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Publication Date

1985-10-01

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To be presented at the 46th Annual Meeting,
International Water Conference,
Pittsburgh, PA, November 4-7, 1985

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October 1985

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LBL-20252
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**FLUID COMPOSITION IN THE TUBE SHEET CREVICES
OF A NUCLEAR STEAM GENERATOR**

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October 1985

This paper was presented at the 46th Annual Meeting,
International Water Conference, Pittsburgh, Pennsylvania, November 4-7, 1985

We infer the chemical conditions that exist in the tube sheet crevices of a nuclear steam generator, and the probable composition of the crevice fluid. The crevice fluid is usually alkaline in powerplants that use fresh water for cooling, and we are concerned with this case. Sodium silicate and sodium bicarbonate have been reported in the condensate of many powerplants, and sodium acetate, sodium formate, and other organic salts have been reported in some (1). It is likely that silicates and salts of organic acids are present in the crevices of many powerplants, and play an important role in defining the chemical environment in the crevice. Our work and conclusions will be presented in detail in a report now in preparation (2).

Table 1 depicts our approach to the problem, and summarizes some of our conclusions. The condensate and make-up water contain various solutes in trace amounts. The more volatile compounds (carbon dioxide, ammonia, hydrazine) boil away with the water and cannot be concentrated in the crevice. Sodium bicarbonate dissociates to carbon dioxide and sodium hydroxide as the water boils away. The sodium hydroxide ultimately ends up in the crevice liquid. Most of the non-volatile solutes precipitate in the crevice to form the crevice deposits. Quartz and the sodium silicates are the most important crevice minerals, because they buffer the activity of hydroxide ion in the crevice liquid.

Table 1. Outline of Crevice Chemistry

Boil away	H_2O, NH_3, N_2H_4, CO_2
Hydrolyzes to produce OH^-	HCO_3^-
Insoluble oxides	$Fe_3O_4, FeCr_2O_4$
Crevice deposits	Ca, Mg silicates Aluminosilicates Ca phosphates Na_2SO_4

Four possible crevice liquids:

	I	II	III	IV
Solvent	Caustic	Na Acetate, etc.	K silicate liquid	Na Borate liquid
Controls alkalinity	Caustic	Quartz, Na silicates	K silicate liquid	Na Borate liquid
Corrodent	Caustic	?	?	?

The crevice liquid exists at primary temperature and secondary pressure without drying out. Typically, the vapor pressure of the crevice liquid will be about 57% of the vapor pressure of pure water at the same temperature. Few compounds can form a solution concentrated enough to reduce the vapor pressure of water to this degree. We know four classes of compounds that are capable of forming a crevice liquid, and might reasonably be present in the steam generator. These are (1) sodium and potassium hydroxides, (2) sodium acetate and other salts of organic acids, (3) potassium silicate, and (4) sodium borate. The hydroxide and acetate liquids are completely miscible, and may be considered extreme cases of the same liquid. Other, less soluble compounds may be present in the liquid in subordinate amount, including sodium chloride, sodium sulfate, and sodium carbonate.

Because the range of possible crevice liquids is so severely restricted by the requirement of extreme solubility, a wide range of condensate compositions will ultimately "boil down" to one of these four liquids. However, the trace solutes in the condensate will determine the composition of the crevice liquid within this narrow range. In the sodium acetate liquid the ratio of silica to sodium hydroxide entering the crevice will determine the activity of hydroxide ion in the crevice liquid. If the mole ratio $Na_2O : SiO_2$ is less than one, a hydroxide dominated liquid cannot form, because the sodium hydroxide will react with silica to form solid sodium silicates that buffer the crevice liquid.

Thus far we have described the solvents and buffers that dominate the chemistry of the crevice. They set the stage for the corrosion process, but do not necessarily play a direct role. The actual *corrodent* may be quite distinct from the solvent. Sodium hydroxide is "solvent", "buffer", and corrodent all in one. However, sodium acetate is only a solvent, noncorrosive by itself. The following species are known or suspected to be corrodents:

NaOH, KOH
Carbonate
Reduced Sulfur Compounds?
Organic salts?
Divalent Copper?

MOLTEN SALT LIQUIDS

At the temperature of the hot leg crevices in the steam generator, typically 317°C, *NaOH, KOH*, and many organic salts are molten or near their melting points. These compounds are practically miscible with water at this temperature, forming liquids that resemble molten salt as much as they resemble aqueous solutions.

The vapor pressure was determined as a function of water content for a variety of salts and salt mixtures. The experimental procedure and full results are described in detail elsewhere (2,3). In these experiments, a moderately concentrated solution (25-45 weight % solids) was heated to the temperature desired in an autoclave. Then water vapor was slowly released from the autoclave, condensed, and collected in a buret. The temperature and pressure inside the autoclave were periodically recorded. A curve of water vapor pressure vs. water content was derived from these data, and fitted using a thermodynamic model recently proposed for such liquids by Pitzer and Simonson (4). The Pitzer - Simonson Model allows the activity and activity coefficients of individual salts in the melt to be calculated, using parameters derived from the vapor pressure data. We have collected most data needed to define a complete model of the system $H_2O - NaOH - CH_3COONa - NaCl - KOH -$

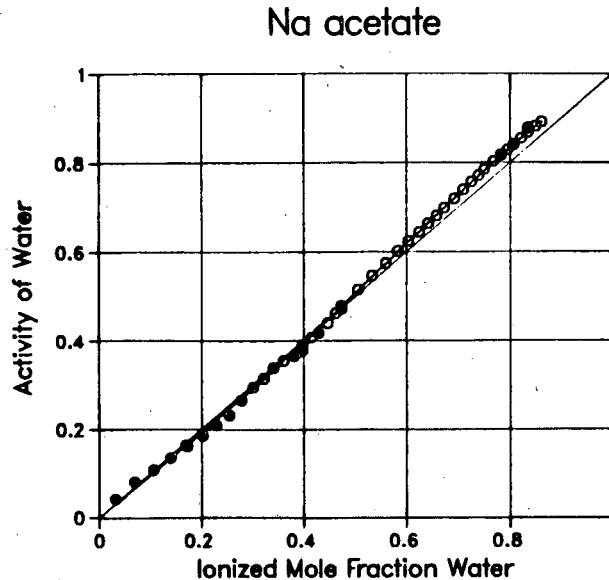


Figure 1. Activity of water in the system $H_2O - CH_3COONa$ at $317^\circ C$. Fitted curves using model proposed by Pitzer and Simonson (4). "Ionized Mole Fraction Water" in Figure 1 is defined as $H_2O / (H_2O + Na^+ + CH_3COO^-)$. The activity of water is the fugacity of water in the solution divided by the fugacity of pure water at the same temperature.

$CH_3COOK - KCl$ at $317^\circ C$. Data for several additional organic salts were also collected.

The activity of water as a function of ionized mole fraction water in the system $H_2O - CH_3COONa$ is presented in Figure 1. All the water activity values were derived from pressure measurements near $317^\circ C$. The activity of water in the crevice liquid is about 0.65. Water and sodium acetate practically form an ideal solution at this temperature.

SODIUM SILICATE

The phase diagram of the system $H_2O - Na_2O - SiO_2$ at $317^\circ C$ is presented in Figure 2. Figure 2 is based on the phase diagram published by Tuttle and Friedman for $300^\circ C$ (5), modified in accordance with our own data for $317^\circ C$. When the mole ratio $Na_2O:SiO_2$ is less than one-half two liquids may coexist, one dilute, the other containing about 75 weight % dissolved solids. There is a narrow field of two liquids (plus vapor) only, and two larger fields of two liquids plus one solid.

Figure 3 shows the activity of water in the sodium silicate system with a mole ratio that corresponds to the dense liquid L_2 . The stirrer in our autoclave kept spinning without impediment and temperature remained stable down to 21% water content. This observation is consistent with Figure 3, which predicts the charge to remain completely liquid down to 21% water content. This observation is not consistent with the alternative phase diagram proposed by Rowe *et al.* (6), which predicts a completely solid charge below 27% water content.

Above 27 weight % water content, the activity of water is practically constant. A dilute liquid of constant composition coexists with a dense liquid throughout this

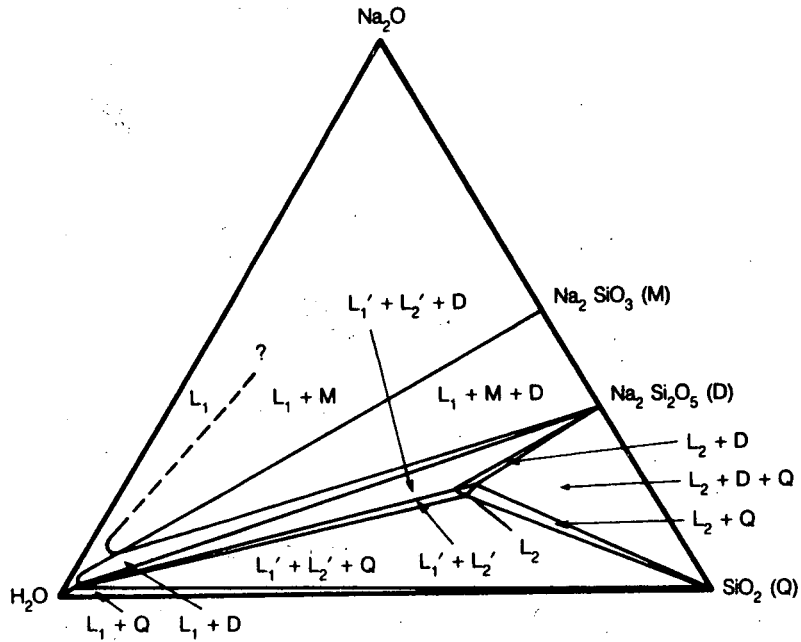


Figure 2. Phase diagram for the system $H_2O - Na_2O - SiO_2$ at $317^\circ C$. Redrawn following phase diagram for $300^\circ C$ published by Tuttle and Friedman (5). Composition triangle in terms of weight percentages of the components indicated. L_1 and L_2 refer to the single liquid fields in the diagram. L_1' and L_2' are the loci of compositions within L_1 and L_2 which may coexist as immiscible liquids.

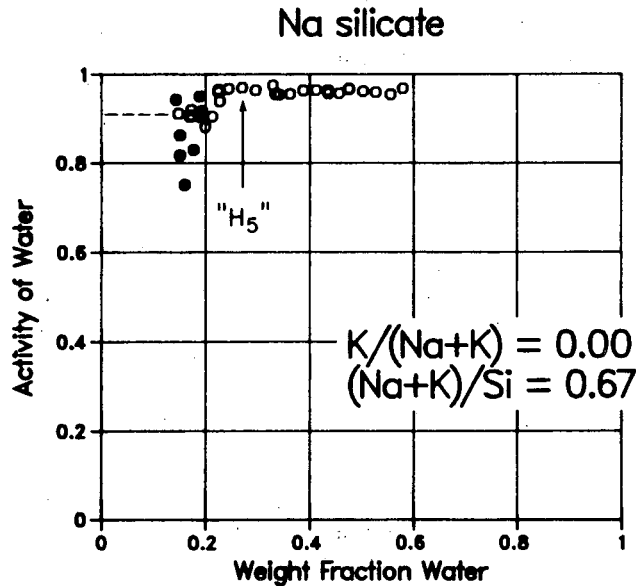


Figure 3. Activity of water in the system $H_2O - (Na_2O)_{0.33}SiO_2$. Dashed line represents our estimate of the activity of water at the eutectic point $SiO_2 - Na_2Si_2O_5 - L_2$. Note the composition variable is weight fraction water. H_5 is the approximate composition of "sodium trisilicate pentahydrate" which Rowe *et al.* (6) suppose to exist in the sodium silicate - water phase diagram.

Table 2. Sodium Silicate Liquids

	L_1'	M-D eutectic	L_2'
Na_2O	1.7	9.2	19
SiO_2	0.9	4.4	54
H_2O	97.4	86.4	27

range. Between 27% and 21% water content the charge consists of the heavy liquid only, and the activity of water drops slightly as water is removed from it. At about 21% water precipitation of quartz and sodium disilicate began, indicated by unstable temperature, groans and rattles from the stirrer, and much larger scatter in the data. The stirrer locked up and the experiment was ended shortly thereafter. In this region the charge consists of quartz, sodium disilicate, and a dense liquid of constant composition and water activity. Our estimate of the true, equilibrium water activity is sketched in Figure 3. The approximate composition of the heavy liquid L_2 estimated from Figure 3 is presented in Table 2.

The water rich pole of the phase diagram is particularly important. There is the dilute liquid L_1' that coexists with the dense liquid L_2' , and a eutectic liquid that coexists with sodium disilicate and sodium metasilicate. We have determined the composition of L_1' and the M-D eutectic in rocking furnace experiments that are described below. Average values from two sets of experiments are presented in Table 2.

The vapor pressure lowering ability of the sodium silicate system is defined by the two lighter liquids. At a $Na_2O:SiO_2$ mole ratio less than one-half, L_2' can coexist with L_1' , but the vapor pressure of L_2' cannot be lower than the vapor pressure of L_1' , which is fairly dilute. At $Na_2O:SiO_2$ mole ratios between one-half and one, the M-D eutectic is the densest liquid that can exist in the system, and it is not concentrated enough to support substantial superheat. Clearly, sodium silicate alone cannot depress the vapor pressure of water enough to form a crevice liquid.

If the mole ratio $Na_2O:SiO_2$ is less than one-half, a sodium silicate liquid will always dry out to produce a mixture of quartz and sodium disilicate in the crevice. If this mole ratio is between one-half and one, the liquid will dry out to a mixture of sodium disilicate and sodium metasilicate. In other words, the M-D eutectic liquid is the most alkaline liquid that can exist with $Na_2O:SiO_2$ mole ratio less than one. A concentrated caustic solution can form *only* if this mole ratio is greater than one, corresponding to the upper left side of the phase diagram.

POTASSIUM SILICATE

Potassium silicate behaves very differently from sodium silicate. The activity of water in potassium silicate with $K_2O:SiO_2 = 0.48$ is presented in Figure 4. The charge remained liquid until the very end of the experiment, and the liquid retained about 8 weight % water at zero gauge pressure. This mole ratio approximately corresponds to one of the eutectic points in the phase diagram of the potassium silicate - water system (7). The approximate water content and water activity of the

eutectic composition are indicated in Figure 4. At water content less than 17 % (the eutectic composition), the liquid is supersaturated, but precipitation of solids is slow, and the charge remains liquid until the end of the experiment.

Clearly, potassium silicate is able to support enough superheat to form a crevice liquid by itself. As with the sodium silicate system, a *KOH* - rich solution cannot be produced unless the mole ratio $K_2O:SiO_2$ is greater than one. With either sodium or potassium, the silica in the system buffers the alkalinity. As with sodium silicate, a domain of two coexisting liquids probably occurs in the potassium silicate - water phase diagram. Coexistence of immiscible liquids is suggested, because the activity of water is nearly one when water content is only 50% in Figure 4.

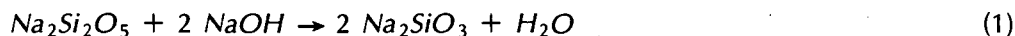
SODIUM BORATE

Figure 5 shows the activity of water in molten sodium borate as a function of ionized mole fraction water. The curve is fitted well by the Pitzer - Simonson Model, and shows a strong positive deviation, suggesting impending separation into two phases. Clearly, sodium borate also supports enough superheat to produce a crevice liquid. If the mole ratio $NaOH:B(OH)_3$ is less than one, all of the *NaOH* will react with boric acid to produce borate anions, and the concentration of OH^- in the liquid will be relatively small. The data in Figure 5 were plotted assuming that all borate anions in the solution are anhydrous; assuming hydrated anions would have produced an implausibly distorted plot. This observation suggests that borate anions are in fact anhydrous.

CREVICE BUFFERS

With the proper acid - base ratio, sodium borate and potassium silicate will form a dense, superheated liquid phase containing little free hydroxide ion. In effect, the borate and silicate anions buffer the alkalinity of the liquid. These liquids probably are not alkaline enough to be corrosive unless other corrodents are present. Adding boric acid to the condensate would produce a sodium borate liquid in the crevice. In fact, addition of boric acid to the condensate has been advocated as a means to prevent crevice corrosion (8).

In most cases, there is little or no boric acid in the condensate, and sodium far exceeds potassium. If silica is present in the crevice together with sodium salts of organic acids, the silica will react with *NaOH* to form some mixture of quartz and sodium silicates. The crevice liquid will be a mixture of the molten organic salt with water. If the mole ratio of $NaOH : SiO_2$ is between one and two, sodium disilicate and sodium metasilicate (Na_2SiO_3) will precipitate together, and the activity of sodium hydroxide in the crevice liquid will be controlled by reaction (1):



If the mole ratio $NaOH : SiO_2$ is less than one, quartz and sodium disilicate ($Na_2Si_2O_5$) will precipitate together, and the activity of *NaOH* in the crevice liquid will be controlled by reaction (2):



If the mole ratio $NaOH : SiO_2$ is greater than 2, all the silica will precipitate as sodium metasilicate, and any *NaOH* remaining will accumulate in the crevice liquid together with sodium acetate. The alkalinity of the crevice liquid will be determined

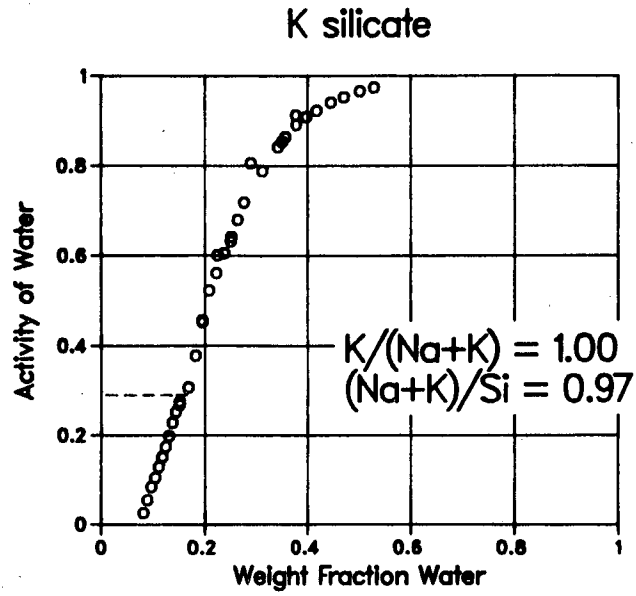


Figure 4. Water activity in the system $H_2O - (K_2O)_{0.48}SiO_2$ near $317^\circ C$. Dashed line represents our estimate of the activity of water in the corresponding eutectic liquid.

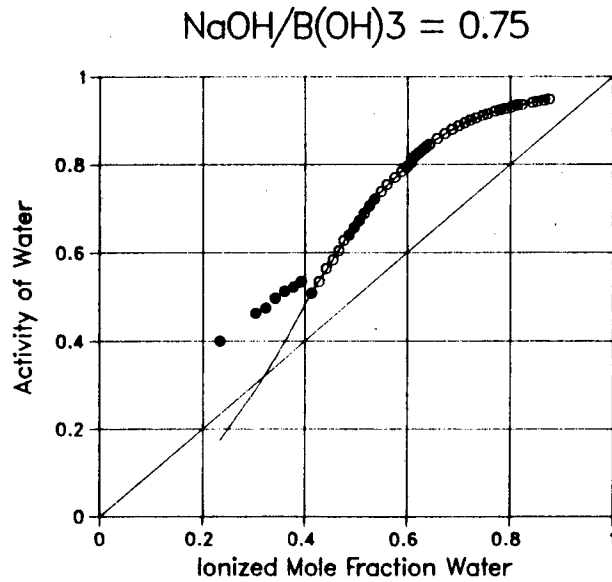
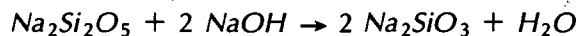


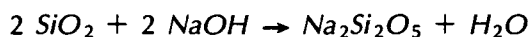
Figure 5. Water activity in the system $H_2O - (NaOH)_{0.75}B(OH)_3$ near $317^\circ C$. The composition variable is ionized mole fraction water, as in Figure 1. Curve fitted using the Pitzer - Simonson Model, as in Figure 1.

Table 3. Summary of a_{NaOH} Buffers at 317°C

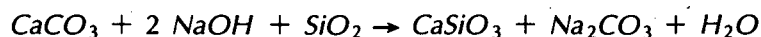
(1) $\log a_{NaOH} = -2.82$



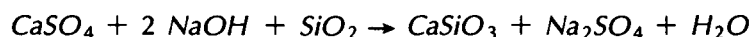
(2) $\log a_{NaOH} = -3.64$



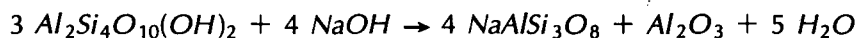
(3) $\log a_{NaOH} = -4.39$



(4) $\log a_{NaOH} = -5.94$



(5) $\log a_{NaOH} = -8.78$



by the mole ratio of this remaining $NaOH$ to sodium acetate. A crevice liquid with sodium hydroxide activity greater than the value corresponding to buffering reaction (5-1) is possible *only* if the mole ratio of $NaOH : SiO_2$ is greater than 2.

These buffering reactions determine the activity of $NaOH$ in solution. The activity of $NaOH$ is independent of gross solution composition; if the activity of $NaOH$ in the crevice fluid deviates from the value buffered by the coexisting solids, the solids will react with the fluid until the correct value is established.

The values of a_{NaOH} corresponding to buffer reactions (1) and (2) at 317°C were determined experimentally. Two determinations were made for either buffer with a sodium acetate - water melt as the solvent. Two additional determinations were made for either buffer using water alone as the solvent. In all experiments, the solids were equilibrated with the given solvent, and the liquid phase was separated from the solids while hot. The liquid phase was chemically analyzed, and a_{NaOH} at 317°C was calculated using the thermodynamic model of the system $H_2O - NaOH - CH_3COONa$ discussed above. Values of a_{NaOH} for either buffer obtained in the sodium acetate melt agreed well corresponding values obtained with water as the solvent. Averaged values of a_{NaOH} determined for reactions (1) and (2) are presented in Table 3.

Other silicate compounds are frequently present in the crevice, and may buffer the crevice fluid at other, less alkaline values of a_{NaOH} . Values of a_{NaOH} for several other reactions were calculated using thermodynamic data from published compilations, and included in Table 3. The last reaction involves the mineral pyrophyllite. Three other reactions involving pyrophyllite and its close relative kaolinite are possible, with $\log a_{NaOH} = -8.81, -8.97, \text{ and } -9.17$.

The activity of a neutral compound, e.g. $NaOH$ may be calculated, and the values summarized in Tables 4 are fairly reliable. The activity of a single ion - in this case OH^- - cannot be calculated rigorously. In order to estimate the pH of these solutions, we must estimate $[OH^-]$. In a moderately concentrated solution the activity coefficient of OH^- will approximately equal the mean activity coefficient of

Table 4. Estimates of pH and related values at 317°C

Buffer Reaction	log a_{NaOH}	In H_2O		In H_2O-CH_3COONa	
		log[OH ⁻]	pH	log[OH ⁻]	pH
(1)	-2.82	-0.61	10.74	-1.50	9.85
(2)	-3.64	-1.00	10.35	-2.28	9.07
(3)	-4.39			-3.01	8.34
(4)	-5.94			-4.56	6.79
(5)	-8.78			-7.40	3.95

NaCl at the same temperature and ionic strength (the so-called Liu-Lindsay approximation). The pH values for the two sodium silicate buffer reactions in water calculated using this approximation are presented in Table 4. In this case, Na^+ , OH^- , silicic acid, and silicate anions are the only solute species in the aqueous phase. The Liu-Lindsay approximation cannot be applied to the sodium acetate - water melt which has very high ionic strength. Recall that water and sodium acetate form a nearly ideal solution (Figure 1). It is reasonable to assume that the activity coefficient of sodium ion in a sodium acetate - water melt will approximately equal the mean activity coefficient of sodium acetate, which is approximately one and may be calculated using the Pitzer - Simonson Model. Knowing a_{NaOH} and the activity coefficient of sodium ion, we can readily estimate $[OH^-]$. These values are also presented in Table 4.

While the buffer reactions fix the values of a_{NaOH} , the corresponding pH will vary with the composition of the solution. For example, the pH values of the two sodium silicate buffers in the sodium acetate melt are substantially lower than in water. This difference is a consequence of the common ion effect. The sodium acetate melt contains a large amount of sodium, and the activity of sodium ion is correspondingly high. With a_{NaOH} fixed by the buffer, increasing a_{Na^+} will necessarily decrease a_{OH^-} and pH.

ORGANIC COMPOUNDS IN THE CREVICE

Acetate and formate are commonly the major organic anions present in the condensate and water from crevice flushing. In some cases lactate and oxalate have also been reported. Benzene sulfonate and phenoxide may also occur, as their precursors will occasionally be present in the make-up water (ion exchange resin beads and humic acid, respectively).

Acetate and its homologues are easily produced by decomposition of humic acid or fatty acids under hydrothermal conditions. Humic acid may penetrate the

Table 5. Reactivity of Organics in the Crevice Environment

Acetate, Propionate		Practically stable
Benzoate Benzene sulfonate		Stable?
Phenoxide		Polymerizes
Formate	→	CO_3^{-2} , CO , H_2
Oxalate	→	CO_3^{-2} , CO
Lactate	→	Polyacrylate fragments

makeup water demineralizers in colloidal form. Linseed oil used for rust-proofing will react as a fatty acid, and quickly decompose producing acetate. Lactate may be derived from polyacrylamide based water treatment chemicals (this hypothesis needs to be tested). Other water treatment chemicals containing organic acids may give a variety of organic anions, some of which will decompose further to give acetate. Benzene sulfonate may be formed by decomposition of ion exchange resins.

The origin of formate is unknown. Perhaps formate is produced by partial reduction of bicarbonate. The most likely reducing agents are hydrazine, other AVT chemicals, and iron (in the tube sheet).

In the alkaline crevice fluid formate and oxalate will rapidly react with hydroxide to produce carbonate and hydrogen. The reaction of formate with hydroxide is so rapid at high temperature that formate should be near chemical equilibrium with carbonate and hydrogen. Carbonate is known to be corrosive. Therefore, formate and oxalate may be considered indirectly corrosive. Decomposition of formate may be a major source of carbonate in the crevice, as carbonate itself is probably too volatile to accumulate there by condensate evaporation.

Acetate is thermodynamically unstable under crevice conditions, but the decomposition of acetate is very slow, and acetate is practically stable. While acetate is a fairly strong complexing agent for metal ions at high temperature, there is no evidence that acetate is corrosive. As discussed above, sodium acetate will tend to decrease corrosion to some extent by reducing the alkalinity of the crevice fluid. All told, acetate may do more good than harm in the crevice environment.

Lactate will partially dehydrate to acrylate in the crevice, and acrylate will polymerize to some extent, forming short chain, disordered polyacrylate anions. Small polyacrylates will probably be good chelating acids, and are potentially corrosive. We also found diglycolate among the decomposition products of sodium polyacrylate, and diglycolate is also a chelating agent.

The apparent reactivity of organic compounds in the crevice environment is summarized in Table 5.

DISCUSSION

We have gained a useful understanding of the conditions needed for a crevice liquid to exist, and what determines how alkaline this liquid will be. We believe that corrosive agents other than hydroxides, silica and organic salts must play a role in crevice corrosion. The presence of organic anions and silica in the condensate argues against a strongly alkaline crevice environment. In many cases, there may be insufficient caustic in the crevice fluid to account for the corrosion observed, and another explanation must be sought.

Among the organic compounds, only acetate, formate, and lactate are quantitatively important, and acetate is innocuous. Formate decomposes to produce carbonate, which is corrosive. If formate is an important ion in the condensate or carbonates are found in the crevice, remedial measures should focus on eliminating formate and bicarbonate from the condensate. Hydrazine and other AVT compounds should be screened for ability to reduce carbonate to formate and selected accordingly. The possible corrosiveness of decomposition products of lactate also deserves some attention.

In principle, acetic acid or silica might be added to the condensate in order to decrease the alkalinity of the crevice liquid, but this benefit must be balanced against possible harmful effects elsewhere in the system. Adding boric acid to the condensate will cause a sodium borate liquid to form in the crevices, likewise reducing alkalinity but with less likelihood of harmful side effects.

The activity of $NaOH$ in a sodium acetate liquid will be controlled by the sodium silicate buffer system in many cases. Therefore, adding silica will always enable the alkalinity of a sodium acetate liquid to be reduced to that of the sodium disilicate - quartz buffer. In the absence of acetate, adding silica will cause the crevice fluid to dry up completely.

ACKNOWLEDGEMENTS

This work was supported by the Steam Generator Project Office of the Electric Power Research Institute under Contract S302-5 through the U.S. Department of Energy Contract DE-AC03-76SF00098.

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This report was done with support from the Department of Energy. Any conclusions or opinions expressed in this report represent solely those of the author(s) and not necessarily those of The Regents of the University of California, the Lawrence Berkeley Laboratory or the Department of Energy.

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