

ZERO LIQUID DISCHARGE OPERATION OF COOLING TOWERS AT A MAJOR STEEL MILL

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ABSTRACT

Two new cooling towers were started up on ZLD water chemistry at a major steel mill to minimize discharge waste treatment costs and reduce water use. High TDS (total dissolved solids) water chemistry that results from ZLD required new scale and corrosion inhibition technology to protect galvanized and steel surfaces. After two years operation, galvanized tubes and mild steel in systems show outstanding corrosion protection, control of “white rust” and no scale at TDS up to 150,000 in tower water. New technology that produces inhibitor chemistry from source water silica permits operation at high TDS without scale or corrosion, and provides biostatic water chemistry. Water treatment costs are less than traditional chemical treatment, and chemical handling and storage is eliminated.

Keywords: Zero Liquid Discharge, ZLD, Total Dissolved Solids, high TDS, silica inhibitor chemistry, corrosion inhibition, scale inhibition, cooling tower, green chemistry.

INTRODUCTION

In 1984, California Steel Industries reopened operation of the prior Kaiser Steel facility that was commissioned in the 1940’s. They installed additional waste water recovery and reuse systems in 1992, and has continued to optimize systems to conserve water and avert wastewater discharge issues. The site currently reuses their own treated sewage as makeup to cooling water systems. The local southern California community still lacks abundant water resources or wastewater discharge infrastructure to serve this operation, and CSI has continued to explore and develop alternatives to manage its own water use and wastewater discharge requirements in coordination with area municipalities and regulatory authorities.

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They installed new evaporative coolers at two different site locations in 2006 to provide secondary cooling of closed cooling loops for air compressor equipment. No wastewater discharge collection lines were in place for tower blowdown, and previous tower blowdown had to be collected in wastewater collection pits or tanks from which their tank truck could haul this water to their in-plant waste processing system. Use of a zero liquid discharge approach for cooling tower water treatment allowed them to avert either significant wastewater hauling cost or capital cost investment to install a wastewater discharge collection system.

The EC West location installed one cross flow fluid cooling tower to support three air compressors. The EC 10 location installed one counterflow fluid cooling tower to support a single air compressor. Both cooling towers were constructed with galvanized tube bundles and galvanized housing. The crossflow unit included a 304 SS basin.

The typical operating temperature drop across the crossflow tube bundle ranges from 5 to 15 °F with a 340 GPM evaporative cooling water circulating flow, and one to two operational air compressor load. The average temperature drop across the counterflow tube bundle is 24 to 28 °F with a 115 GPM evaporative cooling water circulating flow, and the operational load of the single air compressor. Prior to replacement of these two systems, loss of adequate tower water cooling caused the air compressors to experience high temperature trip outs, with frequent seal failures and oil changeouts from overheating. Scaled evaporative condenser tube bundles in the old cooling towers caused the closed loop cooling water temperatures to run 10 to 20 °F higher than design operating conditions.

Makeup water to the cooling towers is provided from both municipal and well sources using the same aquifer, and have equivalent water quality. The dissolved mineral content of this source water presented significant scaling potential from both hardness and silica, and corrosion from total dissolved solids (TDS) content. Providing reliable chemical treatment control for these systems to prevent scale, corrosion and biological fouling was difficult with varying operational loads and required discharge (blowdown) of 30 to 40% of makeup water to avoid concentration of hardness toward scale forming water chemistry.

ZLD / Silica Chemistry

Application of a new patented and licensed corrosion and scale inhibition technology was well suited for this situation, because it permitted ZLD operation of the cooling towers^{1,2}. The process eliminates scaling potential by removing hardness with ion exchange pretreatment (softening). Silica in the source water, that is normally associated with scale deposition, is modified by the silica chemistry control process to non scaling forms of silica. This modified silica chemistry also provides outstanding corrosion protection to system metals. The silica corrosion inhibition mechanism is so effective that cooling water can now be concentrated to TDS levels that would not have been permitted with traditional chemical inhibitors.

This technology also provides opportunity to implement “green chemistry”, since use and discharge of environmentally restricted organic, phosphate and heavy metal containing chemicals used in traditional water treatment are eliminated. With higher tower water concentration, even use of biocidal agents may be eliminated or reduced due to impeded biological proliferation of fresh water organisms by high TDS concentration in tower water. ZLD operation permits discharge issues to be averted.

Discussion of the silica chemistry and the mechanisms of scale inhibition and corrosion inhibition were previously presented in a paper at Corrosion 2007¹. However, a substantial list of references has been provided in this paper for information on silica chemistry behavior, and development of the currently applied technology^{1-3, 9-14}. It is interesting to note that much was known about the corrosion inhibiting properties of silica in other applications outside of high TDS cooling water treatment, and in the steel industry in particular. Amorphous silica coatings have been studied as an alternative means to protect galvanized steel from white rust in sheet production and shipping, and is used to coat and protect the inside of stainless tubing used for hydrochloric acid dispensing from chloride attack⁴⁻⁵. It is also used to coat and provide additional corrosion protection for galvanized steel parts used in automotive parts manufacture⁶.

ZLD / Salt Solubilities

One of the chemical abnormalities of this technology is often difficult for professionals trained in traditional water treatment technology to accept. This being the relatively unlimited concentration of TDS in ZLD cooling tower water, as contributed by makeup water sources, without scale or precipitation. Most realize that use of softened makeup water permits concentration of water in low pressure boilers to relatively high TDS levels and alkalinity before reaching limiting concentrations that affect foaming or carryover. Cooling towers, utilizing this treatment chemistry, do not have such limitations for foaming or insoluble salt formations. Some slight foaming tendencies may be experienced at lower TDS concentrations, but foaming also subsides at higher TDS concentrations. Certainly, operators of water vapor recovery evaporators are familiar with operation at 15% or greater dissolved solids ranges, although they rely on recycle of suspended solids for adsorption and precipitation in the process.

However, most would not have anticipated that TDS concentrations exceeding 15% TDS (150,000 mg/L), with pH greater than 9 and approaching 10, could be maintained without scale or precipitation of low solubility polyvalent metal salts of calcium and magnesium. Some want to apply the numerous scaling indexes to this chemistry, but such indexes become irrelevant with control at low residuals of calcium and magnesium and with increased solubility that results from high TDS (or ionic strength) and non common (or foreign) ion effects. Replacement of calcium and magnesium ions in the makeup with monovalent sodium ion and concentration of natural alkalinity (or addition of caustic that is converted to carbonate alkalinity by carbon dioxide adsorbed by the cooling tower) results in highly soluble sodium carbonate as the major controlled chemistry in the tower water. Thus, high concentrations of sodium ions become the non common ion to low solubility polyvalent metal compounds such as calcium carbonate or calcium phosphate, and increase their solubility. As the sodium concentration increases, the solubility of salts such as calcium carbonate, calcium sulfate and calcium phosphate increase.

A common example of this non common ion effect is represented by seawater which is composed by weight at 55.04% chloride, 30.61% sodium, 7.68% sulfate, 3.69% magnesium, 1.16% calcium and 1.10% potassium. Seawater, as well as some oilfield brines, are rich in sodium or monovalent metal ion salts and high in ionic strength. Calcium and magnesium are at significantly greater soluble concentrations in seawater than continental surface waters. **Figure 1** shows that the solubility of calcium sulfate is more than doubled in sodium chloride at

concentration that approaches that found in seawater (25,000 mg/L). Higher tower water TDS concentrations, approaching 150,000 TDS, provides significantly increased solubilities for such calcium salts, even when approaching pH 10 in the tower water. The makeup and cooling tower chemistry material balance data (COC) discussed and presented later in this paper, as illustrated in **Tables 1 & 2** show such solubility impact on ions from the softened water source. One of the most limited solubility compounds at high pH, calcium phosphate, has been found to remain soluble and concentrate to the 50 to 100 mg/L range at pH 10 in these two ZLD systems.

The basic effects on solubility produced by common versus non common ion pairs, ionic strength and respective activity coefficients in high TDS / salt solutions is part of many college undergraduate laboratory educational experiments. The increase in solubility of an electrolyte in a solution of a second electrolyte with no common ions compared with pure water is not an easy concept to grasp because it seems to be counterintuitive. A simple college experiment to dissolve $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ (gypsum) in pure water and in 0.25 M NaCl will show that gypsum dissolves almost completely in the sodium chloride solution, but not in pure water. Determination of mean activity coefficients using measured concentrations and application of the Davies Equation, an extension of Debye–Hückel theory, will yield the non common ion effect on solubility. The basic principle is there are ionic interactions between the solute ions and the solvent ions, which allow for more dissolution because only free ions enter into the expression for the solubility product equilibrium constant. From a simple mathematical point of view, in higher ionic strength solutions, activity coefficients for calcium and sulfate become smaller, and hence the concentrations must be larger to maintain a constant solubility product at equilibrium. A more in depth discussion of such solubility behaviors is provided in a paper by Kester in studies of ion pair effects in seawater ¹⁵.

Application of the Stiff Davis calculation has been used to predict increased solubility impact in oilfield brines, but such brines, like seawater, do not have the higher buffered concentrations of carbonate alkalinity (pH 10) and control at low concentrations of the polyvalent metals (principally calcium and magnesium) provided by this cooling tower treatment process. So no attempt has been made to model the solubility behavior experienced.

Though water treatment specialists recognized that removing scale forming minerals (softening) from cooling water was a practical approach to protect some critical applications, they continued to rely on traditional treatment chemicals, unaware of the opportunity to convert silica from a deposition threat to a resource in corrosion protection and water conservation. This technology was not recognized as a cost effective and reliable process that could easily be employed to control the concentration and equilibrium of amorphous silica in cooling towers, as illustrated in **Figure 2**, or that it would provide outstanding scale and corrosion inhibition. The technology is now available to water treatment professionals and their customer applications through licensing.

EXPERIMENTAL PROCEDURES

Field Studies

Salt use efficient softening equipment was used to produce polished quality soft water and installed prior to startup of the new evaporative condensers at both locations. The design of this softening equipment reduced consumption and handling of salt by more than 50%, as regeneration used only 4 pounds of salt per cubic foot of resin (versus normal 8-12 pounds per cubic foot). This equipment design also produced 50% less regeneration water wastage than normal softening equipment, which was important since the brine waste had to be collected for in-plant waste processing.

Since the makeup source water was of potable quality, no further pretreatment was required beyond sodium cycle softening. Blowdown was eliminated from the tower, and no chemicals or biocides were added to the tower water. The soft makeup and tower water were monitored for TDS, pH, total hardness and soluble silica content. Boiological dip stick cultures and corrosion weight loss testing were conducted with mild steel coupons.

Tower and soft makeup water samples were obtained from the two towers for material balance comparison of chemical concentration (COC) of primary ion chemistries in the softened source water. Corrosion rate studies were conducted with weight loss test specimens via ASTM Standards G4-01 Standard Guide for Conducting Corrosion Tests in Field Applications and G1-03 Standard Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens.

Laboratory Studies

Electrochemical corrosion studies were performed to evaluate how silica inhibitor chemistry would perform at elevated temperatures with various metals¹. Testing was performed at temperatures of 77° F, 130° F, 160° F and 190° F (25° C to 88° C) with tower water test chemistry of 50000 TDS, 9000 chloride, 450 silica and pH 10. The independent study was conducted by Corr Instruments using real time coupled multi-electrode array corrosion probes which also accurately measure localized (pitting) as well as general corrosion rates⁷⁻⁸. The metals tested were carbon steel 1008, 316L SS, aluminum 1100, copper 1100 and zinc. Control testing was conducted with a 0.5 normal solution of sea salt (25000 TDS) without silica chemistry.

Coupled multi-electrode array sensors⁷ were used to measure corrosion rates of mild carbon steel (Type 1008 -- UNS G10080), Copper (CDA 110-UNS C11000), Aluminum (Type 1100, UNS A91100), pure zinc, and stainless steel (Type 316L--UNS S31603) in both the silica-inhibited and the non-inhibited sea salt waters. In a coupled multielectrode sensor, there are multiple miniature electrodes made of materials identical to the component of interest. Statistically, some of the electrodes have the properties that are close to the anodic sites and others have the properties that are close to the cathodic sites of the corroding metal. The resulting electrical currents are measured. The localized corrosion rate is calculated using the current from the most anodic electrode, and the general corrosion rate is calculated using the average anodic currents from all the electrodes in the array⁸. The multi-electrode array sensor probes were made of 1-mm diameter wires of these metals and each has 16 electrodes. During the study, these probes were immersed in the test solutions and connected to a

nanoCorr S-50* multi-electrode corrosion analyzer which gives both localized and general corrosion rates on real time. The corrosion analyzer was also connected to a temperature probe, an oxidation/reduction potential (ORP) probe, and a saturated Ag/AgCl reference electrode, all of which immersed in the same test solution.

RESULTS AND DISCUSSION

Field Study Results

Both the evaporative cooling systems have been operating on ZLD over the last two years with TDS between 25,000 and 150,000 (1X to 6X seawater). Typical soft makeup and system water chemistry analyses are provided in **Table 1 & Table 2**, and show chemistry of concentration (COC) of the principal ions produced in the softened source water. Notably, silica does not show the equivalent level of concentrations (COC) as other soluble ions due to the modification of the majority of the source water chemistry into higher molecular weight polymeric or colloidal forms not measured by the acid molybdate test which only measures soluble silica monomer.

Total hardness levels were controlled below 80 mg/L in the tower water, with 100 to 600 concentrations (COC) of softened source makeup water, as enabled by the polished quality of the pretreatment system. Soft makeup is the only important control requirement for plant operators with ZLD operation, since there are no chemical feed or blowdown control adjustments. Hardness in the tower water must be controlled below levels that would precipitate silica and interfere with the corrosion inhibiting film formation on metals. Thus, for high COC operation, hardness removal equipment must provide excellent quality and reliability. Pretreatment hardness leakage or upsets necessitate blowdown to lower hardness to avoid hardness and silica precipitation. No hardness leakage has occurred over two years with the two CSI pretreatment systems.

Scale Control and Heat Transfer. Water temperatures for the closed cooling water loops has operated at design conditions since startup of the new evaporative condensers, and air compressor maintenance requirements on this equipment has been reduced to expected levels. Inspection of the galvanized tube bundles on both the EC West and EC 10 towers after two years of operation on ZLD show no scale deposition, and galvanized tube surface appearance is comparable to their condition when installed with no galvanized white rust as shown in **Figures 3 & 4**. The EC 10 unit ran at the highest closed loop operating water temperature of 110 °F, with a 28 °F temperature drop across the tube bundle. It has also ran at the highest chemical concentration (582 COC) at 146,000 TDS.

Corrosion Inhibition. Carbon steel corrosion rates of < 0.005 mpy were measured by weight loss analysis on 61 day coupon exposure, showing only slight color variation from an unexposed (control) coupon as shown in **Figure 5**. Both coupons were cleaned and weighed using ASTM Standards G-4-01 and G1-03. A galvanized coupon was also installed, with 60 day exposure of the coupon for visual examination as shown in **Figure 6**, since the acid cleaning procedure for weight loss measurement strips the galvanized film from the coupon. The galvanized coupon surface appearance was equivalent to a non-exposed coupon. With

* Trade name of Corr Instruments, LLC, San Antonio, Texas, USA

the outstanding corrosion protection of steel with this technology, use of galvanized surface tube bundles is not necessary, thus providing opportunity for lower equipment cost for this type of cooling service.

Biological Monitoring and Control. No biological organisms were detected by biological dip stick cultures with plate counts of 10^0 after 48 hour incubation, and no bio growth was found in either system during the study.

Wastewater Discharge Reduction. The EC West and EC 10 systems were able to reduce (eliminate) blowdown discharge by 534,000 and 330,000 GPY respectively, and now produce less than 9,000 and 5,000 GPY from respective softener regeneration waste water. This reduction eliminates the necessity for daily waste haul pickups from each tower location's collection point to pickup only once per month for this source.

TDS Buildup on Drift Eliminators. Evaporation of dissolved salts (TDS) in tower water that is spashed from the basin results in buildup on drift eliminators in some tower designs with either ZLD or traditional chemical treatment. This buildup is accelerated by the higher TDS concentration in ZLD tower water. However, the ZLD salts are highly soluble since hardness has been removed from the source water, and they are easily washed back into the tower system. This build up is insignificant in most tower designs, as it was with the counterflow tower, while aesthetically undesirable in others. Buildup was experienced with the cross flow tower due to tendency for tower basin water splash onto the side eliminators. This was initially managed with periodic pressure wash with soft water supply, but later with installation of low volume soft water misting sprays outside the eliminators. The mister operation was tied to fan operation and timer to maximize spray water capture into the tower basin and limit its makeup contribution. The mister sprays have eliminated the buildup.

Laboratory Corrosion Inhibition Study Results

Figure 7 and **Figure 8** show the results of the evaluations with carbon steel and aluminum. Corrosion was mitigated to very low levels at each of the temperatures in the study for all metals. Carbon steel corrosion rates are comparable to those expected for stainless steel. Aluminum, which is amphoteric and soluble at pH 10, showed very low corrosion at all temperatures. Localized (pitting) corrosion was equally mitigated with all metals (pitting is typically 10X or greater than general corrosion rates). **Table 3** summarizes results for all metals tested with silica inhibitor chemistry and non-inhibited control results.

The study results in **Table 3** indicate that all of these metal types and their various alloys commonly utilized in cooling water contact applications are well protected by silica chemistry at higher water temperatures and high TDS. Thus, lower cost alternatives for heat transfer metal selection are available with use of this corrosion inhibitor chemistry.

SUMMARY & CONCLUSIONS

Averting the discharge handling costs for cooling tower blowdown was the principal justification for implementing ZLD in these systems. However, other benefits included improved performance and reliability of the air compressor equipment, reduced tower fan operation (from improved heat transfer), and reduced operator maintenance. Eliminating

chemical storage, handling and discharge was also beneficial from the environmental and safety perspective. Most significant is that the cost savings and system performance provided with this new approach did not have to be at the expense of water conservation or environmental impact.

This ZLD approach will facilitate additional opportunities to manage water use and wastewater challenges by addressing scale, corrosion and biological control issues that impact cooling water equipment energy efficiency, maintenance, and process operational reliability. It also provides ROI through water conservation and wastewater discharge reduction savings. Four additional cooling tower systems have been converted to ZLD at CSI since this study was undertaken.

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FIGURES & TABLES

Figure 1 – Increased Solubility of Calcium Salt in Sodium Chloride

Calcium sulfate (gypsum) solubility increases with increasing sodium chloride.

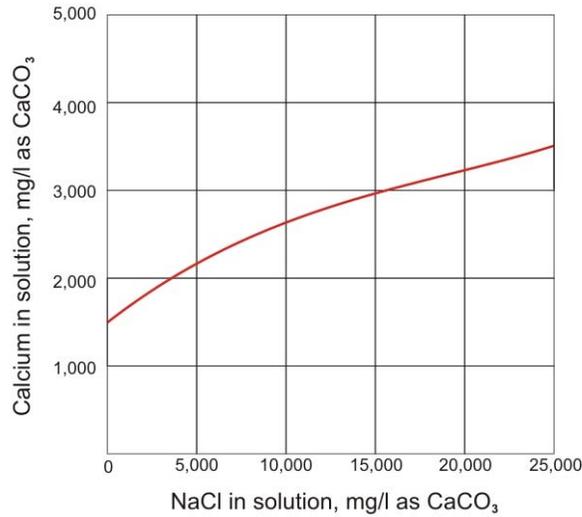


Figure 2 – Relationship between Soluble, Insoluble and Polymerized Silica Species at Varying pH and Concentration.

Species In Equilibrium with Amorphous Silica

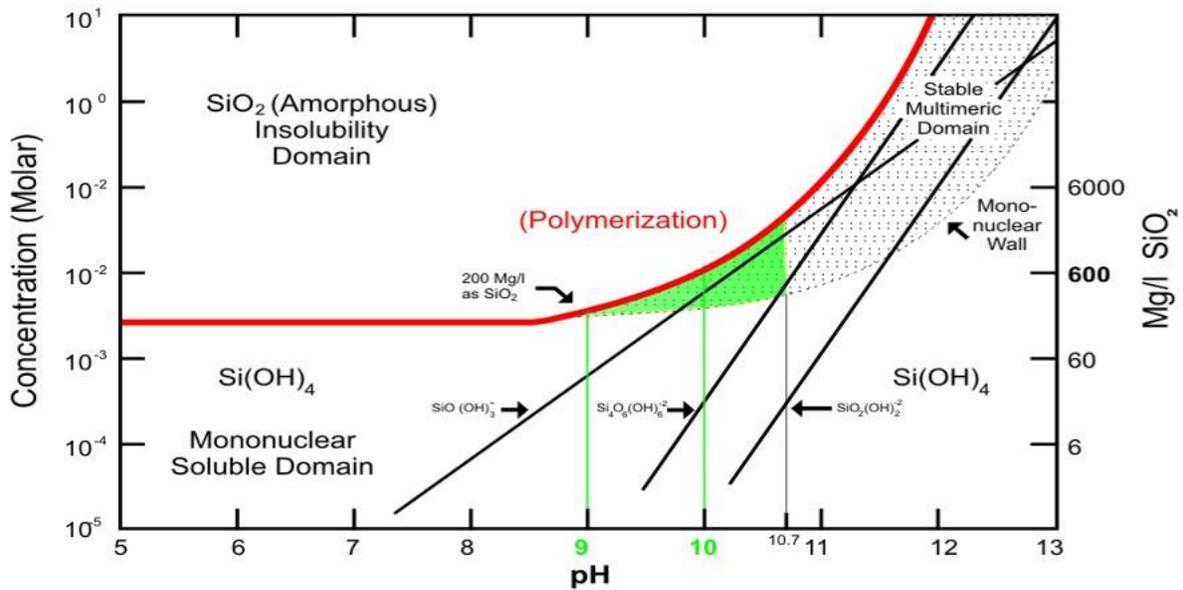


Figure 3 – EC West Galvanized Tubes (24 months Service)



Figure 4 – EC 10 Galvanized Tubes (20 Months Service)



Figure 5 - # 1652 CS (61 days, 0.017 mpy), # 1664 CS (control, 0.013 mpy)

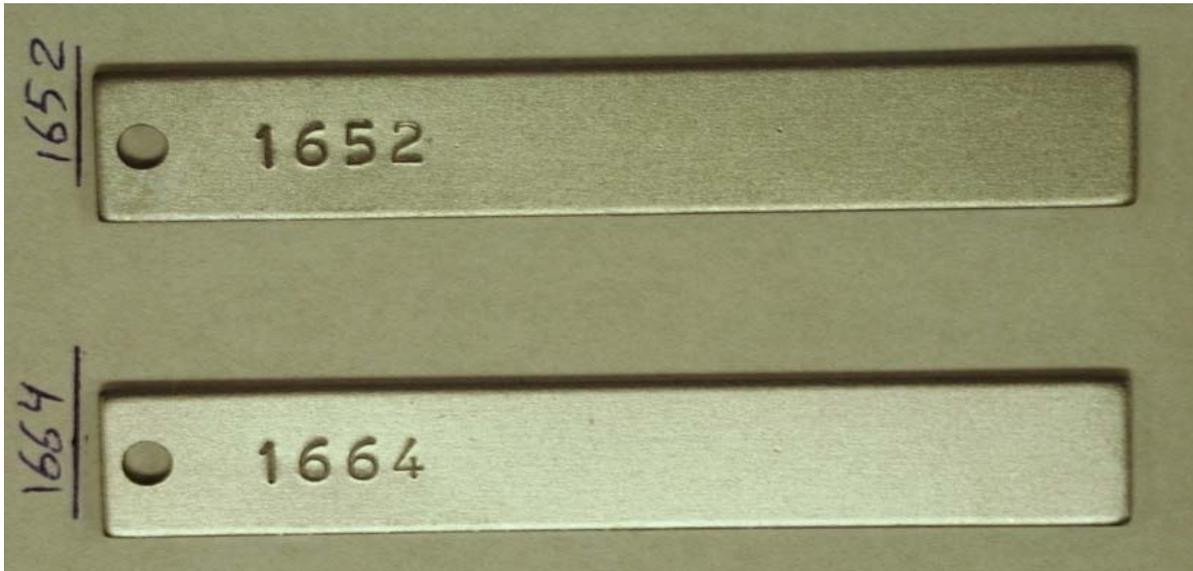


Figure 6 – Galvanized Coupon # 234 exposed 60 days.



Figure 7 - Localized and General Corrosion Rates of Carbon Steel 1008 in High TDS / High Silica Water

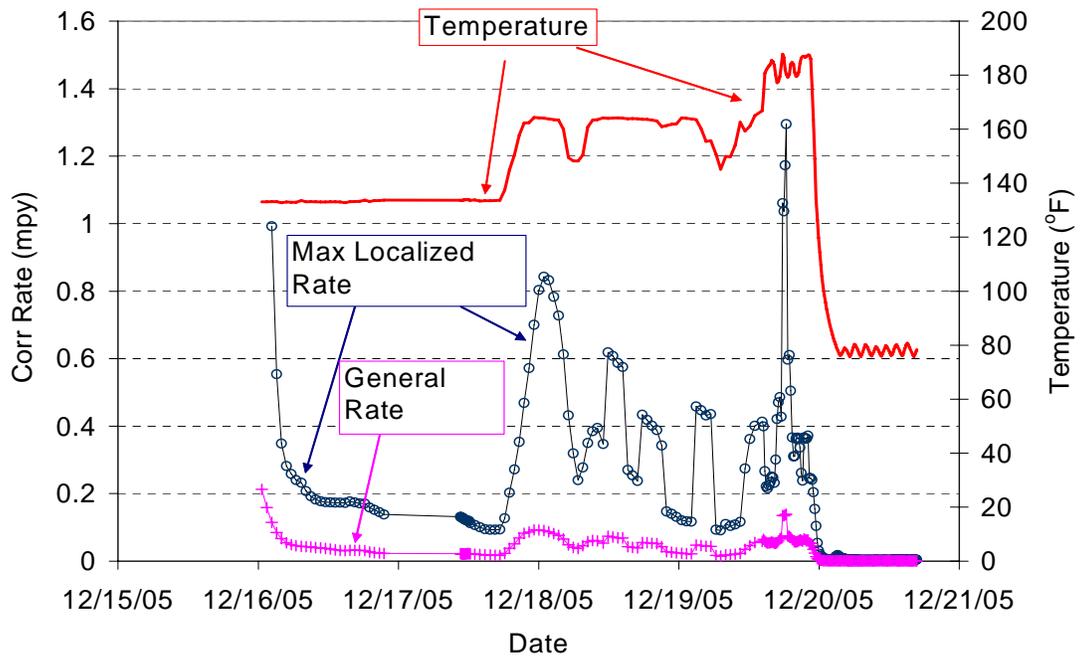


Figure 8 - Localized and General Corrosion Rates of Aluminum 1100 in High Silica / High TDS Water

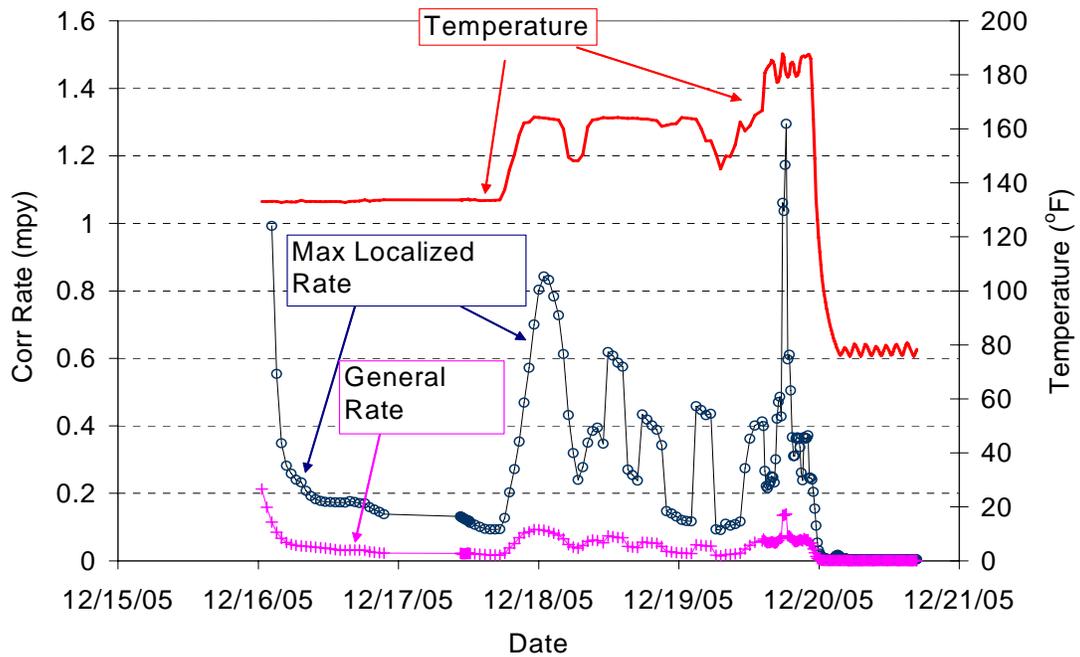


Table 1 – EC West Tower Water Chemistry

EC West Tower / Soft Makeup Chemistry (COC) Ratios			
Sample / Tests	Tower	Soft MU	COC
TDS, mg/L (NaCl Myron L 6P)	141,000	251	562
pH	10.05	7.58	
Copper, mg/L Cu	0.6	0.0015	
Iron, mg/L Fe	ND	ND	
Zinc, mg/L Zn	ND	ND	
Silica, mg/L SiO ₂	960	30	32
Calcium, mg/L CaCO ₃	35	<0.1	
Magnesium, mg/L CaCO ₃	15	<0.1	
Phosphate, mg/L PO ₄	82	0.15	547
Nitrate, mg/L NO ₃	2450	4.5	544
Sodium, mg/L Na	141,000	250	564
Sulfate, mg/L SO ₄	9,995	18	555
Chloride, mg/L NaCl	21,000	38	552
Tot. Alkalinity, mg/L CaCO ₃	67,000	120	558
(COC) = Concentration of Chemistry			

Table 2 – EC 10 Tower Water Chemistry

EC 10 Tower / Soft Makeup Chemistry (COC) Ratios			
Sample / Tests	Tower	Soft MU	COC
TDS, mg/L (NaCl Myron L 6P)	146,000	251	582
pH	10.07	7.58	
Copper, mg/L Cu	0.7	0.0015	
Iron, mg/L Fe	ND	ND	
Zinc, mg/L Zn	ND	ND	
Silica, mg/L SiO ₂	1,050	30	35
Calcium, mg/L CaCO ₃	62	<0.1	
Magnesium, mg/L CaCO ₃	16	<0.1	
Phosphate, mg/L PO ₄	89	0.15	593
Nitrate, mg/L NO ₃	2590	4.5	575
Sodium, mg/L Na	145,000	250	580
Sulfate, mg/L SO ₄	10,260	18	570
Chloride, mg/L NaCl	22,400	38	589
Tot. Alkalinity, mg/L CaCO ₃	69,400	120	578
(COC) = Concentration of Chemistry			

Table 3 - The Effect of Silica and Temperature on Corrosion Rates in High (50,000 mg/L) TDS Waters

Metals	Test Solution	° F	° C	General (mpy)	Max Loc (mpy)
CS 1008	Sea Salt	77	25	-	60
CS 1008	Tower / Silica	77	25	0.02	0.1
CS 1008	Tower / Silica	130	55	0.1	0.2
CS 1008	Tower / Silica	160	71	0.2	0.4
CS 1008	Tower / Silica	190	88	0.2	1.9
SS 316 L	Sea Salt	77	25	-	0.04
SS 316 L	Tower / Silica	77	25	< 0.0015	< 0.005
SS 316 L	Tower / Silica	130	55	< 0.01	< 0.01
SS 316 L	Tower / Silica	160	71	< 0.01	< 0.01
SS 316 L	Tower / Silica	190	88	< 0.01	0.013
AL 1100	Sea Salt	77	25	-	20
AL 1100	Tower / Silica	77	25	< 0.05	< 0.1
AL 1100	Tower / Silica	130	55	0.002	0.009
AL 1100	Tower / Silica	160	71	< 0.05	0.2
AL 1100	Tower / Silica	190	88	< 0.060	0.37
Zn	Sea Salt	77	25	8	80
Zn	Tower / Silica	77	25	< 0.05	< 0.01
Zn	Tower / Silica	130	55	< 0.2	0.4
Zn	Tower / Silica	160	71	-	2.0
CU 110	Sea Salt	77	25	-	0.4
CU 110	Tower / Silica	77	25	< 0.05	< 0.2
CU 110	Tower / Silica	130	55	< 1.0	3.0
CU 110	Tower / Silica	160	71	-	4.0