

Potential for the Localized Corrosion of Alloy 22 Waste Packages in Multiple-Salt Deliquescent Brines in the Yucca Mountain Repository

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ABSTRACT

It has been postulated that the deliquescence of multiple-salt systems in dust deposits and the consequent localized corrosion in high-temperature brines could lead to premature failure of the Alloy 22 waste packages in the Yucca Mountain repository. EPRI has developed a decision tree approach to determine if the various stages leading to waste package failure are possible and whether the safety of the repository system could be compromised as a result. Through a series of arguments, EPRI has shown that it is highly unlikely that the multiple-salt deliquescent brines will form in the first place and, even if they did, that they would not be thermodynamically stable, that the postulated brines are not corrosive and would not lead to the initiation of localized corrosion of Alloy 22, that even if localized corrosion did initiate that the propagation would stifle and cease long before penetration of the waste package outer barrier, and that even if premature waste package failures did occur from this cause that the safety of the overall system would not be compromised.

EPRI concludes, therefore, that the postulated localized corrosion of the waste packages due to high-temperature deliquescent brines is neither a technical nor a safety issue of concern for the Yucca Mountain repository.

INTRODUCTION

Absorption of water vapor by hygroscopic salts in dusts deposited onto waste packages (WP) in a repository for nuclear wastes at Yucca Mountain (YM), Nevada has been postulated to lead to the formation of deliquescent brines at temperatures that are well above the boiling point of pure water at local atmospheric pressures [1]. Localized corrosion of the Alloy 22 waste package outer barrier could occur if surface temperatures are above a threshold value for the initiation of localized corrosion, and if such brines are acidic Cl^- solutions having low concentrations of certain anions (*e.g.*, NO_3^- , $\text{HCO}_3^-/\text{CO}_3^{2-}$) that are known to inhibit the onset of corrosion if present in sufficiently high concentrations [2,3].

A previous analysis of possible deliquescence [4] conclusively demonstrated that localized corrosion of Alloy 22 by Ca-Cl brines would not occur for a variety of reasons. Subsequent to that study, a new set of deliquescent salts in the system $\text{NaNO}_3\text{-KNO}_3\text{-NaCl-H}_2\text{O}$ were proposed as possibly leading to formation of a deliquescent brine at temperatures near 200°C [5,6]. Accordingly, EPRI has conducted a new study of this revised deliquescent salt system with the objective to provide an independent technical analysis of the potential for aggressive deliquescent brines to form, persist, and initiate as well as sustain localized corrosion of Alloy 22 under anticipated repository conditions.

EPRI has approached this issue through the use of a decision tree that defines a number of questions, each of which must be answered in the affirmative for the deliquescence of salts on the WP surface to pose a safety concern (Fig. 1). The first two questions relate to the possible formation and stability of multiple-salt deliquescent brines on the WP surface in the repository. If such deliquescent brines form and are stable, they may not be corrosive to the highly corrosion-resistant Alloy 22 WP material. Therefore, the next three questions in the decision tree relate to whether these brines will be corrosive, whether localized corrosion will initiate, and, if so, will corrosion continue to propagate or will it stifle. The final question is, even if deliquescence leads to early WP failure by localized corrosion, is the safety of the repository system significantly compromised? A single "no" response to any of these questions means that the deliquescence of multiple-salt systems on the WP surface is neither a key technical nor a safety issue for the YM repository.

DECISION TREE APPROACH

The decision tree approach used by EPRI in 2004 to address the issue of localized corrosion due to the deliquescence of simple divalent cation chloride salts [4,7] is extended here to the deliquescence of multiple-salt systems at potentially higher temperatures.

Can the proposed multiple-salt deliquescent brines form?

The DOE have recently considered the possible formation of deliquescent multiple-salt systems from minerals present in dust on the WP surface [1]. DOE used an abstraction procedure based on the chemistry of solutions leached from dust samples obtained from the Exploratory Studies Facility (ESF) to identify three different salt assemblages that could be present in dusts initially deposited onto WP surfaces:

- Assemblage A; $\text{NaCl}(s) + \text{KNO}_3(s)$,
- Assemblage B; $\text{NaCl}(s) + \text{KNO}_3(s) + \text{NaNO}_3(s)$, and
- Assemblage C; $\text{NaCl}(s) + \text{KNO}_3(s) + \text{NaNO}_3(s) + \text{Ca}(\text{NO}_3)_2(s)$.

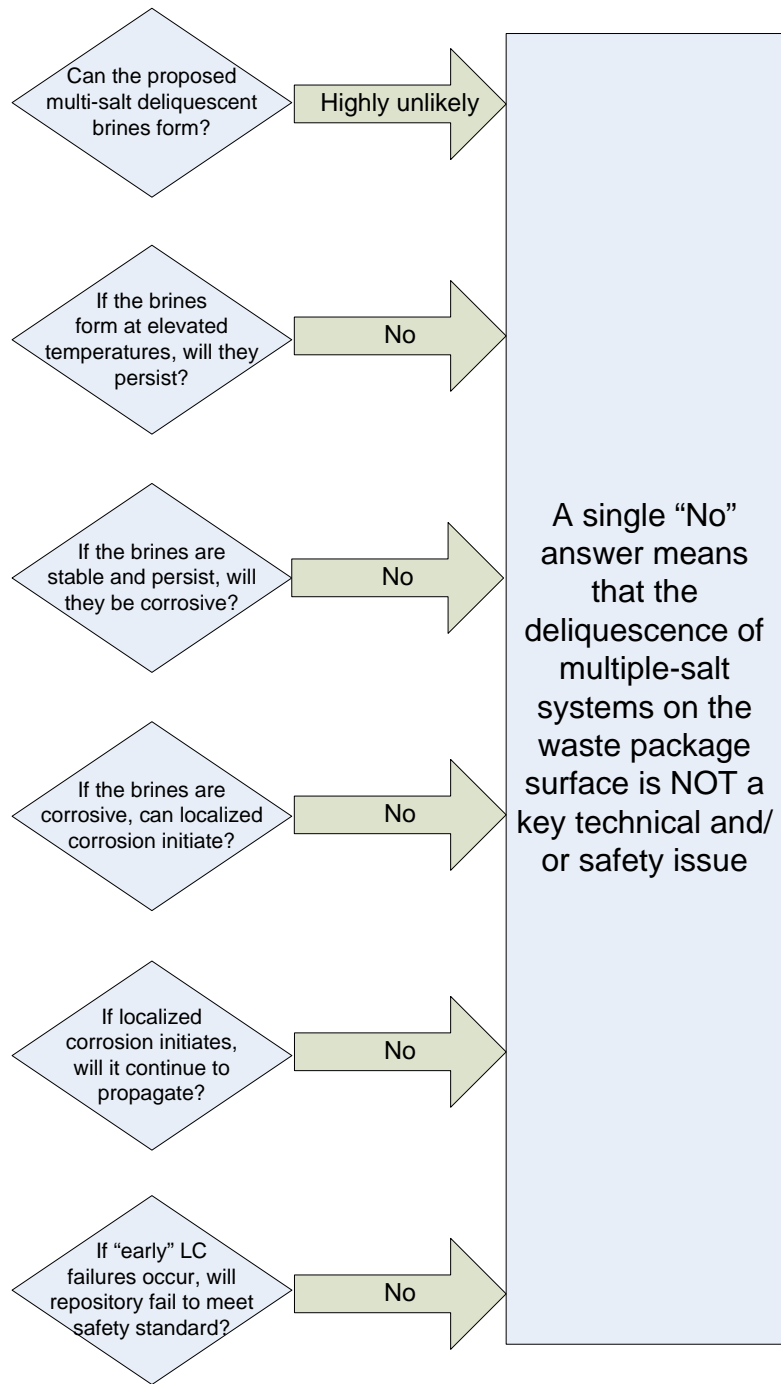


Fig. 1. Decision tree approach used in EPRI's analysis of the safety consequences of the postulated deliquescence of multiple-salt systems on the waste package surface

These dusts also contain other salt and non-salt minerals, which could affect the stability and corrosivity of deliquescent solutions [1,4]. Recent experimental evidence suggests that deliquescent brines of the pure assemblages A, B and C are stable at high temperatures [1,6,8].

No direct observations have been made of the mineralogy of soluble salts in dusts at Yucca Mountain [1,9]. Similar to the DOE methodology, EPRI has therefore used a geochemical modeling approach to “reconstitute” the mineralogy of these dusts based on the chemistry of solutions leached from dust samples. EPRI has applied an evaporative concentration model to DOE dust leachate data assuming two limiting conditions (Fig. 2):

1. open-system behavior with respect to $\text{CO}_2(\text{g})$ and $\text{O}_2(\text{g})$ only, and
2. open-system behavior with respect to all gases, including $\text{HNO}_3(\text{g})$, $\text{HCl}(\text{g})$, $\text{HF}(\text{g})$ and $\text{HBr}(\text{g})$.

Both models, referred to here as EPRI-1 and EPRI-2, are based on calculations using the EQ3/6 thermodynamic code. The second model (EPRI-2) differs from the first in that it allows acid degassing of the brine solution.

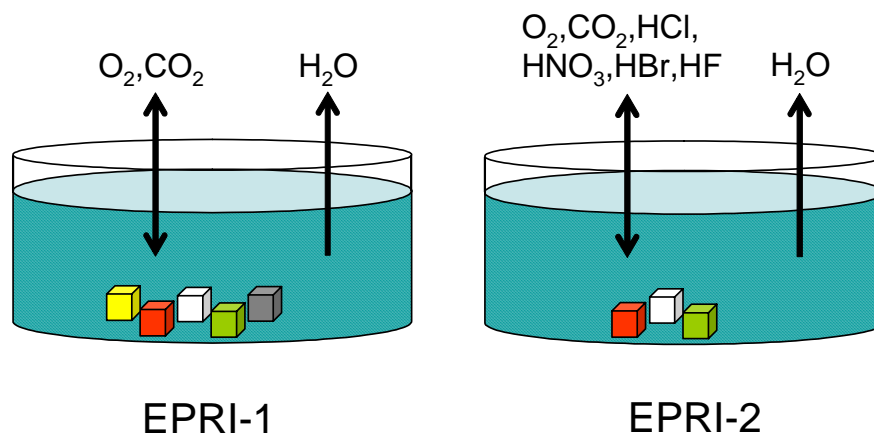


Fig. 2. Schematic diagram illustrating constraints in the EPRI-1 and EPRI-2 models used to simulate the evaporation of dust leachates to dryness at 25°C . EPRI-1 assumes: 1) $p_{\text{CO}_2(\text{g})}$ and $p_{\text{O}_2(\text{g})}$ are fixed at atmospheric values, and 2) H_2O evaporates continuously and irreversibly to the terminal eutectic. The EPRI-2 model is similar to EPRI-1, but also assumes that the partial pressures of the acid gases $\text{HCl}(\text{g})$, $\text{HNO}_3(\text{g})$, $\text{HBr}(\text{g})$ and $\text{HF}(\text{g})$ are fixed at values determined by the chemistry of the leachate prior to evaporation. Minerals, including salts, are assumed to precipitate or dissolve under equilibrium control in both models depending on the extent of evaporation. Fewer minerals will be present at dryout in EPRI-2 than in EPRI-1 in accordance with the phase rule and the additional constraints imposed in EPRI-2 on $p_{\text{HCl}(\text{g})}$, $p_{\text{HNO}_3(\text{g})}$, $p_{\text{HBr}(\text{g})}$ and $p_{\text{HF}(\text{g})}$.

Table I shows the resulting mineral assemblages from the “numerical evaporation” of various DOE dust leachate solutions. The predicted mineral assemblages differ between the two models. If the system is assumed to be open to only CO₂ and O₂ (EPRI-1 model), then the precipitation of halite and various nitrate salts is predicted, consistent with DOE’s mineral assemblages A, B, and C above. However, if the system is assumed to be also open with respect to gaseous HNO₃, HCl, HF, and HBr (EPRI-2 model) then no highly deliquescent nitrate salts are predicted to form and the equilibrium relative humidity (RH) for the eutectic mixture is very high (of the order of 80-85%). This difference in behaviour is significant because, in the absence of deliquescent nitrate salts, the WP surface will not become wetted during the hottest part of the thermal pulse. EPRI considers the second model to be more realistic, since it treats all gaseous species similarly and does not assume that the system is open to some gases but not others.

EPRI concludes, therefore, that it is highly unlikely that the proposed multiple-salt deliquescent brines will form on the WP surface (Fig. 1).

Table I. Calculated Eutectic Mineral Assemblages Resulting from Numerical EQ3/6 Evaporation of DOE Dust Leachates

Mineral	Formula	P07*		P14*		P22*		S82c*	
		EPRI-1	EPRI-2**	EPRI-1	EPRI-2**	EPRI-1	EPRI-2**	EPRI-1	EPRI-2
Anhydrite	CaSO ₄	x		x		x		x	
Calcite	CaCO ₃	x	x	x	x	x	x	x	x
Nitrocalcite	Ca(NO ₃) ₂ :4H ₂ O	x							
Fluorite	CaF ₂	x		x					
Halite	NaCl	x		x		x		x	
KBr	KBr	x		x		x		x	
Niter	KNO ₃	x		x		x		x	
Sepiolite	Mg ₄ Si ₆ O ₁₅ (OH) ₂ :6H ₂ O	x	x	x	x	x	x	x	x
SiO ₂ (am)	SiO ₂	x	x	x	x				
Soda Niter	NaNO ₃	x		x		x		x	
Glauberite	Na ₂ Ca(SO ₄) ₂		±	x	±	x		x	
Huntite	CaMg ₃ (CO ₃) ₄					x	x	x	x
Sellaite	MgF ₂					x		x	
Syngenite	K ₂ Ca(SO ₄) ₂ :H ₂ O		x		±		±		
Thenardite	Na ₂ SO ₄		x		x		x		x
Arcanite	K ₂ SO ₄		±		±		x		x
Pirssonite	Na ₂ Ca(CO ₃) ₂ :2H ₂ O				±		±		x
Darapskite	Na ₃ NO ₃ SO ₄ :H ₂ O								
Relative Humidity (%)		38.9	84.0-84.9	54.8	82.2-84.9	54.8	82.2-84.0	54.8	82.1

* Refer to DOE dust leachate solutions [10]

** An “x” indicates that the mineral is present in the equilibrium assemblage. The symbol ± denotes that the indicated mineral does not form if another precipitates in its place. In such cases, for leachate P07 these minerals are glauberite/arcanite, for leachate P14 they are glauberite/arcanite and pirssonite/syngenite, and for leachate P22 they are pirssonite/syngenite. These differences in mineral paragenesis result in small variations in relative humidity at the eutectic.

If the brines form, will they persist?

Even if the mineral assemblages A, B, and C could exist on the WP surface, EPRI considers that deliquescent brines formed from these assemblages would not be thermodynamically stable. Acid-gas volatilization would cause these solutions to lose HCl and HNO₃ (as noted above), and the resultant sulfate- and/or carbonate-dominated solutions would dry out.

Acid degassing has been considered by both EPRI [4,11] and DOE [1], although the latter conservatively assumes that the rate of degassing may be limited because of the increase in pH of the brine solution and because of slow kinetics of pH-buffering by ancillary minerals in the dust. However, EPRI believes there are a number of reasons why acid degassing will be sustained, including: 1) the fact that some of the relevant non-salt minerals (notably carbonates) are known to dissolve or precipitate rapidly even at low temperatures [12], 2) the fine-grain size of minerals in dusts should greatly accelerate mineral dissolution reactions through associated increases in reactive surface area [13], 3) the rates of these reactions are known to increase rapidly with increasing temperature [14], and 4) recent, unpublished¹ high-temperature (up to 180°C) tests by the Center for Nuclear Waste Regulatory Analyses (CNWRA) show that high, sustained rates of acid degassing from simulated deliquescent brines can occur.

In EPRI's opinion, therefore, if the proposed multiple-salt deliquescent brines did form, they would not persist, primarily because of acid degassing (Fig. 1).

If the brines are stable and persist, will they be corrosive?

Even if these multiple-salt deliquescent brines did form and were stable on the WP surface, they would not be corrosive from the standpoint of localized corrosion.

To illustrate this point, let us consider the three-salt mineral assemblage B (NaCl(s), KNO₃(s), and NaNO₃(s)) proposed by DOE [1]. Fig. 3 illustrates the temperature-time and corresponding RH-time profiles for an average and the hottest WP in the YM repository. Also marked on these profiles are the points at which a deliquescent brine could exist, with points a/a', b/b', and c/c' corresponding to conditions at which halite (NaCl(s)), niter (KNO₃(s)), and soda niter (NaNO₃(s)) co-exist (points a and a' for the average and hottest WP, respectively), at which halite and niter co-exist (points b and b'), or which only halite is present (points c and c'). At each of these points in time, small areas on the WP surface in which these salts are in intimate contact will be wetted by a saturated brine in equilibrium with the ternary, binary, or single salt. As the temperature decreases and the RH increases, the saturated brine will become more dilute until the next solid phase completely dissolves. Thus, at points a and a', parts of the WP surface could be wetted by a saturated solution in equilibrium with solid NaCl(s), KNO₃(s), and

¹ Presented at the September 2006 Workshop on Corrosion conducted by the Nuclear Waste Technical Review Board (NWTRB) in Las Vegas, Nevada. Available at: <http://www.nwtrb.gov/meetings/2006/sept/pabalan.pdf>

NaNO₃(s). As the temperature decreases and RH increases, the brine solution would become more dilute until at points b and b' the NaNO₃(s) would have completely dissolved and only solid NaCl(s) and KNO₃(s) would persist. With increasing RH the KNO₃(s) would eventually also dissolve, at points c and c'. The compositions of the brine solutions at these three points in the evolution of the deliquescent brine are given in Table II.

The deliquescent brines are characterized by a high [NO₃⁻]:[Cl⁻] molal ratio (Table II). Nitrate is known to act as an inhibitor for the initiation of localized corrosion [2-4]. Table II also gives the predicted corrosion potential (E_{CORR}) and crevice re-passivation potential (E_{RCREV}) for each of these brines, based on models developed by the DOE [15]. In all cases, the value of E_{RCREV} vastly exceeds the value of E_{CORR}, indicating that these solutions are not corrosive in terms of localized corrosion.

The extent to which these environments are non-corrosive is illustrated in Fig. 4, which shows a plot of the critical temperature for localized corrosion as a function of the [NO₃⁻]:[Cl⁻]. The critical temperature for localized corrosion is the temperature at which E_{CORR} exceeds E_{RCREV}, a criterion for the initiation of localized corrosion, for a given [NO₃⁻]:[Cl⁻]. This curve divides the figure into a corrosion zone and a zone in which the [NO₃⁻]:[Cl⁻] is sufficient to inhibit localized corrosion. The environmental conditions at points a/a', b/b', and c/c' (indicated by the blue square and red circle symbols) fall well within the zone of protection.

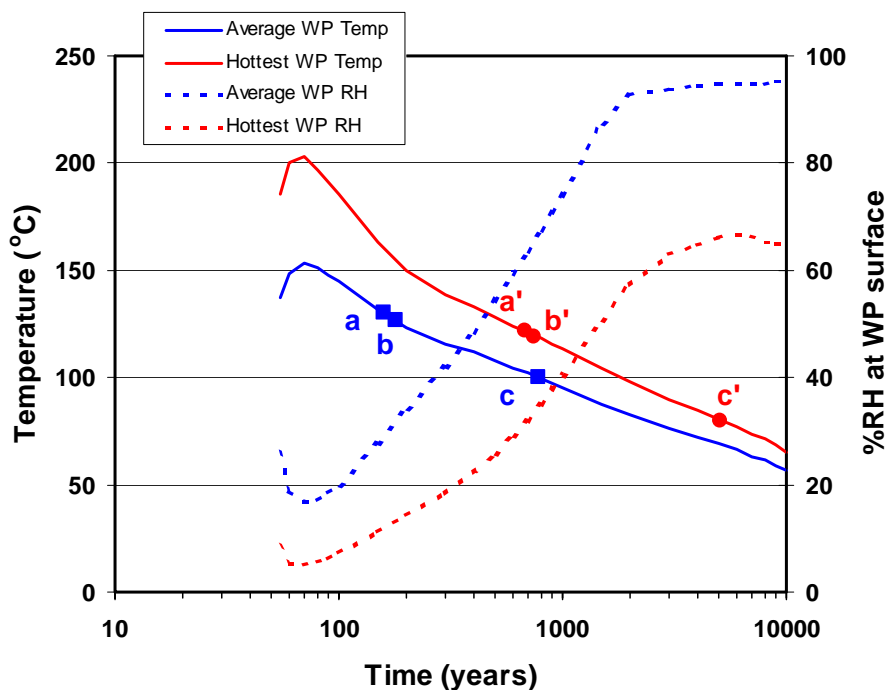


Fig. 3. Predicted times at which various brines will exist on the waste package surface for salt assemblage B.

Therefore, EPRI maintains that, even if such deliquescent brines were stable on the WP surface, they are not corrosive in terms of localized corrosion of the Alloy 22 WP material.

A similar analysis can be performed for any other postulated mineral assemblage.

Table II. Predicted Compositions of Brine Solutions on the Waste Package Surface for Mineral Assemblage B for the Points Illustrated in Fig. 3 and the Corresponding Corrosion and Localized Corrosion Repassivation Potentials (Cl^- and NO_3^- concentrations in mol/kg)

Brine	t (yrs)	T ($^{\circ}\text{C}$)	rh (%)	pH	Cl^-	NO_3^-	$\text{NO}_3^-/\text{Cl}^-$	E_{CORR} (mV_{SSC})	E_{rcrev} (mV_{SSC})
<i>Average waste package</i>									
<i>a</i>	160	130.0	29.2	5.42	0.52	27.23	52.4	352	>1000
<i>b</i>	180	126.8	30.1	5.39	0.53	27.03	51.0	351	>1000
<i>c</i>	780	99.8	66.5	5.36	3.97	7.95	2.0	257	>1000
<i>Hot waste package</i>									
<i>a'</i>	680	121.6	31.4	5.35	0.55	26.71	48.6	349	>1000
<i>b'</i>	750	119.3	33.8	5.34	0.76	24.73	32.5	340	>1000
<i>c'</i>	5100	80.0	66.5	5.29	3.94	7.87	2.0	248	>1000

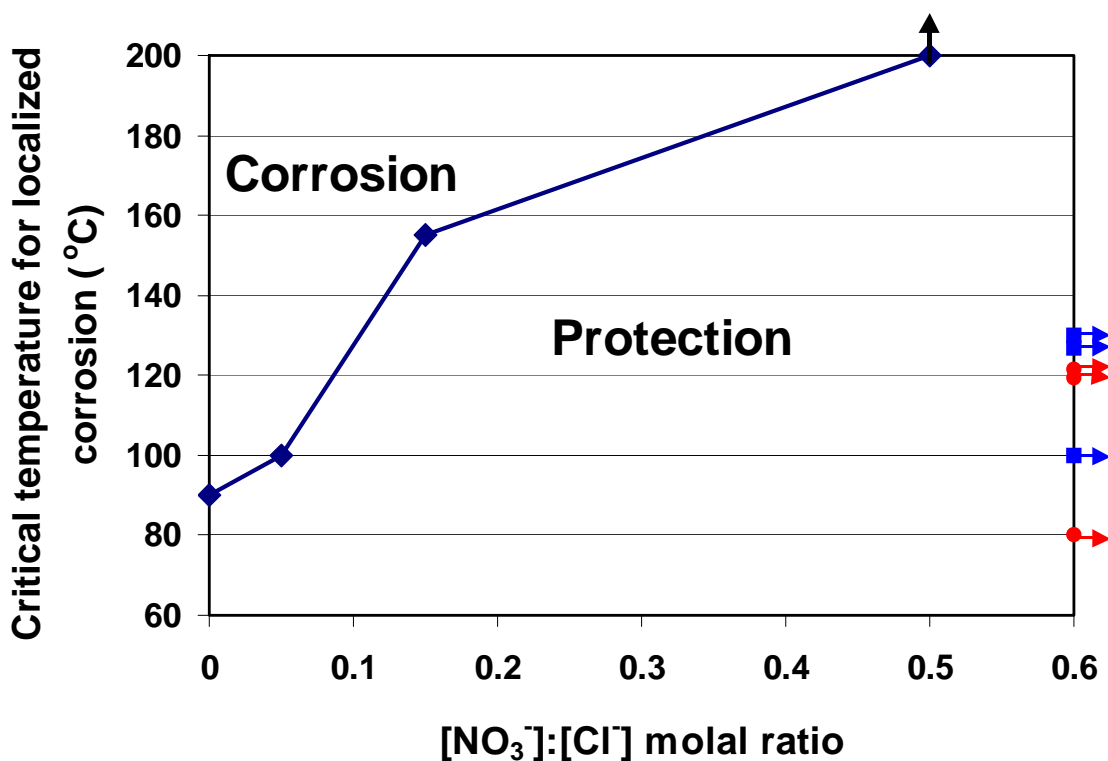


Fig. 4. Comparison of the predicted temperatures and nitrate:chloride ratios of deliquescent solutions derived from salt assemblage B with the critical temperature for the initiation of localized corrosion

If the brines are corrosive, can localized corrosion initiate?

In addition to the non-corrosiveness of the environment, further arguments can be made in support of the claim that localized corrosion will not initiate on the WP surface. First, the nature of the surface deposits will not serve to spatially separate the locations of the anodic and cathodic reactions, a pre-requisite for the initiation of localized corrosion. Porous dust and other types of deposit on the WP surface will not effectively restrict the access of atmospheric O₂ to the Alloy 22, so that the differential [O₂] cell required to separate anodic and cathodic sites cannot be established. The effective depletion of the [O₂] under a porous dust deposit was shown to be <0.01% of the bulk concentration in the case of deliquescent CaCl₂ brines [4,7]. Updating these calculations for the higher temperature and higher concentrations (and, therefore, greater salting-out effects) for deliquescent multiple-salt systems indicates that the maximum O₂ depletion is still <1% of the bulk concentration, even at temperatures as high as 180°C.

Second, DOE have argued that capillary action in the dust deposits coupled with the small volumes of solution and the droplet-like characteristics of the deliquescent solutions imposes severe restrictions on the ability to sustain separate anodic and cathodic sites [1]. Capillary action will tend to retain the small liquid volumes inside the pores of the dust deposit, away from direct contact with the WP surface. Furthermore, even if the droplets of deliquescent solution contact the surface, it is argued that the small size will limit the spatial separation of anodic and cathodic sites, in a similar argument to that given above.

Therefore, EPRI concludes that even if stable corrosive deliquescent brines did form on the WP surface, the characteristics of the dust deposits and small volumes of solution would preclude the initiation of localized corrosion.

If localized corrosion initiates, will it continue to propagate?

Alloy 22, along with many other metals, exhibits a strong tendency to stifle during localized corrosion [1,4]. EPRI has long argued that this property should be accounted for in lifetime predictions of the Alloy 22 WP. Recent experimental evidence from both the DOE [1] and CNWRA [16] confirm this tendency for stifling of the localized corrosion of Alloy 22. A complete understanding of the stifling mechanism is not yet available, but several possible reasons have been suggested, including:

1. potential (iR) drop down the crevice
2. mass-transport effects
3. loss of critical crevice chemistry by catalysis of H⁺ reduction
4. loss of critical crevice chemistry by the reduction of NO₃⁻ (in NO₃⁻:Cl⁻ mixtures)
5. negative shift in E_{CORR} upon LC initiation

The loss of critical crevice chemistry by the reduction of NO_3^- ions to NH_4^+ may be particularly important in nitrate-rich systems at elevated temperature.

Mathematically, the effect of stifling is represented in the WP failure model by the expression

$$D = kt^n \quad (1)$$

where D is the depth of corrosion as a function of time t, n is the time exponent and k is a temperature-dependent growth constant the value of which also depends on the nature of the environment. Based on measurements of the time-exponent for the decrease in localized penetration [1,4,16], estimates of the extent of crevice propagation suggest that the corrosion will cease at depths $\ll 25\%$ of the WP wall thickness.

For these reasons, EPRI concludes that, even if localized corrosion should initiate, stifling of the propagation reaction will occur well before the WP outer barrier has been penetrated (Fig. 1).

If “early” localized corrosion failures occur, will the repository fail to meet the safety standard?

In an earlier assessment, EPRI considered the consequences of WP failures from localized corrosion resulting from the deliquescence of salts. It was concluded that even if all WP failed at the time of emplacement the ability of the entire system to meet the regulatory dose requirements was not compromised. These calculations indicate the strength of the multi-barrier approach inherent in the design of the YM repository.

EPRI concludes, therefore, that even if premature failures from localized corrosion were to occur, the YM repository would still meet the regulatory dose requirements (Fig. 1).

CONCLUSIONS

In summary, a detailed analysis of the various stages in the postulated failure of Alloy 22 waste packages due to localized corrosion in high-temperature, multiple-salt deliquescent brines indicates that

- such multiple-salt deliquescent brines are highly unlikely to form,
- such brines would not be stable if formed,
- such brines, if they are stable, would not be corrosive for Alloy 22,
- localized corrosion would not initiate under dust deposits in the presence of such brines,
- penetration of the container of the waste package outer barrier would not occur, even if localized corrosion is sustained,
- early, localized penetration of waste packages would not compromise the ability of a repository at Yucca Mountain to comply with established regulatory safety criteria, and lastly,
- EPRI concludes, therefore, that the postulated localized corrosion of the waste packages due to high-temperature deliquescent brines is neither a technical nor a safety issue of concern for the Yucca Mountain repository.

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