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Report on
**SCALE DEPOSITION IN
WATER DISTRIBUTION SYSTEMS**
for
**STATE ELECTRICITY BOARD
ICELAND**

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Sheppard T. Powell
Consulting Chemical Engineer
Baltimore, Md.

October, 1946

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October 17, 1946

E. G. Eggertson, Inc.,
27 William Street,
New York 5, N. Y.

Dear Sirs:

We are transmitting herewith an original and two copies of our report in which is submitted our findings regarding the causes for the excessive scale deposition that is occurring in the various distribution systems in Iceland.

We have reviewed several water analyses and scale analyses with which you provided us at the time you were in our office. We also have had the scale sample from the one-half inch pipe and four inch pipe analyzed gravimetrically and by X-ray as you requested. These data have been assembled in the accompanying report and have formed the basis for the various conclusions which we have reached regarding the causes of scale deposition.

Due to the lack of complete information it has been impossible to make firm recommendations regarding the manner in which the various water supplies should be treated in order to correct the existing conditions. Therefore, we have proposed that a study be undertaken, by means of pilot plant equipment, to determine the scaling characteristics of the various supplies and the rate at which scale is formed in the several systems. In addition, we believe that close attention should be given to the matter of corrosion. Apparently this is quite severe, judging by the high iron oxide content of the several samples of scale and the samples of pipe which we inspected.

As you requested, we have prepared two reports, this one covering the matter of the proposed new heat exchangers to the Reykjavik Power Plant, the scale formations from the various waters throughout Iceland and some general conclusions regarding the difficulties in the City of Reykjavik. The second report is concerned primarily with the problems that have been encountered in the distribution systems supplying well water to Reykjavik. As a result, there is a certain amount of duplication in the two reports; however, the second report has been prepared

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in such a manner that it may be transmitted to the City if that is your desire and will cover only the problem encountered in the capital.

We shall be pleased to send you any further information which you may desire with regard to this problem.

Very truly yours,


Sheppard T. Powell

STP:k

REPORT ON
SCALE DEPOSITION IN WATER DISTRIBUTION SYSTEMS
FOR
STATE ELECTRICITY BOARD
ICELAND

In accordance with your request at the time of our meeting in my office on August 29th, I am writing you my views regarding the problems we discussed. As you requested, we have had the samples of scale you left with us analyzed by chemical analysis and by X-ray. There were two samples; one was deposited in a one-half inch diameter pipe carrying hot water, the other was deposited in a four-inch diameter pipe carrying both steam and hot water.

In reviewing the problem of deposit formation in the various pipe lines throughout Iceland, we find that it is necessary to make the following assumptions due to lack of complete information at this time: (a) That the analyses with which we have been provided are typical of the conditions existing in the particular systems from which they were taken; (b) That the scale found in the small pipe studied was similar to that found at Reykjavik; (c) That ground water contamination of this supply may have occurred and the latter condition was largely responsible for the scale which deposited in the four-inch diameter line, because it upset the

ionic balance of the water and caused precipitation of the calcium carbonate in large amounts. It is indicated, also, that oxygen has been entrained or absorbed by the water entering the distribution systems either at open storage tanks, through air lifts or from other unknown sources. The assembly of additional data will confirm or modify our findings predicated on the information supplied to us.

CONCLUSIONS

(1) The sample of scale from the one-half inch diameter pipe which was analyzed indicates that severe corrosion had occurred in the system. This probably was due to the presence of oxygen and the resulting iron oxide may have aided in the deposition of silica.

(2) The sample of scale from the four-inch diameter pipe which was analyzed indicates that the solubility of calcium carbonate was exceeded resulting in a heavy carbonate scale formation. We believe the water which was supposed to have caused the deposit was not representative of normal conditions that exist. Possibly, the quality of the water has been altered since taking the sample or contamination from an outside source has occurred to change the water characteristics.

(3) The condition which should occur, at the power plant, from reheating the Reykjavik town supply in heat exchangers may be less serious than originally anticipated. If the samples given us were typical, it is indicated that the major detriment to operation of the heat exchangers should be corrosion. The use of Admiralty tubes, naval brass tube sheets and welded steel shell should minimize the possibility of serious corrosion. The presence of

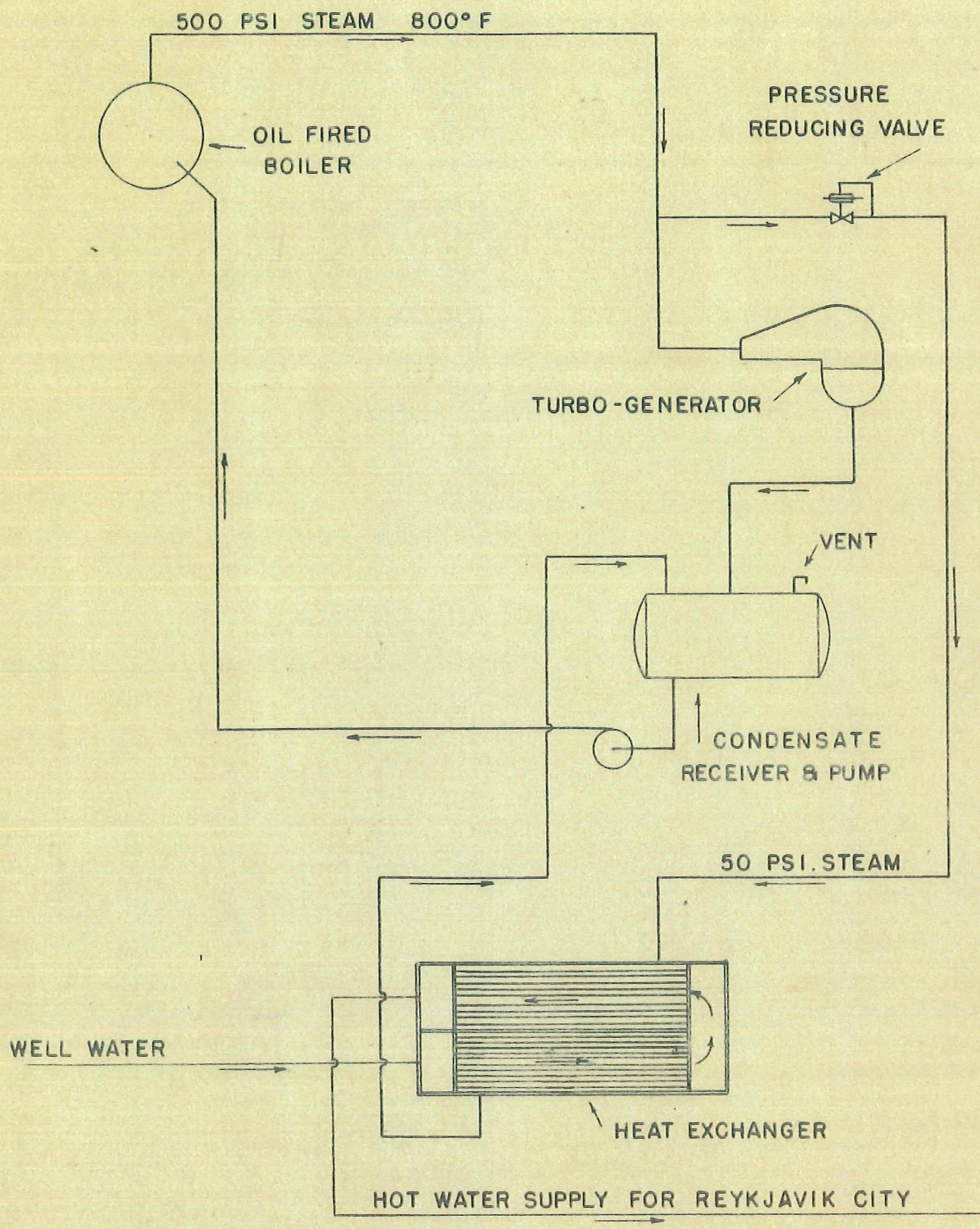
large amounts of iron oxide in the deposit indicates the heat exchangers could be cleaned with inhibited acid at off-peak periods if deposits, resulting from corrosion of the piping system, are precipitated in the exchangers.

(4) To assure the availability of the heat exchangers for reheating the town of Reykjavik heating supply, facilities and connections should be provided which will permit ready acid cleaning of the proposed heat exchangers in the event such exchangers are installed (Figures 1 and 3). If this plan should be followed we shall be pleased to forward information regarding the care that should be observed in carrying out this operation.

(5) An alternate plan to the use of the heat exchangers now proposed might be adopted if it is found on further investigation that excessive silica is deposited from the water supply. In this plan open coil heaters would be substituted for the heat exchangers. Steam for heating would pass through the coils and the water would flow on the outside. Thus, any silica deposit would be readily accessible for mechanical removal (Figure 2).

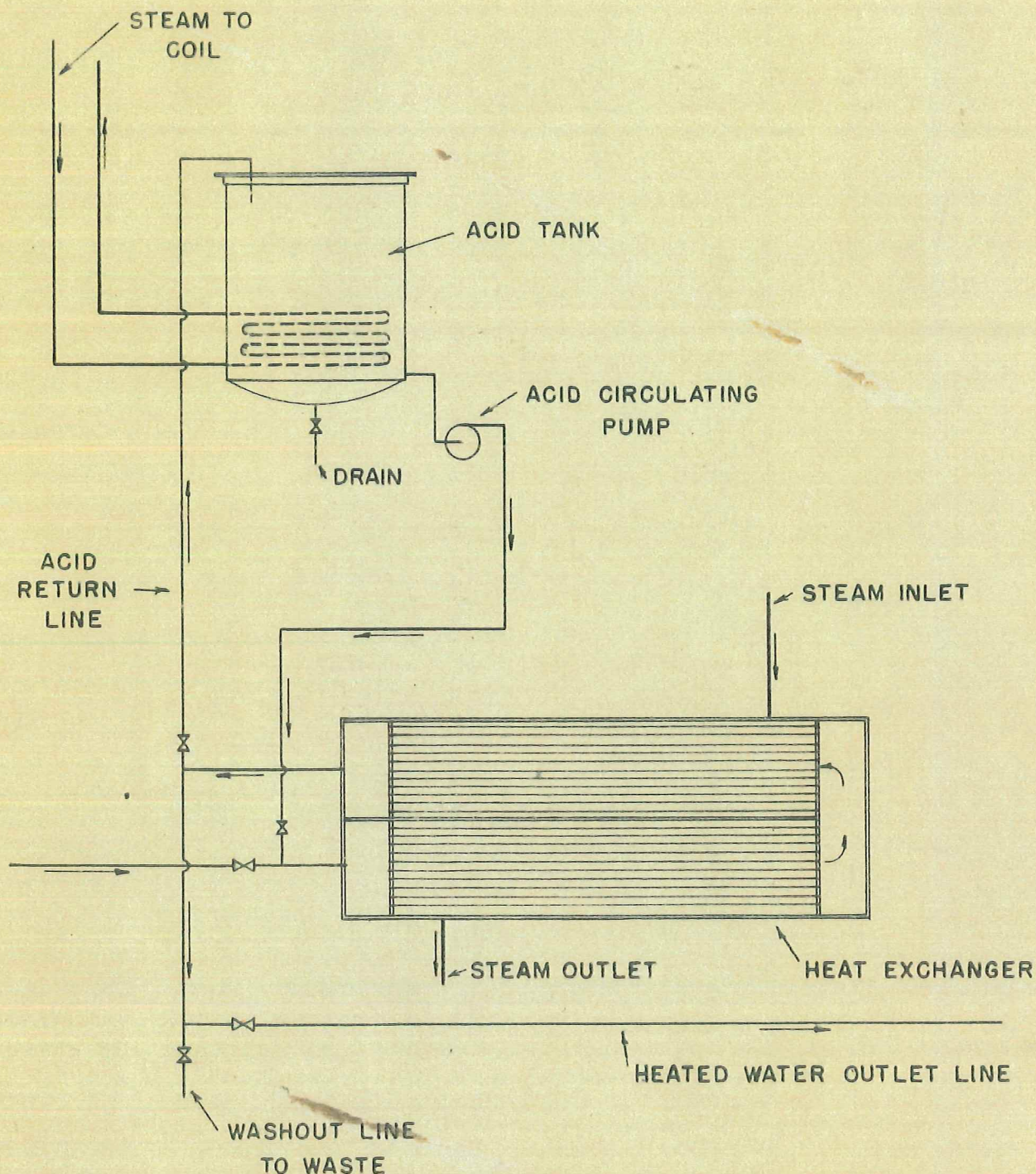
(6) Firm recommendations cannot be made on the incomplete data in our possession. It is our opinion that a study program should be initiated immediately, on a pilot plant scale, to determine the characteristics of the water supply that is to be reheated at the power plant. An outline of a program is attached hereto illustrating the type of data we believe should be obtained and the suggested equipment required to carry out this program.

SCHEMATIC ARRANGEMENT OF EQUIPMENT USING TWO-PASS HEAT EXCHANGERS FOR REHEATING WATER SUPPLY TO CITY OF REYKJAVIK



S.T. POWELL
B-368

SCHEMATIC ARRANGEMENT OF EQUIPMENT FOR ACID
CLEANING THE HEAT EXCHANGERS, USED FOR REHEATING
THE WATER SUPPLY TO CITY OF REYKJAVIC



Sheppard T. Powell

(7) In the case of the four-inch diameter pipe, in which calcium carbonate was precipitated, it is possible this condition may be corrected by the use of a hot-lime process softener. Installation of such a unit would permit precipitation of the calcium carbonate in a location where the sludge could be removed without harm to the system. To reduce the quantity of chemicals purchased, the softener would be designed to operate under pressure. Probably, it would be necessary to recover the calcium carbonate sludge and calcine it in a rotary kiln. This would convert the precipitated calcium carbonate to calcium oxide which could be slaked and reused. If the treated water required a reduction in the pH value this could be effected by recovery and use of the carbon dioxide in the kiln gases. It would be necessary in this latter case, as in the previous ones, to conduct certain pilot plant studies to determine the exact method required to treat this water and the extent of treatment necessary to most effectively condition the supply. This is shown on the program outline attached hereto.

(8) Regarding the small cities scattered throughout Iceland, we note that the water analyses are similar in their chemical characteristics. However, the supplies contain variable amounts of hardness, sulfides and silica. The one analysis of scale which was made available to us indicates that the predominant constituent in the deposit is silica. However, the sample contained appreciable iron oxide and calcium which may have aided in the deposition of silica in the piping. It is necessary to study these supplies more thoroughly before firm

recommendations can be made. This probably should be held in abeyance until the major water supplies have been studied more carefully because the solution of these problems may be satisfactory for the smaller supplies with only minor adjustments.

DISCUSSION

The deposit found in the small pipe was suspected of containing high concentrations of silica. However, the chemical analysis showed it to be 72-1/2 per cent iron oxide, and only 14 per cent silica, the balance consisting of traces of various other elements. We believe that this scale was caused primarily by corrosion of the piping system, resulting from the presence of oxygen in solution in the water. The condition can be corrected by deaeration of the water to eliminate oxygen and prevention of resolution of this gas in the system.

Attached hereto is a photograph (Exhibit A) showing the sections of the sample of pipe after being split longitudinally. One view shows the scale deposit in the pipe and the other is the same sample after removal of deposit with inhibited acid. It will be noted that appreciable pitting of the tube surface occurred. The type of water from which this scale was deposited contained an appreciable amount of hardness, 245 ppm, no sulfides, and 73 ppm of silica. The water was labeled Vatn frá Reykjanesi (harka 13.5°). The X-ray analyses confirm the chemical analyses both of which are given in Table I.

The scale sample from the four-inch line was a white, hard deposit, believed to be silica. The chemical analysis, a copy of which is attached, Table II, showed that the main con-

stituent in this scale was calcium carbonate. There was a trace of sulfate and chloride, a small amount of manganese, iron and aluminum but only 0.64 per cent of silica. It is our understanding that the scale was deposited from the water sample marked Borhola 2, Reykjakoti. This water contained relatively little hardness, some sulfide and about 135 ppm of silica. The deposit laid down could not have been precipitated by this water according to the water analysis furnished us. We suspect this sample is not representative of the supply actually in use, and it is indicated that contamination may have occurred from other sources.

We are attaching to this letter Tables I and II showing the analyses of the scale and the waters which we understand caused the deposition of these two scales. The analysis of the water which precipitated the scale in the one-half inch diameter piping was used to calculate the Langelier Scaling Index, and it was found that this water had the highest index of any of those which we studied. A high index generally indicates rapid precipitation of scale forming solids. The index adjusted for temperature effects varied from 1.8 to 2.8 at temperatures of 190, 167, 104 and 207° Fahrenheit. These represent the temperature of the water that might be expected at the well, at the point of delivery of the supply to the distribution system and the water as wasted. The temperature of 204° is that predicted if well water were reheated in the heat exchangers at the power plant.

We would have expected this water to lay down a heavy calcium carbonate scale judging by the scale index. However, the scale analyses indicated that only a trace of calcium was present.

TABLE I

ICELAND - STATE ELECTRICITY BOARD

ANALYSES OF SCALE SAMPLE REMOVED FROM 1/2" DIAMETER PIPE AND THE
WATER WHICH CAUSED THIS DEPOSIT

Constituents	As	Water Sample No. 8	Constituents	As	Scale Sample No. 9
Calcium	Ca	98.00	Silica	SiO ₂	14.20
Magnesium	Mg	-	Iron oxide	Fe ₂ O ₃	72.54
Sodium & Potassium	Na	217.00	Aluminum oxide	Al ₂ O ₃	0.20
Chloride	Cl	400.00	Calcium oxide	CaO	tr.
Sulphate	SO ₄	45.00	Magnesium oxide	MgO	0.16
Nitrate	NO ₃	+	Copper	Cu	0.00
Sulfide	S ₃	-	Chloride	Cl	tr.
Bicarbonate	HCO ₃	8.30	Sulfur tri-oxide	SO ₃	tr.
Carbonate	CO ₃	60.30	Carbonate	CO ₃	tr.
Hydroxide	OH	-	Loss on ignition		8.68
Silica	SiO ₂	73.00	Manganese	Mn	0.08
Aluminum	Al	+	Loss at 100°C		5.80
Iron	Fe	3.00			
Ammonia	NH ₃	tr.	Spectroscopic Analysis:	Fe	high
Sum		<u>904.60</u>		Si	.5-5.0
As Calcium Carbonate				Mn	.1-1.0
Calcium Hardness		245.00		Cu, Ca	.01-.1
Magnesium Hardness		-	X-ray Analysis:	Mg	.001-.01
Total Hardness		245.00	Iron Oxide		
Sodium & Potassium		<u>471.50</u>	Cronstedtite	Fe ₃ O ₄	80%
Total Cations		<u>716.50</u>		2 FeO.Fe ₂ O ₃	
Total Residue		840.00		SiO ₂ .2H ₂ O	20%
pH		9.2			
Corrected Saturation Index					
190°F		1.82			
167°F		2.05			
104°F		2.83			
207°F		1.80			

(8) Vatn frá Reykjanesi (Harkv 13.50°)

Note: Similar to City water - actual water that deposited scale.

TABLE II

ICELAND - STATE ELECTRICITY BOARD

ANALYSES OF SCALE SAMPLE REMOVED FROM 4" DIAMETER PIPE AND THE
WATER WHICH CAUSED THIS DEPOSIT

Constituents	As	Water Sample No. 10	Constituents	As	Scale Sample No. 11
Calcium	Ca	7.00	Silica	SiO ₂	0.64
Magnesium	Mg	tr.	Iron Oxide	Fe ₂ O ₃	0.58
Sodium & Potassium	Na	102.00	Aluminum oxide	Al ₂ O ₃	tr.
Chloride	Cl	68.00	Calcium oxide	CaO	54.86
Sulphate	SO ₄	27.00	Magnesium oxide	MgO	tr.
Nitrate	NO ₃	+	Chloride	Cl	tr.
Sulfide	S	13.40	Sulfur tri-oxide	SO ₃	tr.
Bicarbonate	HCO ₃	+	Carbonate	CO ₃	60.00
Carbonate	CO ₃	43.40	Loss on ignition		0.60
Hydroxide	OH		Manganese		0.21
Silica	SiO ₂	135.00			
Aluminum	Al	+	Loss at 100°C		0.06
Iron	Fe	2.00			
Ammonia	NH ₃	+	Spectroscopic Analysis:		
Sum		<u>397.80</u>		Ca	high
As Calcium Carbonate				Mn	.5-5.0
Calcium Hardness		17.50		Mg	.1-1.0
Magnesium Hardness		-		Si	.05-.5
Total Hardness		17.50		Al, Fe, Sr	.01-.1
Sodium & Potassium		<u>220.70</u>		Ba, Cu	.001-.01
Total Cations		<u>238.20</u>	X-ray Analysis:		
Total Residue		445.00	Calcium Carbonate	CaCO ₃	90%
pH		9.4	Magnesium Silicate	Mg ₃ Si ₂ O ₇	10%
Corrected Saturation Index				2 H ₂ O	
190°F		0.62			
167°F		0.68			
104°F		0.72			
207°F		0.61			

(10) Borhola II Reykjakoti - Efnagreining & hveravatni

The apparent reason for failure of this water to deposit calcium scale seems to be that some inhibiting substance, possibly silica, retarded the scaling which would occur normally.

Calculations were made from the analysis of the water which deposited the calcium carbonate scale in the four-inch diameter pipe to determine its scaling characteristics. This water showed a normal index compared with the values obtained for the other waters and would have been expected to precipitate only a small amount of calcium carbonate. However, the analyses showed the deposit to consist of almost 100 per cent calcium carbonate. It is obvious that the latter type of deposits could not have accumulated from the water represented by the analysis given to us.

We recalculated the several analyses of the various waters which were given us. These have been placed on an ionic basis in order to determine if the anions and cations balanced. The group of water and scale analyses shown on Table III are those you informed us were representative of the water supplies in several small cities in Iceland. The analyses were labeled Spytir, Reykjakoti, and Svampur. The analysis of these waters showed they contained only a small amount of calcium hardness (10 ppm and 17 ppm), appreciable quantities of sulfide (84 ppm and 86 ppm), and a large amount of silica (310 ppm and 272 ppm). We also have calculated the Langelier Index which is indicative of the scale that will be precipitated from waters. This was done at the same temperatures as those in the analyses discussed elsewhere, namely, 190, 167, 104 and 207° Fahrenheit. The indices calculated from these data show that the waters should be nearly stable in so far as calcium carbonate is

TABLE III

ICELAND - STATE ELECTRICITY BOARD

ANALYSES OF WATER AND SCALE SAMPLES REPRESENTATIVE

OF THE CONDITIONS EXISTING IN THE SMALL CITIES

Constituents	As	Water Sample No. 1	Water Sample No. 2	Constituents	As	Scale Sample No. 3 Quantitative Analyses	Scale Sample No. 3 Qualitative Spectroscopic Analyses
Calcium	Ca	4.00	7.00	Water	H ₂ O	7.70	
Magnesium	Mg	tr.	+	Magnesium	MgO	0.82	
Sodium & Potassium	Na	762.00	241.00	Calcium Oxide	CaO	5.60	
Chloride	Cl	58.10	170.00	Iron Oxide	Fe ₂ O ₃	12.80	
Sulphate	SO ₄	68.00	70.00	Silica	SiO ₂	62.00	
Nitrate	NO ₃	+	+	Loss at red heat		<u>10.42</u>	
Sulfide	S	26.80	27.50	Total		99.34	
Bicarbonate	HCO ₃	+	+	Net Loss		0.66	
Carbonate	CO ₃	75.00	87.00				
Hydroxide	OH	-					
Silica	SiO ₂	310.00	272.00				
Aluminum	Al	+	+	Sodium			Na
Iron	Fe	tr.	2.00	Potassium			K
Free Ammonia	CO ₂ NH ₃	+	+	Calcium			Ca
		tr.	tr.	Magnesium			Mg
Sum		<u>703.90</u>	<u>876.50</u>	Silicon			Si
				Boron			B
As Calcium Carbonate				Iron			Fe
Calcium Hardness		10.00	17.50	Chromium			Cr
Magnesium Hardness		tr.	tr.	Lead			Pb
Total Hardness		<u>10.00</u>	<u>17.50</u>	Nickel			Ni
pH		9.4	9.3	Manganese			Mn
Corrected Saturation Index				Tin			Sn
190°F		0.69	0.96	Bismuth			Bi
167°F		0.73	0.97	Cobalt			Co
104°F		0.73	0.94	Titanium			Ti
207°F		0.69	0.95				

- (1) (Spytir) (Tekio ur hvernum sjalfum) - Efnagreining á hveravatni. Vatn frá Gufudal. Hnverageroi.
- (2) (Svampur)- Efnagreining á hveravatni - Vatn frá Gufudal, Hverageroi.
- (3) Utfelling i rori frá Gufudal - Reykjakoti

concerned. There will be a general tendency, however, to precipitate more scale with a marked decrease in temperature.

Included with the information you gave us was an analysis of scale presumably caused by one of these supplies. In this analysis the silica amounted to 62 per cent, the iron oxide to 13 per cent and the calcium oxide was approximately 6 per cent. Qualitative, spectroscopic analyses you had made confirmed these results. In the latter case the iron oxide and calcium deposited may have caused the silica to be precipitated due to adsorption or occlusion by the precipitated calcium.

The analyses labeled as typical of the various waters scattered throughout Iceland are shown on Table IV. They appear to be similar to the previous supplies discussed in that the waters are relatively soft and contain variable amounts of silica, the limits ranging from 75 to 452 ppm. Sulfides were present in two of the supplies but were not reported in the other two. The samples in question were labeled Borhola I Reykjakoti, Borhola I Norourreykjun, Vatn fra', Kristnesi and Vatn fra' Laungalandi. There were no scale analyses which had been caused by these waters. However, as with the previous supplies, we calculated the Langelier Index for each at various temperatures. The analysis of the Norourreykjun has the lowest index of any of the waters studied. It is indicated from this that calcium scale would not be precipitated in the lines carrying this water but corrosion might occur. All of these supplies, as in the case of the first two discussed, appear to be very nearly in equilibrium but so adjusted that they would deposit more calcium scale at a lower temperature when the water

TABLE IV

ICELAND - STATE ELECTRICITY BOARD

ANALYSES OF TYPICAL WATER SAMPLES

Constituents	As	Water Sample No. 4	Water Sample No. 5	Water Sample No. 6	Water Sample No. 7
Calcium	Ca	5.00	4.00	9.00	8.00
Magnesium	Mg	tr.	2.00	3.00	2.00
Sodium & Potassium	Na	246.00	60.00	44.00	63.20
Chloride	Cl	207.00	18.20	16.00	23.60
Sulphate	SO ₄	53.00	33.00	48.00	34.00
Nitrate	NO ₃	+	+	+	tr.
Sulfide	S	22.70	3.30	+	+
Bicarbonate	HCO ₃	+	+	+	+
Carbonate	CO ₃	78.00	46.00	35.00	58.20
Hydroxide	OH				
Silica	SiO ₂	452.00	82.00	75.00	110.00
Aluminum	Al	tr.	+	+	+
Iron	Fe	1.00	tr.	2.00	1.60
Free	CO ₂	tr.	+	-	-
Ammonia	NH ₄	+	-	tr.	tr.
Sum		<u>1064.70</u>	<u>248.50</u>	<u>232.00</u>	<u>300.60</u>
As Calcium Carbonate					
Calcium Hardness		12.50	10.00	22.50	20.00
Magnesium		tr.	8.20	12.30	8.25
Total Hardness		12.50	18.20	34.80	28.25
Sodium & Potassium		<u>535.50</u>	<u>130.00</u>	<u>95.90</u>	<u>137.55</u>
Total Cations		<u>548.00</u>	<u>148.20</u>	<u>130.70</u>	<u>165.80</u>
Chloride		292.00	26.60	22.50	33.20
Sulphate		55.10	34.50	50.00	35.40
Nitrate		+	+	+	tr.
Sulfide		71.00	10.30	+	+
Mineral Acid Ions		418.10	71.40	72.50	68.60
Bicarbonate		-	-	-	-
Carbonate		130.00	76.80	58.20	97.20
Hydroxide		-	-	-	-
Total Anions		<u>548.10</u>	<u>148.20</u>	<u>130.70</u>	<u>165.80</u>
Total Residue		1100.00	200.00	250.00	275.00
pH		9.48	9.32	9.5	9.4
Corrected Saturation Index					
190°F		0.88	0.36	0.70	0.74
167°F		0.90	0.39	0.76	0.94
104°F		0.92	0.43	0.96	0.96
207°F		0.85	0.35	0.67	0.88

(4) Borhola I Reykjakoti - Efnagreining & hveravatni

(5) Borhola I Norourreykjum - Efnagreining & hveravatni. Vatn ur Mosfellsdal

(6) Vatn frá Kristnesi

(7) Vatn frá Laugalandi

cooled.

The analyses from the Reykjavik heating plant system are shown in Table V. These were recalculated and found to contain a small amount of hardness, moderate amounts of silica compared to the other waters and relatively small amounts of sulfide. An analysis of the scale showed it to consist of 66 per cent iron oxide, 13 per cent silica and a small amount of magnesium. These two water samples when checked to determine the Langelier Index were found to have a normal index for these waters.

The scale which was deposited by this water was examined spectroscopically and the results confirmed the analytical determination. Apparently in this instance, corrosion was an important factor in the formation of deposits.

TABLE V
 ICELAND - STATE ELECTRICITY BOARD
 ANALYSES OF SCALE AND WATER FLOWING THROUGH THE REYKJAVIK
 DISTRIBUTION SYSTEM

Constituents	As	Water Sample No. 12	Water Sample No. 13	Constituents	As	Scale Sample No. 14 Quantitative Analyses	Scale Sample No.14 Qualitative Spectroscopic Analyses
Calcium	Ca	8.00	9.40	Silica	SiO ₂	13.03	
Magnesium	Mg	-	-	Iron Oxide	Fe ₂ O ₃	65.78	
Sodium & Potassium	Na	46.80	43.20	Magnesium Oxide	MgO	3.65	
Chloride	Cl	24.30	25.20	Potassium	K ₂ O	2.80	
Sulphate	SO ₄			Water	H ₂ O	3.60	
Nitrate	NO ₃	+	+	Loss at red heat		6.70	
Sulfide	S	1.41	1.32	Total		98.25	
Bicarbonate	HCO ₃	-	-	Residue		1.75	
Carbonate	CO ₃	49.20	46.20	Iron			Fe
Hydroxide	OH	-	-	Silicon			Si
Silica	SiO ₂	84.00	82.00	Potassium			K
Manganese	Mn	tr.	tr.	Magnesium			Mg
Ammonia	NH ₃	tr.	tr.	Manganese			Mn
Iron	Fe	tr.	0.06	Tungstun			W
Sum		<u>213.71</u>	<u>207.38</u>	Bismuth			Bi
As Calcium Carbonate				Cadmium			Cd
Calcium Hardness		20.00	23.50	Boron			B
Magnesium		-	-	Calcium			(Ca)
Total Hardness		20.00	23.50	Cobalt & Nickel			(Co)(Ni)
Sodium & Potassium		<u>101.50</u>	<u>93.85</u>	Aluminum			(Al)
Total Cations		<u>121.50</u>	<u>117.35</u>	Zirconium			Zr
Total Residue		236.00	230.00				
Color		10.00	10.00				
pH		9.3	9.2				
Organic Substance		1.30	1.40				
Corrected Saturation Index							
190°F		0.70	0.66				
167°F		0.74	0.71				
104°F		0.76	0.69				
207°F		0.70	0.66				

(12) (13) - Two samples of water from Reykjavik heating plant. Borings Nos. 7 and 17.
 (14) - Utfelling i vori frd' Hitaveitv Reykjavikor

PROGRAM FOR INVESTIGATION OF WATER CONDITIONS
CAUSING EXCESSIVE SCALE PRECIPITATION
IN PIPE LINE DISTRIBUTION SYSTEMS

(1) Analytical data should be obtained to show the dissolved constituents contained in this water. Sufficient samples should be collected so any variations in quality can be evaluated.

(2) Dissolved oxygen should be determined at frequent intervals. It is desirable to determine if oxygen is present in the existing system and, if so, in what quantities. It also is important to find if it is present in larger quantities at one time than at another and whether it fluctuates in different sections of the City. Attached herewith is a procedure for making oxygen determinations. This may have to be modified if it is found that sulfides, iron or other interfering substances are present in appreciable quantities in the water. It is suggested that reference be made to the American Public Health Association standard methods of water analysis and that the analyst be guided by the information contained herein.

(3) It is suggested that a scale testing apparatus similar to that shown on the attached prints, A-261, A-266 or AA-171, should be constructed. The cross-section through which the water flows should be so adjusted as to approximate the velocity which will normally exist in the tubes of the proposed

new heat exchangers. It probably would be desirable to construct one of these units of steel and one of the same material from which the heat exchanger tubes are to be fabricated. It also would be advisable to install equipment for running the modified Marble Test, which is attached, to ascertain the scale forming qualities of this water. Sufficient data should also be collected so that calculations can be made to determine the Langelier Scaling Index. Control of the pH of this water could be guided by the results obtained from the calculated index and the Marble Test Index in order to determine if control of the scale forming properties of this water are feasible by simple pH adjustment rather than a complete treating plant.

(4) If it is found that oxygen is contained in the water supply steps should be taken to effect its removal prior to discharging the water through the scale testing apparatus. In this way the corrosive action of the water may be studied both before and after deaeration. Thus, it can be determined if oxygen is the main factor responsible for the incrustation in the lines at the present time or if silica will continue to deposit regardless of the other conditions existing. In addition, should calcium carbonate scale be deposited, it would be possible to adjust the pH and thereby control the latter condition. Such data would show if the proposed heat exchangers will be satisfactory or if it is advisable to consider the installation of open atmospheric heaters, coil type, using steam in the tubes and water outside, a type of construction which would permit ready cleaning.

I - PIPE SECTION CUT LENGTHWISE TO SHOW
ACCUMULATION OF SCALE DEPOSIT PARTLY
REMOVED.

II - PIPE SECTION CUT LENGTHWISE TO SHOW
PITTING FROM CORROSION AFTER REMOVING
SCALE WITH INHIBITED ACID

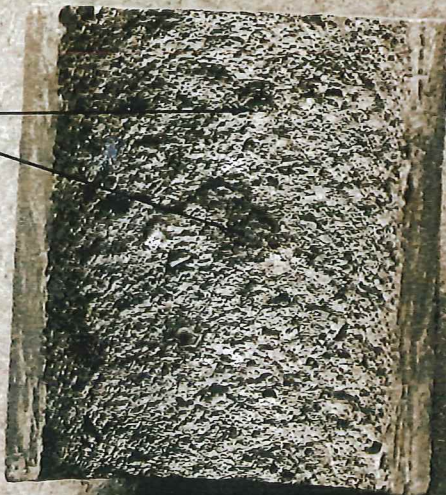
1

SCALE DEPOSIT



2

CORROSION PITS



MODIFIED MARBLE TEST FOR SATURATION INDEX DETERMINATION

When inhibitors are present in the water the Calculated Langelier Index indicates a higher scaling value than is actually the case. For cases such as this it is advisable to use the marble test method to determine the Saturation Index. The following method can be used to determine the Saturation Index directly, with the only test required being pH.

The theory of the method is that if a water that is not in equilibrium is brought in contact with calcium carbonate the water will attain equilibrium. If it is under-saturated with respect to calcium carbonate, which is the same as having a negative saturation index, the water will dissolve sufficient calcium to saturate the solution, making the saturation index zero. If the water is super-saturated, which is the same as a positive saturation index, the excess calcium carbonate will precipitate, causing the saturation index to be zero.

The method is as follows:

1. Determine the pH of the sample as collected at room temperature. This is the actual pH (pH_a).
2. Place 200 ml. of the sample in a 500 ml. Erlenmeyer flask and add sufficient powdered calcium carbonate to saturate the sample (about a teaspoonful). Shake the flask with a swirling motion to mix the calcium carbonate and water at frequent intervals for five minutes.

3. Using a medium filter paper, filter about 20 cc. of the water in the flask to waste; then, using the same filter paper, filter enough of the sample for a pH determination. This pH will be the saturation pH (pH_s) at room temperature.
4. Determine the saturation index at room temperature using the formula:

$$S.I. = pH_a - pH_s$$

EXAMPLE:

The water used for cooling heat exchangers is regulated to control scale deposition. The temperature in to the equipment is 70° F. and the outlet temperature is 120°F. What is the saturation index at 70°F. and 120°F.?

Using the method just described, the following results are obtained at a room temperature of 80°F.:

$$pH \text{ actual at } 80^\circ F. (pH_a) = 8.0$$

$$pH \text{ of saturation } (pH_s) = 7.8$$

$$\text{Saturation Index} = + \underline{\quad .20}$$

INSTRUCTIONS FOR MAKING THE TEST FOR OXYGEN IN FEEDWATER

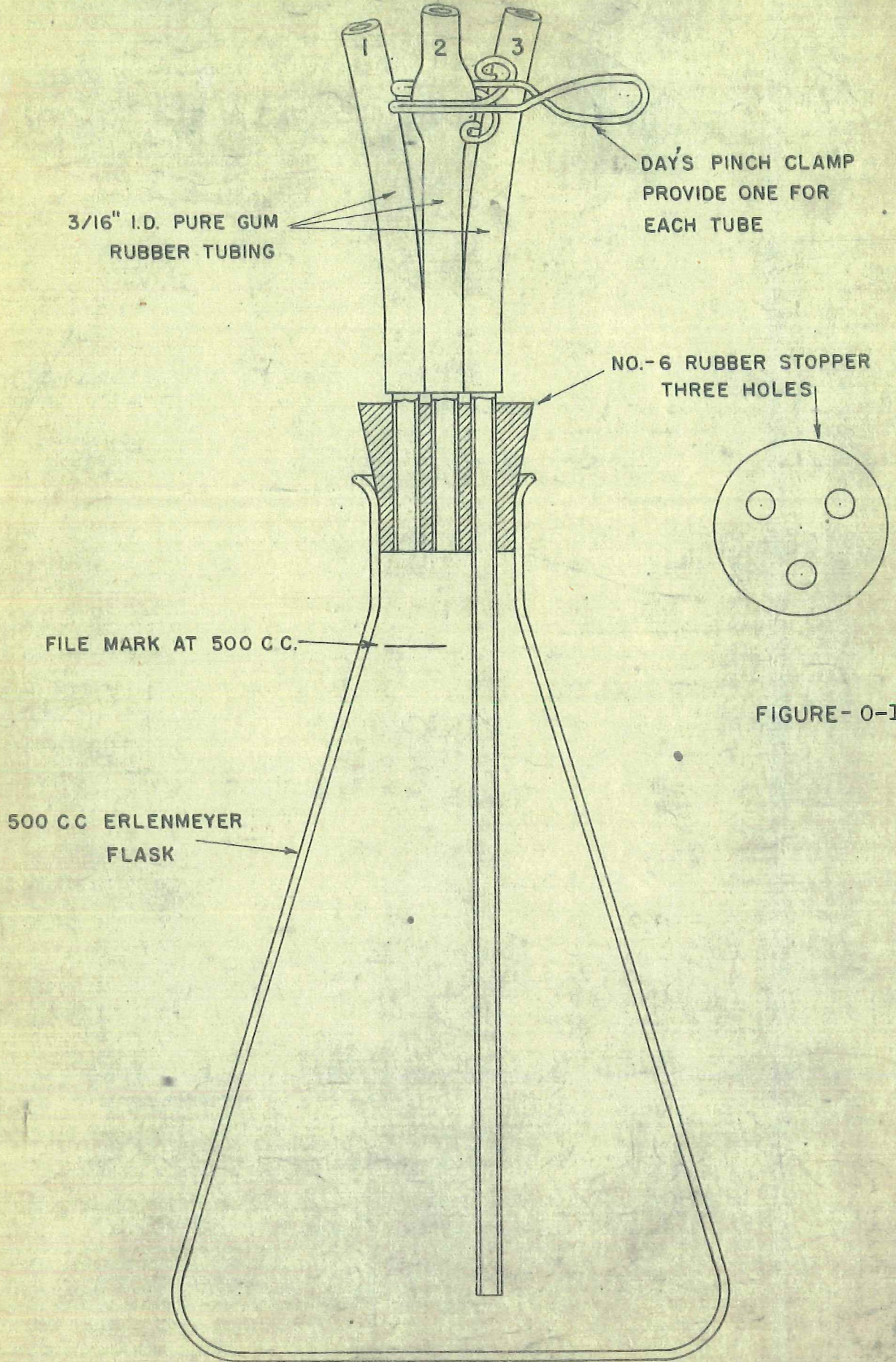
The Winkler test, described below, is the recognized standard procedure for determining dissolved oxygen in water. Boiler feedwater should be so treated that the dissolved oxygen is completely removed, and when this test is made no more than a trace of oxygen should ever be found in the water. The accurate determination of dissolved oxygen in amounts between 0 and 0.1 cc. per liter is a delicate procedure which requires extreme care.

COLLECTION OF SAMPLE

The feedwater should be passed through a cooling coil such as is shown in Figure 0-1, which will cool it to 70°F., if possible. If it cannot be cooled down to this temperature because the cooling water is at a high temperature, it will be necessary, at a later step in the analysis, to immerse the sampling flask in ice water. All valves in the sampling line and coil ahead of the outlet valve on the coil should be wide open and the flow should be throttled by the outlet valve so that a minimum time of thirty seconds is required for filling the sample flask (Figure 0-2).

Attach the rubber inlet tube (No. 3) of the sampling flask to the outlet nipple in the bottom of the cooling coil, using an extra length of rubber tubing and a glass connector. Remove the pinch clamps from all three tubes of the sampling flask and, with the stopper removed, flood the flask in order to remove bubbles of air. Insert the stopper in such a manner that no bubbles are trapped on its lower side. Even minute visible air bubbles will influence the results and the sampling must be begun over if they

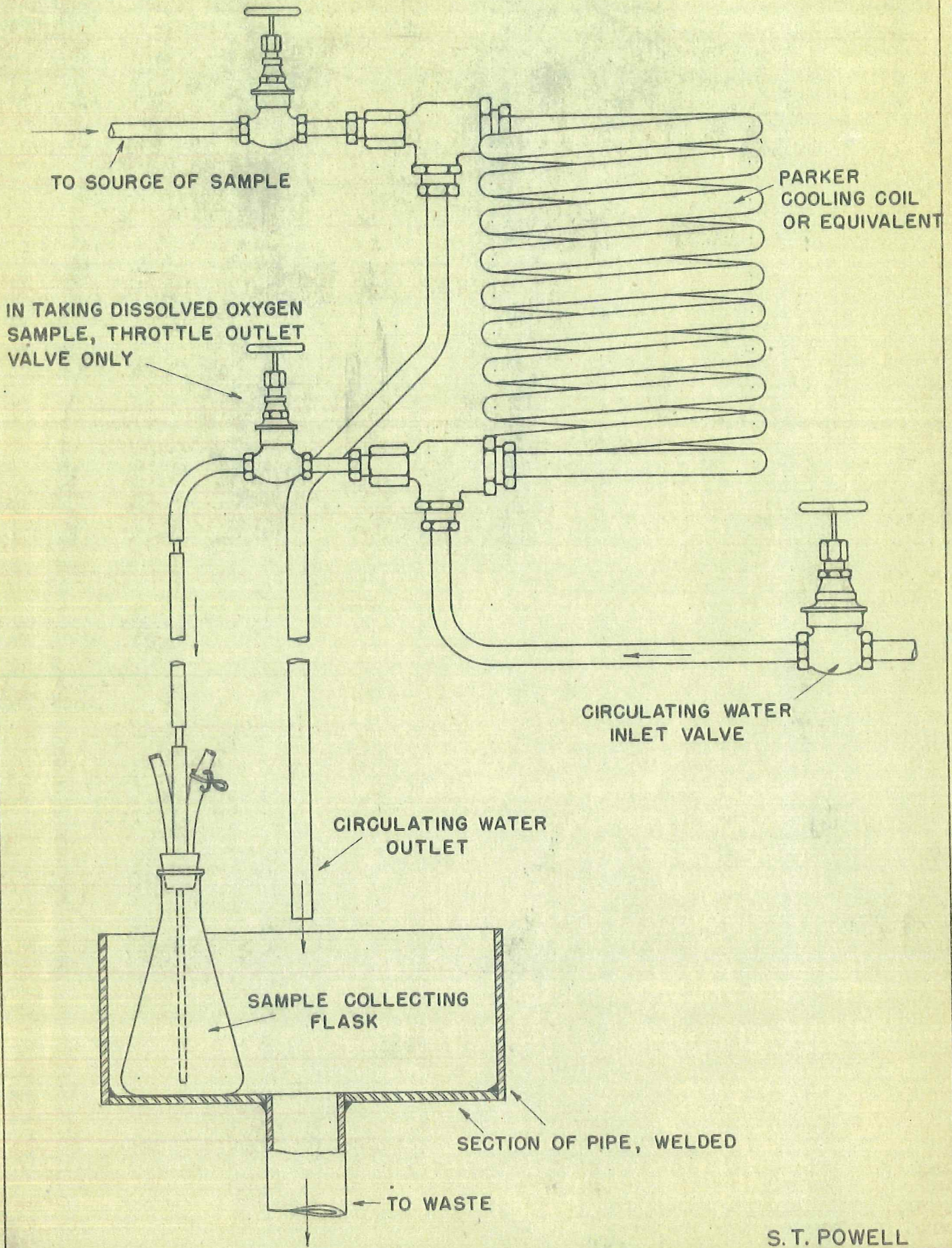
SAMPLING FLASK FOR DISSOLVED OXYGEN TEST



S.T. POWELL

A-194

ARRANGEMENT OF COOLING COIL AND SAMPLING FLASK FOR DISSOLVED OXYGEN TEST



S. T. POWELL
A - 220

are discovered at any time. Permit the water to overflow through the rubber tubes Nos. 1 and 2 for not less than five minutes, then disconnect the flask from the sampling cock of the cooling coil and place one of the pinch clamps on tube No. 3.

ADDING THE REAGENTS

The flask should now be full of water, which should also extend up to the top of the three rubber tubes on the stopper. Fill a 2 ml. pipette with manganous chloride solution and insert the tip into rubber tube No. 1, making sure that there are no air bubbles in the top of the pipette or in the rubber tube. Allow 2 ml. of the solution to flow through the tube into the flask. When this is done it will displace water from the flask out through tube No. 2.

Disconnect the flask from the pipette and insert the tip of a second 2 ml. pipette containing sodium hydroxide-potassium iodide solution into tube No. 2. Never add Solution No. 1 and Solution No. 2 through the same tube, or with the same pipette. Add 2 ml. of the solution, which will displace water through tube No. 1.

Now place pinch clamps on all of the tubes below the water level, so that no air bubbles are caught below the clamps. Rinse off the tubes and the stopper and invert the flasks several times to mix thoroughly. Set the flask aside until the precipitate settles out, which will require 3 to 4 minutes.

Remove the clamps from tubes No. 2 and 3 and add 2 ml. of sulphuric acid solution through tube No. 3. Replace the clamps, being careful not to retain air bubbles, and invert to mix thoroughly. The precipitate will be dissolved by the acid. When the contents of

the flask are clear the sample is fixed and there is no further danger of contamination by exposure to air. However, the sample must be cooled to 70° F., or lower, before completing the test. Insert a thermometer in the flask and, if necessary, immerse it in ice water to cool it. This is important, since the test is not accurate at temperatures above 70° F.

TITRATING THE SAMPLE

Use the excess of treated sample above the 500 ml. calibration on the flask to rinse out at 750 ml. porcelain casserole. Then transfer the remaining 500 ml. sample to the casserole, and add 3 ml. of starch solution. Titrate with N/40 sodium thiosulphate solution until the disappearance of the blue color. Each ml. of N/40 thiosulphate required for this change is equivalent to 0.28 ml. per liter of dissolved oxygen in the sample.

EXAMPLE

Suppose that a 500 ml. sample of feedwater required 0.4 ml. of N/40 thiosulphate to discharge the blue color. Then $0.4 \times 0.28 = 0.11$ ml. per liter of dissolved oxygen present in the sample.

NOTES CONCERNING THIS TEST

- (1) When the manganous chloride and alkaline potassium iodide are added a white precipitate will be noticed, which is manganous hydroxide.
- (2) If oxygen is present a brown-colored precipitate will appear in a quantity depending on the amount of oxygen present.
- (3) The addition of sulphuric acid dissolves the brown precipitate and sets free an amount of iodine equivalent to the quantity of oxygen in the sample.

(4) The solution at this time will be yellow in color, the depth of the color depending upon the amount of oxygen present.

(5) If oxygen is present and starch is added to the solution at this stage, the color will change to blue, the intensity of which depends on the amount of oxygen present.

TOLERANCE

With care, this method will give reproducible results for quantities of oxygen as small as 0.01 ml. per liter.

PRECAUTIONS TO BE OBSERVED IN MAKING THIS TEST

The determination of dissolved oxygen in amounts less than 0.05 ml. per liter is an extremely delicate one and the accuracy of the results will depend largely on the accuracy of the technique of the analyst. The following precautions should be taken in order to assure accurate results:

(1) The temperature of the sample as collected should be 70° F. or less.

(2) Only chemicals which are free from oxidizing impurities should be used.

(3) Water for making test solutions should be free from nitrites, sulphites, organic matter, and ferrous or ferric iron.

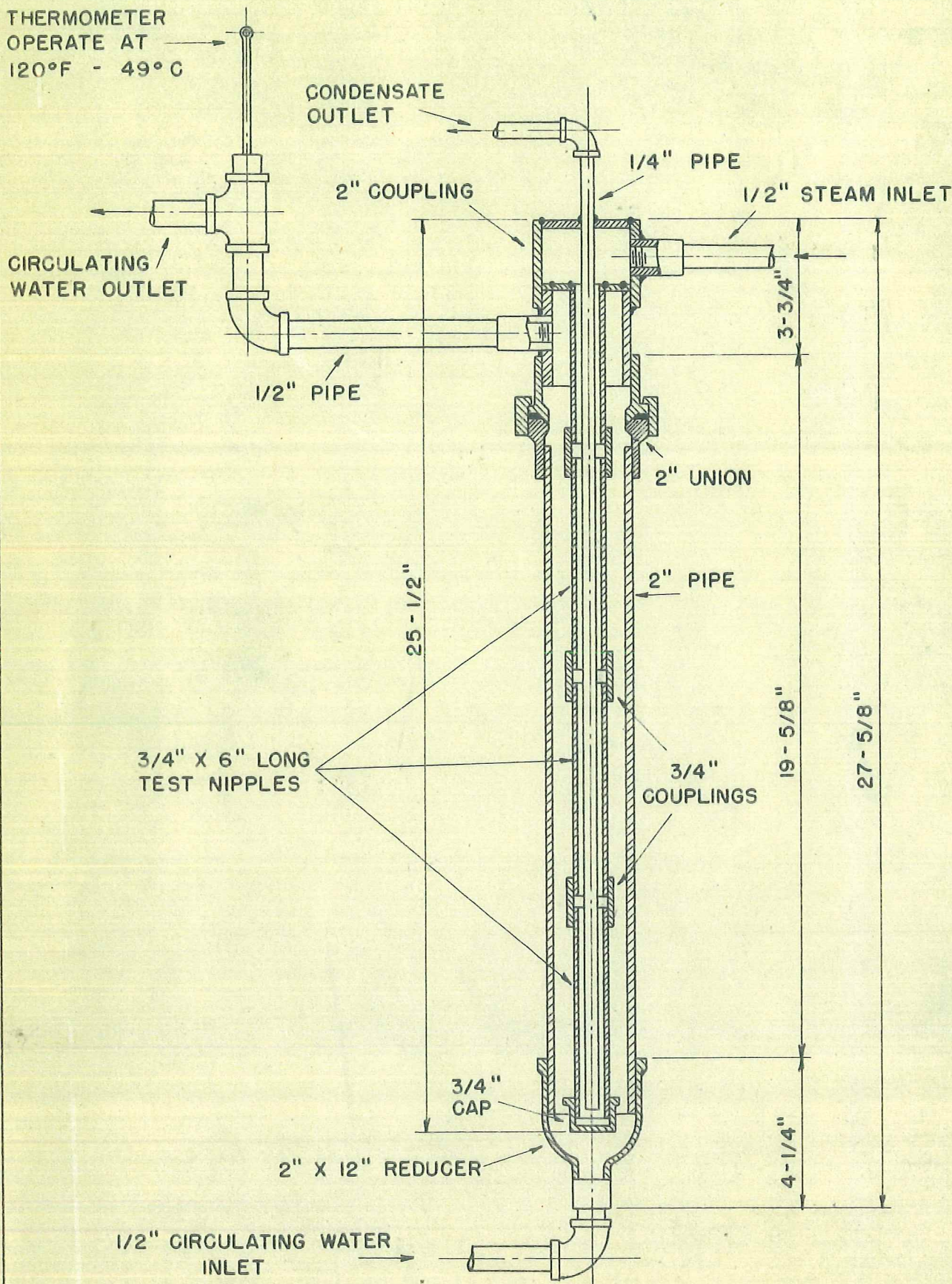
(4) The sample should be drawn through copper tubing and all joints should be braced wherever possible.

(5) Sampling cocks and unions should be made tight by shellacking.

(6) New lines should be flushed out for four hours before using.

(7) Red lead used for making joints will introduce a large error. Lines containing such joints should be flushed out

CORROSION TESTING UNIT

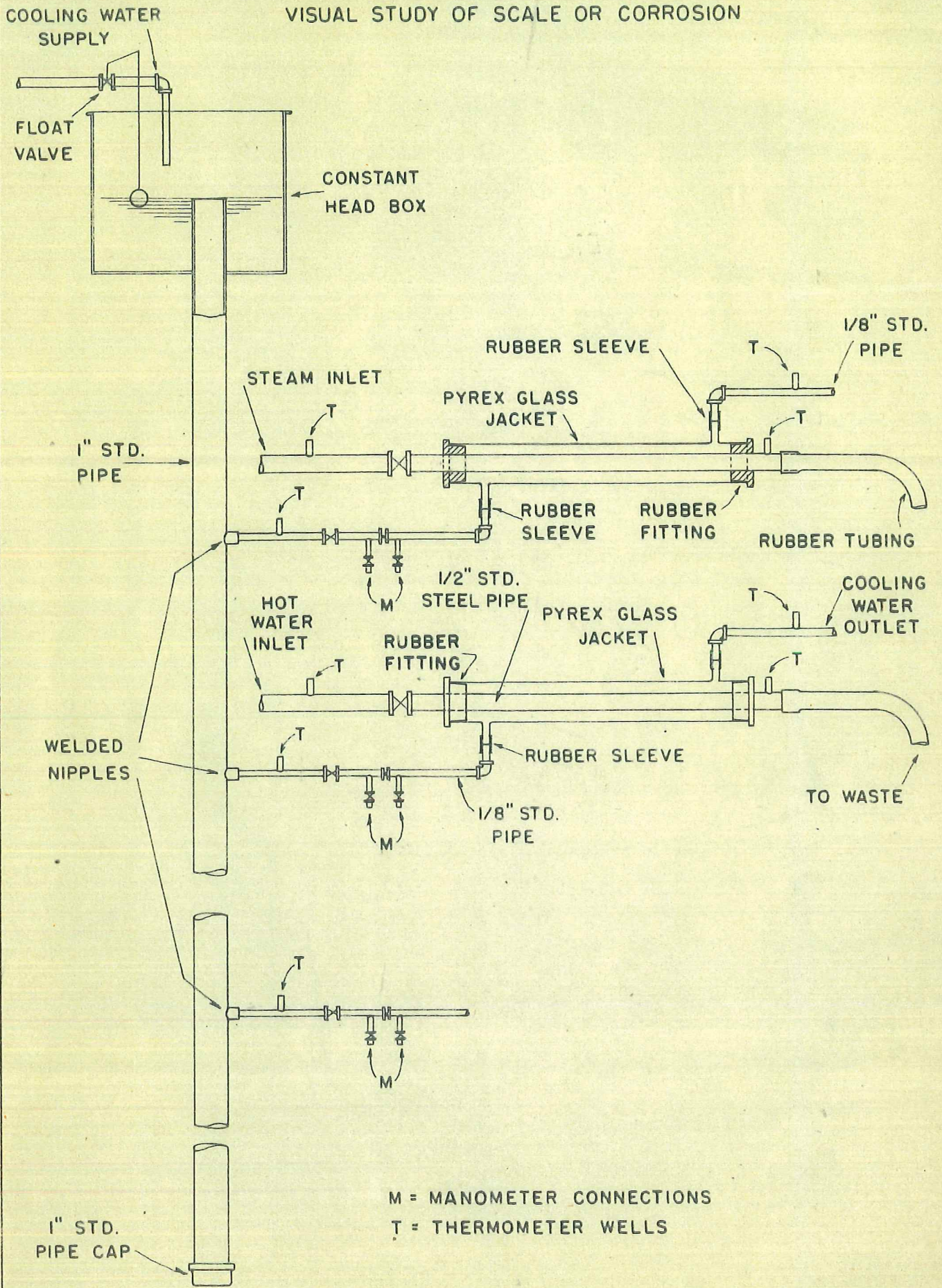


ORIGINAL DESIGN BY DOW CHEMICAL CO. FREEPORT, TEX.

S.T. POWELL

A - 261

SCHEMATIC FLOW DIAGRAM OF TEST ASSEMBLY FOR VISUAL STUDY OF SCALE OR CORROSION



S.T. POWELL

one or more days before a sample is drawn for the test.

(8) New rubber tubing should not be used, since appreciable errors are introduced by dissolved sulphur.

TESTS FOR INTERFERENCE IN DISSOLVED OXYGEN DETERMINATION

Collect another 500 ml. sample of the water to be tested without observing the precautions described above. Add the oxygen reagents in following order: alkaline potassium iodide, sulphuric acid, manganous chloride. Mix after each addition, being careful that the alkaline solution is thoroughly neutralized with acid before adding the manganous solution. Transfer to a casserole, add starch and stir. If the sample turns blue, oxidizing substances are present, which will give high oxygen results. This may be nitrites, iron, lead oxide or other contaminants. Unless its source can be found and eliminated, titrate with thiosulphate to determine the exact amount required to destroy the blue color, and use this as a correction to be deducted when determining dissolved oxygen.

If no blue color develops, indicating the absence of positive interference, add one drop of iodine solution (approximately N/40). If a blue color does not develop, negative interference is present and the results for oxygen will be low. The necessary correction can be estimated by continuing to add iodine from a burette until a blue color develops and then titrating with thiosulphate an equal amount of iodine solution, added to distilled water.

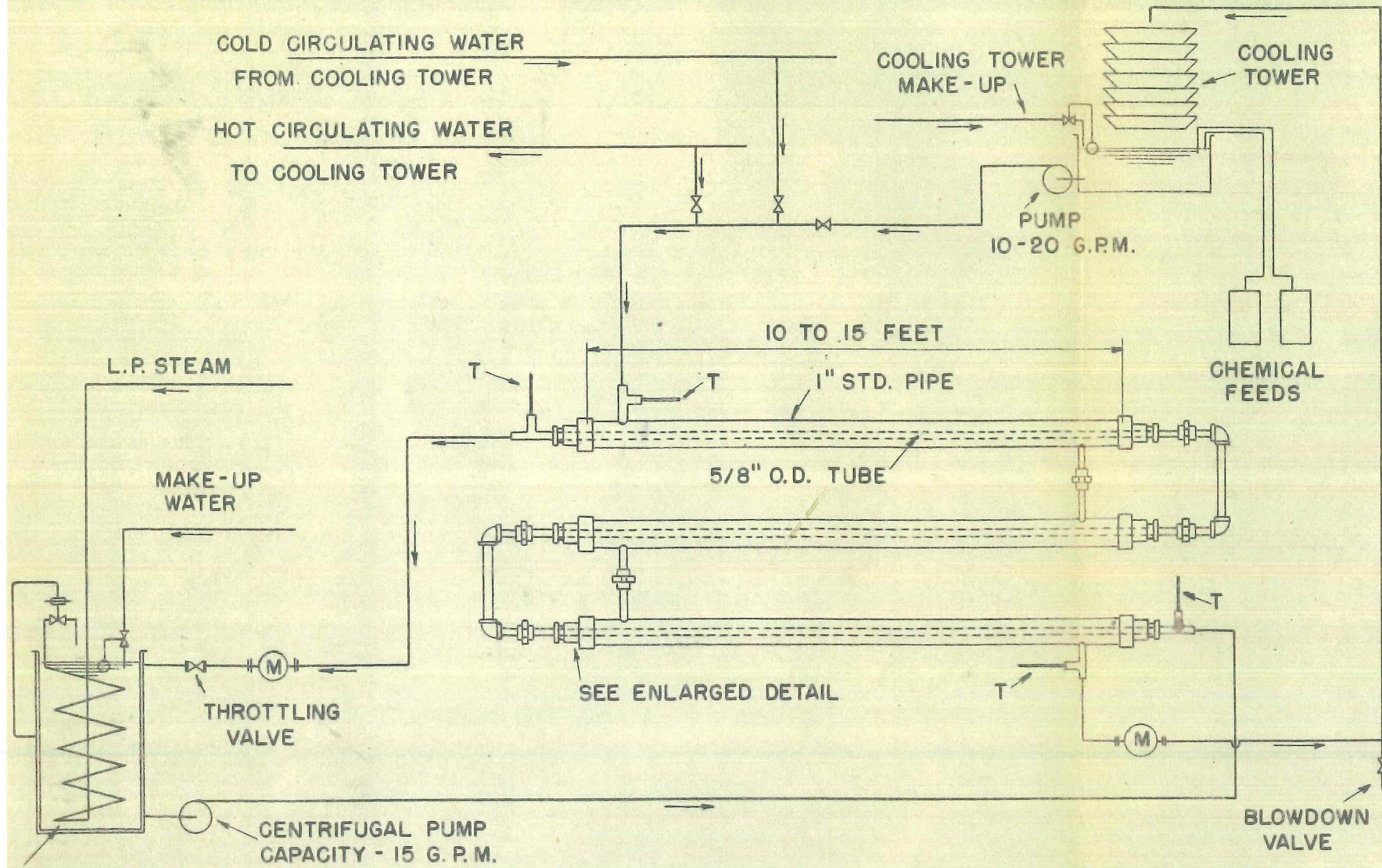
REAGENTS REQUIRED:

- (1) Manganous chloride solution. Dissolve 425.5 grams of crystalline manganous chloride, $MnCl_2 \cdot 4H_2O$, in one liter of distilled water.

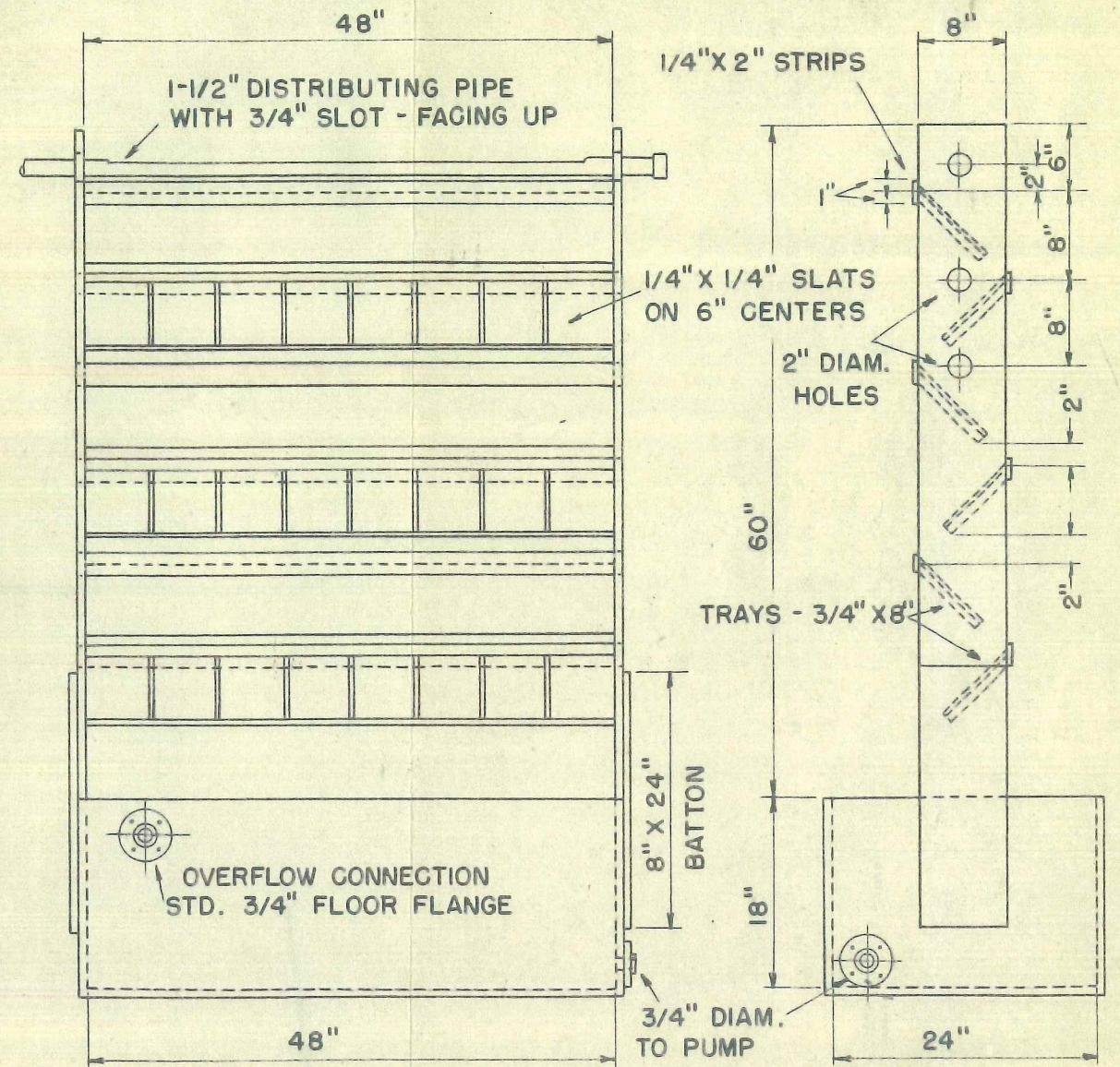
- (2) Alkaline Potassium Iodide. Dissolve 700 grams of c.p. potassium hydroxide, and 150 grams of c.p. potassium iodide, in separate portions of distilled water.
- (3) Sulphuric Acid, 1:1. Mix c.p. concentrated sulphuric acid (sp. gr. 1.84) with an equal volume of distilled water, pouring the acid into the water in a small stream and stirring constantly.
- (4) Starch Indicator Solution. Dissolve 5 grams of arrowroot starch in 50 ml. of distilled water to make a thin paste and then pour into 200 ml. of boiling water and continue boiling for 4 or 5 minutes. When cold add a few drops of chloroform and transfer to a dark glass-stoppered bottle. Starch solution does not keep well and should be made fresh every week.
- (5) Standard N/40 Sodium Thiosulphate Solution. This solution is not permanent, and must be replaced every two weeks, or calibrated weekly by titrating an N/40 standard solution of potassium dichromate, potassium permanganate or standard iodide iodate solution used for the sulphite test. To prepare the thiosulphate solution, dissolve 6.205 grams of chemically pure recrystallized sodium thiosulphate in water and dilute to one liter. The water used must be distilled and freshly boiled and cooled.



SCHEMATIC ARRANGEMENT OF PILOT PLANT FOR SCALE & CORROSION STUDIES



NOTE: 1-1/2" DISTRIBUTION PIPE MAY BE LOCATED AT VARIOUS ELEVATIONS IN ACCORDANCE WITH AMOUNT OF COOLING REQUIRED



DETAIL OF COOLING TOWER TO BE CONSTRUCTED OF 7/8" DRESSED LUMBER - REDWOOD, GYPRESS, OR CEDAR

CONTACT HEATER MAY BE USED IN PLACE OF HEATING COIL

NOTE: LENGTH & NUMBER OF TUBES DEPENDENT ON RANGE OF HEAT TRANSFER USED. DUPLICATE INSTALLATIONS SHOULD BE PROVIDED TO COMPARE TREATMENTS. M = VOLUMETRIC METER T = THERMOMETER

