

CONF-790679--1

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SILICA CONTROL AND MATERIALS TESTS AT THE SALTON SEA GEOTHERMAL FIELD

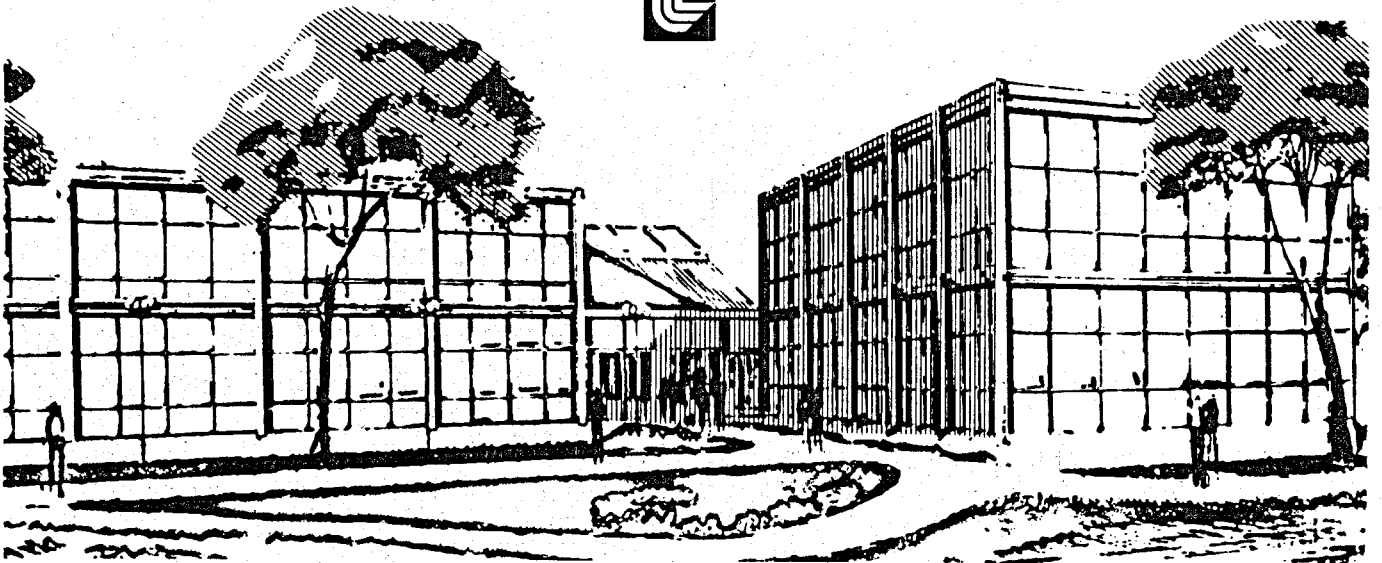
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June 7, 1979

MASTER

Third Annual EPRI Geothermal Conference
Monterey, California
June 26-29, 1979

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SILICA CONTROL AND MATERIALS TESTS AT THE SALTON SEA GEOTHERMAL FIELD

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INTRODUCTION

The Lawrence Livermore Laboratory maintains and operates a test facility near Niland, California, in the Imperial Valley for field studies on SSGF brine chemistry, scale and solids control, materials, and injection. This paper is an overview of recent work in silica control and materials testing.

SILICA CONTROL

Recently a number of commercial organic and inorganic chemicals have been tested as inhibitors of silica precipitation and scaling. The first bench-scale tests¹ conducted off-line revealed that many compounds containing the ethylene oxide moiety, $-\text{CH}_2-\text{CH}_2-\text{O}-$, were effective in stabilizing colloidal silica in geothermal brine. These initial measurements also gave an indication of the classes of compounds that were promising for further testing in a test apparatus designed especially for brine treatment studies at the Niland test site.

The types of additives and the tests conducted are classified into two groups, supported by separate programs in the Division of Geothermal Energy, DOE.

- (1) Proprietary chemicals - the identity of which in many cases is proprietary. The evaluation of these products was sponsored by the Industrial Support Program and, as such, the test objectives and schedule were closely integrated with the 1978-79 test plan for the SDG&E/DOE Geothermal Loop Experimental Facility.
- (2) Generic chemical compounds - these were selected on the basis of their organic functional groups and possible activity toward silica. This work was sponsored by the Technology Development Program of DGE.

This present paper is a review of the highlights of the results of tests of proprietary and generic chemical additives. A brief summary of an experiment on the use of sludge seeding for scale control is also presented. More detailed descriptions of these results are also available.²⁻⁴

Test Apparatus

The system for testing the scale control additives is shown schematically in Figure 1. In this apparatus, two-phase fluid from the geothermal well is first passed through a wellhead centrifugal separator. The steam is discarded and single-phase brine is obtained at nearly wellhead temperature and pressure. The brine is then divided into two parallel channels for the testing of the scale control additives. The brine in each channel is flashed from $\sim 200^\circ\text{C}$ temperature to 125°C in flash vessels and then passed to an atmospheric receiver. A third "delay stage" provides brine at atmospheric pressure, $90-100^\circ\text{C}$, and aged ~ 10 minutes with respect to its input.

Brine flow is maintained in each channel at 7.0 gpm (~ 1 lb/sec). Flow is monitored with an orifice meter with a plate that is cleaned periodically. Additive solution (at about 0.5-1.0% strength) is metered into the brine approximately eight feet upstream of the first test specimen using high pressure pumps equipped with pulsation dampeners. The flow is monitored and maintained in the range of 0.02 to 0.04 gpm by means of turbine flow meters. The accuracy of maintaining a desired concentration of additive was determined by means of a cesium-ion tracer study and found to be $\pm 10\%$.

Experimental Procedure

The scaling tendency of the brine is measured at various points in the system by the use of specimen pipe spools, flat coupons, (mild steel, Teflon, and Hastelloy), small diameter tubing, and perforated disks. Corrosion rates of materials exposed to the brine are estimated using Petrolite Instruments three-electrode electrochemical equipment.

To measure the effect of the additives on the rate of precipitation of silica, brine samples are collected for study from the 125° sampling ports. As collected, the brine flashes to 105°C . It is then placed in air-tight, Viton-gasketed, 130-ml, screw-cap glass bottles and incubated at 90°C . At appropriate intervals after sampling, the bottles are opened and the contents filtered

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SCALE-CONTROL TEST SYSTEM

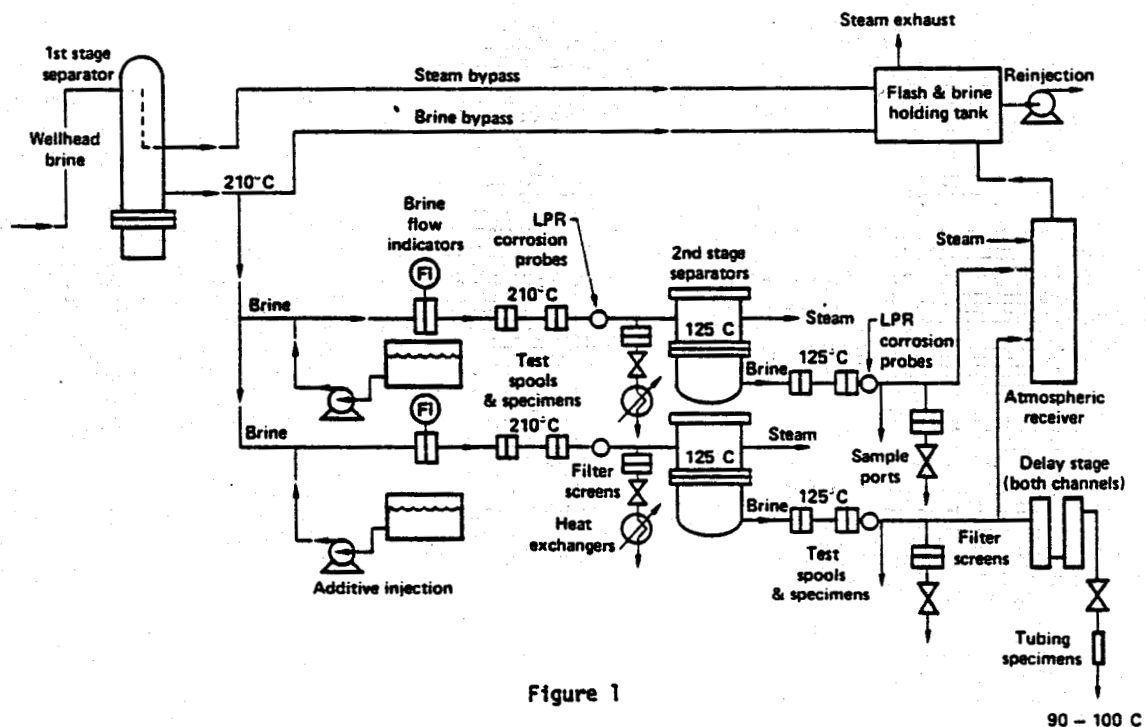


Figure 1

90 - 100 C

through fine-porosity glass crucibles. The silica remaining in the filtrate is measured by atomic absorption spectrophotometry using the method of standard additions. The collected solids are dried in air at 105°C and weighed as a measure of suspended solids concentration of the brine. Measurement of the silica in this manner has been shown to yield values for the total concentration of silica (monomeric, polymeric, and particulate <1 μm in size) not retained by the filter. The initial concentration of silica in the brine sampled at the effluent port was determined in samples immediately stabilized by acidification with hydrochloric acid.

Results of Proprietary Additive Tests²

Table 1 lists the proprietary additives that were examined for use as antiscalants in geothermal brine, together with some information supplied by the manufacturer regarding their composition. These were obtained for testing in response to an industry-wide solicitation, and virtually represents the state of the art (Fall, Winter, 1978) in commercial mixtures for scale control in boilers and other geothermal applications. Two of the mixtures, Cortron R-16 and the

Austral-Erwin oil additive, were somewhat different because they were designed to prevent the adherence of the silica scale, rather than its nucleation. Cortron R-16 also is reported to function as a corrosion inhibitor. These additives were evaluated using brine from the Magmax No.1 well at the Salton Sea Geothermal Field. The average properties of this brine when flashed to atmospheric pressure were the following: Chloride, 4.1 Mol/l; total dissolved solids, ~22%; pH 6.0; density at 25°C, 1.16 g/cm³; and SiO₂, 470 mg/kg. The durations of the scaling tests were from 64 to 113 h.

Table 2 summarizes the results of the tests of the proprietary additives. Four of the additives obtained later in the investigation (Polysperse Plus, Geomate 259, XFS-43075, and Visco 3744) were evaluated only as precipitation inhibitors. At 200°C, the rate of accumulation of scale (primarily heavy-metal sulfides) from the untreated brine was of the order of .05 mils/h, thus it was difficult to obtain a quantitative measure of the scaling rate here. The effect of Thermosol APS is less than the estimated experimental uncertainty. Although shown only as a 0% reduction factor in Table 2, several additives appeared

Table 1. Proprietary Additives Evaluated for Use as Antiscalants in Hypersaline Geothermal Brine.

<u>Compound</u>	<u>Company</u>	<u>Chemical Type</u>	<u>Concentration Tested, ppm</u>
Betz 419	Betz Laboratories	Acrylic Polymer + Phosphonate	20
Polysperse Plus	Betz Laboratories	-	20
Geomate 256	Dearborn Chemical	Polymer + Phosphonate	35
Geomate 259	Dearborn Chemical	-	40
CL-165	Calgon	Polymer Mixture	18
Drewspers 747	Drew Chemical	Polymer + Phosphonate	15
Thermosol APS	Far Best	Phosphonate	20
S-404	C-E Natco	Polymer	18
SC-210	Southwest Specialty Chemicals	Carboxylic Acid Polymer	10
Cortron R-16	Champion Chemicals	Filming Amine	120, 20
XFS-43075	Dow Chemical	-	40
Visco 3744	Nalco	Acrylic Polymer + Surfactant	20
Oil Additive	Austral-Erwin	Cottonseed, Tall Oils + Other Additives	40

to increase the thickness of scale compared to that formed from the control -- untreated brine. The formation of such a "pseudoscale" (see Reference 5) would, of course, not be acceptable in plant operation. In general, visual inspection indicated that none of the additives had a beneficial effect on the high temperature scale, but a more accurate assessment must await experiments employing much longer exposure times.

At 125°C, the scaling rate ranged from .1 to .4 mils/h. The scale formed at this temperature and below is predominantly silica. As can be seen in Table 2, some of the additives did clearly inhibit the formation of scale on mild steel at 125°C. SC-210 and the Drewspers 747, were probably the best, but neither was an unqualified success. As mentioned above scaling rates were measured on three materials: mild steel (AISI Type 1009), Teflon (TFE), and Hastelloy C-276. The latter two surfaces do not corrode and, therefore, yield scaling rates that are probably more representative of the long-term behavior of the medium. None of the proprietary additives significantly reduced the accumulation of scale on the Teflon and Hastelloy coupons. In contrast, the initial scaling rates on mild steel result from both scaling and corrosion processes. The shorter the exposure, the more important the corrosion process will be in relation to the deposition of the silica scale. Among all of the additives, probably only SC-210 warrants further testing. It reduced scaling the most at 125°C, and its electrochemical corrosion-

rate data (higher than the control values) indirectly indicated antiscalant activity.²


Finally as noted in Table 2, none of the additives had a measurable effect on the rate of precipitation of silica from the effluent brine. This suggests that if the additives are to have an effect on the scaling rates, they would have to function by influencing the adherence of the scale rather than its rate of growth from the brine.

Results of Sludge Seeding Tests²

Another approach to scale control that has been attempted by other investigators (see, for example, Reference 6) is to add to the fluid a finely divided solid upon which the scale-forming compound deposits in preference to the plant surfaces. The rationale is similar to the addition of seed crystals to promote precipitation in crystallization processes. Although the scale with which we are dealing is predominantly amorphous silica, by providing a large ratio of seed-to-plant-surface area, reduction of the degree of supersaturation and hence the scaling tendency of the brine should be attainable. Also, to be a viable technique, the seed substance must remain fluidized and pass through the plant equipment with minimal holdup.

For Salton Sea geothermal brines the ideal seed material would be colloidal silica. Closely approaching such material is the wet sludge obtained from the sedimentation of the

Table 2. Performance of Proprietary Additives Evaluated as Antiscalants in Hypersaline Geothermal Brine

Additive	% Reduction in scale on mild steel				Precipitation 90°C
	200°C		125°C		
	Pipe Spool	Coupon	Pipe Spool	Coupon	
Betz 419	0	0	0	52	Negative 
Polysperse Plug	-	-	-	-	
Geomate 256	0	0	0	32	
Geomate 259	-	-	-	-	
CL-165	0	0	0	32	
Drewperse 747	0	0	30	50	
Thermosol APS	30	30	0	50	
S-404	0	0	-	21	
SC-210	0	0	80	42	
Cortron R-16	0	0	0	0	
XFS-43075	-	-	-	-	
Visco 3744	-	-	-	-	
Oil Additive	0	0	0	0	

effluent brine from the GLEF prior to injection. This sludge is of mud-like consistency and is a finely-divided precipitate composed primarily of silica, with lesser amounts of iron compounds and metal sulfides, i.e., all of the usual ingredients of geothermal scale. Using sludge as a seed material appeared promising because earlier work⁷ on developing an effluent process for this brine demonstrated the effectiveness of pre-precipitated solids contact as a means of rapidly promoting the precipitation of silica.

A short-duration seeding test was conducted in which wet sludge containing 20% by weight solids was metered into the brine at 200°C in the same manner as the chemical additives. Sludge flow rates of 0.30 and 0.15 gpm were used. Because of the accumulation of the sludge itself on all of the test specimens, it was impossible to measure directly the extent, if any, of scale reduction obtained. However, our measurements of the levels of "dissolved" silica in the brine in the presence and the absence of the sludge showed a significant effect. At high temperature (200°C) and a sludge flow rate of 0.15 gpm, the seeding process apparently reduced the level of dissolved silica from 453 to 416 mg/kg which may not be a significant change; but from 516 to 306 or 396 (depending on the measurement technique²) at 125°C, which is a substantial reduction in the degree of supersaturation. From the limited data obtained, it does appear that this method of scale control has promise and further tests should be conducted.

Results of Tests of Generic Organic Compounds^{3,4}

As discussed above, a parallel program of testing is being conducted in which specific organic compounds are being evaluated systematically as potential scale control agents. The principal line of this investigation is first to screen compounds as inhibitors of the precipitation of silica at 90°C, and then, if they show promise, to test them more thoroughly in a scaling test.

Thus far, over 60 substances representing several different classes of organic compounds have been examined by injection into the brine at 200°C in the test system and measurement of the kinetics of silica precipitation in the brine held at 90°C. The classes of compounds found to be most effective as precipitation inhibitors are listed in Table 3. As was speculated previously,¹ the oxyethylene moiety clearly appears to be the source of activity in many of the substances that interact with colloidal silica, possibly because of hydrogen bonding to the silanol groups of the silica particles.⁸ However, two nitrogen-based compounds, the polyethylene imines and polyethyloxazolines, also show activity. Polyethylene imine is $(-CH_2CH_2NH-)_n$, very similar in structure to the polyoxyethylenes.

Only limited data have been obtained thus far on the effects of the generic compounds on brine scaling rates. In a 100-h test of Natrosol 250LR,² it was found that at

Table 3. Classes and Examples of Organic Compounds that Inhibit the Precipitation of Silica from Hypersaline Geothermal Brine. (Injection at 200°C; Observation at 90°C)

<u>Class</u>	<u>Trade Name Name</u>	<u>Chemical Name</u>	<u>Manufacturers</u>
Hydroxyethylcelluloses	Natrosol 250LR	Hvdroxyethylcellulose	Hercules
Ethoxylated Amines	Ethomeen C/25	Polyoxyethylene(15) cocoamine	Armak
Ethoxylated Quaternary Ammonium Compounds	Ethoquad 18/25	Methylpolyoxyethylene(15) Octadecylammonium Chloride	Armak
Polyoxyethylenes	Carbowax 14,000	Polyoxyethylene, M.W. = 14,000	Union Carbide
Polyoxyethylene-Polyoxypropylene Copolymers	Pluronic F38	Polyoxyethylene-Polyoxypropylene Copolymer, M.W. = 5,000	BASF-Wyandott
Polyethylene Imine	Corcat P-200	Polyethylene imine, M.W. = 20,000	Cordova
Polyethyloxazaline	XD-8779.00	Polyethyloxazaline, M.W. = 60,000	Dow Chemical

125°C it reduced the scale on the mild steel coupons by ~50%, but not at all on the pipe spools and inert coupons -- similar in performance to the best proprietary additives. A short duration scaling test of Carbowax 14,000 revealed that it significantly reduced the soft, dendritic silica scale that is formed at 90°C, but increased the quantity of scale at 125°C. At the higher temperatures^{3,4} many of these substances have limited solubility and, hence, activity toward scale inhibition. We have obtained compounds with similar structure identified to be active toward silica and that are more soluble at the higher temperatures for further scaling tests in the near future. Attempts will be made to optimize their performance at the higher temperatures.

MATERIALS TESTS

The LLL materials test program includes both downwell and surface exposure of potential well casing and plant component materials for SSGF brines. Downwell tests have consisted of exposure of coupons and a 1.66" diameter test string in wells supplying brine to the GLEF. The purpose is to determine the API grade of steel best suited for well casing application and to evaluate the composition dependent variations within API specifications that influence corrosion rates. This work is funded by the Industrial Support Program of DGE. Results of these exposures are presented below.

Work funded by the Technology Development Program of DGE include a new test apparatus, consisting of removable 12" diameter spools that are installed in a bypass loop off the

Magnamax #1 production line. The capabilities of this new test facility and the scope of the first scheduled test are also presented. In addition, a second string of well casing materials is in preparation.

Well Casing Materials

Our concern is directed toward finding a cost-effective material which will survive for 20-30 years as a well casing in hypersaline geothermal fields. Our previous work^{9,10} with acidified geothermal brines in connection with surface piping materials suggested that low to intermediate alloy steels containing chromium and molybdenum have significantly lower corrosion rates than carbon steel. Downwell environmental conditions resemble the acidified brine conditions because of the high dissolved CO₂ content downwell.

The first downwell corrosion tests were conducted in summer 1978 when we placed a fixture of corrosion coupons at a depth of 1800 feet in Magnamax I well. The temperature at this depth was 260°C. Specimens were exposed for 3 months. The results indicated that carbon steels (AISI 1018 and ASTM A106B) corrode at rates of 20-400 mils/year (calculated from the weight loss), and that the corrosion rate is dependent on the steel microstructure. The carbon steel specimens suffered very severe localized corrosion. The principal corrosion product was Fe₃O₄. By comparison, two commercial alloy steels, ASTM A387 Grade 5 (5-1/2 Cr-21 Mo) and Grade 9 (9 Cr-1 Mo) showed corrosion rates of 4-5 mils/year. These alloys were slightly pitted. Included in the

test were high-performance alloy materials. Of these, type 430 stainless steel exhibited moderate to heavy corrosion attack (corrosion rates greater than 20 mils/year), while 26 Cr-1 Mo showed only light pitting (corrosion rate about 1 mil/year). Alloys 29 Cr-4 Mo, Inconel 600, Hastelloy B, Hastelloy C-276, MP 35N and commercial purity Ti showed no discernible corrosion attack and negligible weight loss.

The specimens were cylindrical, 1/4-inch diameter and approximately 1-1/2-inch long. They were insulated with Teflon from one another and the support fixture. The assembly of coupons rested inside a perforated Teflon sleeve. While the perforated sleeve permitted exposure to the geothermal brine, relatively stagnant conditions existed around the corrosion specimens. Thus, the corrosion rates are relative and serve as a basis of comparison from one alloy to another. Higher rates would be expected under flowing conditions. The exposed specimens were encrusted with PbS-rich scale.

The corrosion test fixture was maintained at the 1800 ft. level by an API J-55 carbon steel, 1-1/4-inch well casing string. The string was comprised of 30-ft. sections of this material with upset ends. Several sections of this string suffered severe corrosion. The most attacked sections were from the lower depths. Also, the attack was most severe near the upset ends. The attack caused perforation of the casing string (0.140-inch wall) in many places. Metallographic analysis performed after exposure indicated that the corrosion was most severe in locations where there was an abrupt change in microstructure. The change in microstructure occurred near the upset ends, because the tube is reheated during the upsetting operation and is usually not subsequently heat treated to restore the original normalized structure. Pipe sections which were microstructurally more uniform from location to location suffered much less corrosion.

This result suggests that galvanic effects occur between different locations on a pipe. High conductivity of the brine favors this kind of corrosion. We are pursuing an investigation of microstructural effects on corrosion rate by performing different heat treatments on API steels.

Recently, we exposed a 1700 ft. string for 3 months in Woolsey I well. The string was comprised of three different API steels: J-55, N-80, and C-75. Again, most of the corrosion was confined to the bottom 200 ft. of string. Of the three steels, J-55 was the most attacked and the attack occurred mostly near the upset ends. The N-80 and C-75 appeared relatively unattacked. Our analysis of the

string is not complete at this time, but the improved performance of the N-80 and C-75 may be due to post heat treatment after the upset operation. N-80 is normalized and C-75 is quenched and tempered.

We are preparing to expose an additional 1800 ft. string in Magmax I well for a 6-month period. The string will be comprised of API, ASTM, and AISI carbon and alloy steels. In addition, we will heat treat "pups" (3-ft. lengths) of API J-55 and N-80 steels to obtain different microstructures. The ASTM steels include steels with 1/2 Mo, 1 Cr-1/2 Mo, 2-1/4 Cr-1 Mo, 5-1/2 Cr-1/2 Mo, 7 Cr-1/2 Mo, and 9 Cr-1 Mo compositions.

Plant Materials

Our work to date indicates that iron-base materials undergo both general and localized corrosion in contact with geothermal brines and steams. While alloying additions (Cr and Mo) improve the general corrosion resistance of the steel, the effect on localized corrosion is not as clear. Little information is available in the open literature concerning the performance of alloy steels in anaerobic, moderately acid (due to CO₂) conditions. Results from our work with CH₄ acidified brines indicates formation of wide shallow pits on Cr - Mo alloy steels. The key question is how these pits grow with time - do they grow laterally and coalesce resulting in a more or less generalized wastage or do they grow into the pipe wall resulting in perforation. Do the pits become inactive with exposure time? Pitting corrosion observations are generally made only a few times during the course of, or only at the end of, short duration tests. Longer time estimates are generally based on linear extrapolation, which could lead to erroneously low estimates, and thereby eliminate useful materials.

We are in the process of measuring the localized corrosion rate of 3 carbon steels and 5 alloy steels. Corrosion coupons will all be initially exposed to wellhead (approx. 225°C), two-phase brine in a 12-inch by-pass loop off the Magmax #1 production line. The test loop can be isolated with WKM valves to permit removal of the coupons which are mounted in 5-3' long spools. In operation the specimens will be exposed to full I-well flow. During shutdowns, the bypass will be drained and bathed with nitrogen to minimize air leakage. Specimens will be withdrawn periodically for weight loss and pit depth determinations. These withdrawals will occur over a 6 month accumulated exposure period.

This facility will give us the capability to evaluate time dependent factors and the effect of other metallurgical variables on localized corrosion in Niland brines.

ACKNOWLEDGEMENTS

The chemical testing in this work was carried out by C. H. Otto, Jr., S. B. Deutscher, R. Lim, A. D. Kupfer, L. Abrego, C. O. Pruneda, and W. P. Frey. Metal specimens and fixtures are prepared by R. E. Garrison and W. P. Frey. A. Goldberg collaborates in the corrosion analysis. Metallurgical examination of specimens is done by E. O. Snell.

Work performed under the auspices of the U. S. Department of Energy by the Lawrence Livermore Laboratory under contract number W-7405-ENG-48. Reference to a company or product name does not imply approval or recommendation of the product by the University of California or the U. S. Department of Energy to the exclusion of others that may be suitable.

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"Work performed under the auspices of the U.S. Department of Energy by the Lawrence Livermore Laboratory under contract number W-7405-ENG-48."

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