^{++} addition was an additional variable. In a run in which H_2O_2 was adjusted to $2 \times 10^{-5} \text{N}$ and in which other additions of 2 ppm $\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$, 35 ppm SO_4^{--} and 0.26 ppm Fe^{++} were made, the rate of pressure drop increase was much greater than that encountered with additions of ferrous and peroxide alone. It would seem from these last two tests that Fe^{+++} is the dangerous form of iron as regards film deposition and that oxidation of Fe^{++} to Fe^{+++} must occur before formation of a film. It also appears that the rate of oxidation in non-deaerated water is comparatively slow. The results of these tests are as follows:

TABLE VI

Tests With Ferrous Ion Added as FeSO_4

Run No.	H_2O_2 $\text{Nx}10^5$	Fe^{++} ppm	Hrs Oper.	Film Formation Mils/hr.	Increase in ΔP -p/hr.	Decrease in ΔT -°/hr.	Decrease in h_w -°/hr.
40	1.9	0.05(2)	33.9	0.01 - 0.06	0.2	0.2	0.7
51-2	1.9	0.05(37)	15.3	0.01 - 0.13	0.2	0.5	1.6
51-1	2.0	0.05(66)	15.3	0.01 - 0.13	0.2	0.3	1.2
43	0.0	0.2(2)	40.7	0.02 - 0.11	0.1	0.3	1.0
42	2.0	0.2(2)	36.0	0.02 - 0.13	0.5	0.6	1.5
53-2	2.0	0.2(37)	33.5	0.05 - 0.16	0.5	0.06	1.5
53-1	1.9	0.2(66)	33.5	0.05 - 0.16	0.4	0.6	1.6
120-2	3.1	0.26(39)	17.5	-	3.1	1.6	3.7

Note: Figures in parentheses show time lag, in seconds, before specimens.

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Iron films - velocity effect:

At a velocity of 5 ft./sec. films were formed that were very thick and of large particle size. This effect is possibly due to the fact that at lower flow rates, resulting in lower turbulence, it is necessary that particles grow larger before they are thrown out of the stream. Since the stagnant water film thickness is less at higher flow rates, it is possible that the large particles formed at 20 ft./sec. are brushed off by the moving water thus leaving only small particles in the film.

TABLE VII

Tests with Ferric Ion Added and Varying Water Velocities

Run No.	Fe ⁺⁺⁺ ppm	Velocity ft./sec.	Film Formation Mils/hr.	Increase in ΔP -%/hr.	Decrease in ΔT -%/hr.	Decrease in h_w -%/hr.
21-1	0.01	5	0.02 - 0.40	1.8	1.2	2.1
20-1	0.01	10	0.04 - 0.15	2.5	0.8	1.2
Ave.*	0.01	20	0.03 - 0.20	1.8	1.4	3.2

* Average of 6 runs (18-1, 26-2, 27-1, 27-2, 28-2, 29-2)

Iron films - heat flux effect:

Data on effect of heat-flux are incomplete. It is indicated, however, that film formation is slower under reduced heat flux for a given velocity. In one test under accelerated film forming conditions comparing a heat flux of 20 Kw/ft. with that of 35 Kw/ft., it was found that at the higher power level the rate of increase in pressure drop was approximately 3 times greater than that at 20 Kw/ft. This comparison is based on the overall length of tube with water temperature rises of 26 and 39°C at the lower and higher heat fluxes, respectively.

Iron films - demineralized versus raw water:

The rates of film formation in raw Chicago city water were studied, since with the exception of high chloride content, this water approximates in many respects that at "W" more closely than the demineralized water generally used. Raw water of 6.5 pH with peroxide adjusted to $2 \times 10^{-5} M$ one second before the test specimens produced no film or pressure drop increase. Addition of 0.05 ppm Fe⁺⁺⁺ to this water gave a hard, thin film with no increase in pressure

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drop. Films formed under similar conditions using demineralized water showed an increase in pressure drop over a like time interval. Results are given.

TABLE VIII

Tests Comparing Addition of Ferric Ion as $\text{Fe}_2(\text{SO}_4)_3$ to both City and Demineralized Water

Run No.	Fe^{+++} ppm	H_2SO_4 ppm	Film Formation mils/hr.	Increase in ΔP -%/hr.	Decrease in ΔT -%/hr.	Decrease in h_w -%/hr.
Ave.*	0.0	0.0	0.01 - 0.07	0.20	0.2	0.7
64-2	0.05	2.0	0.03 - 0.07	0.40	0.3	0.6
65-2	0.05	2.0	0.02 - 0.05	0.23	0.2	0.7
65-1**	0.0	0.0	0.00 - 0.03	0.04	0.1	0.41
65-2**	0.05	2.0	0.01 - 0.03	0.00	0.1	0.2

* Average of 12 runs with demineralized water containing peroxide adjusted to $2 \times 10^{-3} \text{N}$. runs are 12-1, 12-2, 13-1, 14-1, 15-1, 16-1, 17-1, 18-2, 30-2, 31-2, 32-2, and 33-2.

** Unfiltered, chlorinated, Chicago city water was employed, whereas the other runs used demineralized water.

Aluminum films - aluminum sulfate:

Because of the possible presence of Al^{+++} in "W" water from the use of filter alum as a flocculating agent, several tests were made to study the effect of Al^{+++} concentration in demineralized water and to compare film formation in city and demineralized water. Additions of 0.035 to 0.35 ppm Al^{+++} as the sulfate to demineralized water caused greater rates of pressure drop increase with increasing Al^{+++} concentrations. The aluminum films appeared to form mainly at the entering or cold end of the tube and were of a transparent gelatinous nature. A single test showed that for 0.1 ppm of Al^{+++} added to raw city water, the rate of pressure drop increase was only 10% of that obtained by adding the same concentration to demineralized water. Replacement of filter alum with ferrisul in the water treating plant at "W" caused Al^{+++} studies to become secondary to those on Fe^{+++} and Cr^{+++} , thus the Al^{+++} studies were stopped. All of the following runs

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were made at a pH of 6.5 with H_2O_2 adjusted to $2 \times 10^{-5} N$

TABLE IX

Tests Comparing Addition of Aluminum Ion as $Al_2(SO_4)_3$ to both
Demineralized and City Water

Run No.	Hrs. Oper.	Al^{+++} ppm	Film Formation Mils/hr.	Increase in ΔP -%/hr.	Decrease in ΔT -%/hr.	Decrease in h_w -%/hr.
66-1*	36.3	0.037	0.00 - 0.04	0.2	0.2	0.5
68-2*	55.3	0.050	0.00 - 0.05	0.4	0.1	0.4
68-1	8.8	0.064	0.00 - 0.09	1.4	0.2	0.8
67-2*	15.6	0.10	0.00 - 0.07	1.5	0.0	0.3
66-2*	14.7	0.32	0.00 - 0.38	2.4	0.5	1.4
69-2**	62.0	0.10	0.01 - 0.02	0.2	0.2	0.4

* Runs with 35 ppm SO_4^{--} added as Na_2SO_4 .
** Raw city water test.

Chromic films:

In iron film studies researchers from other laboratories had reported that silica from sodium silicate would prevent the deposition of a film. Because of the abrasive action of the silicate, however, addition of an inhibitor, such as $Na_2Cr_2O_7 \cdot 2H_2O$ was found necessary by CMX.

In April, 1944, preliminary tests, to determine the effect of possible reduction products of dichromate under radiation, were made with 0.03 and 0.35 ppm Cr^{+++} added. At the higher concentration a green film was obtained, while at the lower concentration the film was a dull brown. When other laboratory workers showed that dichromate underwent a reduction to chromic ion in the presence of pile radiation, film studies on Cr^{+++} assumed a role of major importance. Tests were made to evaluate the effects of pH, $Na_2Cr_2O_7 \cdot 2H_2O$, H_2O_2 , SO_4^{--} , Fe^{+++} and chemical reducing agents on the formation of chromic films.

The rate of formation of the chromic film caused by 0.35 ppm Cr^{+++} was identical in 6.5 pH demineralized water with or without the addition of 35 ppm SO_4^{--} . Addition of 2 ppm $Na_2Cr_2O_7 \cdot 2H_2O$ to demineralized water containing

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0.35 ppm Cr^{+++} had no retarding effect on the formation of the film. The results of these tests were as follows:

TABLE X

Tests Studying the Effect of Dichromate and Sulfate
on Chromic Ion Added as $\text{Cr}_2(\text{SO}_4)_3$

Run No.	Hrs. Oper.	Cr^{+++} ppm	$\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$ ppm	SO_4^{--} ppm	Increase in ΔP -%/hr.	Decrease in ΔT -%/hr.	Decrease in h_w -%/hr.
54-1	24.9	0.03	0.0	0.0	0.4	0.4	1.2
(54-2 (11.7 (72-1 (24.9 (73-2		0.35	0.0	0.0	1.5	0.9	2.8
74-2	26.5	0.35	0.0	35.0	1.0	0.9	2.1
72-2	9.0	0.35	2.0	0.0	2.1	0.7	3.2
(77-2 (8.5 (93-2 (25.7 (94-2		0.14	2.0	35.0	1.4	1.8	4.0
(74-1 26.5 (76-2		0.35	2.0	35.0	1.1	0.8	2.0

All tests made in 6.5 pH demineralized water with H_2O_2 adjusted to $2 \times 10^{-5} \text{N}$.

A run at $1 \times 10^{-3} \text{N}$ H_2O_2 with 6 ppm $\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$ gave a rate of pressure drop increase similar to that obtained when no additions other than peroxide were made. Runs made with additions of 2 ppm $\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$ gave an average rate of pressure drop increase similar to that found in tests with 0.03 ppm Cr^{+++} . These runs show that peroxide does not convert the dichromate to a film forming stage, at least under the test conditions. Results are given on the following page.

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TABLE XI

Tests Studying the Effect of Dichromate, Peroxide and Sulfate
on Film Formation in 6.5 pH Demineralized Water

Run No.	Hrs. Oper.	$\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$ ppm	H_2O_2 $\text{Nx}10^5$	SO_4^{--}	Increase in ΔP -%/hr.	Decrease in ΔT -%/hr.	Decrease in h_w -%/hr.
(55-1 (55-2	38.4	6.0	100	0.0	0.05	0.02	0.10
(86-1 (86-2 (87-1 (87-2	9.1-38.0	2.0	2.0	33.0	0.5	0.6	1.8
(88-1 (88-2 (89-1 (89-2							
(91-2 (92-1 (85-2	11.3-41.5	2.0	0.0	35.0	1.1	0.7	1.9

For similar pH and reagent conditions, the rate of chromic film formation was lower in raw Chicago city water than in demineralized water. The film produced when 0.35 ppm Cr^{+++} was added to raw water was a more brilliant brown in color and gave only 30% of the pressure drop increase obtained when a similar concentration was added to demineralized water. The tests are reported in the following table:

TABLE XII

Tests Comparing Addition of Chromic Ion as
 $\text{Cr}_2(\text{SO}_4)_3$ to both City and Demineralized Water

Run No.	Hrs. Oper.	Cr^{+3} ppm	Increase in ΔP -%/hr.	Decrease in ΔT -%/hr.	Decrease in h_w -%/hr.
70-1	12.5	0.34	0.4	0.6	2.0
71-2	51.6	0.66	0.5	0.6	1.2
65-1	38.3	0.0	0.04	0.1	0.4
(54-2 *(72-1 (73-2	11.7-24.9	0.35	1.5	0.9	2.8

* Average of 3 demineralized water runs. The other runs are with raw city water. All these runs were made at pH of 6.5 with H_2O_2 adjusted to $2 \times 10^{-5} \text{N}$.

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Chromic films - pH effect:

With an addition of 0.14 ppm Cr^{+++} to 4.6 pH demineralized water containing $\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$ and 35 ppm SO_4^{--} , the rate of pressure drop was only 10 to 20% of that obtained at a pH of 5.3 and 6.4. Water at a pH of 7.2-7.7 containing added Cr^{+++} , dichromate, peroxide and sulfate gave a fast rate of film buildup similar to that at a pH of 6.4. Because of the variable nature of the iron pickup in the lines to the specimens under the low pH conditions, the above results are to be interpreted only as general conclusions. Film analyses showed the iron content of the films to increase from 8.8 to 26.0% as the pH was varied from 6.5 to 4.5 (see Table XIII in Appendix). For the same pH range the chromium content decreased from 20.0 to 12.7%. No pitting or weight loss was observed upon examination of the aluminum specimens exposed to the low pH water containing dichromate, but extended exposure tests were not made. The following table contains results on these tests:

TABLE XIV

Tests Studying the Effect of pH on Chromic Ion Added as $\text{Cr}_2(\text{SO}_4)_3$

Run No.	Hrs. Oper.	pH	Cr^{+++} ppm	Increase in ΔP -%/hr.	Decrease in ΔT -%/hr.	Decrease in h_w -%/hr.
(97-1						
(97-2	(9.5					
(98-1	(40.2	7.5	0.13	1.3	1.3	2.8
(98-2						
(113-2						
110-1	50.2	7.5	0.073	0.7	0.8	1.5
110-2	50.2	7.5	0.03	0.5	0.6	1.2
77-2	25.7	6.4	0.15	0.7	1.5	2.6
119-2*	30.7	6.4	0.14	0.5	0.8	1.9
77-1	25.7	5.3	0.14	0.7	0.8	1.8
78-1	72.2	4.6	0.13	0.1	0.2	0.5

Runs made in demineralized water with H_2O_2 adjusted to $2 \times 10^{-5} \text{M}$.

* Ionic Cr^{+++} solutions were insured by use of H_2SO_4 .

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In the runs reported on the previous page at a pH of 4.5 the chromium predominated in the film; a single test with Fe^{+++} and no Cr^{+++} added gave no pressure drop increase at the low pH. The rate of pressure drop increase in runs with both chromic and ferric added ions was greater than with chromic alone, thus indicating that the iron was being carried down with the chromium. Under similar water conditions an increase in the time lag before the specimen for either Cr^{+++} or Fe^{+++} reagent also caused an increase in pressure drop when the reagents were being added either singularly or together. A comparison of tests with iron and chromic films at a pH of 4.5 and 7.8 showed that the pressure drop increase was 50% greater at the lower pH. The results are shown in the following table:

TABLE XV

Tests Studying the Effect of Added $\text{Fe}_2(\text{SO}_4)_3$ at pH 4.5 and 7.8
on Chromic Ion Added from $\text{Cr}_2(\text{SO}_4)_3$

Run No.	pH	Cr^{+++} ppm	Fe^{+++} ppm	Hrs. Oper.	Increase in ΔP -%/hr.	Decrease in ΔT -%/hr.	Decrease in h_w -%/hr.
79-2	4.6	0.0	0.0	54.0	0.1	0.2	0.5
78-1	4.6	0.12(2)	0.0	72.2	0.1	0.2	0.5
(80-1 (82-1 (83-1	4.5	0.0	0.05(35)	5.3-6.6	0.8	0.7	2.1
81-1	4.5	0.0	0.05(2)	7.2	0.0	0.0	0.0
81-2	4.6	0.14(39)	0.05(2)	7.2	2.1	1.1	7.2
84-1	4.6	0.13(2)	0.05(2)	25.3	1.3	1.2	2.5
(80-2 (82-2 (83-2	4.5	(2) 0.14(2) (2)	0.05(39)	5.5-6.6	2.8	1.3	4.5
(101-1 (101-2	7.9	0.13(2) (2)	0.05(39)	37.5	1.2	1.2	2.1
(102-1 (102-2	7.8	0.08(2) (2)	0.05(39)	33.0	1.4	1.1	2.2

Numbers in parentheses indicate time lag of reagents before specimens (seconds).
Runs were made in demineralized water with additions of 35 ppm SO_4^{--} and 2.0 ppm $\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$ and had the H_2O_2 adjusted to $2 \times 10^{-5} \text{N}$.

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Simulation of reduction of dichromate by radiation in the pile was attempted through addition of 14 ppm elon* to 6.5 pH demineralized water having added dichromate and sulfate. Samples withdrawn at the outlet of the tubes, or 2.5 seconds after elon addition, indicated a substantial reduction of dichromate. The rate of pressure drop increase was about the same with or without elon addition. This indicated that the time of contact in the tube was either insufficient for Cr^{+++} to deposit as a film under the run conditions or the reduction of dichromate was not accurately indicated by the analyses (colorimetric using diphenyl carbazide). Analyses of the chromic films formed when elon was added gave 13% Cr at the cold end and 16% at the hot end of the test specimen. The film in the control run, no elon added, had 5% and 6% Cr, respectively, at the cold and hot ends of the tube. The results of these tests were as follows:

TABLE XVI

Tests Studying the Effect of Forming Chromic Ion in the
Annulus from Reduction of Dichromate with Elon*

Run No.	Hrs. Oper.	Elon ppm	Increase in ΔP -%/hr.	Decrease in ΔT -%/hr.	Decrease in h_w -%/hr.
85-2	11.3	0.0	2.1	0.7	2.7
85-1	11.3	7.0	2.0	1.1	3.3
91-2	29.9	0.0	0.8	0.9	1.9
91-1	29.9	13.2	0.5	0.7	1.6
92-1	41.5	0.0	0.4	0.4	1.1
92-2	41.5	13.4	0.5	0.6	1.2

Runs were made in 6.5 pH demineralized water with additions of 2.0 ppm $\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$ and 35 ppm SO_4^{--} , and without peroxide being added. *Elon, also known as metol, a chemical reducing agent (para methyl amino phenol sulfate).

Since the treated water at "W", after being flocculated and filtered contains ca 18 ppm of Ca and 5 ppm of Mg, tests were made with Ca^{++} and Mg^{++} to evaluate the effect of run duration and oxalic acid on these ions at low pH and the effect of silica on deposition of these ions at high pH.

Calcium and magnesium films:

During attempted removal of iron films with oxalic acid CMX had encountered a film of calcium oxalate, calcium silicate and calcium sulfate when the pH exceeded 3.5. At M1 the specimens treated with 3.5 pH demineralized water having

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additions of 35 ppm SO_4^{--} , 2 ppm $\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$, 18 ppm Ca^{++} , 50 ppm oxalic acid and H_2O_2 adjusted to $2 \times 10^{-5}\text{N}$ had no film and the tests showed no decrease in heat transfer after exposures of 10 and 17 hours. Appreciable pitting corrosion was found along the tubes.

The above additions in a pH range of 4.3 to 4.9 (run 105-2) produced a very rapid film build-up as shown in Figure 6. Lowering of the pH to 4.1 or below caused some reduction in pressure drop probably because the calcium carbonate in the film being more soluble than the calcium oxalate tended to dissolve. Examination of the specimens disclosed the film to be a white granular substance. The films analyzed 70 to 80% calcium oxalate. Insufficient sample prevented analysis for CO_2 ; however, a study of the available amount of Ca showed that it was principally tied up as the oxalate.

As a further investigation of film formation caused by the presence of Ca^{++} and Mg^{++} in the water, tests were made with 0 up to 40 ppm of silicate added as sodium silicate to 8.5 pH demineralized water containing additions of dichromate, sulfate and peroxide. In runs (106, 107, 114) with 18 ppm Ca^{++} and no Mg^{++} added no pressure drop increase occurred when the pH remained at 8.5 to 8.7; however, when the pH rose above 9.0, an immediate rapid rate of pressure drop increase took place even where no silicate was added. The white brittle scale formed on the tubes analyzed ca 40% calcium. The low content of silicon in the film lead indicates that the film was principally calcium carbonate. See Table XVII for the film analyses and Figures 7 and 8 for the effect of pH on pressure drop.

A further study of the effect of high pH on deposition of a combined Ca and Mg film was made in the above water containing both 18 ppm Ca^{++} and 5.8 ppm Mg^{++} (run 111). A study of Figures 9 and 10 showed that no pressure drop increase was observed in the pH range of 8.2-8.7 with or without silicate; however, increasing the pH to 8.9-9.3 immediately caused a rapid pressure drop rise in the run without silicate while no change in pressure drop was observed with added silicate. The white, granular, brittle scale from the run with no silica was quite thick and partially discolored with iron. Analyses of the film can be found in Table XVII. At a pH above 9.5 a fast pressure drop increase took place with silica. A similar film somewhat whiter in color was found on the latter specimen, thus indicating that the silicate reduced the deposition of iron. It would seem that the addition of 40 ppm silicate prevents deposition of a Ca and Mg film below a pH of 9.3. These tests are tabulated in the table on the following page.

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TABLE XVIII

Tests Studying the Effect of Silica on Calcium and
Magnesium Films at High pH

Run No.	Hrs. Oper.	pH	Ca ⁺⁺ ppm	Mg ⁺⁺ ppm	SiO ₂ ppm	Increase in ΔP -%/hr.	Decrease in ΔT -%/hr.	Decrease in h_w -%/hr.
107-2	11.1	8.2-8.7	13.9	0.0	0.0	1.1	1.7	-
107-2A	2.3	9.2-9.4	15.9	0.0	0.0	9.4	3.2	-
114-2	5.0	9.0-9.7	18.6	0.0	0.0	24.1	4.4	11.8
106-2	14.5	8.0-9.0	17.5	0.0	9.9	0.0	0.4	1.1
106-2A	5.0	8.6-9.3	17.5	0.0	9.9	8.6	0.4	2.0
106-1	14.5	8.0-9.0	17.5	0.0	20.7	0.2	0.4	1.5
106-1A	5.0	8.6-9.3	17.5	0.0	20.7	7.7	0.9	3.3
107-1	11.1	8.3-8.7	18.4	0.0	38.6	0.3	0.2	-
107-1A	2.6	9.3-9.6	18.4	0.0	38.6	5.0	2.6	-
114-1	10.0	9.1-10.2	18.5	0.0	41.0	11.0	1.8	5.3
112-2	26.0	9.0-10.5	0.0	5.9	0.0	1.0	1.7	3.0
112-1	26.0	9.9-10.5	0.0	5.9	35.0	0.9	0.9	2.2
111-2A	4.0	8.5-9.3	15.7	5.1	0.0	12.5	3.3	9.7
111-2	10.0	7.7-8.6	15.7	5.1	0.0	0.0	0.4	1.5
111-1A	4.3	9.5-10.1	16.6	5.2	39.0	7.7	4.4	9.6
111-1	26.2	8.0-9.3	16.6	5.2	39.0	0.0	0.3	1.1

Runs were made with demineralized water containing $2 \times 10^{-5} N$ H₂O₂, 2 ppm Na₂Cr₂O₇ · 2H₂O and 35 ppm SO₄²⁻.

During part A of the above runs the pH was purposely increased to the indicated range.

Figures were not calculated for those runs in which a dash appears.

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With the addition of 5 ppm Mg^{++} to the above water and no Ca^{++} added (Run 112) no pressure drop increase was found in the pH range of 7.6 to 9.0 either with or without silicate; however, purposely increasing the pH to 9.2-9.9 caused pressure drop increases of 2.0%/hr. in 19 hours of test without silicate and 1.1%/hr. in 15 hours with 40 ppm silicate. Analyses of the white films found on both specimens (Table XVII) showed the film to be magnesium silicate and/or magnesium carbonate. There was no indication of a rapid pressure drop increase as had been experienced at 9.5 pH in previous tests having additions of Ca^{++} or Ca^{++} and Mg^{++} to the water. Figures 11 and 12 showed the effect of pH on pressure drop.

Iron and chromium films - removal:

Interspersed with the runs on film formation were tests to determine means of removal of films which had already been deposited on the specimens. The main work on removal of iron films was carried out at other sites. Results from two tests, in which 50 ppm oxalic acid at 3.5 pH were used to remove an iron and an iron and chromium film deposited from 4.5 pH demineralized water containing 0.14 ppm Cr^{+++} and 0.05 ppm Fe^{+++} and 2.0 ppm H_2SO_4 in addition to the regular reagents, showed that the pressure drop was restored to its initial value only in the case of the run with no trivalent chromium. The pressure drop in the other case was lowered only 1/3 of the rise caused by the film. Results during both the film formation and removal periods are shown in the following table:

TABLE XIX

Tests Studying the Effectiveness of Oxalic Acid in Removal of
Iron and Iron and Chromic Films

Run No.	pH	Hrs. Oper.	Cr^{+++} ppm	Fe^{+++} ppm	$C_2H_2O_4 \cdot 2H_2O$ ppm	Increase in ΔP -%/hr.	Decrease in ΔT -%/hr.	Decrease in h_w -%/hr.
83-1	4.3	6.5	0.0	0.06	0.0	1.0	1.2	3.7
83-1A	4.0	4.2	0.0	0.0	43.0	1.0*	0.0	0.0
83-2	4.3	6.5	0.14	0.05	0.0	3.5	1.7	6.1
83-2A	3.8	4.2	0.0	0.0	42.0	1.1*	0.4**	1.7**

Runs were made with demineralized water containing 35 ppm SO_4^{--} , 2.0 ppm $Na_2Cr_2O_7 \cdot 2H_2O$ and with H_2O_2 adjusted to $2 \times 10^{-5} N$.

The part A of each run was that portion of the test in which film removal treatment was being carried out.

* Decrease.

** Increase.

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Because of the tenacity of the chromic films, as shown by the fact that protectors were not completely effective in preventing film deposition, particular stress at ML was placed on finding a means for removal of these films. Studies of the effectiveness of $(\text{NH}_4)_2\text{S}_2\text{O}_8$ (Run 97) and alkaline H_2O_2 (Runs 98, 101 and 102) in removal of Cr^{+++} films and of oxalic acid (Runs 85 and 86) in removal of $\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$ films were made.

In all instances the chromic films were deposited from 7.5 pH demineralized water (non-added iron 0.10-0.20 ppm) containing 0.14 ppm Cr^{+++} and the usual additions of dichromate, sulfate and peroxide. Additions of 2 ppm $(\text{NH}_4)_2\text{S}_2\text{O}_8$ at 7.7 pH were not effective in removing the deposited chromic film. Increasing the $(\text{NH}_4)_2\text{S}_2\text{O}_8$ additions to 10 ppm caused an increase rather than a decrease in pressure drop.

Six deposited chromic films were removed by water at a pH of 7.5 having additions of 40 to 190 ppm H_2O_2 . The removal treatments were of 6 to 7 hours duration. Evaluation of the data from tests for determining optimum quantities of peroxide and optimum time of treatment in removal of chromic films was made. Examination of the film remaining on the specimen after exposure to water having 39.5 and 91 ppm H_2O_2 showed that the greenish brown color persisted in the film, thus indicating that traces of Cr^{+++} still remained. After treatment with 131 and 152 ppm H_2O_2 , the film was reddish brown, thus indicating that all or most of the Cr^{+++} had been removed. All the specimens were bare of any film at the hot or outlet end. This characteristic was common to all concentrations of peroxide used for removal. (Table XX).

From the 6 runs with peroxide additions in the range of 40 to 190 ppm, a treatment period of 2 to 3 hours appeared to be the optimum since after this time the pressure drop returned to its initial value at a much slower rate. Within the ability to duplicate results in laying down the films for subsequent removal tests, the data showed no trend toward increased rate of removal over the range of 40 to 190 ppm H_2O_2 .

Results of these tests have been tabulated in Table XXI (appendix) and in the table on the following page.

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TABLE XXII

Tests Determining the Optimum Amounts of H_2O_2
Required for Removal of Chromic Films

Run No.	Hrs. Oper.	H_2O_2 ppm	Increase in ΔP -%/hr.	Decrease in ΔT -%/hr.	Decrease in h_w -%/hr.
101-1	37.5	0.0	1.0	1.2	2.1
101-1A	6.3	39.5	2.2*	5.9**	15.4**
102-1	33.0	0.0	1.5	1.1	2.2
102-1A	7.1	91	2.9*	3.9**	10.8**
98-2	9.5	0.0	2.0	1.9	4.9
98-2A	7.5	102	3.1*	2.3**	8.7**
101-2	37.5	0.0	1.3	1.2	2.1
101-2A	6.3	131	2.5*	8.3**	25.0**
102-2	33.0	0.0	1.3	1.2	2.2
102-2A	7.1	152	3.1*	7.5**	9.2**
98-1	40.2	0.0	0.8	0.8	1.8
98-1A	6.0	189	2.0*	4.3**	11.7**

* Decrease.

** Increase.

Part A is during the removal period.

Runs were made with 7.5 pH demineralized water containing sulfate and dichromate.

Removal of films formed by dichromate and the usual additions to 6.5 pH demineralized water with 50 ppm oxalic acid at a 3.5 pH required a longer period of treatment, 9 hours, than indicated by other investigators to bring the pressure drop back to within one pound of its original value. The h_w

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was restored to ca 80% of its original value. The results of these tests are listed in the following table:

TABLE XXIII

Tests Studying the Effectiveness of Oxalic Acid on
Removal of Films Deposited by Dichromate

Run No.	Hrs. Oper.	pH	$\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$ ppm	$\text{C}_2\text{H}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$ ppm	Increase in ΔP -%/hr.	Decrease in ΔT -%/hr.	Decrease in h_w -%/hr.
85-2	11.2	6.6	1.9	0.0	2.1	0.7	2.7
85-2A	6.7	4.8	1.1	55.4	1.9*	0.3**	2.0**
86-1	21.5	6.4	1.8(43)	0.0	0.4	0.5	1.7
86-1A	9.1	3.4	1.9	53.6	1.2*	1.7**	1.6**
86-2	21.5	6.4	1.8(2)	0.0	0.5	0.7	2.0
86-2A	9.1	3.5	1.9	47.2	1.4*	1.5**	5.0**

Parentheses indicate seconds time lag before the specimens.

* Decrease.

** Increase.

Part A is during removal period.

Runs were made with demineralized water containing sulfate, dichromate and peroxide.

Aluminum films - removal:

Studies on the removal of aluminum films were put into the background since again the use of Ferrisul in place of filter alum for water treatment at "W" assured that only on abnormal occasions, would the content of process water reach 0.03 ppm Al. According to conversations with J.P. Howe in August, 1944, substantiated by our work, it would be necessary to consider the Al film problem at this concentration. (MUC - J. P. Howe 268, Treatment of Cooling Water at "W" for 105-B. Contains estimate as to probable quantities of Al, Fe and Cr to be found in plant water).

Iron and chromium films - prevention of their formation:

In the discussion of preceding runs we have dealt with problems of film formation and film removal. The third phase of the film work by the Engineering Development Section concerned the study of means of preventing the deposition of Cr^{+++} , Cr^{+++} and iron, dichromate and calcium films by use of suitable protectors.

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The protectors tested had been recommended for their effectiveness by the Corrosion Group of the Metallurgy Section after evaluation in their stirred beaker experiments. SiO_2 added as sodium silicate; ammonium persulfate, $(\text{NH}_4)_2\text{S}_2\text{O}_8$; disodium dihydrogen pyrophosphate, $\text{Na}_2\text{H}_2\text{P}_2\text{O}_7$; meadol, an alkali lignin; and calgon were the protectors investigated. Application of electrical potentials was also used in attempt to prevent formation of iron films.

Silicate was tried as an inhibitor for Cr^{+++} and for Cr^{+++} and Fe^{+++} films. Additions of SiO_2 in the range of 5 to 45 ppm did not prove 100% effective in preventing deposition of a film from 6.5 pH water containing 0.35 ppm added Cr^{+++} , but the silicate did cause a reduction in the rate of pressure drop increase. The addition of 15 ppm SiO_2 to 6.5 pH water decreased the chromium and iron content of the film to 10.6% and 5.8%, respectively. The film from the control run, containing no silica, analyzed 20.0% Cr and 8.8% Fe. The rate of film formation at a pH of 4.7 from demineralized water containing added Cr^{+++} and Fe^{+++} was about the same with or without the addition of silicate. Film analyses (Table XIII, appendix) indicated that silicate had caused a decrease in the percentage of both iron and chromium in the film, thus showing some protective action. The results of these tests are reported in the following table:

TABLE XXIV

Tests Studying the Protective Power of Silicate in Presence
of Chromic and Ferric Ions

Run No.	Hrs. Oper.	pH	Cr^{+++} ppm	Fe^{+++} ppm	SiO_2 ppm	Increase in ΔP -%/hr.	Decrease in ΔT -%/hr.	Decrease in h_w -%/hr.
75-1	28.9	6.5	0.35	0.0	5	0.4	0.3	1.1
75-2	28.9	6.5	0.37	0.0	15	0.6	0.3	1.1
76-1	26.4	6.5	0.35	0.0	48	0.2	0.4	1.2
76-2	26.4	6.4	0.36	0.0	0.0	0.8	0.6	1.7
84-1	25.3	4.6	0.13	0.05	0.0	1.3	1.2	2.5
84-2	25.3	4.7	0.14	0.05	13.1	1.1	1.0	2.2

Runs were made with demineralized water having additions of sulfate, dichromate and H_2O_2 .

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Use of meadol, to inhibit film formation, was tried in 6.5 pH water having added dichromate and sulfate and 0.14 ppm added Cr^{+++} . Addition of 3 ppm meadol caused a higher rate of pressure drop increase than was found in the control run, with no meadol; whereas, 10 ppm meadol gave a lower rate of pressure drop increase than in the control run. The meadol reduced the film thickness in each case, but the thinner film did not result in as much of a decrease of pressure loss as would be expected, probably, because the compact film offered more resistance to flow. Films formed in the runs with added meadol indicated by analysis that the alkali lignin was preventing, in part, the deposition of iron and chromium. With meadol added, iron and chromium in the film analyzed 7% and 6%, respectively, and without meadol the corresponding figures were 11% and 17%. The results of these tests are tabulated:

TABLE XXV

Tests Studying Additions of Meadol to Prevent Deposition of
Chromic Films

Run No.	Hrs. Oper.	Cr^{+++} ppm	Meadol ppm	Increase in ΔP -%/hr.	Decrease in ΔT -%/hr.	Decrease in h_w -%/hr.
93-1	12.2	0.12	2.7	2.1	1.8	4.6
93-2	12.2	0.13	0.0	1.3	2.1	4.7
94-1	8.5	0.13	10.1	1.1	0.5	2.9
94-2	8.5	0.13	0.0	2.1	1.7	4.8

Runs were made with 6.5 demineralized water containing sulfate and dichromate but without peroxide.

Meadol - An alkali lignin by-product from the sulfate paper industry (Lignin MRM, Mead Corp., Ohio).

A series of tests with and without the protective action of $\text{Na}_2\text{H}_2\text{P}_2\text{O}_7$ for preventing deposition of chromic ion and iron films was completed. A study of the chromic-iron films deposited in the internally heated equipment showed that the iron submitted more readily to protection than did Cr^{+++} . With a protector to iron-chromic ion ratio of 20 to 1 (Run 108) and with ratios of iron to Cr^{+++} in the water of 2 to 1 and 6 to 1, the film analyses revealed iron-chromium ratios of 0.6 to 1 and 1 to 1, respectively. Reducing the protector ratio to 5 to 1 (Run 109) and using water containing iron-chromic ratios of 2 to 1 and 7 to 1, the iron-chromium ratios in the films were 0.4 to 1 and 0.8 to 1, respectively.

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The control run (run 110) showed that when $\text{Na}_2\text{H}_2\text{P}_2\text{O}_7$ was absent, iron and Cr^{+++} would deposit in the film in about the same ratios to each other as they were in the water. This confirmed the belief expressed in runs 108 and 109 concerning the fact that iron was protected more by $\text{Na}_2\text{H}_2\text{P}_2\text{O}_7$ than was Cr^{+++} . Pressure drop and h_w data showed that both 20 to 1 and 5 to 1 protector to metallic ion ratios were beneficial in prohibiting film formation with the 20 to 1 ratio being decidedly better. The following table contains the results of these runs:

TABLE XXVI

Tests Studying the Use of $\text{Na}_2\text{H}_2\text{P}_2\text{O}_7$ for Prevention of
Chromic and Iron Films

Run No.	Cr^{+++} ppm	$\text{Na}_2\text{H}_2\text{P}_2\text{O}_7$ ppm	$\text{Fe}/\text{Cr}^{+++}$ in Bulk H_2O	$\text{Na}_2\text{H}_2\text{P}_2\text{O}_7/\text{Fe}+\text{Cr}^{+++}$ in Film	Fe/Cr	Increase in ΔP -%/hr.	Decrease in ΔT -%/hr.	Decrease in h_w -%/hr.
108-1	0.07	4.5	2	21	0.6	0.2	0.1	0.5
108-2	0.03	3.6	6	16	1.0	0.4	0.1	0.4
109-1	0.074	1.0	2	5	0.4	0.2	0.2	0.8
109-2	0.028	0.85	7	4	0.8	0.2	0.1	0.6
110-1	0.073	0.0	1.1	0	1.1	0.7	0.8	1.5
110-2	0.03	0.0	4	0	2.0	0.5	0.6	1.2

Time of tests 49 to 55 hours. Runs were made with 7.5 pH demineralized water containing sulfate, dichromate and H_2O_2 .

From preliminary results by other researchers, CMX indicated interest in the use of $(\text{NH}_4)_2\text{S}_2\text{O}_8$ to minimize reduction of dichromate to chromic ion under conditions of pile radiation. Tests resulting from this interest included studies to determine if ammonium persulfate would have any adverse effects on film formation in the presence of SiO_2 , Cr^{+++} or Fe^{++} . The use of $(\text{NH}_4)_2\text{S}_2\text{O}_8$ did not prevent chromic film formation in a "W" annulus, but it did not have any adverse effects with silicate or dichromate. In water containing 0.2 to 0.3 ppm iron the persulfate increased the rate of iron film formation. It may be concluded that persulfate had essentially no effect on Cr^{+++} film formation when the iron was protected by silicate. The studies with chromic ion were made in part with 0.14 ppm Cr^{+++} added from a $\text{Cr}_2(\text{SO}_4)_3$ solution maintained by H_2SO_4 below a pH of 2 to insure that the chromium would be added in the

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ionic rather than colloidal form. Tabulation of these runs follow:

TABLE XXVII

Tests Studying the Use of $(\text{NH}_4)_2\text{S}_2\text{O}_8$ for Prevention of

Chromic Films

Run No.	Hrs. Oper.	pH	$(\text{NH}_4)_2\text{S}_2\text{O}_8$ ppm	Cr^{+++} ppm	Increase in ΔP -%/hr.	Decrease in ΔT -%/hr.	Decrease in h_g -%/hr.
113-2	32.3	7.5	0	0.14	0.8	1.1	2.3
113-1	32.3	7.5	1.9	0.14	1.1	1.1	2.1
115-1	28.9	7.5	4.6	0.14	1.5	1.1	2.4
115-2	28.9	7.5	9.5	0.14	1.3	1.5	2.7
116-1	55.3	6.5	5.5	0.0	0.3	0.2	0.6
116-2	55.3	6.5	0.0	0.0	0.2	0.2	0.8
117-1**	50.5	6.5	4.6	0.0	0.3	0.3	1.0
117-2**	50.5	6.5	0.0	0.0	0.3	0.2	0.9
119-1	30.7	6.4	4.9	0.13*	1.2	1.2	2.4
119-2	30.7	6.4	0.0	0.14*	0.5	0.8	1.9
77-2	25.7	6.5	0.0	0.15	0.7	1.5	2.6
121-1'	14.8	6.4	5.6	0.16*	0.2	1.1	3.2
121-2'	14.8	6.5	0.0	0.15*	0.2	0.6	2.2
122-1'	31.9	6	4.6	0.14*	0.1	0.8	2.0
122-2'	31.9	6.5	0.0	0.14*	0.1	0.6	1.6

* $\text{Cr}_2(\text{SO}_4)_3$ solution made up with H_2SO_4 so that chromium would be in ionic rather than colloidal form.

** No dichromate added to either section.

Runs were made with demineralized water containing sulfate, dichromate and peroxide.

* 30 ppm SiO_2 were added to these runs.

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The use of calgon was studied as a prohibitor of calcium film formation during iron film removal by oxalic acid. Both 20 and 50 ppm calgon were found to be 100% effective in preventing deposition of a calcium film removal at a pH of 4.2 to 4.9 in the presence of 18 ppm Ca^{++} , 50 ppm oxalic acid, 2 ppm $\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$ and with H_2O_2 adjusted to $2 \times 10^{-5}\text{M}$. However, iron film removal at this pH was very slow as less than 10% of the pressure drop increase was recovered in 9 hours treatment. Results are tabulated as follows:

TABLE XXVIII

Tests Studying the Use of Calgon in Prevention of
Calcium Oxalate Films

Run No.	Hrs. Oper.	Ca^{++} ppm	$\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$ ppm	Calgon ppm	Increase in ΔP -%/hr.	Decrease in ΔT -%/hr.	Decrease in h_w -%/hr.
124-1	21.0	15	48	44	0.0	0.1	0.4
124-2	21.0	15	44	18	0.1	0.1	0.6
125-1	30.3	0.0	0.0	0.0	1.4	1.1	2.3
125-1A	9.8	9.8	50	18	0.3*	2.0**	4.2**
125-2	30.3	0.0	0.0	0.0	0.9	1.2	2.3
125-2A	9.8	9.8	43	50	0.3*	1.0**	1.6**

Runs were made with demineralized water at pH 4.5 (except for 125-1 and 125-2 - film formation occurring at 6.5) containing sulfate, dichromate and peroxide. Parts A are during the film removal periods.

* Decrease.

** Increase.

Iron films - prevention by application of electrical potentials:

Attempt was made to determine the charge of the iron colloid by application of a potential across the annulus. This would permit studies of prevention of film formation by giving the specimen and ribbed tube a charge opposite to that found on the colloid. The apparatus used was full scale equipment as previously described in this report. Tests were run with applied potentials as high as 3 volts. At this potential electrolytic corrosion began to be severe and no higher voltages were tried. Changing of the polarity or potential did not make any difference in film deposition. The results of these cataphoretic tests indicate the possibility that the mechanism of the formation of these films involves the agglomeration of the colloid in the bulk of the water with subsequent deposition on the specimen and ribbed tube surfaces regardless of the charge on these surfaces. This possibility is substantiated in part by the fact that additions of electrolytes promote deposition of the film and that larger particles are produced at lower velocities.

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Conclusions:

Film formation:

It must be realized in drawing conclusions from the data and results presented herein that the water used in these tests differs appreciably from that used in tests at CMX and that there are many variables involved in these tests, some of which are unknown and others, though known, cannot be controlled. The latter include iron pickup in the pipe lines, turbidity in the water and variations in time lag for the added reagents. It is believed, however, that the general principles and variables involved in film formation studies here were used to advantage in the studies at CMX. Results from single tests were considered of value in indicating trend only.

On the basis of the preliminary tests in which the water was preheated prior to the specimens, it was concluded that in such a system film formation is dependent upon the time required for the colloidal particles to increase to a size at which they will plate out. The deposition of the film is a cyclic process depending upon iron pickup, time for particle size growth, point of application of heat and size of particles required for deposition.

Film formation in 6.5 pH demineralized water containing only added peroxide and a small amount of iron pickup from the pipes occurred at a very slow rate. Additions of Fe^{+++} added simultaneously with 2 ppm H_2SO_4 increased the rate of film formation. Additions of Fe^{+++} added simultaneously with chloride ion as sodium chloride increased the rate of film formation, but the effect was less than with sulfate. Ferrous additions gave pressure drop increases much lower than those with Fe^{+++} . These tests indicate that Fe^{+++} is the dangerous form of iron as regards film deposition and that oxidation of Fe^{++} to Fe^{+++} must occur before formation of a film.

Single run studies of the effects of various anions upon rate of pressure drop increase showed that the $\text{SO}_4^{=}$ and NO_3^- gave rates 2 to 3 times greater than those produced by Cl^- , $\text{Fe}(\text{CN})_6^{=}$ and $\text{Cr}_2\text{O}_7^{=}$ for similar equivalents of the anions.

Based on single runs at low velocities the rate of iron film formation is faster and the films are thicker than those formed at high velocities. The coarse films formed at low flows do not decrease the heat transfer coefficients as much as films of corresponding thickness formed under high velocities.

Preliminary tests at constant velocity indicate that iron film formation is slower under reduced heat flux.

Single tests showed that the rate of pressure drop increase was less for Fe^{+++} , Cr^{+++} and Al^{+++} films in raw city water than in demineralized water.

Additions of 0.035 to 0.35 ppm Al^{+++} as the sulfate to demineralized water caused greater rates of pressure drop increase with increasing Al^{+++} concentrations. The aluminum films appeared mainly at the entering or cold end of the tube.

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Sulfate ion has no effect on chromic film formation in 6.5 pH demineralized water. Dichromate has no retarding effect on formation of Cr^{+++} films. The thickness of Cr^{+++} and Fe^{+++} films increased toward the outlet or higher temperature end of the tube.

The rate of pressure drop from chromic films deposited from 4.6 pH demineralized water was only 10 to 20% of that obtained at pH of 5.3, 6.4 and 7.5. In runs at low pH (4.6) the rate of film formation with both ferric and chromic added ions was greater than with either chromic or ferric alone. Tests with iron and chromic films at pH of 4.5 and 7.8 showed that pressure drop was 50% greater at the lower pH.

Film removal:

In use of oxalic acid for removal of iron films care must be taken to keep the pH of the process* water below 4.0 to avoid deposition of calcium films.

A pH above 9.0 in the process water must be avoided since deposition of calcium or magnesium films will occur with or without the presence of silicate. The critical pH is about 0.3 of a pH unit higher when silicate is added.

The use of 50 ppm oxalic acid did not entirely remove a chromic and iron film which had been deposited at a pH of 4.5. Alkaline $(\text{NH}_4)_2\text{S}_2\text{O}_8$ was not effective in removal of chromic films deposited at 7.5 pH.

Chromic film removal with alkaline H_2O_2 was successful. A study of pressure drop data showed the optimum period of treatment to be 2 to 3 hours. The pressure drop data indicated no trend toward increased rates of removal over a range of 40 to 190 ppm H_2O_2 . However, film analyses showed less chromium in the remaining film at higher H_2O_2 concentrations.

Film prevention:

Silicate was not 100% effective in preventing deposition of Cr^{+++} films at pH 6.5 or Cr^{+++} and Fe^{+++} films at pH 4.6.

Meadol does prevent in part the deposition of iron and Cr^{+++} films at pH 6.5.

A study of the protection of Cr^{+++} and iron in internally heated equipment by $\text{Na}_2\text{H}_2\text{P}_2\text{O}_7$ showed that iron submitted more readily than did the Cr^{+++} .

Calgon was completely effective in preventing deposition of calcium films at a pH of 4.2-4.7 while removing iron films with oxalic acid. The disadvantage of this treatment lies in the fact that iron removal in this pH range is very

* This applies to "W" flocculated and filtered water which contains about 20 ppm Ca and 5 ppm Mg.

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slow but aluminum corrosion is, on the other hand, somewhat less severe than at a pH of 3.5, the usual range for oxalic acid treatment.

The use of an electrical potential across the annulus did not prevent the deposition of a film regardless of direction of current flow. This method, therefore, was considered impractical.

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FIG. #1

CATAPHORETIC
PIEZOMETER CONN.

UPSTREAM WATER END
FIG. #2

MUC TECHNICAL DIV. ENGINEERING DEVELOPMENT SECTION UPSTREAM WATER END FIG. #2		
DRAWN <i>WLP.</i>	DATE 11-22-44	ENG SKETCH 91
CHECK	SCALE NONE	
APPR. <i>WLP.</i>

FIG. 3

REAGENT ADDITION SYSTEM

MUC TECHNICAL DIV.		
ENGINEERING DEVELOPMENT SECTION		
PIEZOMETER RING FOR		
CATAPHORETIC CELL FIG #4		
DRAWN TRP.	DATE 11-14-44	ENG.
CHECK	SCALE:	SKETCH
APPR. <i>ACM</i>	NONE	92

CE-2524
FIG. #4

Fig. 5 - Electrical Contact Micrometer

Method of Operation: The micrometer and its two V shaped blocks, as shown above, are clamped to the slug or specimen tube which has a film to be measured. The film must be kept in a thoroughly moist condition but without excess water. The leads of a milliammeter (supplied current from a small battery) are inserted at the electrical connections. The micrometer spindle is then slowly advanced toward the film surface while the operator watches for the first deflection of the milliammeter. At this point the spindle is stopped and the first micrometer reading is taken. The spindle is again advanced until a much larger deflection of the milliammeter is observed. A second micrometer reading is taken, which when subtracted from the first gives a thickness measurement of the film.

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Figure 6.
Pressure Drop and pH versus Elapsed Time.

Low flow rate from 17½ hrs. to end of run.

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FIG. #7+8

Figure 7.
Pressure Drop and pH
versus Elapsed Time.

Similar results found
on Run 107-1.

The pH was purposely
increased at $4\frac{1}{2}$, at 8
and at $12\frac{1}{2}$ hours.

Figure 8.
Pressure Drop and pH
versus Elapsed Time.

The pH was purposely
increased at 5, 8
and $12\frac{1}{2}$ hrs.

At 13 hrs. maximum pressure
drop was reached. Flow
could no longer be main-
tained.

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Figure 9.
Pressure Drop and pH versus Elapsed Time.

FIG #9+10

pH purposely raised at $10\frac{1}{2}$ and $26\frac{1}{2}$ hrs. and purposely lowered at 15 hours.

Figure 10.
Pressure Drop and Ph versus Elapsed Time.

pH purposely raised for first $9\frac{1}{2}$ hrs.

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Figure 11.
Pressure Drop and pH versus Elapsed Time.

FIG 11+12

pH purposely raised at 13 and at 24 hrs.

Figure 12.
Pressure Drop and pH versus Elapsed Time

pH purposely raised at 13 and at 24 hrs.

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Table I

Average Analysis of Water Used in Film Studies

	Raw City H ₂ O Sept. 21, 1943	Demineralized City Water *	At "W", Finished Water from Filter Plant Clear Well	CMX-Filtered River Water 1943-1944 Average
<u>Fe</u> , ppm - - - - -	0.11	0.16**	0.05	0.03-0.10
<u>Cu</u> , ppm - - - - -	0.02	0.02	0.0	0.01-0.02
<u>Mg</u> , ppm - - - - -	11.0	0.08	4.0	5.5 -6.0
<u>Ca</u> , ppm - - - - -	32.8	0.32	20.0	19.0-23.0
Total Solids, ppm	154.0	66.6	-	84-98
<u>SiO₂</u> ppm - - - - -	3.4	3.9	-	5-7
<u>SO₄⁼</u> , ppm - - - - -	14.6	1.8	20.0	12-22
<u>CO₃⁼</u> , ppm - - - - -	0.0	0.0	0.0	0.0
<u>HCO₃⁼</u> , ppm - - - - -	69.5	70.0	-	40
<u>Cl⁻</u> , ppm - - - - -	4.7	0.7	1.2	0.2-0.6
pH - - - - -	8.1	6.5	7.8	7.8
Conductivity, reciprocal ohms	1.8×10^{-4}	9.6×10^{-5}	-	1.8×10^{-4}

Footnote: - The underlined symbols refer to those substances which were varied by addition of reagents during film studies.

* This is the analysis of the water after pH adjustment, but before addition of any reagents.

** Fe content in water leaving the demineralizers is about 0.06 ppm; the remaining Fe comes from pickup in the lines to the specimens.

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Table XIII
Film Analyses

Table XIII

Run Section	Reagents Added, ppm							pH	Film Deposit Analysis (%) ^{oo}					Time Hrs.	
	Cr ⁺³	Na ₂ Cr ₂ O ₇ ·2H ₂ O	H ₂ O ₂ x10 ⁵ N	Fe ⁺³	SiO ₂ ^o	Special Reagents	SO ₄ ⁼		Fe	Al	Cr	Si	CO ₂		
93-1	0.12	2.5	-	-	-	2.7 Meadol	35.8	6.5	7.2		6.4	0.3		12.2	Chromic film meadol protected.
93-2	0.13	1.9	-	-	-	-	35.8	6.5	11.0		14.3	1.3		12.2	
94-2	0.13	2.0	2.2	-	-	-	35.0	6.6	12.4	9.9	42.2	1.2		8.5	
85-1	-	1.9	-	-	-	7.0 Elon	35.4	6.6	30.0		7.2	1.3		11.3	Chromic films with and without elon.
91-1 In	-	1.9	-	-	-	13.2 Elon	35.0	6.5	20.6		13.0			29.9	
91-1 Out	-	1.9	-	-	-	13.2 Elon	35.0	6.5	22.1		16.0	2.1		29.9	
91-2 In	-	1.9	-	-	-	-	35.0	6.5	31.1		5.5			29.9	
91-2 Out	-	1.9	-	-	-	-	35.0	6.5	28.9		6.6	1.2		29.9	
92-1 In	-	1.9	-	-	-	-	32.4	6.4	30.6		17.0			41.5	
92-1 Out	-	1.9	-	-	-	-	32.4	6.4	30.4	13.6	5.9	3.9		41.5	
92-2 In	-	1.9	-	-	-	13.4 Elon	32.4	6.4	24.1		13.4			41.5	
92-2 Out	-	1.9	-	-	-	13.4 Elon	32.4	6.4	30.6	4.8	16.5	1.5		41.5	
83-1	-	1.9	2.3	0.06 ¹	-	-	33.4	4.3	13.0		9.3	3.3		6.6	-Ferric film formed at low pH and partially removed with 43 ppm oxalic acid at pH of 4.0.
80-2	0.16	2.2	1.1	0.05 ¹	-	-	36.2	4.7	14.3		7.4	1.5		5.6	
81-2	0.14	1.7	1.3	0.05 ¹	-	-	33.0	4.6	15.6		9.2	0.5		7.2	
82-2	0.12	1.4	2.4	0.05 ¹	-	-	26.9	4.4	18.0		6.5	1.3		5.5	
84-1	0.13	1.9	2.0	0.05 ¹	-	-	35.5	4.6	25.0	18.7	7.9	4.4	13.0	25.3	
84-2	0.14	1.9	1.6	0.05 ¹	13.1	-	35.5	4.7	25.0		10.8	1.2		25.3	Ferric and/or chromic films with and with- out silica at vary- ing pH.
75-1	0.36	1.9	2.0	-	5.1	-	27.9	6.5	5.1	10.4	50.0	4.7		26.4	
75-2	0.37	1.9	1.9	-	15.2	-	27.9	6.5	5.8	5.3	10.6	2.8	15.0	28.9	
79-2	-	1.6	1.8	-	-	-	29.6	4.6	38.4		3.0	2.2		55.3	Dichromate films at varying pH.
123-1	-	3.8	-	-	-	-	-	6.5	32.8	1.2	8.6			78.0	
123-2	-	3.8	-	-	-	-	-	6.5	35.8	14.1	9.7			72.0	
70-1	0.34	-	2.1	-	-	-	-	6.5	6.2	13.1	18.6	0.6		12.5	City water runs.
71-1	0.66	-	2.0	-	-	-	-	6.5	5.6	2.3	22.3	0.3	26.4	51.6	
74-1	0.36	2.0	2.3	-	-	-	33.1	6.4	6.4	0.5	25.0	2.8	18.9	26.5	Chromic films with and without dichromate, varying pH, and city vs. demineralized water
74-2	0.36	-	2.3	-	-	-	33.1	6.4	5.9	1.8	25.6	1.9	20.6	26.5	
78-1	0.12	1.6	2.0	-	-	-	30.0	4.6	26.0	11.1	12.7	1.8		72.2	
78-2	0.14	2.0	2.0	-	-	-	31.0	5.2	11.5	12.9	14.8			72.2	
77-1	0.14	1.9	1.9	-	-	-	26.0	5.3	16.7	4.2	16.1	2.3	24.7	25.7	
71-2	0.15	1.9	1.5	-	-	-	26.0	6.4	8.8	9.2	20.0	1.9	20.5	25.7	Ferric and chromic films formed with and without ammo- nium persulfate.
97-1	0.13	4.1	2.3	-	-	-	32.0	7.4	9.5	6.7	22.1			28.8	
115-1	0.14	1.9	2.0	-	-	4.6(NH ₄) ₂ S ₂ O ₈	31.0	7.5	4.6	31.9	3.7			28.9	
115-2	0.14	1.9	2.9	-	-	9.5(NH ₄) ₂ S ₂ O ₈	31.9	7.5	6.5	22.6	6.9			28.3	
77-2	0.14	1.9	1.5	-	-	-	26.0	6.5	8.8	20.0	9.2	1.9		25.7	
119-2	0.14*	1.8	3.1	-	-	-	33.8	6.5	15.4	21.6	5.8			30.7	
119-1	0.14*	1.8	2.9	-	-	5.0(NH ₄) ₂ S ₂ O ₈	33.8	6.5	16.1	18.3	6.9			30.7	
121-2	0.14*	2.2	3.0	-	30.6	-	34.3	6.5	7.5	23.3	4.3			14.8	
121-1	0.14*	2.2	3.1	-	29.4	5.6(NH ₄) ₂ S ₂ O ₈	34.3	6.5	11.5	19.8	4.5			14.8	
122-2	0.14*	1.7	2.9	-	28.6	-	28.6	6.5	9.2	23.9	2.5			31.9	
122-1	0.14*	1.7	2.7	-	26.6	4.6(NH ₄) ₂ S ₂ O ₈	26.6	6.5	14.3	18.6	1.2	8.5		31.9	
120-2	-	1.9	3.1	0.26 ¹¹	-	-	35.6	6.5	36.0	5.8	6.0			17.5	
120-1	-	1.9	2.9	0.25 ¹¹	-	5.1(NH ₄) ₂ S ₂ O ₈	35.6	6.5	35.0	6.9	5.7			17.5	
113-1	0.14	1.9	2.4	-	-	2.0(NH ₄) ₂ S ₂ O ₈	28.8	7.5	4.3	30.7	16.9		16.1	32.4	
113-2	0.14	1.9	2.3	-	-	-	28.8	7.5	5.3	29.1	15.9		15.7	32.4	

^{oo} Sample not large enough for analyses where spaces are blank.^o SiO₂ from sodium silicate - Type N - Philadelphia Quartz Co.* Cr₂(SO₄)₃ made up in presence of H₂SO₄ to keep chromium in ionic form.¹¹ Ferrous iron added.¹ All runs using ferric iron had 2 ppm of H₂SO₄ added.

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TABLE XVII

Analyses of Ca^{++} Films Formed at Low pH and of
 Ca^{++} and/or Mg Films Formed at High pH

Run No.	Hrs. Oper.	Reagents Added (ppm)					Film Analyses (%) ¹						
		Ca^{++}	Mg^{++}	$\text{H}_2\text{C}_2\text{O}_4$	SiO_2^*	pH	Ca	Mg	Fe	Al	Si	CO_2	$\text{C}_2\text{O}_4^{--}$
105-1	19.5	16.5	-	44.2	-	4.5	24.2	-	0.3	-	-	-	46
105-2	19.5	16.5	-	47.0	-	4.5	22.4	-	0.2	-	-	-	61
106-1	5.0	17.5	-	-	20.7	>9	35.7	-	0.07	-	-	-	-
106-2	5.0	17.5	-	-	9.9	>9	38.9	-	0.05	1.5	-	-	-
107-1	2.6	18.4	-	-	38.6	>9	38.1	-	0.09	1.1	0.38	-	-
107-2	2.3	18.9	-	-	-	>9	38.4	-	0.15	0.6	0.17	-	-
111-1	4.3	16.6	5.2	-	39.0	>9	34.7	0.3	0.2	0.7	6.7	16.5	-
111-2	4.0	15.7	5.1	-	-	>9	39.6	0.3	0.1	1.7	0.13	13.0	-
112-1	26.0	-	5.9	-	35.4	>9	1.4	7.2	0.3	0.6	17.8	5.1	-
112-2	26.0	-	5.9	-	-	>9	14.9	15.3	0.5	5.5	0.8	19.6	-
114-2	5.0	18.7	-	-	-	>9	39.5	-	0.05	-	0.11	13.4	-
114-1	10.0	18.5	-	-	41.2	>9	39.9	-	0.04	-	0.41	8.8	-

¹

A blank space in the analyses indicates no data available.

* SiO_2 from sodium silicate - Type N - Philadelphia Quartz Co.

The runs were made with water containing sulfate, dichromate and peroxide.

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TABLE XX

Analyses of Chromic and Iron Films After Alkaline Peroxide Removal Treatment

Run No.	Hrs. Oper.	Conditions During Formation					Film Analyses (%)		
		Cr ⁺³ ppm	Fe ⁺³ ppm	H ₂ SO ₄ ppm	pH	H ₂ O ₂ ppm	After Treatment Fe	Cr	Al
101-1	43.8	0.14	0.05	2	7.5	40	10.8	20.6	14.4
101-2	43.8	0.14	0.05	2	7.5	132	15.5	13.3	16.7
102-1	40.1	0.08	0.05	2	7.5	91	11.3	12.2	12.6
102-2	40.1	0.08	0.05	2	7.5	152	15.8	8.9	13.9

Runs were made with demineralized water containing sulfate, dichromate and peroxide.

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TABLE XXI

Results of Removal of Chromic Films with Alkaline Peroxide

H ₂ O ₂ ppm	Before Film Buildup	Before Film Removal	After Film Removal	Increase from Film	Recovered by Film Removal	% Recovery of ΔP Increase at Hourly Periods of Film Removal						
						1	2	3	4	5	6	7
39	33	46	37	13	9	23	61	61	61	69	69	-
91	30	48	38	18	10	33	43	50	55	55	55	55
102	56	69	52	13	17	23	61	61	61	77	115	130*
131	29	43	36	14	7	36	50	50	50	50	50	-
152	32	46	36	14	10	42	50	57	64	71	71	71
189	48	65	58	17	7	0	32	61	41	41	41	-

H ₂ O ₂ ppm	Before Film Buildup	Decrease in Temperature Rise, °C		Decrease from Film	Recovered by Film Removal	% Recovery of ΔT Decrease at Hourly Periods of Film Removal						
		Before Film Removal	After Film Removal			1	2	3	4	5	6	7
39	50	27	37	23	10	13	26	34	34	39	43	-
91	46	29	37	17	8	29	35	41	47	47	47	47
102	57	47	55	10	8	10	40	20	10	40	70	80*
131	48	27	41	21	14	33	52	57	57	67	67	-
152	49	33	46	16	13	12	19	63	75	81	81	81
189	47	30	38	17	8	6	41	41	41	47	47	-

* The water flow was reduced during the last portion of the run giving fictitious pressure drop results.

Run No.		Sect.	CHEMICAL CONDITIONS		BULK AVERAGE		Conductivity of Soln. RATIO	POINT OF ADDITION HOURS	TIME OF RUN HOURS	VELOCITY FT./SEC.	Δ P INCREASE/HOUR				h _s	U		h _w		% h _w PER HOUR	KILOWATTS/(FT.) ²		FILM THICKNESS		FILM DESCRIPTION	H.E. TO SPEC. TIME REAGENTS	INLET TEMP. °C	CATAPHORETIC UNIT																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																										
			pH	H ₂ O ₂ M/L	SPECIAL REAGENTS	PPM. H ₂ O ₂ SO ₄					PPM. TOTAL SO ₄	1/3	2/3	3/3		OVERALL	START	STOP	START		STOP	START	STOP	MILS						MILS/HR	STRUCTURE	STABILITY																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																						
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Run No.		SECT.	CHEMICAL CONDITIONS			BULK AVERAGE		CONDUCTIVITY OF SOLN. RE-10 ³	POINT OF ADDITION OF REAGENTS	TIME OF RUN HOURS:MIN:SEC	ΔP				h _s	U		h _w	% h _w PER HOUR	KILOWATTS (FT)		FILM THICKNESS		FILM DESCRIPTION		H.E. SPEC. TYPE	INLET TEMP °C	CATAPHORETIC UNIT	REMARKS	
			pH	H ₂ O ₂ N/10 ³	SPECIAL REAGENTS	DPPM Fe+++	DPPM Fe++				1/3	2/3	3/3	AVERAGE		START	STOP			START	STOP	START	STOP	MILS	MILS / HR					STRUCTURE
12	1	6.5	1.2	BLANK	-	-	9.4	2	126.0	20				0.04				5270		41.6	34.0	6.3	0.05	F (FINE)	S (STANDARD)	0	4.3		pHS - FILM THICKEST NEAR CENTER	
	2	6.5	1.4	BLANK	-	-	9.0	2	121.5	20				0.09				5280		41.0	33.3			F	S	0	4.3		" - " " " " " "	
13	1	6.5	2.0	BLANK	-	-	9.3	2	52.8	20	Pressure Gages	0.2	5650	1973	1780	5160	4000	0.40	40.4	36.5	3	0.06	F	S	0	3.1		" - SOME FILM FORMED		
	2	6.5	2.0	0.2 ppm. Fe+++	0.2	2.5	8.6	4	56.5	20	At Fractional	?	3620	1630	1445	4940	3560	0.55	34.6	31.5			F, D	S	0	3.0		" - ΔP ROSE RAPIDLY, BROUGHT TO NORMAL BY VARYING FLOW		
14	1	6.5	2.0	BLANK	-	-	9.4	2	24.1	20	Lengths Were	0	5390	1950	1870	5190	4670	0.28	38.4	38.4	0.2-1.0	0.01-0.4	F	S	0	3.5		" - VERY THIN FILM AT INLET		
	2	6.4	2.0	0.05 ppm. Fe+++	0.05	2.1	9.4	4	24.1	20	Not Installed	1.0	3920	1700	1180	5040	2190	3.56	34.7	25.8	3	0.12	F, D	S	0	3.3		" - UNIFORM FILM DISTRIBUTION		
15	1	6.5	2.0	BLANK	-	-	9.4	2	26.0	20	Before Run 20	0.2	5520	1960	1730	5170	3820	1.00	39.0	35.5			F, SLINEY	LOOSE	0	3.6		" - FILM MAX. AT 3/4 L. FROM INLET		
	2	6.5	2.0	0.05 ppm. Fe+++	0.05	2.1	9.4	4	26.0	20		1.6	4460	1800	1140	5110	1940	2.38	36.5	25.8			?	?	0	3.6		" - NO FILM DATA		
16	1	6.6	2.0	BLANK	-	-	13.0	2	17.5	20		0	5810	1970	1820	4990	4120	1.00	38.6	36.2	0.3-1.8	0.02-0.4	F, SLINEY	LOOSE	0	3.2		" - FILM MAX. AT 3/4 L. FROM INLET		
	2	6.5	2.3	10 ppm. Fe+++	1.0	4.6	12.0	4	17.5	20		1.0	3790	1640	1520	4750	3860	1.08	33.8	30.9	0.7-2.1	0.04-0.12	D (DENSE)	S	0	3.0		" - " " " " " " " "		
17	1	6.5	2.0	BLANK	-	-	12.0	2	44.5	20		0	5460	1920	1770	4930	4150	1.22	37.7	36.8	0.5-1.0	0.01-0.02	F, SLINEY	LOOSE	0	3.2		" - UNIFORM FILM DISTRIBUTION		
	2	6.5	1.9	0.01 ppm. Fe+++	0.01	2.0	12.0	4	44.5	20		0.9	4140	1710	1226	4800	2295	1.74	35.0	27.3	0.5-2.7	0.01-0.06	D	S	0	3.1		" - FILM MAX. AT OUTLET		
18	1	6.4	2.0	0.01 ppm. Fe+++	0.01	2.0	8.8	2	12.7	20		3.9	4710	1820	1130	4930	1860	4.92	36.8	25.6	0.5-1.50	0.04-0.12	D	S	0	4.0		" - FILM MAX. AT OUTLET		
	2	6.4	2.1	BLANK	-	-	9.4	2	12.7	20		0	4650	1800	1740	4890	4450	0.71	36.2	35.0	0.3	0.03	F, SLINEY	LOOSE	0	4.0		" - UNIFORM FILM DISTRIBUTION		
19	1	6.7	1.8	0.01 ppm. Fe+++	0.01	2.0	50	7.8	20		2.5	3280	1556	1455	4940	3970	4.91	32.7	31.2	0.5-2.0	0.07-1.4	D	S	0	4.3		" - FILM MAX. AT OUTLET			
	2	6.6	2.1	0.01 ppm. Fe+++	0.01	2.0	4	7.8	20		0	4310	1747	1605	4880	4050	2.19	35.0	34.8	0.5-2.0	0.07-1.4	D	S	0	3.6		" - FILM MAX. AT 3/4 L. FROM INLET			
20	1	6.6	2.0	0.01 ppm. Fe+++	0.01	2.0	9.0	2	46.8	10	1.2	1.7	4.4	2.5	2890	1190	765	2790	1210	1.21	16.9	10.7	2.0-7.0	0.04-0.15	D, COARSE	S	0	5.7		" - " " " " " " " "
	2	6.6	2.0	0.01 ppm. Fe+++	0.01	2.0	9.0	4	46.8	20	1.2	0.1	1.4	0.8	4280	1746	1060	4870	1743	1.38	35.6	23.8	2.0-4.2	0.04-0.09	D	S	0	4.3		" - " " " " " " " "
21	1	6.5	2.0	0.01 ppm. Fe+++	0.01	2.0	8.6	2	31.5	5	1.1	3.2	2.1	1.8	1640	730	378	1595	528	2.12	8.7	5.4	0.6-14.0	0.02-0.40	D, COARSE	S	0	6.0		" - FILM MAX. AT LAST 1/3 ON TOP
	2	6.5	2.0	0.01 ppm. Fe+++	0.01	2.0	8.7	4	31.5	20	0.5	1.1	2.4	1.3	4430	1763	1100	4870	1820	1.98	35.6	24.0	1.5-4.4	0.05-1.4	D	S	0	4.7		" - FILM MAX. AT 3/4 L. FROM INLET
22	1A	6.6	1.9	0.2 ppm. Fe+++	0.2	2.5	2	4.6	20	2.1	4.3	15.4	7.1	3920	1680	1313	4890	2700	9.60	33.9	27.9					0	4.5		" -	
	1B	6.6	1.9	0.2 ppm. Fe+++	0.2	2.5	2	2.8	20	1.8	5.6	17.6	8.1	5150	1880	1393	4940	2570	16.35	36.2	29.1					0	5.0		" -	
	2	6.7	2.0	0.01 ppm. Fe+++	0.01	2.0	4	11.5	20	0.5	3.5	2.8	2.0	5220	1910	1620	4980	3400	2.76	37.4	33.2					0	5.5		" -	
23	1A	6.4	1.9	0.2 ppm. Fe+++	0.15	2.4	5.9	2	6.0	20	1.8	5.3	15.7	7.6	4850	1953	1250	4870	2020	9.73	36.8	26.2					0	4.5		" -
	1B	6.4	1.9	0.2 ppm. Fe+++	0.15	2.4	5.9	2	2.5	20	0.0	7.3	24.8	11.0	3860	1660	1427	4810	3260	12.30	32.7	29.1					0	4.0		" -
	1C	6.4	1.9	0.2 ppm. Fe+++	0.15	2.4	5.9	2	5.5	20	0.0	2.9	1.5	1.1	3510	1613	1337	4610	2830	7.09	32.0	28.5	11-3.9	0.2-0.6	D	S	0	4.5		" - FILM MAX. AT OUTLET
	2	6.4	1.8	0.01 ppm. Fe+++	0.01	2.0	6.1	4	18.8	20	1.1	0.0	2.3	1.4	5310	1910	1253	4990	2105	3.09	37.4	27.3	0.6-3.6	0.03-0.19	D	S	0	4.5		" -
24	1	6.5	1.8	0.01 ppm. Fe+++	0.01	2.0	7.0	14	18.1	20	1.0	0.9	2.6	1.5	4240	1745	1117	4930	1910	3.39	35.6	24.7	0.9-8.0	0.05-0.44	D	S	0	4.5		" - FILM MAX. ON TOP AT 3/4 L. FROM INLET
	2	6.5	1.8	0.01 ppm. Fe+++	0.01	2.0	7.0	4	18.1	20	0.3	0.6	1.6	2.2	4020	1696	1220	4870	2290	2.93	34.5	26.7	1.9-3.6	0.10-0.20	D	S	0	4.5		" - FILM MAX. ON TOP AT OUTLET.
25	1	6.5	2.1	0.01 ppm. Fe+++	0.01	2.0	7.5	0	20.2	20	0.3	0.8	3.8	1.7	4410	1770	1233	4930	2230	2.94	35.0	26.7	0-3.5	0-0.17	D	S	0	5.0		" -
	2	6.5	2.0	0.01 ppm. Fe+++	0.01	2.0	7.5	4	20.2	20	0.8	2.2	2.0	1.6	4930	1857	1350	4970	2480	2.68	36.9	28.5	2.0-4.0	0.10-0.20	D	S	0	5.0		" -
26	1	6.5	2.1	0.05 ppm. Fe+++	0	2.1	8.6	2	32.3	20	0.6	1.0	2.0	1.2	4060	1707	993	4930	1600	2.10	34.4	22.5	1.0-6.0	0.03-0.19	D	S	0			" -
	2	6.5	2.1	0.01 ppm. Fe+++	0.01	2.0	8.6	2	32.3	20	0.9	1.2	1.5	1.2	3980	1690	1037	4890	1730	2.01	33.8	23.1	2.2-6.8	0.07-0.21	D	S	0			" -
27	1	6.4	2.1	0.01 ppm. Fe+++	0.01	2.0	8.3	2	36.7	20	0.0	0.4	0.8	0.4	2205	1210	942	4200	2120	1.34	19.0	15.4	1.0-7.2	0.03-0.19	LIGHT-D	S	0			" -
	2	6.6	2.1	0.01 ppm. Fe+++	0.01	2.0	8.2	2	36.7	20	0.7	1.6	0.9	1.0	4190	1725	1070	4860	1780	1.73	34.7	23.8	1.3-6.3	0.03-0.17	D	S	0			" -
28	1	6.4	2.1	0.05 ppm. Fe+++	0	2.1	50	7.0	20	0.0	0.6	6.0	2.3	3180	1507	1160	4690	2430	6.02	31.7	25.8	1.6-6.3	0.20-0.30	D	S	0			" -	
	2	6.5	1.9	0.01 ppm. Fe+++	0.01	2.0	8.3	2	7.0	20	1.3	2.3	3.0	2.2	3890	1670	1263	4800	2510	5.96	33.8	26.4	0.6-4.5	0.09-0.63	D	S	0			" - F ₂ ⁺⁺⁺ ADDED BY MISTAKE FOR PART OF RUN
29	1	6.5	2.0	0.05 ppm. Fe+++	0	2.1	8.3	50	14.3	20	0.0	2.8	2.8	1.7	3830	1650	1050	4760	1777	4.34	34.4	23.7	1.2-5.8	0.09-0.95	D	S	0			" -
	2	6.5	2.1	0.01 ppm. Fe+++	0.01	2.0	7.4	2	14.3	20	1.5	3.1	1.7	1.8	5750	1960	1437	4970	2570	3.34	38.3	30.3	0.3-5.3	0.02-0.41	D	S	0			" -
30	1	6.5	2.1	0.01 ppm. Fe+++	0.01	2.0	9.2																						" -	
	2	6.5	2.0	BLANK	-	-	9.2																						" -	
	2	6.5	2.0	BLANK	-	-	9.2																						" -	

scale 104

[illegible]

Table XXXII. 54
Legend CB-2524

Run and Section	Chemical Conditions			Conductivity $\times 10^5$	Time of Run Hours	Inlet Temp. °C	Percent Increase per Hour				ΔT °C	h_s	U		h_w		h_w % Dec. per Hour	Kilowatts per Foot		Film	
	pH (s)	H_2O_2 $\times 10^4$	Special Reagents - ppm				1/3	2/3	3/3	Overall			Start	Stop	Start	Stop		Start	Stop	Mils per Hour	Description
51 I	6.6	2.1	0.05 Fe ⁺⁺ (66); 0.09 H ₂ SO ₄ (66)	8.2	15.3	6	0.0	0.31	0.34	0.23	0.33	4330	1760	1630	4940	4050	1.18	35.6	33.3	0.01-0.13	F, S, Red Brown
II	6.6	1.9	0.05 Fe ⁺⁺ (37); 0.09 H ₂ SO ₄ (37)	7.8	15.3	6	0.0	0.35	0.50	0.24	0.52	5240	1905	1700	5030	3820	1.57	37.2	34.2	0.01-0.13	F, S, " "
52 I	6.4	2.2	0.2 Fe ⁺⁺ (2); 2.0 HCl(2)	6.8	5.4	6	1.22	0.83	4.35	1.97	0.34	3280	1545	1532	4820	4690	0.50	32.0	31.3	0-0.93	D, U, Red Br. Tan
II	6.4	2.8	0.2 Fe ⁺⁺ (2); 2.0 HCl(2)	6.8	5.4	6	1.59	5.25	6.85	4.28	0.0	4130	1720	1740	4950	5080	0.0	34.1	34.1	0-0.61	D, U, Red Br. Tan
53 I	6.6	1.9	0.2 Fe ⁺⁺ (66); 0.3 H ₂ SO ₄ (66)	7.5	33.5	7	0.0	0.41	0.75	0.41	0.63	4200	1675	1220	4940	2240	1.64	31.6	26.8	0.05-0.16	D, S, Red Br.
II	6.5	2.0	0.2 Fe ⁺⁺ (37); 0.3 H ₂ SO ₄ (37)	7.2	33.5	6	0.13	0.15	0.83	0.53	0.06	4550	1790	1300	4970	2410	1.54	35.5	27.7	0.05-0.16	D, S, Red Br.
54 I	6.5	2.3	0.03 Cr ⁺⁺ (2)	6.5	24.9	6.5	0.24	0.33	0.57	0.39	0.42	4700	1858	1590	5030	3460	1.25	36.3	32.5	0.02-0.10	F, S, Mud Br.
II	6.5	2.2	0.35 Cr ⁺⁺ (2)	6.2	24.9	6.5	0.73	0.80	2.00	1.07	0.92	3540	1600	1130	4860	1850	2.48	32.6	24.9	0.04-0.21	D, Gray Gr.
55 I	6.5	98.	5.7 Na ₂ Cr ₂ O ₇ ·2H ₂ O(2)	7.2	38.8	6	0.0	0.12	0.13	0.09	0.04	3790	1660	1630	4890	4650	0.13	33.9	33.3	0-0.04	—
II	6.5	98.	5.9 Na ₂ Cr ₂ O ₇ ·2H ₂ O(2)	7.2	38.0	6	0.0	0.0	0.0	0.0	0.0	4570	1800	1788	4970	4850	0.06	35.7	35.7	0-0.03	—
56 I	6.5	2.1	1.3 SO ₄ ²⁻ (5)	7.3	41.3	7	0.35	0.0	0.0	0.04	0.04	3340	1565	1528	4880	4550	0.16	32.0	31.6	0-0.02	F, S, Orange Tan
II	6.5	2.2	1.3 SO ₄ ²⁻ (5)	7.3	41.3	7	0.12	0.0	0.20	0.09	0.08	5350	1925	1845	5060	4540	0.25	36.9	35.7	0-0.02	F, S, Orange Tan
57 I	6.5	2.0	14.8 SO ₄ ²⁻ (5)	11.0	24.8	7	0.0	0.0	0.19	0.0	0.07	3660	1638	1593	4930	4550	0.31	33.3	32.7	<0.01	—
II	6.5	2.0	14.8 SO ₄ ²⁻ (5)	11.0	24.8	7	0.0	0.19	0.0	0.0	0.07	4590	1814	1836	5020	5210	0.0	35.7	36.2	<0.01	—
58 I	6.5	1.9	47.6 SO ₄ ²⁻ (5)	16.8	19.8	8	0.0	0.0	0.0	0.0	0.0	3395	1580	1580	4920	4920	0.0	32.2	32.2	0-0.02	F, S, Red
II	6.5	2.3	47.6 SO ₄ ²⁻ (5)	17.2	19.8	9	0.0	0.0	0.39	0.0	0.09	4670	1830	1775	5080	4650	0.43	35.1	34.5	0-0.01	F, S, Red
59 I	6.5	1.9	14.0 SO ₄ ²⁻ (2)	9.8	25.8	9	0.29	0.0	0.0	0.0	0.0	3820	1673	1665	5010	4940	0.06	33.4	33.4	0-0.04	F, S, Red Br.
II	6.6	2.0	49.3 SO ₄ ²⁻ (2)	17.8	25.8	9	0.0	0.0	0.28	0.07	0.07	4780	1850	1775	5080	4570	0.39	35.1	34.6	0-0.04	F, S, Red Br.
60 I	6.5	1.9	3.7 SO ₄ ²⁻ (2)	2.2	19.5	10	0.0	0.0	0.27	0.09	0.0	3820	1680	1675	5060	5000	0.06	33.4	33.4	0-0.04	F, S, Red Tan
II	6.5	1.9	7.3 SO ₄ ²⁻ (2)	7.9	19.5	9	0.24	0.0	0.0	0.0	0.17	4680	1835	1795	5110	4810	0.30	35.7	34.6	0-0.04	F, S, Red Tan
61 I	6.5	2.8	2 Cl ⁻ (5); 2 Cl ⁻ (2); 2 SO ₄ ²⁻ (2)	8.7	33.8	9	0.0	0.0	0.14	0.05	0.16	3600	1632	1520	4990	4080	0.54	32.7	30.9	0-0.04	F, S, Lt. Red Tan
II	6.5	1.9	2 Cl ⁻ (5); 2 SO ₄ ²⁻ (2)	8.0	33.8	9	0.0	0.14	0.0	0.05	0.05	4570	1812	1753	5060	4640	0.24	34.6	33.9	0-0.03	F, S, Dk. Red Tan
62 I	6.5	2.8	2.0 SO ₄ ²⁻ (2)	8.6	35.8	9	0.0	0.12	0.0	0.0	0.10	3800	1672	1615	5010	4530	0.27	33.4	32.2	0-0.03	F, S, Br.
II	6.5	1.9	2.0 SO ₄ ²⁻ (2)	8.2	35.8	9	0.0	0.14	0.0	0.0	0.14	5230	1915	1770	5110	4190	0.50	35.7	33.9	0-0.03	F, S, Br.
63 I	6.5	1.9	2.0 H ₂ SO ₄ (2)	7.2	37.7	11	0.19	0.0	0.28	0.14	0.14	4140	1750	1620	5130	4150	0.54	33.9	32.2	0.01-0.04	F, S, Red Br.
II	6.5	2.0	0.05 Fe ⁺⁺ (2); 2.0 H ₂ SO ₄ (2)	7.2	37.7	11	0.24	0.14	0.38	0.23	0.20	4400	1635	1464	5030	3700	0.71	31.5	29.2	0.02-0.05	F, S, Red Tan
64 I	6.5	2.0	0.05 Fe ⁺⁺ (2); 4 Cl ⁻ (2); 2.0 H ₂ SO ₄ (2)	7.2	40.2	10	0.18	0.40	0.50	0.38	0.33	3140	1530	1272	4970	3020	0.98	30.9	26.7	0.04-0.07	D, S, Red Br.
II	6.5	1.9	0.05 Fe ⁺⁺ (2); 2.0 H ₂ SO ₄ (2)	7.2	40.1	10	0.23	0.35	0.77	0.40	0.27	3980	1720	1546	5060	3850	0.57	33.4	29.8	0.03-0.07	D, S, Red Br.
65 I	6.7	2.0	City Water - no additions	25.0	38.3	10	0.19	0.11	0.0	0.04	0.10	3550	1620	1530	5010	4220	0.41	32.2	30.9	0-0.03	F, S, Red Tan
II	6.7	2.0	City Water + 0.05 Fe ⁺⁺ (2); 2.0 H ₂ SO ₄ (2)	27.0	38.3	10	0.0	0.13	0.0	0.0	0.09	3960	1740	1630	5080	4620	0.24	33.9	32.7	0.01-0.03	F, S, Red
66 I	6.6	1.9	0.037 Al ⁺⁺ (2); 35.9 SO ₄ ²⁻ (5)	17.1	36.3	10	0.21	0.12	0.15	0.15	0.15	3650	1643	1530	5030	4080	0.52	32.7	30.9	0-0.04	F, Red Br.
II	6.6	1.9	0.32 Al ⁺⁺ (2); 37.4 SO ₄ ²⁻ (5)	16.0	14.7	10	3.3	2.2	1.0	2.4	0.47	4490	1805	1650	5110	4030	1.44	34.6	32.2	0-0.38	D, U, Colorless
67 I	6.5	1.9	0.10 Al ⁺⁺ (2); 31 SO ₄ ²⁻ (5)	14.5	15.6	11	1.7	1.9	0.45	1.5	0.0	3580	1630	1600	5030	4760	0.34	31.5	31.5	0-0.07	D, U, Colorless
II	6.5	2.1	0.06 Al ⁺⁺ (2)	14.5	8.8	10	2.4	1.4	0.60	1.4	0.23	3510	1610	1573	5010	4650	0.81	32.2	31.5	0-0.09	D, U, Colorless
68 I	6.5	2.0	0.050 Al ⁺⁺ (2); 33.4 SO ₄ ²⁻ (5)	16.9	55.3	11	0.54	0.36	0.14	0.38	0.13	4020	1720	1590	5060	4080	0.35	33.7	30.9	0-0.05	F, S, Red Br.
69 I	6.6	2.1	City Water + 0.10 Al ⁺⁺ (2)	28.5	62.0	12	0.07	0.0	0.50	0.15	0.15	3720	1665	1477	5110	3670	0.45	32.2	29.2	0.01-0.02	F, Red Bronze
70 I	6.5	2.1	City Water + 0.34 Cr ⁺⁺ (2)	26.9	12.5	12	2.0	0.0	0.76	0.42	0.61	3320	1573	1425	5060	3780	2.0	30.9	28.6	0.05-0.25	D, S, Dull Gr. Br.

Under Chemical Conditions:-

Figure in parenthesis indicates point of reagent addition, e.g.:
(5) added at storage
(1), (2) etc. - time lag
in seconds from addition point to specimen.

Under Film

Description:-

D-Dense. Br-Brown
F-Fine. Gr-Green
S-Stable.
Sl-Slimy.
U-Unstable.
T-Tenacious

Run and Section	Chemical Conditions		Conductivity $\times 10^{-5}$	Time of Run Hours	Inlet Temp. °C	ΔP Percent Increase per Hour				ΔT % Dec. per Hour	h_s	U		h_w		h_w % Dec. per Hour	Kilowatts per Foot		Film	
	pH(s)	Special Reagents - ppm				1/3	2/3	3/3	Overall			Start	Stop	Start	Stop		Start	Stop	Mils per Hour	Description
71 II	6.5	City Water + 0.66 Cr ⁺³ (2)	25.1	51.6	14	0.83	0.0	1.1	0.48	0.56	3400	1605	1048	5160	1900	1.2	30.9	22.0	0.02-0.38	D.T. Gray Gr.
72 I	6.6	0.35 Cr ⁺³ (2)	7.9	11.7	13	0.45	1.5	4.8	2.5	1.4	3800	1690	1363	5170	2500	4.4	32.7	27.4	0.03-0.57	D.S. Br. Gr.
II	6.7	0.35 Cr ⁺³ (2); 2 Na ₂ Cr ₂ O ₇ · 2 H ₂ O(2)	9.8	9.0	16	0.97	1.6	4.8	2.1	0.67	3250	1573	1400	5200	3690	3.2	29.8	27.9	0.04-0.53	D.S. Br. Gr.
73 II	6.5	0.34 Cr ⁺³ (2)	6.5	11.8	14	0.35	0.41	3.2	1.0	0.34	2950	1490	1390	5080	4070	1.7	29.2	27.9	0-0.35	D.S. Olive Gr.
74 I	6.4	0.36 Cr ⁺³ (2); 2.0 Na ₂ Cr ₂ O ₇ · 2 H ₂ O(43); 33.1 SO ₄ ⁻² (5)	1.4	26.5	15	0.0	0.68	4.0	1.4	0.96	3920	1723	1200	5270	2260	2.2	32.7	24.4	0.02-0.37	D.S. Olive Gr.
II	6.4	0.36 Cr ⁺³ (2); 33.1 SO ₄ ⁻² (5)	1.4	26.5	15	0.0	0.83	3.5	1.0	0.87	3560	1645	1167	5200	2260	2.1	30.9	23.7	0.03-0.18	D.S. Olive Gr.
75 I	6.5	0.36 Cr ⁺³ (2); 5.5 SiO ₂ (35); 1.9 Na ₂ Cr ₂ O ₇ · 2 H ₂ O(43); 27.9 SO ₄ ⁻² (5)	14.5	28.9	15	0.0	0.33	0.66	0.35	0.34	3080	1530	1344	5160	3520	1.1	29.8	26.7	0.01-0.20	D.T. Olive Gr.
II	6.5	0.37 Cr ⁺³ (2); 15.5 SiO ₂ (39); 1.9 Na ₂ Cr ₂ O ₇ · 2 H ₂ O(47); 27.9 SO ₄ ⁻² (5)	15.1	28.9	15	0.31	0.0	2.3	0.55	0.34	3180	1555	1355	5160	3480	1.1	29.8	26.7	0.01-0.16	D.T. Olive Gr.
76 I	6.5	0.35 Cr ⁺³ (2); 48.5 SiO ₂ (39); W	16.4	26.4	15	0.0	0.45	0.25	0.22	0.38	3120	1540	1354	5160	3520	1.2	29.8	26.7	0-0.08	F.T. Lt. Gr.
II	6.4	0.36 Cr ⁺³ (2); W	15.7	26.4	15	0.33	0.72	1.6	0.79	0.57	2970	1498	1203	5110	2780	1.7	28.6	24.4	0.04-0.25	D.S. Olive Gr.
77 I	5.3	0.14 Cr ⁺³ (2); W	8.5	25.7	15	0.21	0.62	1.5	0.70	0.78	7860	2255	1673	5540	2970	1.8	39.3	31.5	0.01-0.17	D.S. Dk. Red Br.
II	6.4	0.15 Cr ⁺³ (2); W	14.1	25.7	16	0.0	0.70	1.8	0.70	1.5	7410	2219	1237	5540	1860	2.6	38.1	24.4	0.04-0.28	D.L. Dk. Gr. Br.
78 I	4.6	0.12 Cr ⁺³ (2); W	5.5	72.2	15	0.07	0.19	0.23	0.14	0.19	3090	1532	1305	5160	3260	0.51	29.7	25.6	0-0.03	D.S. Dk. Red Br.
II	5.2	0.14 Cr ⁺³ (2); W	6.1	12.3	15	0.71	1.1	4.1	1.6	0.0	3050	1546	1553	5160	5530	0.0	29.7	29.7	0.01-0.35	D.S. Dk. Red Br.
79 II	4.6	0.02 Cr ⁺³ (2); W	5.2	54.0	17	0.08	0.0	0.15	0.07	0.17	7140	2200	1937	5580	4150	0.46	38.1	34.5	0-0.65	F.S. Red Tan
80 I	4.7	0.05 Fe ⁺³ (35); 2 H ₂ SO ₄ (35); W	6.3	5.3	18	0.95	0.0	3.3	1.1	0.70	3440	1630	1550	5520	4570	2.5	30.3	29.2	0-0.17	F.S. Red Br.
II	4.7	0.05 Fe ⁺³ (39); 2 H ₂ SO ₄ (39); 0.16 Cr ⁺³ (2); W	6.7	5.8	18	0.0	4.1	13.0	4.1	1.7	6620	2152	1830	5580	3950	5.6	36.9	33.4	0-0.47	D.S. Red Br.
81 I	4.5	0.04 Fe ⁺³ (2); 2 H ₂ SO ₄ (2); W	7.2	7.2	17	0.0	0.0	1.2	0.0	0.0	3000	1512	1512	5220	5220	0.0	29.2	29.2	0-0.06	F.S. Red Br.
II	4.6	0.05 Fe ⁺³ (2); 2 H ₂ SO ₄ (2); 0.14 Cr ⁺³ (39); W	7.8	7.2	17	0.0	2.5	5.3	2.1	1.1	7150	2198	1925	5550	4080	7.2	37.6	34.6	0-0.47	D.S. Red Br.
82 I	4.4	0.05 Fe ⁺³ (35); 2 H ₂ SO ₄ (35); W	7.2	5.5	16	0.9	0.0	1.3	0.36	0.34	3470	1630	1625	5250	5190	0.2	30.9	30.3	0-0.18	D.S. Red Br.
II	4.4	0.05 Fe ⁺³ (39); 2 H ₂ SO ₄ (39); 0.12 Cr ⁺³ (2); W	8.6	5.5	16	0.83	0.83	1.5	0.97	0.57	7080	2190	2090	5540	4960	1.9	38.1	36.9	0-0.56	D.S. Red Br.
83 I	4.3	0.06 Fe ⁺³ (35); 2 H ₂ SO ₄ (35); W	1.0	6.6	16	0.85	0.0	2.7	1.0	1.2	3070	1533	1398	5200	3920	3.7	29.8	27.4	—	—
II	4.0	42.9 H ₂ C ₂ O ₄ · 2 H ₂ O(2); X	—	4.3	16	0.0	-1.4	-1.8	-0.97	0.0	3070	1398	1395	3920	3910	0.0	27.4	27.4	—	Dk. Red Br.
83 II	4.3	0.05 Fe ⁺³ (39); 2.7 H ₂ SO ₄ (39); 0.14 Cr ⁺³ (2); W	1.0	6.6	16	1.3	2.8	11.0	3.5	1.7	6210	2085	1770	5490	3310	6.1	36.9	32.7	—	—
IIA	3.8	41.6 H ₂ C ₂ O ₄ · 2 H ₂ O(2); X	—	4.3	16	-0.94	0.0	-2.6	-1.1	-0.42	6210	1770	1730	3310	3550	-1.7	32.7	32.2	—	Dk. Red Br.
84 I	4.6	0.05 Fe ⁺³ (2); 2 H ₂ SO ₄ (2); 0.13 Cr ⁺³ (2); W	11.1	25.3	16	0.67	1.0	2.7	1.3	1.2	5660	2019	1245	5460	2040	2.5	36.3	24.9	0.03-0.18	D.S. Dk. Red Br.
II	4.7	0.05 Fe ⁺³ (2); 2 H ₂ SO ₄ (2); 0.14 Cr ⁺³ (2); 13.1 SiO ₂ (39)	10.5	25.3	16	0.34	0.75	3.2	1.1	0.95	3910	1724	1230	5290	2310	2.2	32.2	24.4	0.02-0.21	D.L. Dk. Red Br.
85 I	6.6	7.0 Elon(P); 1.9 Na ₂ Cr ₂ O ₇ · 2 H ₂ O(43); 35.4 SO ₄ ⁻² (5); X	15.1	11.3	17	0.84	2.5	3.2	2.0	1.1	5030	1935	1605	5490	3450	3.3	34.5	30.3	0.05-0.30	D.S. Red Br.
II	6.6	1.9 Na ₂ Cr ₂ O ₇ · 2 H ₂ O(47); 35.4 SO ₄ ⁻² (5); X	—	11.3	17	1.2	2.1	4.0	2.1	0.72	3100	1540	1362	5220	3620	2.7	29.2	26.7	—	—
IIA	4.8	1.2 Na ₂ Cr ₂ O ₇ · 2 H ₂ O(47); 55.4 H ₂ C ₂ O ₄ (2); 43.5 SO ₄ ⁻² (5)	14.9	6.7	16	7.2	-1.8	-3.7	-1.9	-0.33	3100	1362	1423	3620	4100	-2.0	26.7	27.4	—	Removed
86 I	6.4	W	11.8	21.5	16	0.22	0.26	0.88	0.42	0.47	2980	1505	1288	5170	3280	1.7	29.2	26.2	—	—
IIA	3.4	53.6 H ₂ C ₂ O ₄ (2); 37.5 SO ₄ ⁻² (5); X	—	9.1	14	-0.82	0.96	-1.8	-1.2	-1.7	2980	1288	1433	3280	4430	-1.6	26.2	28.6	—	Red. Br.
II	6.4	1.9 Na ₂ Cr ₂ O ₇ (2); 27.5 SO ₄ ⁻² (5)	11.5	21.5	15	0.19	0.37	1.2	0.46	0.74	6410	2103	1625	5460	3090	2.0	37.6	31.5	—	—
IIA	3.5	17 H ₂ C ₂ O ₄ (2); 1.9 Na ₂ Cr ₂ O ₇ (2); 87.5 SO ₄ ⁻² (5); X	15.1	9.1	13	-0.75	-1.5	-2.6	-1.4	-1.5	6410	1625	1820	3090	3880	-5.0	31.5	33.9	—	Red Br.
87 I	6.5	1.9 Na ₂ Cr ₂ O ₇ (2); 25.8 SO ₄ ⁻² (5)	15.1	38.0	14	0.13	0.29	0.40	0.26	0.29	2570	1380	1198	5010	3230	0.93	27.4	24.4	0.01-0.07	D.S. Red Br.
II	6.5	W	14.4	38.0	14	0.21	0.23	1.7	0.50	0.59	6310	2090	1485	5420	2630	1.4	37.6	27.2	0.03-0.08	D.S. Red Br.
88 I	6.5	1.9 Na ₂ Cr ₂ O ₇ (2); 30.7 SO ₄ ⁻² (5)	14.8	9.1	13	0.0	0.42	0.61	0.30	0.92	3220	1562	1373	5110	3520	2.5	30.9	27.4	0-0.22	D.S. Red Br.
II	6.4	1.9 Na ₂ Cr ₂ O ₇ (2); 30.7 SO ₄ ⁻² (5)	14.9	12.6	14	0.43	1.4	4.0	1.4	1.0	5870	2010	1742	5390	3720	3.4	36.9	33.4	0.02-0.20	D.S. Red Br.
89 I	6.5	W	16.2	26.6	14	0.17	0.21	0.93	0.36	0.54	5920	2045	1652	5390	3310	1.4	36.9	31.5	0-0.08	D.S. Red Br.
II	6.5	W	12.1	26.6	14	0.0	0.19	1.9	0.45	0.54	7410	2205	1752	5460	2320	1.5	38.6	32.7	0.01-0.12	D.S. Red Br.

Under Chemical Conditions:-

Figure in parenthesis indicates point of reagent addition, e.g. (s) added at storage (p) added at specimen thru 1st piezometer ring. (1), (2) etc. - time lag in seconds from addition point to specimen.

All runs have $2 \times 10^{-5} N$ H₂O₂ added 1 sec. before specimen except those designated by X.

W = ca. 35 ppm SO₄⁻² added at storage and 2 ppm Na₂Cr₂O₇ · 2 H₂O added 43 or 47 sec. before spec.

Under Film Description:-

D-Dense. Dk-Dark. T-Tenacious. Lt-Light S-Stable. Br-Brown F-Fine. Gr-Green L-Loose.

Run and Section	Chemical Conditions		Conduc- tivity 10 ⁻⁵	Time of Run Hours	Inlet Temp. °C	Percent Increase per Hour				ΔT % Dec. per Hour	h _s	U		h _w		h _w % Dec. Hour	Kilowatts per Foot		Film		Legend
	pH(s)	Special Reagents - ppm				1/3	2/3	3/3	Overall			Start	Stop	Start	Stop		Start	Stop	Mils per Hour	Description	
90+91 I	6.4	1.9 Na ₂ Cr ₂ O ₇ (43); 3.5 SO ₄ ²⁻ (5); 13.2 Elon(2); X	18.8	29.9	14	0.15	0.53	0.97	0.50	0.70	5950	2050	1500	5390	2750	1.6	36.9	29.2	0-0.13	D, Sp. Red Br.	Under Chemical Conditions:- Figure in parenthesis indicates point of reagent addition, e.g.: (s) added at storage (1), (2) etc. - time lag in seconds from addition point to specimen. All runs have 2x10 ⁻⁵ N H ₂ O ₂ added 1 second before spec. except those designated by X. W = ca. 35 ppm SO ₄ ²⁻ add- ed at storage and 2 ppm Na ₂ C ₂ O ₄ ·2H ₂ O add- ed 43 or 47 seconds before specimen.
II	6.4	1.9 Na ₂ Cr ₂ O ₇ (47); 3.5 SO ₄ ²⁻ (5); X	18.1	29.9	14	0.67	0.53	1.40	0.80	0.94	7460	2210	1448	5460	2370	1.9	38.6	27.9	0.02-0.13	D, S, Red Br.	
92 I	6.5	1.9 Na ₂ Cr ₂ O ₇ (43); 3.2 SO ₄ ²⁻ (5); X	16.5	41.5	15	0.24	0.41	0.55	0.38	0.39	3030	1518	1200	5130	2700	1.1	29.2	24.4	0.01-0.08	D, S, Red Br.	
II	6.4	1.9 Na ₂ Cr ₂ O ₇ (47); 3.2 SO ₄ ²⁻ (5); 13.4 Elon(2); X	17.4	41.5	15	0.12	0.60	0.91	0.50	0.55	5810	2040	1450	5420	2610	1.2	36.3	27.9	0.01-0.14	D, C, Dk. Red Br.	
93 I	6.5	2.5 Na ₂ Cr ₂ O ₇ (43); 0.12 Cr ³⁺ (2); 3.6 SO ₄ ²⁻ (5); 2.7 Meado(1); X	17.6	12.2	18	1.10	1.30	1.40	2.10	1.80	5000	1935	1340	5490	2430	4.6	34.6	26.7	0.07-0.36	D, T, V, Dk. Br.	
II	6.5	1.9 Na ₂ Cr ₂ O ₇ (47); 0.13 Cr ³⁺ (2); 3.6 SO ₄ ²⁻ (5); X	17.3	12.2	17	0.0	1.10	3.80	1.30	2.10	6250	2100	1387	5510	2350	4.7	36.3	27.4	0.07-0.41	D, S, Lt. Br.	
94 I	6.6	0.13 Cr ³⁺ (2); 10.1 Meado(143); W; X	14.0	8.5	17	1.10	0.66	1.80	1.10	0.50	2850	1470	1345	5170	3880	2.9	27.9	26.7	0.01-0.27	D, V, Dk. Br.	
II	6.5	0.13 Cr ³⁺ (2); W;	13.7	8.5	16	1.20	1.50	3.90	2.10	1.70	4570	1850	1495	5340	3180	4.8	33.4	28.6	0.01-0.44	D, Lt. Br.	
97 I	7.4	0.13 Cr ³⁺ (2); W	26.0	28.8	18	0.58	1.64	3.19	1.51	1.18	3050	1800	1060	5270	2060	2.1	33.3	21.8	0.02-0.16	D, Lt. Gr. Br.	
II	7.2	0.11 Cr ³⁺ (2); W	—	20.0	18	0.47	1.50	2.90	1.40	1.40	3900	1735	1190	5360	2080	3.1	31.5	22.6	—	—	
IIA	7.8	9.7 (NH ₄) ₂ S ₂ O ₈ (40); W; X	29.0	5.5	18	1.60	0.0	0.97	0.80	0.69	3900	1140	1114	2080	1970	0.1	22.6	22.0	0.09-0.85	D, Lt. Br. Gr.	
98 I	7.6	0.11 Cr ³⁺ (2); W	27.0	40.3	19	0.45	0.62	2.20	0.85	0.85	2700	1440	869	5270	1550	1.76	27.9	17.8	—	—	
IA	7.7	19.0 H ₂ O ₂ (2); W	26.0	6.0	20	-0.65	-0.83	-5.30	-2.00	-4.30	2700	869	1130	1550	2630	-11.7	17.8	22.6	—	—	
98 II	7.5	0.14 Cr ³⁺ (2); W	29.2	9.5	19	0.0	3.10	5.70	2.00	1.90	4950	1930	1480	5510	2970	4.9	33.9	27.9	—	—	
IIA	7.7	10.2 H ₂ O ₂ (2); W	—	7.5	18	-1.20	-3.10	-5.30	-3.10	-2.30	4950	1480	1845	2970	4900	-8.7	27.9	32.7	—	—	
101 I	7.9	0.13 Cr ³⁺ (2); 0.05 Fe ³⁺ (35); 2 H ₂ SO ₄ (35); W	29.0	37.5	25	0.19	1.30	2.10	1.00	1.23	3895	1695	1303	5620	1187	2.1	30.8	16.7	—	—	
IA	7.8	39.5 H ₂ O ₂ (2); W	—	6.3	24	-1.10	-1.10	-4.30	-2.20	-5.70	3895	795	1190	1187	2335	-15.4	16.7	22.8	—	—	
101 II	7.9	0.13 Cr ³⁺ (2); 0.05 Fe ³⁺ (39); 2 H ₂ SO ₄ (39); W	29.0	37.5	25	1.10	1.60	1.20	1.30	1.20	3800	1660	784	5580	1170	2.1	29.6	18.7	—	—	
IIA	7.8	13.1 H ₂ O ₂ (2); W	—	6.3	24	0.0	-4.00	-3.70	-2.50	-8.30	3800	784	1320	1170	2970	-25.0	16.7	25.3	—	—	
102 I	7.8	0.08 Cr ³⁺ (2); 0.05 Fe ³⁺ (35); 2 H ₂ SO ₄ (35); W	32.4	33.0	24	0.76	1.50	2.40	1.50	1.10	3080	1510	855	5500	1460	2.2	28.3	17.9	—	—	
IA	7.8	9.1 H ₂ O ₂ (2); W	—	7.1	24	-0.75	-2.80	-4.70	-2.90	-3.90	3080	855	1150	1450	2560	-10.8	17.9	22.8	—	—	
102 II	7.8	0.08 Cr ³⁺ (2); 0.05 Fe ³⁺ (39); 2 H ₂ SO ₄ (39); W	32.0	33.0	25	0.50	1.80	1.80	1.30	1.18	3680	1655	908	5600	1485	2.2	30.2	18.5	—	—	
IIA	7.8	15.2 H ₂ O ₂ (2); W	—	7.1	24	-1.00	-4.40	-3.50	-3.10	-7.50	3680	908	1520	1485	4300	-9.2	18.5	28.3	—	No Film	
103+104 I	3.5	18.4 Ca ²⁺ (43); 4.5 H ₂ C ₂ O ₄ (2); W	29.0	10.3	24	0.66	0.0	0.0	0.0	0.0	4030	1720	1720	5600	5600	0.0	31.4	31.4	—	No Film	
II	3.5	1.1 Ca ²⁺ (47); 4.5 H ₂ C ₂ O ₄ (2); W	29.0	12.0	24	0.0	0.0	0.0	0.0	0.01	4260	1765	1760	5660	5650	0.0	32.7	31.4	—	White Gran.	
105 I	4.8	16.5 Ca ²⁺ (43); 4.4 H ₂ C ₂ O ₄ (2); W	21.0	19.5	24	0.42	1.00	0.0	0.50	0.43	3240	1550	1380	5510	3030	1.6	28.9	26.5	—	White Gran.	
II	4.1	16.5 Ca ²⁺ (47); 4.7 H ₂ C ₂ O ₄ (2); W	24.0	14.5		No Calculations															
106 I	7.9	17.5 Ca ²⁺ (43); 20.75 O ₂ (35); W	46.0	5.0	24	3.30	5.30	17.0	7.70	0.91	3120	1420	1330	4350	3620	3.3	27.1	25.9	—	Brittle White	
II	7.9	17.5 Ca ²⁺ (47); 9.9 S ₂ O ₈ (39); W	43.0	5.0	24	3.10	6.30	16.7	8.60	0.42	3940	1610	1550	4720	4240	2.0	29.6	28.9	—	Brittle White	
107 I	7.3	18.4 Ca ²⁺ (43); 38.6 S ₂ O ₈ (35); W	42.0	2.6	24	0.0	0.0	22.1	5.00	2.6	Not Calculated									Brittle White	
II	7.2	18.9 Ca ²⁺ (47); W	43.0	2.3	24	3.00	7.00	15.5	9.40	3.2	Not Calculated									Brittle White	
108 I	7.4	0.07 Cr ³⁺ (2); 4.5 Na ₂ H ₂ P ₂ O ₇ (35); W	28.0	48.7	24	0.35	0.0	0.29	0.21	0.13	3060	1510	1380	5490	4120	0.5	28.3	26.5	—	F, T, Red Br.	
II	7.4	0.03 Cr ³⁺ (2); 3.6 Na ₂ H ₂ P ₂ O ₇ (39); W	28.0	48.7	24	0.0	0.35	0.68	0.35	0.13	3620	1635	1570	5550	4370	0.4	30.2	28.3	—	F, S, Red Gr.	
109 I	7.5	0.074 Cr ³⁺ (2); 1.03 Na ₂ H ₂ P ₂ O ₇ (35); W	32.0	55.0	23	0.0	0.36	0.52	0.25	0.24	3010	1512	1322	5445	3160	0.8	28.3	24.7	—	F, S, Red Br.	
II	7.5	0.028 Cr ³⁺ (2); 0.85 Na ₂ H ₂ P ₂ O ₇ (39); W	31.0	55.0	23	0.17	0.22	0.18	0.18	0.10	3250	1545	1370	5460	3740	0.6	28.9	26.5	—	F, S, Gr. Br.	
110 I	7.5	0.073 Cr ³⁺ (2); W	33.0	50.2	23	0.33	0.40	1.65	0.66	0.82	3510	1610	854	5525	1370	1.5	30.2	17.9	—	D, S, Gr.	
II	7.5	0.03 Cr ³⁺ (2); W	33.0	50.2	23	0.60	0.40	0.60	0.53	0.58	4250	1760	1150	5000	2085	1.25	32.0	22.8	—	D, S, Gr.	
111 I	7.5	16.6 Ca ²⁺ (43); 5.2 Mg ²⁺ (43); 39.5 O ₂ (35); W	42.0	4.3	23	6.50	4.20	14.5	7.70	4.40	2930	1320	1065	3830	2250	9.6	25.8	21.0	—	Brittle White	
II	7.5	15.7 Ca ²⁺ (47); 5.1 Mg ²⁺ (47); W	41.0	4.0	23	5.00	12.5	20.0	12.5	3.30	3240	1440	1225	4640	2550	9.7	27.7	24.0	—	Brittle Br. Wh.	

Run and Section	Chemical Conditions		Conductivity 152x10 ⁶	Time of Run Hours	Inlet Temp. °C	ΔP				ΔT % Dec. per Hour	h _s	U		h _w		h _w % Dec. per Hour	Kilowatts per Foot		Film Description
	pH(s)	Special Reagents-ppm				1/3	2/3	3/3	overall			start	stop	start	stop		start	stop	
112 I	8.9	5.9 Mg ⁺⁺ (43); 35 SiO ₂ (35); W	41.0	39.8	23	0.32	1.15	1.44	0.90	0.94	2835	1445	1040	5430	2260	2.25	27.7	21.0	Brittle White
II	9.0	5.9 Mg ⁺⁺ (47); W	41.0	39.8	23	0.0	1.54	1.92	0.96	1.72	3200	1540	760	5470	1180	3.01	29.0	16.0	Brittle Red White
113 I	7.5	0.13 Cr ⁺³ (2); 1.87 (NH ₄) ₂ S ₂ O ₈ (5); W	30.0	32.4	24	0.77	0.84	1.20	1.10	1.10	2880	1460	910	5450	1670	2.10	27.1	17.2	F.S.L. Gr.
II	7.4	0.15 Cr ⁺³ (2); W	30.0	32.4	24	0.28	0.84	1.50	0.83	1.10	3180	1535	872	5510	1475	2.30	28.3	17.9	F.S.L. Gr.
114 I	9.4	18.5 Ca ⁺⁺ (43); 41 SiO ₂ (35); W	50.0	10.0	24	0.30	1.30	27.5	11.0	1.80	2960	1480	1140	5460	2570	5.3	28.4	22.2	Brittle White
II	9.3	18.6 Ca ⁺⁺ (47); W	44.0	5.0	24	1.80	51.2	24.4	24.1	4.40	2980	1485	1100	5460	2230	11.8	27.7	21.6	Brittle Red White
115 I	7.4	0.14 Cr ⁺³ (2); 4.6 (NH ₄) ₂ S ₂ O ₈ (5); W	32.0	28.9	25	0.90	1.10	2.70	1.50	1.10	2840	1385	873	5460	1635	2.4	26.5	17.9	F.S.L. Gr.
II	7.4	0.14 Cr ⁺³ (2); 9.5 (NH ₄) ₂ S ₂ O ₈ (5); W	—	29.3	25	1.40	1.60	0.90	1.30	1.50	2840	1445	720	5460	1130	2.7	26.5	14.8	F.S.L. Gr.
116 I	6.4	W; 5.5 (NH ₄) ₂ S ₂ O ₈ (5)	25.5	55.3	24	0.16	0.0	1.10	0.27	0.16	2900	1465	1295	5460	3690	0.6	27.7	25.3	F, Red Br.
II	6.4	W	21.5	55.3	24	0.18	0.18	0.16	0.18	0.24	3180	1535	558	5470	3070	0.8	28.3	24.6	F, Red Br.
117 I	6.5	4.6 (NH ₄) ₂ S ₂ O ₈ ; 30.4 SO ₄ ²⁻ (s)	18.0	50.5	24	0.17	0.44	0.34	0.30	0.27	3060	1505	1170	5460	2680	1.0	27.7	24.0	F.S. Red Br.
II	6.5	30.4 SO ₄ ²⁻ (s)	18.0	50.5	24	0.0	0.44	0.50	0.28	0.25	3390	1590	1300	5540	3120	0.87	29.6	25.9	F.S. Red Br.
118 I	6.4	27.6 SiO ₂ (35); 4.5 (NH ₄) ₂ S ₂ O ₈ (5); W	17.0	46.2	20	0.0	0.0	0.0	0.0	0.14	3060	1495	1390	5320	4200	0.45	28.9	27.1	Thin strip Red Br.
II	6.4	29.1 SiO ₂ (39); W	16.0	46.2	20	0.0	0.0	0.24	0.08	0.09	3400	1570	1500	5340	4600	0.30	27.6	28.3	Thin strip Red Br.
119 I	6.4	0.13 Cr ⁺³ (2); 4.9 (NH ₄) ₂ S ₂ O ₈ (5); W	17.0	30.7	22	0.59	0.90	3.30	1.20	1.20	2560	1365	775	5320	1340	2.4	26.5	16.7	F.S. Red Br.
II	6.4	0.14 Cr ⁺³ (2); W	16.0	30.7	22	0.30	0.36	0.72	0.46	0.75	2600	1375	1000	5320	2190	1.9	20.5	20.3	F.S. Red Br.
120 I	6.3	0.25 Fe ⁺² (35); 5.0 (NH ₄) ₂ S ₂ O ₈ (5); W	16.0	17.5	22	3.40	2.40	12.3	4.90	2.00	2520	1350	830	5320	1530	4.1	26.5	17.3	D.S. Red Br.
II	6.3	0.26 Fe ⁺² (39); W	16.0	17.5	22	1.80	2.90	4.70	3.10	1.60	3000	1485	990	5390	1920	3.7	28.3	20.3	D.S. Red Br.
121 I	6.4	0.16 Cr ⁺³ (2); 30 SiO ₂ (35); 5.6 (NH ₄) ₂ S ₂ O ₈ (5); W	18.0	14.8	22	0.0	0.75	0.0	0.25	1.10	3120	1520	1210	5420	2850	3.2	28.9	24.0	F.S. Br. Gr.
II	6.5	0.15 Cr ⁺³ (2); 30 SiO ₂ (39); W	17.0	14.8	22	0.0	0.85	0.0	0.24	0.59	3000	1500	1320	5890	3620	2.2	28.3	25.9	F.S. Br. Gr.
122 I	6.6	0.14 Cr ⁺³ (2); 27 SiO ₂ (35); 4.4 (NH ₄) ₂ S ₂ O ₈ (5); W	17.0	31.9	22	0.0	0.0	0.63	0.12	0.82	2940	1420	1010	5390	2020	2.0	28.3	20.9	F.S. Br. Gr.
II	6.5	0.14 Cr ⁺³ (2); 29 SiO ₂ (39); W	17.0	31.9	21	0.0	0.0	0.39	0.11	0.60	3160	1520	1340	5360	3000	1.6	28.9	23.4	F.S. Br. Gr.
124 I	4.4	44 Calgon(35); 15 Ca ⁺⁺ (43); 48 H ₂ C ₂ O ₄ (2); W	25.0	21.0	15	0.0	0.0	0.0	0.0	0.09	3450	1570	1520	5170	4700	0.43	31.5	30.8	No Film
II	4.4	18 Calgon(39); 15 Ca ⁺⁺ (47); 44 H ₂ C ₂ O ₄ (2); W	21.0	21.0	15	0.0	0.0	0.60	0.14	0.10	3020	1460	1400	5080	4390	0.65	28.9	28.3	No Film
125 I	6.4	0.18 Fe ⁺² (5); W	15.0	30.3	15	0.55	0.91	3.50	1.40	1.10	2830	1415	885	5080	1610	2.3	28.9	19.4	—
IIA	4.5	18 Calgon(35); 9.8 Ca ⁺⁺ (43); 50 H ₂ C ₂ O ₄ (2); W	19.0	9.8	16	0.0	-0.79	-1.60	-0.27	-2.00	2830	885	1050	1610	2260	-4.2	19.4	22.8	D.S. Red Br.
125 II	6.5	0.17 Fe ⁺² (5); W	15.0	30.3	15	0.0	1.10	2.00	0.89	1.20	3000	1460	885	5110	1560	2.3	29.6	19.4	—
IIA	4.6	50 Calgon(39); 9.8 Ca ⁺⁺ (47); 43 H ₂ C ₂ O ₄ (2); W	22.0	9.8	16	0.0	-0.79	-0.79	-0.17	-0.79	3000	885	960	1560	1810	-1.6	19.4	21.0	D.S. Red Br.

Under Chemical Conditions:-

Figure in parenthesis indicates point of reagent addition, e.g.:

(s) added at storage.

(p) added at specimen.

(1), (2), etc. - time lag in

seconds from addition point to spec.

All runs have 2×10^{-5} N H₂O₂ added 1 sec. before spec.

W = ca. 35 ppm SO₄²⁻ added at storage and 2 ppm

Na₂Cr₂O₇·2H₂O added 43 or 47 sec. before specimen.

*Cr₂(SO₄)₃ made up with acid (H₂SO₄) to keep Cr⁺⁺⁺ ionic.

Under Film Description:

D-Dense. Br.-Brown.

F-Fine. Gr.-Green.

S-Stable. Wh.-White.

Sl.-Slimy.

May 30, 1944

SECRET

To: V. D. Donihee
From: R. C. Olson

Run No. 74 will be as follows: -

Purpose: This run is to determine if $\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$ added 43 seconds before the specimen will affect Cr^{+++} film formation with $\text{Cr}_2(\text{SO}_4)_3$ added 2 seconds before the specimen. The water has been adjusted to 35 ppm $\text{SO}_4^{=}$.

<u>Conditions:-</u>	<u>Both Sections</u>	<u>Desired</u>	<u>Obtained</u>
Water - - - - -	From demineralizer	-	
Water Velocity - - - - -	20 ft./sec.	20 ft./sec.	
pH - - - - -	6.5 adjusted at storage	6.43	
Chlorides - - - - -	0 - 1 ppm non-added	0.7	
Peroxide - - - - -	$2 \times 10^{-5}\text{N}$	2.3	
Steam Pressure Inlet - - - - -	50 lbs./sq.in.	50 lbs./sq.in.	
Water Pressure Inlet - - - - -	85 lbs./sq.in.	85 lbs./sq.in.	
Water Temperature Inlet - - - - -	As from anionic	15°C	
Water Temperature Outlet - - - - -	Variable	-	
$\text{SO}_4^{=}$ from Na_2SO_4 added at storage - - - - -	35 ppm	33.1 ppm	
(Section I) Cr^{+++} from $\text{Cr}_2(\text{SO}_4)_3$ - - - - -	0.35 ppm*	0.36 ppm	
$\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$ added 43 seconds before specimen - - - - -	2.0 ppm	2.0 ppm	
(Section II) Cr^{+++} from $\text{Cr}_2(\text{SO}_4)_3$ - - - - -	0.35 ppm*	0.36 ppm	

*Addition through Saran 2 seconds before specimen.

TECHNICAL DIVISION
Engineering Development Section

ACC:kb

R. C. Olson

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CORROSION ENGINEERING DEVELOPMENT

Date: May 20, 1944

Section No. II - Run No. 74

Time	Water									Water			Steam				Solution Addition
	Flow in. Hg.	Pressures, psi.				Delta Pressures				Temp. °C			Press. psi		Temp. °C		
		In	1/3	2/3	Out	1/3	2/3	3/3	ΔP	In	Out	ΔT	In	Out	In	Out	
4:30 PM	9.1	85	60	42	30	25	18	12	55	15	67	52	50	28	148	132	H ₂ O ₂ (1 sec.) anal. for at half hour intervals and addition was adjusted accordingly.
5:00 PM	9.5	85	62	44	32	23	18	12	53	15	67	52	50	28	148	132	
5:30 PM	9.5	85	62	44	32	23	18	12	53	15	67	52	50	28	148	132	
6:00 PM	9.5	85	64	45	34	21	19	11	51	15	67	52	50	31	150	133	
6:30 PM	9.5	85	64	44	33	21	20	11	52	15	67	52	50	32	153	133	
7:00 PM	8.5	85	64	44	33	21	20	11	52	15	67	52	50	32	153	133	
7:30 PM	8.5	85	64	44	33	21	20	11	52	15	67	52	50	32	153	133	
8:00 PM	9.5	85	64	44	33	21	20	11	52	15	66	51	50	32	150	133	
8:30 PM	9.5	85	64	44	33	21	20	11	52	15	66	51	50	32	150	133	
9:00 PM	9.5	85	63	44	33	22	19	11	52	15	65	50	50	31	149	133	
9:30 PM	9.5	85	63	44	32	22	19	12	53	15	65	50	50	32	150	133	
10:00 PM	9.5	85	63	44	32	22	19	12	53	15	65	50	50	32	150	133	
10:30 PM	9.5	85	63	44	32	22	19	12	53	15	65	50	50	32	150	133	
11:00 PM	9.5	85	63	44	31	22	19	13	54	15	63	48	50	29	147	133	
11:30 PM	9.3	85	63	43	30	22	20	13	55	14	63	49	50	31	149	133	

Recorded by: T. Wayne Perry

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Corrosion Engineering Development
Record of Analysis

P.D. No. 74

May 20 1944

Time	Section I			Section II			Treater House				Remarks
	pH	Table H ₂ O ₂	Cl	pH	Table H ₂ O ₂	Cl	An. pH	An. Cl ⁻	Sto. pH	Sto. Cl ⁻	
Standard	6.50	2x10 ⁻⁵ N	0-1	6.50	2x10 ⁻⁵ N	0-1	-	0-1	6.20	0-1	H ₂ O ₂ *
5:00 PM	6.41	-	0.6	6.40	3.47	0.4	6.02	0.3	6.18	0.2	
5:30 PM											
6:00 PM	6.35	-	0.3	6.33	2.37	0.3	5.54	0.2	6.11	0.2	
6:30 PM											
7:00 PM	6.39	-	0.3	6.42	2.12	0.2	5.71	0.2	6.20	0.15	
7:30 PM											
8:00 PM	6.39	-	0.3	6.40	1.88	0.2	5.83	0.6	6.18	0.6	
8:30 PM											
9:00 PM	6.48	-	0.9	6.54	1.71	0.9	5.88	1.1	6.30	1.1	
9:30 PM											
10:00 PM	6.44	-	2.0	6.51	1.27	1.8	5.80	2.4	6.28	2.3	
10:30 PM											
11:00 PM	6.31	-	0.8	6.37	2.67	0.8	5.60	0.2	6.11	0.15	

Recorded by: Benjamin Struminski

* Analyses for Section I from May 20, 5:00 PM to May 21, 7:00 AM were not made because of dichromate interference. H₂O₂ readings after 7:00 AM have been corrected for the dichromate interference.

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Corrosion Engineering Development
Record of Analysis

Exhibit A-3

May 21, 1944

P.D. No. 74

Time	Section I			Section II			Treater House				Remarks
	pH	Table H ₂ O ₂	Cl	pH	Table H ₂ O ₂	Cl	An. pH	An. Cl ⁻	Sto. pH	Sto. Cl ⁻	
Standard	6.50	2x10 ⁻⁵ N	0-1	6.50	2x10 ⁻⁵ N	0-1	-	0-1	6.2	0-1	pH meter standard- ized every 3 hours.
12:00 M	6.35	-	0.50	6.37	1.65	0.50	5.60	0.50	6.12	0.50	
12:30 AM											
1:00 AM	6.10	-	1.8	6.20	2.03	1.2	5.50	1.0	5.83	1.0	
1:30 AM											
2:00 AM	6.20	-	2.0	6.21	3.1	1.8	5.50	1.5	5.90	1.5	
2:30 AM											
3:00 AM	6.30	-	2.0	6.20	1.5	1.6	5.60	1.6	5.87	1.3	
3:30 AM											
4:00 AM	6.15	-	1.3	6.20	1.6	2.3			5.90	0.50	
4:30 AM							5.0	0.75			
5:00 AM	6.00	-	1.3	5.90	2.12	0.5	5.20	0.25	5.70	0.25	
5:30 AM											
6:00 AM	6.55	-	1.6	6.55	3.1	0.51	5.35	0.25	6.60	0.25	
6:30 AM											
7:00 AM	6.40	-	1.0	6.50	3.1	0.20	5.20	0	6.00	0	
Recorded by: Walter L. Spear											
8:00 AM	6.80	2.38	0.6	6.75	5.50	0	5.80	1.0	10.0	0.2	
8:30 AM											
9:00 AM	6.60	1.43	0.2	6.40	1.54	0.2	5.40	0.5	6.15	0	
9:30 AM											
10:00 AM	6.55	1.38	0	6.40	1.62		5.32	0.2	6.02	0	
10:30 AM											
11:00 AM	6.55	2.30	0	6.45	1.65	0	5.10	0	6.10	0	
11:30 AM											
12:00 N	6.55	0.63	0	6.42	1.48	0	5.60	0	6.22	0	
12:30 PM											
1:00 PM	6.60	1.76	0	6.52	2.03	0	5.62	0.5	6.20	0.2	
1:30 PM											
2:00 PM	6.65	1.87	1.3	6.50	2.46	1.0	5.32	0.7	6.12	0.7	
2:30 PM											
3:00 PM	6.65	1.21	1.1	6.50	2.20	0.9	5.30	0.6	6.12	0.7	
Recorded by: Robert L. East											
4:00 PM	6.50	0.83	1.1	6.53	2.03	1.1	5.53	1.1	6.29	1.1	
4:30 PM											
5:00 PM	6.44	2.15	1.6	6.51	1.99	1.5	5.50	1.7	6.23	1.6	
5:30 PM											
6:00 PM	6.53	2.42	2.4	6.61	2.45	2.6	5.88	4.2	6.49	4.1	
Shutdown 6:30 PM Recorded by: Benjamin Struminski											

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CORROSION ENGINEERING DEVELOPMENT

Date: May 21, 1944

Section No. 11 - Run No. 74

Time	Flow in. Hg.	Water									Water			Steam				Solution Addition
		Pressures, psi.				Delta Pressures					Temp. °C			Press. psi.		Temp. °C		
		In	1/3	2/3	Out	1/3	2/3	3/3	ΔP	In	Out	ΔT	In	Out	In	Out		
12:00 PM	9.5	85	63	44	30	22	19	14	55	15	63	48	50	31	150	134	Cr ₂ (SO ₄) ₃ (2 sec.) and Na ₂ SO ₄ (storage) were added stoichiometrically with check of reagent flow rates being made at half hour intervals.	
12:30 AM	9.4	85	63	43	29	24	20	14	56	15	62	47	50	30	149	137		
1:00 AM	9.5	85	63	43	29	22	20	14	56	15	62	47	50	31	149	138		
1:30 AM	9.5	85	63	42	29	22	21	13	56	15	62	47	50	30	148	137		
2:00 AM	9.5	85	63	42	29	22	21	13	56	15	62	47	50	30	149	137		
2:30 AM	9.5	85	63	42	29	22	21	13	56	15	61	46	49	31	148	137		
3:00 AM	9.5	85	63	43	29	22	20	14	56	15	62	47	52	32	149	138		
3:30 AM	9.5	85	63	43	29	22	20	14	56	15	62	47	50	31	148	137		
4:00 AM	9.4	85	63	43	29	22	20	14	56	15	62	47	52	33	149	138		
4:30 AM	9.5	85	63	42	23	22	21	14	57	15	60	45	49	30	148	137		
5:00 AM	9.4	85	73	45	23	22	20	15	57	15	60	45	49	30	147	136		
5:30 AM	9.4	85	62	41	23	23	21	15	59	15	60	45	49	30	148	134		
6:00 AM	9.5	85	61	40	23	24	21	17	62	15	60	45	54	30	150	135		
6:30 AM	9.5	85	61	40	23	24	21	17	62	15	59	44	50	31	148	135		
7:00 AM	9.5	85	61	40	23	24	21	17	62	15	59	44	54	32	151	138		
7:30 AM	9.5	85	61	40	22	24	21	18	63	15	58	43	50	30	150	134		
Recorded by: Lyle G. Rulon																		
8:00 AM	9.4	85	60	38	20	25	22	18	65	15	59	44	50	30	151	134		
8:30 AM	9.4	85	60	38	20	25	22	18	65	15	59	44	50	30	152	136		
9:00 AM	9.5	85	60	38	20	25	22	18	65	14	59	45	50	30	151	135		
9:30 AM	9.5	85	60	38	19	25	22	19	66	14	59	45	50	30	152	135		
10:00 AM	9.5	85	60	38	19	25	22	19	66	14	59	45	50	30	153	134		
10:30 AM	9.5	85	60	38	19	25	22	19	66	14	59	45	50	30	152	133		
11:00 AM	9.5	85	60	38	19	25	22	19	66	14	57	43	50	30	153	137		
11:30 AM	9.5	85	60	38	19	25	22	19	66	14	58	44	50	30	152	135		
12:00 N	9.5	85	60	38	19	25	22	19	66	15	58	43	50	30	152	135		
12:30 PM	9.5	85	60	38	18	25	22	20	67	15	58	43	50	30	151	133		
1:00 PM	9.5	85	60	38	18	25	22	20	67	15	57	42	50	30	152	133		
1:30 PM	9.5	85	60	38	18	25	22	20	67	14	57	43	50	30	151	134		
2:00 PM	9.5	85	60	38	18	25	22	20	67	15	58	43	50	30	152	134		
2:30 PM	9.5	85	60	38	17	25	22	21	68	15	57	42	50	30	151	134		
3:00 PM	9.5	85	60	37	16	25	23	21	69	15	57	42	50	30	152	134		
3:30 PM	9.5	85	60	37	16	25	23	21	69	15	58	43	50	30	151	133		
Recorded by: G. R. Wilde																		
4:00 PM	9.5	85	60	37	16	25	23	21	69	15	58	43	50	30	151	133		
4:30 PM	9.5	85	60	37	16	25	23	21	69	15	58	43	50	30	151	133		
5:00 PM	9.5	85	60	37	15	25	23	22	70	15	54	39	50	30	147	133		
5:30 PM	9.5	85	60	37	15	25	23	22	70	15	55	40	50	30	148	133		
6:00 PM	9.5	85	60	38	15	25	22	23	70	15	55	40	50	30	147	133		

Shut Down

Recorded by: T. Wayne Perry

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SECTION #1
PRESSURE DROP & TEMPERATURE RISE CURVES
RUN NUMBER 74

Date- May 22, 1944
By- R. J. Rosenberg

LEGEND
— Total Pressure Drop
○ — Total Temperature Rise
□ — Pressure Drop 1st 3rd
△ — Pressure Drop 2nd 3rd
● — Pressure Drop 3rd 3rd

PRESSURE DROP ACROSS SPECIMEN LBS. PER SQ. IN.

TEMPERATURE RISE ACROSS THE SPECIMEN DEGREES C.

Robbison
May 22, 1948

SECTION #2
PRESSURE DROP & TEMPERATURE RISE CURVES
RUN NUMBER 74

Date May 22, 1944
By R. J. Rosenberg

LEGEND
 — Total Pressure Drop
 — Total Temperature Rise
 — Pressure Drop 1st 3rd
 — Pressure Drop 2nd 3rd
 — Pressure Drop 3rd 3rd

PRESSURE DROP ACROSS SPECIMEN LBS PER SQ. IN.

80
64
48
32
16
0

TEMPERATURE RISE ACROSS THE SPECIMEN DEGREES C.

Exhibit A-6
CE-2524
65

12:00MN 8:00AM 4:00PM 12:00MN 8:00AM 4:00PM 12:00MN 8:00AM 4:00PM 12:00MN
 5/20/44 5/21/44 5/22/44
 Time of Run

P.D. 74-SECTION II

0.35 ppm Cr^{+++} , High SDg water

RSD
7/2/74

Section 2 - Start Up

$$h_w = 150 (1 + 0.011 \times t_{av.}) \frac{v^{0.8}}{D^{0.2}}$$

$$= 150 (1 + 0.011 \times 105.8) \frac{10.95}{0.683}$$

$$= 2404 \times 2.164$$

$$= 5200 \left(\frac{1}{h_w} = 1.92 \times 10^{-4} \right)$$

$$Q = 21.0 \times 60 \times 8.33 \times 1.8 \times 52$$

$$= 18,900 \times 52$$

$$= 9.83 \times 10^5$$

$$Kw/ft. = \frac{Q}{3413 \times 9.33} = \frac{9.83 \times 10^5}{31800} = 30.94$$

$$\Delta t_m = \frac{67.1}{\ln \frac{212.5}{145.4}} = \frac{67.1}{\ln 1.461} = 176.8 \text{ } ^\circ\text{F}$$

$$U = \frac{9.83 \times 10^5}{3.38 \times 176.8} = 1645$$

$$\frac{1}{U} = \frac{1}{h_w} + \frac{1}{k} + \frac{1}{h_s}$$

$$\frac{1}{h_s} = \frac{1}{1645} - (1.35 \times 10^{-4} + 1.92 \times 10^{-4}) = (6.08 - 3.27) \times 10^{-4} = 2.81 \times 10^{-4}$$

$$h_s = 3560$$

$$T \text{ out water} = 67$$

$$^{\circ}\text{C} = 67$$

$$^{\circ}\text{C} = 152.6 \text{ } ^\circ\text{F}$$

$$T \text{ in water} = 15$$

$$^{\circ}\text{C} = 15$$

$$^{\circ}\text{C} = 59 \text{ } ^\circ\text{F}$$

$$\frac{52}{41 \text{ } ^\circ\text{C}} \quad \frac{2 \mid 82}{41 \text{ } ^\circ\text{C}} \quad \frac{2 \mid 211.6}{105.8 \text{ } ^\circ\text{F Av.}}$$

$$P \text{ in steam} = 50 + 15 = 65 \text{ psi} = 298 \text{ } ^\circ\text{F} \quad \text{Reading } 148 \text{ } ^\circ\text{C}$$

$$P \text{ out steam} = 28 + 15 = 43 \text{ psi} = 271.5 \text{ } ^\circ\text{F} \quad 132 \text{ } ^\circ\text{C}$$

$$298$$

$$152.6$$

$$145.4$$

$$271.5$$

$$59$$

$$212.5$$

$$145.4$$

$$67.1$$

Section 2 - Shut Down

$$Q = 18,900 \times 40$$

$$= 7.55 \times 10^5$$

$$Kw/ft. = \frac{7.55 \times 10^5}{21800} = 23.7$$

$$\Delta t_m = \frac{48.5}{\ln \frac{215.5}{167}} = \frac{48.5}{\ln 1.288} = 191.8 \text{ } ^\circ\text{F}$$

$$U = \frac{7.55 \times 10^5}{3.38 \times 191.8} = 1167$$

$$\frac{1}{h_w} = \frac{1}{1167} - (1.35 \times 10^{-4} + 2.81 \times 10^{-4})$$

$$\frac{1}{h_w} = (8.58 - 4.16) \times 10^{-4} = 4.42 \times 10^{-4}$$

$$h_w = 2260$$

$$T \text{ out water} = 55$$

$$^{\circ}\text{C} = 131 \text{ } ^\circ\text{F}$$

$$T \text{ in water} = 15$$

$$^{\circ}\text{C} = 59 \text{ } ^\circ\text{F}$$

$$40 \Delta t \text{ } ^\circ\text{C}$$

$$P \text{ in steam} = 50 + 15 = 65 \text{ psi} = 298 \text{ } ^\circ\text{F} \quad \text{Reading } 147 \text{ } ^\circ\text{C}$$

$$P \text{ out steam} = 30 + 15 = 45 \text{ psi} = 274.5 \text{ } ^\circ\text{F} \quad 133 \text{ } ^\circ\text{C}$$

$$298$$

$$131$$

$$167$$

$$274.5$$

$$59$$

$$215.5$$

$$167$$

$$48.5$$

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Delta Pressure and Temperature Calculations68
Exhibit A-6Run No. 74Date: May 23, 1944Δt Decrease

Δt at startup 52 °C Run time = 26.5 hours
 Δt at shutdown 40
 Δt decrease 12 °C

$$\% \Delta t \text{ decrease} = \frac{12 \times 100}{52} = 23$$

$$\% \Delta t \text{ decrease/hr} = \frac{23}{26.5} = 0.87$$

ΔP Increase

	1st 3rd	2nd 3rd	3rd 3rd	Total
ΔP at startup psi	25	18	12	55
ΔP at shutdown	<u>25</u>	<u>22</u>	<u>23</u>	<u>70</u>
ΔP increase psi	0	4	11	15

% Increase% Increase/hr.

1st 3rd =	$\frac{0}{25} \times 100 = 0$	$\frac{0}{26.5} = 0$
2nd 3rd =	$\frac{4}{18} \times 100 = 22$	$\frac{22}{26.5} = 0.83$
3rd 3rd =	$\frac{11}{22} \times 100 = 92$	$\frac{92}{26.5} = 3.5$
Total =	$\frac{15}{55} \times 100 = 27$	$\frac{27}{26.5} = 1.0$

$$\% \text{ Decrease } h_w = \frac{5200 - 2260 \times 100}{5200} = \frac{2940}{5200} \times 100 = 56.5$$

$$\% \text{ Decrease } h_w/\text{hr} = \frac{56.5}{26.5} = 2.1$$

Calculated by: John Mann

Recorded in log book #380B
Pages 25 and 27

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ENGINEERING DEVELOPMENT SECTION
Summary Sheet - Film Formation Studies at MUC
Run No. 74 Test Section No. I

(69)
Exhibit A-9

CE - 2524

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Duration of Run 26.5 Hrs. Start-up Date 5/20/44-4:00 PM Shut-down Date 5/21/44-6:30 PM								TUBE SPECIFICATION							
								Tube		Type	Length	I.D.	O.D.		
								Specimen		2S	8.54"	1.000"	1.384"		
Ribbed		2S	8.54"	1.547"	1.687"										

START-UP*								SHUT-DOWN*							
Pressures lbs/sq in				Pressure Drop				Pressures lbs/sq in				Pressure Drop			
In	1/3	2/3	Out	1/3	2/3	3/3	Ttl.	In	1/3	2/3	Out	1/3	2/3	3/3	Ttl.
85	70	48	34	15	22	14	51	85	70	44	15	15	26	23	70

INCREASE IN PRESSURE DROP OVER START-UP*												STEAM CONDITIONS	
Increase				Percent Increase				% Increase/hour					
1/3	2/3	3/3	Ttl.	1/3	2/3	3/3	Ttl.	1/3	2/3	3/3	Ttl.		
0	4	15	19	0	18.	107.	37.2	0.0	0.68	4.0	1.4	lbs. ga.	°C.

START-UP			SHUT-DOWN			Decrease In			In	50	150
Temperatures °C.			Temperatures °C.			Temperature Rise					
In	Out	T	In	Out	T	Amt.	%	%/hr.	Out	37	136
15	70	55	15	56	41	14	25	0.96			

		h_w	h_a	U	kw/hr/ft.	Shut-down						
Start-up		5270	3920	1723	32.7							
Shut down		2260		1200	24.4							
% Decrease		57										
% Dec./hr.		2.2				<table border="1" style="width:100%; border-collapse: collapse;"> <tr> <td>In</td><td>50</td><td>150</td></tr> <tr> <td>Out</td><td>35</td><td>138</td></tr> </table>	In	50	150	Out	35	138
In	50	150										
Out	35	138										

FILM MEASUREMENTS 9(in mils)***							ADDED REAGENTS		
	2ft.	2 1/2 ft.	4 1/2 ft.	6 1/2 ft.	8 1/2 ft.	Av.	Reagent	Pt. of Addn	Bulk Av.
Top	0.8	1.8	2.1	2.5	09.8	3.6	pH (Adjustment)	Storage	6.43
Side	0.5	1.7	1.8	1.5	1.5	1.5	H ₂ O ₂ N x 10 ⁻⁵	1 sec.	-
Side	0.7	1.2	1.8	2.2	2.2	2.9	Water gpm	- - -	20.0
Bottom	0.9	1.3	1.4	1.5	1.5	1.5	Cr ⁺⁺⁺	2 sec.	0.362ppm
Av.	0.7	1.5	1.5	1.9	1.9	2.4	Na ₂ Cr ₂ O ₇ · 2H ₂ O	43 sec.	2.01ppm
	Inlet		Av.		Outlet		SO ₄ ⁼	storage	33.1ppm

Film Formation Mils/hr.				.02	.09	.37
-------------------------	--	--	--	-----	-----	-----

TREATER HOUSE		
Anionic	pH 5.53	Cl-0.8) non-
Storage	pH 6.27	Cl-0.72) added
Amt. NaOH 224		gms./hr.

* At fractional distances from water inlet.

** Taken at indicated distances from water inlet.

= Seconds before entering specimen.

Duration of Run 26.5 Hrs. Start-up Date 5/20/44-4:00 PM Shut-down Date 5/21/44-6:30 PM								TUBE SPECIFICATION							
Tube				Type		Length		I.D.		O.D.					
Specimen				2S		8.46'		1.000"		1.324"					
Ribbed				2S		8.46'		1.5-8"		1.687"					

START-UP*								SHUT-DOWN*							
Pressures lbs/sq in				Pressure Drop				Pressures lbs/sq in				Pressure Drop			
In	1/3	2/3	Out	1/3	2/3	3/3	Ttl.	In	1/3	2/3	Out	1/3	2/3	3/3	Ttl.
85	60	42	30	25	18	12	55	85	60	38	15	25	22	23	70

INCREASE IN PRESSURE DROP OVER START-UP*												STEAM CONDITIONS	
Increase				Percent Increase				% Increase/hour					
1/3	2/3	3/3	Ttl.	1/3	2/3	3/3	Ttl.	1/3	2/3	3/3	Ttl.	Start-up	
0	4	11	15	0	22	92	27	0	0.83	3.5	1.0	lbs. ga. °C.	
START-UP				SHUT-DOWN				Decrease In Temperature Rise				In	
Temperatures °C.				Temperatures °C.				Ant. % %/hr.				Out	
In	Out	T		In	Out	T		Ant.	%	%/hr.		Shut-down	
15	67	52		15	55	40		12	23	0.87		lbs. ga. °C.	
				h _w h _s U				kw./hr./ft.				In	
Start-up				5200 3560 1645				30.9				Out	
Shut-down				2260 1167				23.7				In	
% Decrease				56.5								Out	
% Dec./hr.				2.1								In	
												Out	

FILM MEASUREMENTS (in mils)**							ADDED REAGENTS				
	1ft.	2ft.	4ft.	6ft.	8ft.	Av.	Reagent	Pt. of Addn	Date	Av.	Bulk Av.
Top	1.2	2.1	2.2	4.3	4.9	2.9	pH (adjustment)	storage	6.41		
Side	0.9	1.7	1.3	2.4	2.2	1.7	H ₂ O N x 10 ⁻⁵	1 sec.	2.3		3.6
Side	1.2	1.8	1.9	3.1	3.0	2.2	Water gpm				19.9
Bottom	1.0	1.5	1.7	1.6	2.2	1.6	Cr ⁺⁺⁺	2 sec.			0.36ppm
Av.	1.1	1.8	1.8	2.9	3.1	2.1	SO ₄ ⁼	storage			33.1ppm
			Inlet	Av.	Outlet						
Film Formation Mils/hr			.03	.08	.18						
TREATER HOUSE											
Anionic		pH 5.53	Cl- 0.8) non-								
Storage		pH 6.27	Cl- 0.72) added								
Amt. NaOH added		224	gms./hr.								

* At fractional distances from water inlet.
** Taken at indicated distances from water inlet.
= Seconds before entering specimen.

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Chemistry Group C-IV

G. E. Boyd

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REPORT ON ANALYSIS

Sample: Our No. 2323 Your No. Film Deposit EDC 33 P.D. No. 74 - Section I

Source: B. L. Vondra T-II

Date Received: 6/2/44

Constituent Determined	Amount Found	Analyst	Method of Analysis
CO ₂	18.9%	C. Carter	Gravimetric
Si	2.8%	H. Dyas	Gravimetric
Al	0.47% 0.55%	Z. Pojeta	Colorimetric
Fe	6.4%	K. Jensen	Colorimetric
Cr	24.9% 25.0%	W. Schaap	Volumetric

Reported by: R. W. Bane

Date: 6/16/44

Copies to: B. L. Vondra

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Chemistry Group C-IV

G. E. Boyd

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REPORT ON ANALYSIS

Sample: Our No. 2324 Your No. Film Deposit EDC 34 P.D. No. 74 - Section II

Source: B. L. Vondra T-II

Date Received: 6/2/44

Constituent Determined	Amount Found	Analyst	Method of Analysis
CO ₂	20.6%	C. Carter	Gravimetric
Si	1.9)% 1.9)	B. Holt	Gravimetric
Fe	6.0)% 5.8)	"	Colorimetric
Al	1.9)% 1.7)	"	Gravimetric
Cr	25.5)% 25.7)	"	Volumetric

Reported by: R. W. Bane

Date: 6/15/44

Copies to: B. L. Vondra

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Exhibit A-13

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Run 74 - Discussion

Film: Dense, stable, olive green

Both sections had 0.35 ppm Cr^{+++} added 2 seconds before the specimen to water containing 35 ppm $\text{SO}_4^{=}$ added at storage. In addition, 2 ppm $\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$ were added to Section I at a point 43 seconds before the specimen. The results show identical buildups in the presence of 35 ppm $\text{SO}_4^{=}$ as under like additions of Cr^{+++} and $\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$ with no additional $\text{SO}_4^{=}$ added to demineralized water. A tabulation of results for these Cr^{+++} and $\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$ runs follows: -

<u>Run No.</u>	<u>Cr^{+++}</u>	<u>$\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$</u>	<u>$\text{SO}_4^{=}$</u>	<u>%P/hr.</u>	<u>%H₂O/hr.</u>	<u>Mils/hr.</u>	<u>Hrs. Operation</u>
54 II	0.35 ppm	0	0	1.1	2.5	.04(.10).21	24.9
72 I	0.35 ppm	0	0	2.5	4.4	.03(.18).51	11.7
73 I	0.35 ppm	2 ppm	0	1.3	2.4	0(.09).31	11.7
73 II	0.34 ppm	0	0	1.0	1.7	0(.09).35	11.8
74 I	0.36 ppm	2 ppm	35 ppm	1.4	2.2	.02(.09).37	26.5
74 II	0.36 ppm	0	35 ppm	1.0	2.1	.03(.08).18	26.5

Except for Run 72, which had unusually high results, all runs indicate that the buildup to be expected with 0.35 ppm Cr^{+++} addition will be at a rate of 1 to 1.5% Δ P increase/hr. and an average of about 0.1 mils/hr. independent of the presence of $\text{SO}_4^{=}$ or $\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$. The presence of $\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$ shows, in each case, a slightly higher rate of Δ P rise than Cr^{+++} alone but for all practical purposes, this slight difference may be regarded as negligible. $\text{SO}_4^{=}$ in the water from storage does not display any effect but the addition of 35 ppm will be continued to keep the system more similar to "W" conditions.

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