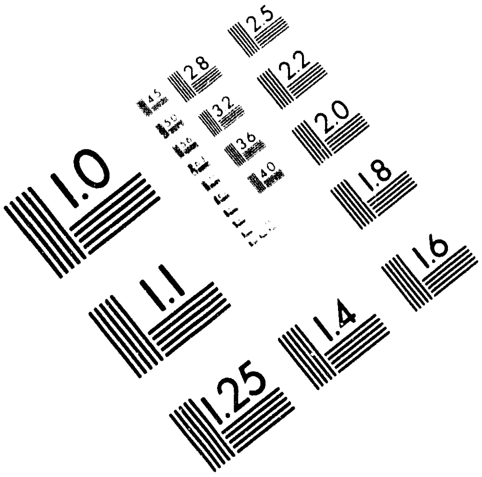

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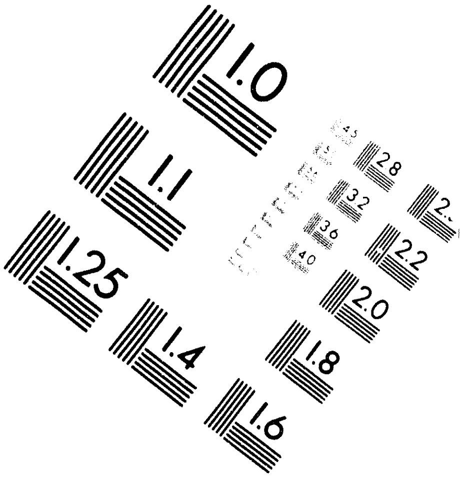

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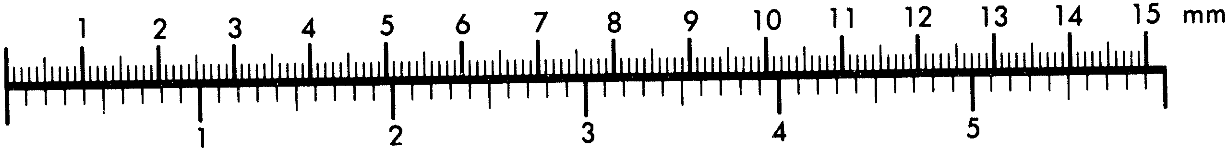

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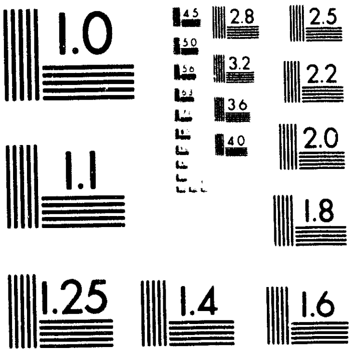

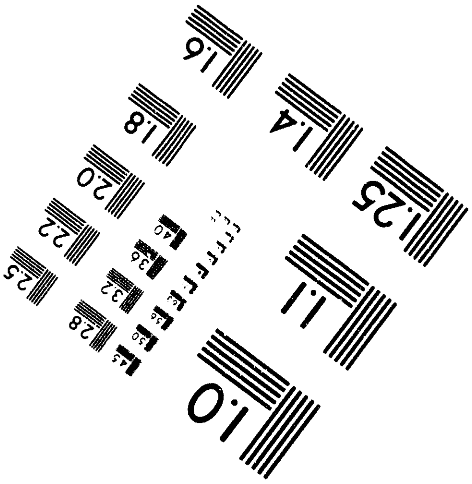

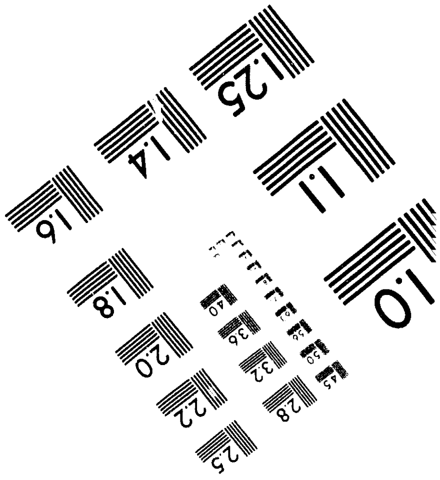

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VAPOR-LIQUID EQUILIBRIUM OF CHLORIDES IN AQUEOUS SYSTEMS TO HIGH TEMPERATURES: APPLICATION TO THE GEYSERS GEOTHERMAL FIELD

J. M. Simonson and Donald A. Palmer

Chemical and Analytical Sciences Division, Oak Ridge National Laboratory,
P.O. Box 2008, MS-6110, Oak Ridge, TN 37831-6110

ABSTRACT

Measurements have been made of the partitioning of chlorides including HCl, NaCl, NH_4Cl, $MgCl_2$, and $CaCl_2$ over wide ranges of solution ionic strength at temperatures to 350°C. Thermodynamic distribution constants K_D have been determined from analyses of the concentrations of species in the coexisting phases using activity coefficients available or estimated from published measurements. These results indicate that it is unlikely that HCl, the single most volatile chloride species at all temperatures considered, is solely responsible for all chloride found in the vapor phase due to the low solution pH required to give the observed levels in steam. Noting that sodium is not found in appreciable levels in steam from the high-temperature reservoir in the northwest Geysers and that ammonia is present in significant amounts as a noncondensible gas, it is shown that a slightly acidic ammonium chloride brine is capable of producing steam having some of the characteristics observed in produced steam from the northwest Geysers.

INTRODUCTION

Steam containing significant concentrations of chloride is produced at The Geysers. This chloride-bearing steam is widespread in the high temperature reservoir found in the Northwest Geysers, although there is some concern that troublesome chloride levels may become more widespread in the normal reservoir of the Central and Southeast Geysers as production continues to deplete the resource. In addition, development of a significant new resource tentatively identified at The Geysers is subject in part to the availability of suitable methods for mitigating the the high chloride and noncondensible gas levels expected in this area. In general, chloride levels in steam higher than a few parts per million have been found to lead to unacceptably high rates of corrosion of well casings, piping, and other components, leading in some cases to loss of production from affected wells. The fate of dissolved solutes in steam at lower temperatures is also of interest, noting that very low concentrations of contaminants in steam have caused problems in the low-pressure turbine systems of fossil-fired power plants arising from corrosion of turbine materials or from deposition of solids onto the turbines. With these considerations in mind, we have carried out a series of experimental measurements of the partitioning of chloride compounds between liquid and vapor phases at temperatures ranging from 50 to 350°C. These studies comprise one part of our overall program at ORNL focusing on the physical chemistry of geothermal systems, and including measurements of volatilities, solubilities, melt-vapor phase behavior, and liquid-vapor and solid-fluid stable isotope distributions.

In our original measurements of the partitioning of HCl (Simonson and Palmer, 1993) it was found that the vapor-phase molalities of HCl over pure HCl(aq) solutions were too small to account completely for the high (to more than 100 ppm) chloride levels observed in some steam samples from The Geysers, particularly considering that the required low pH values for these solutions appear incompatible with the minerals found in the reservoir. Measurements by both emf (Wesolowski and Palmer, in preparation) and volatility (Palmer et al., in preparation) of the hydrolysis of magnesium and calcium ions in solution at high temperature gave further indication that these hydrolyzable cations would not buffer hydrothermal solutions at pH values low enough to give high levels of HCl partitioning to the vapor phase, and that $MgCl_2$ and $CaCl_2$ contribute negligibly to the total chloride in the vapor phase under practical reservoir conditions. Our measurements (Simonson et al., 1994) and available literature data (Khaibullin and Borisov, 1966; Bischoff et al., 1986) have indicated that a modestly-concentrated NaCl brine at slightly acidic pH could account for the chloride levels found in the condensed steam samples from the Northwest Geysers, particularly at high temperatures (≈350°C). However, the sodium content of steam in equilibrium with such brines would be significantly higher than has been observed in these samples.

Moreover, it has been noted that significant levels of ammonia were present as noncondensible gas in these samples. In a study originally motivated by problems encountered in all-volatile-treated fossil-fired steam generator systems and sponsored by the Electric Power Research Institute, we determined (Palmer and Simonson, 1993) the liquid-vapor partitioning behavior of NH_4Cl over solutions containing either excess HCl or NH_3, where the partitioning constants for HCl were measured independently to 350°C (Simonson and Palmer, 1993) and those for NH_3 were modeled over the temperature range of interest based on literature data (Edwards et al., 1978; Jones,

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1963). On the basis of this study of NH_4Cl volatility it is possible to calculate the concentrations of chloride, both as HCl and NH_4Cl, and of ammonia in steam in equilibrium with $NH_4Cl(aq)$ brines over wide ranges of temperature and ionic strength. Thus, given a particular observed steam composition (total chloride + total ammonia) it is possible to determine the composition of brine in equilibrium with this steam at various temperatures.

Clearly the complete system of (brine + vapor + solid) interactions in the Geysers reservoir is significantly more complex than can be treated in a short communciation. The purpose of the present paper is to show that a small part of the overall steam chemistry, *viz.*, the production of steam containing significant concentrations of chloride and ammonia from the high-temperature reservoir in the Northwest Geysers, can be accounted for based on known volatilities of aqueous species through the assumption of an ammonium chloride brine at depth within the reservoir. The treatment presented here is necessarily incomplete; the effects of additional brine salinity (as NaCl) on the chloride partitioning equilibria are not considered, and source terms for other noncondensible gases (e.g., CO_2 and H_2S) found in steam from the Northwest Geysers are not addressed. However, it does demonstrate that the observed chloride and ammonia levels in steam are consistent with the assumption of a slightly acidic ammonium chloride brine at depth within the reservoir, and permits quantitative calculation of the composition of condensates forming at various temperatures from this steam, which can have important implications for the corrosion of various components of the piping and turbine systems at The Geysers. In the following sections we will review briefly the experimental results and data analysis leading to the determination of thermodynamic partitioning constants for the various chloride species, and apply these results to the calculation of the composition of coexisting liquid and vapor phases at both steam-generation and condensate-formation conditions of the Geysers steam cycle.

EXPERIMENTAL

Full details of the experimental methods, including equipment design, sampling of coexisting phases, and analysis of solution compositions, have been presented previously (Simonson and Palmer, 1993; Palmer and Simonson, 1993). The basic method employed is the static equilibration of liquid and vapor phases, with sampling of both phases at carefully controlled rates. The apparatus consists of a stainless steel pressure vessel (HIP Co.) containing a loose-fitting, sealed platinum liner equipped with sampling lines and a platinum thermowell. Pressures on either side (sample or annular) of the liner are equalized by material transfer between the sample and annular spaces through a pressure vessel equipped with a Teflon bag which serves to separate the sample solution from pure water; thus the pressurizing medium in the high-temperature annular space is undersaturated steam. A second water-filled low-temperature pressure vessel equipped with either a small Teflon bag or a polyethylene syringe of appropriate volume is used in conjunction with a high-pressure positive-displacement pump to withdraw condensed vapor samples at accurately controlled rates. Liquid-phase samples are less sensitive to sample rate, and are withdrawn into a syringe at ambient pressure. Compositions of both phases are determined by appropriate combinations of acidimetric titration and ion chromatography.

The liquid-phase compositions used in these experiments were dictated by the compound(s) under study. For HCl the measurements were straightforward. With either HCl(aq) or {HCl + NaCl}(aq) solutions loaded into the apparatus, molalities in the two phases were determined directly by acidimetric titration and ion chromatography, while in runs where NaCl volatility was measured in the presence of NaOH (Simonson and Palmer, 1994) ion chromatography was used to determine both sodium and chloride concentrations and the results compared to indicate charge and mass balance. For the $MgCl_2$, $CaCl_2$ and {$MgCl_2$ + NaCl} runs which will be the subject of a future publication (Palmer and Simonson, in preparation) vapor-phase molalities of hydrogen ion were determined by titration, with the molalities of other cations and chloride found by ion chromatography. The HCl content in these steam samples, coupled with the previously-determined values for HCl partitioning in NaCl media, were used to calculate the extent of hydrolysis of the alkaline earth cations at temperature.

The study of ammonium chloride volatility was complicated significantly by both the hydrolysis of ammonium ion and the high volatility of the hydrolysis products. The hydrolysis reaction

$$NH_4^+(aq) = NH_3(aq) + H^+(aq) \qquad (1)$$

in chloride media gives $NH_3(aq)$ and HCl as reaction products. Clearly the volatility of ammonia is high compared with that of stronger electrolytes in aqueous solutions; also, a comparison of the partitioning constants for HCl and NaCl (Simonson and Palmer, 1994) indicates that HCl partitioning to the vapor is likely to be significantly greater than that of NH_4Cl. Thus, at constant temperature, both the total amounts of the various solutes (NH_3, HCl, and NH_4Cl) and their relative distribution are strongly dependent on both total liquid-phase NH_4Cl molality and solution pH. To address these multiple dependences, measurements were made at varying $NH_4Cl(aq)$ molalities with either excess HCl or NH_3 added to establish nearly constant pH in the liquid phase. The concentration of NH_4Cl in each phase in these experiments was then determined by measuring total nitrogen, chloride, and hydrogen ion in the samples by titration and ion chromatography, calculating expected levels of HCl and NH_3 in the vapor phase samples from the known partitioning constants for these compounds, and calculating the NH_4Cl molality by difference. As would be expected, the precision of the overall determination of the partitioning constant for NH_4Cl was necessarily reduced by the requirements of this indirect method; however, the resulting overall precision in the equilibrium constant values was sufficient to permit the determination of their temperature dependence from 150 to 350°C (Palmer and Simonson, 1993).

DISCUSSION

Treatment of volatility results

The partitioning of a solute between aqueous solution and steam is described by the general reaction for a compound MX as:

$$MX(aq) = MX(vap) \qquad (2)$$

In cases where MX is predominantly a charge-neutral molecule in both liquid and vapor phases (e.g., CO_2 or NH_3) it is straightforward to write an expression for the equilibrium constant for the partitioning reaction:

$$K_D = \frac{m_v(MX)\gamma_v(MX)}{m_l(MX)\gamma_l(MX)} \qquad (3)$$

where the subscripts 'l' and 'v' refer to liquid and vapor phases and m and γ are the molality and activity coefficient of MX in the respective phases. For uncharged solutes in dilute solutions it is reasonable to approximate $\gamma = 1$; this approximation is adopted here for all neutral solutes in both liquid and vapor phases. Equation (3) is equivalent to a Henry's Law representation, changed only by the adoption of an infinite-dilution standard state for the vapor-phase solution; this change is particularly appropriate for low concentrations (or partial pressures) of neutral molecular solutes in steam.

The liquid phase is characterized by significantly higher density and dielectric constant than the vapor at subcritical temperatures. Electrolytes (e.g., NH_4Cl) will therefore be significantly ionized in the liquid phase, but primarily associated to the neutral molecule in steam. For a solute MX which ionizes in the liquid phase, it is convenient to write the partitioning equilibrium constant as

$$K_D(MX) = \frac{m_v(MX)\gamma_v(MX)}{m_l(M^+)m_l(X^-)\gamma_l(M^+)\gamma_l(X^-)} \qquad (4)$$

In equation (4) stoichiometric quantities may be used for the liquid-phase molalities and activity coefficients, making it possible to incorporate available measured values of stoichiometric activity coefficients directly in the determination of K_D from the partitioning measurements. The two equations (3) and (4) for K_D are directly related through the ion-association equilibrium constant in the liquid phase; adoption of the present treatment makes it unnecessary to use potentially ambiguous values for the association equilibrium constant in the determination of distribution constants. It should be noted that K_D as defined for molecular species in equation (3) will approach unity at the solvent critical point, while that for ionizing solutes given by equation (4) will approach the value of the ion association constant at the critical conditions.

Results

For calculations of the composition of coexisting liquid and vapor phases at conditions characteristic of the high-temperature reservoir of the Northwest Geysers we consider here the simplified system (H^+ - NH_4^+ - Cl^- - NH_3 - H_2O). It should be noted that relatively high concentrations of sodium chloride are likely in these brines; this additional salinity will change the molalities of HCl(aq) and NH_4Cl(aq) in equilibrium with steam of a given composition and temperature through both the common-ion effect and potentially large changes in liquid-phase activity coefficients. Also, other components found in steam samples, either as dissolved solutes or noncondensible gases, may introduce important controls on possible brine chemistry within the reservoir, particularly with respect to the effect of borates, carbonates, and sulfides on solution pH. However, since only very low levels of sodium have been found in steam from the Northwest Geysers, the oversimplified system considered here should be directly applicable to the determination of the composition of condensate brine formed from Geysers steam, and will give at least an indication of the levels of NH_4Cl and HCl in the 'source' brine required to yield steam having the measured concentrations of chloride and ammonia. The partitioning reactions which must be considered include:

$$NH_3(aq) = NH_3(vap) \qquad (5)$$
$$H^+(aq) + Cl^-(aq) = HCl(vap) \qquad (6)$$
$$NH_4^+(aq) + Cl^-(aq) = NH_4Cl(vap) \qquad (7)$$

Of these reactions, equations for the distribution constant given by equations (3) and (4) as functions of temperature and solvent density in the liquid and vapor phases have been given previously (Palmer and Simonson, 1993; Simonson and Palmer, 1993). The variation of these equilibrium constants with temperature is shown in Figure 1, with values for NaCl (Simonson et al., 1994) shown for comparison.

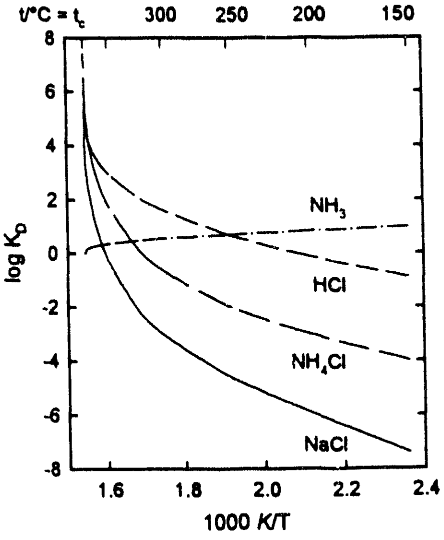

Figure 1. Liquid-vapor partitioning constants K_D for NH_3, HCl, NH_4Cl and NaCl at the saturation vapor pressure.

It is important to note that the relative molalities of NH_3, NH_4Cl, and HCl in steam are dependent on temperature through the relation

$$\frac{m_v(HCl)\,m_v(NH_3)}{m_v(NH_4Cl)} = \frac{K_H K_D(HCl) K_D(NH_3)}{K_D(NH_4Cl)} \tag{8}$$

where the K_Ds are for the species listed and K_H is the equilibrium constant for ammonium ion hydrolysis (equation 1).

There exist no reliable data on the composition of a liquid phase at depth in The Geysers reservoir. Recent modeling studies (Shook, 1994) indicate that a nonzero saturation index is compatible with a conceptual model for the formation of a high-temperature reservoir, and Moore (1992) has found high salinities in some fluid inclusions from The Geysers. Based on averaged values of the chloride and ammonia concentrations in steam from a portion of the high temperature reservoir (Mark Walters, personal communication, 1994), it is possible based on the partitioning equations and the experimentally-determined distribution constants to calculate the composition of a bulk $NH_4Cl(aq)$ brine in equilibrium with steam at a chosen temperature. Calculation of liquid-phase ammonia concentration from the steam composition is straightforward based on equation (2). Calculations for the electrolytes are complicated somewhat by the necessity of maintaining charge balance. Taking the total chloride in steam as known from the wellhead analyses and using the relation given in equation (8), as well as the equilibrium constant for water ionization given by Marshall and Franck (1981) and the liquid-phase charge balance conditions gives for the liquid-phase hydrogen ion molality:

$$m_l^2(H^+)\gamma_l^2(H^+) = \frac{m_v(HCl)K_D(NH_4Cl)}{K_D(HCl)} \times \left\{ \frac{K_D(HCl)K_W\Gamma_1 + m_v(HCl)\Gamma_2}{m_v(HCl)K_D(NH_4Cl) + m_v(NH_4Cl)K_D(HCl)\Gamma_3} \right\} \tag{9}$$

where $\Gamma_1 = \{\gamma_l(H^+)/\gamma_l(OH^-)\}$, $\Gamma_2 = \{\gamma_l(H^+)/\gamma_l(Cl^-)\}$, and $\Gamma_3 = \{\gamma_l(H^+)/\gamma_l(NH_4^+)\}$. Liquid-phase molalities of the other components (NH_4^+, Cl^-, and OH^-) are easily calculated from the hydrolysis, water-ionization and charge-balance relations once $m_l(NH_3)$ and $m_l(H^+)$ are found. The overall calculation is iterative due to the dependence of activity coefficients on solution composition. Activity coefficients of the various ionic species may be calculated at the temperature and ionic strength of interest based on literature values (*cf.* Palmer and Simonson, 1993), or estimated from the extended Debye-Hückel limiting-law. While all the ionic species involved in the various ratios Γ_i are all singly charged and the approximation $\Gamma_i = 1$ may be used in practical calculations, it should be noted that the calculation is sensitive to the values of the activity coefficients, and that the best available values should be used for maximum accuracy.

As an illustration of the application of the measured partitioning thermodynamic properties to the conditions of the high temperature reservoir at The Geysers, the brine composition leading to the formation of saturated (not superheated) steam at 350°C may be calculated based on varying levels typically observed for ammonia and chloride in the steam. We have also calculated the composition of a brine formed at 150°C from the condensation (on expansion and cooling) of an infinitessimal amount of the high-temperature steam. The variation of two brine properties which are particularly important in establishing the corrosivity of these fluids, *viz.*, the pH and total chloride concentration in the liquid phase, are plotted in Figures 2 and 3 against the variation of NH_3 concentration in steam containing 30 ppm chloride and as a function of chloride concentration in steam containing 500 ppm NH_3, respectively. As can be seen in Figure 2, imposing the condition of constant chloride concentration in steam while varying the ammonia concentration implies at both 350 and 150°C a nearly constant liquid-phase chloride molality, with increasing pH in the equilibrium liquid in each case. However, it is important to note the significant shifts in each of these quantities with temperature: for a given steam composition, the equilibrium liquid-phase chloride concentration increases by about three orders of magnitude at 150°C as compared with 350°C, and the pH is lower by more than one unit. Thus steam which is formed at 350°C from a low-salinity, moderate-pH ammonium chloride brine will form condensate at 150°C which has both a high chloride content and low pH.

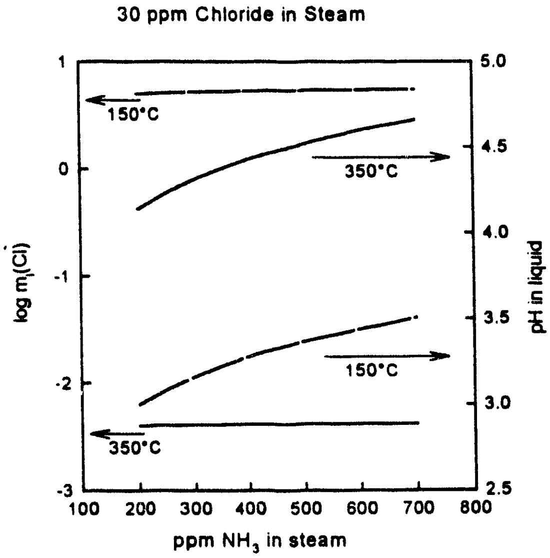

Figure 2. Total chloride molality and pH of ammonium chloride brines in equilibrium with steam having 30 ppm chloride as a function of ammonia concentration.

The variation of brine composition with chloride content in steam having a constant ammonia concentration, shown in Figure 3, is somewhat different from that obtained under constant-chloride conditions. Here the total chloride molality in the liquid phase at constant temperature increases approximately linearly with the increasing chloride in steam, while the equilibrium liquid pH is lowered by the increasing concentrations of both HCl and NH_4Cl required to produce the higher chloride levels in steam. Again the most significant overall changes are due to the differences between the high-temperature 'formation' conditions and the lower 'condensation' temperature. As for the constant-ammonia conditions, the pH at 150°C is lower by more than one unit, while the chloride molality in the equilibrium liquid shows a similar increase of three log units. This behavior is quite general for systems in which steam is formed at high temperature, while condensate forms on expansion and cooling at lower temperature. In all cases where there is no intervening process occurring in steam (e.g., deposition of solid salts or reaction with minerals), the steep decrease in the partitioning constants for NH_4Cl and HCl shown in Figure 1 requires increasing chloride concentration with decreasing temperature for brines in equilibrium with steam of a given composition. The shift to lower pH values at lower temperatures is due to both the relative changes with temperature of the partitioning constants for HCl and NH_4Cl, and to the increase in the partitioning constant for NH_3 as the temperature decreases.

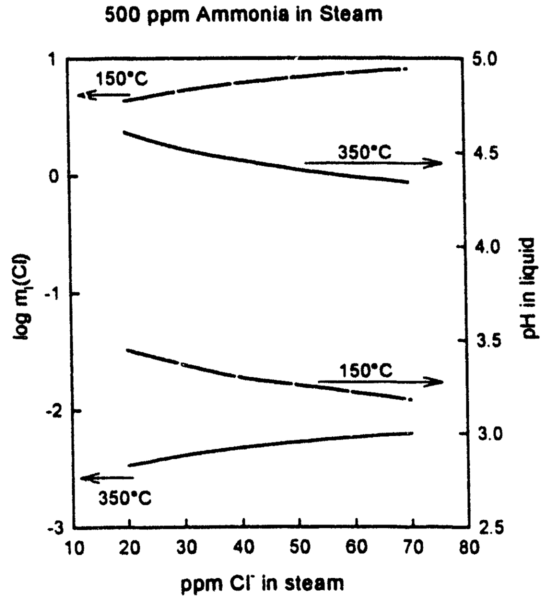

Figure 3. Total chloride molality and pH of ammonium chloride brines in equilibrium with steam having 500 ppm ammonia as a function of chloride concentration.

Considerations in mixed brines

The calculations above have addressed only the concentrations of ammonia and chloride in saturated steam over aqueous ammonium chloride brines. As alluded to above, the actual composition of both liquid and steam in the high-temperature reservoir at The Geysers is significantly more complex than can be addressed here. Important questions remaining to be addressed through both experimental and modeling studies are the effects on steam composition of: (1) a significant NaCl concentration in the reservoir brine; (2) the concentrations of other salts in these brines (e.g., sulfides and carbonates) which are required to produce steam with the observed concentrations of noncondensible gases other than ammonia, and their effect on brine pH; (3) the related questions of the degree of superheating of steam and the effect of these superheating mechanisms (e.g., production of steam from adsorbed fluids) on the equilibrium properties of dense fluids and steam; and (4) the design and development of cost-effective mitigation measures to allow the economic production of steam from the high-temperature reservoir.

It seems likely, based on fluid inclusion studies and the probable formation of the reservoir from a marine environment, that fluids in the high-temperature reservoir contain significant concentrations of sodium chloride. At temperatures above 300°C, NaCl should distribute to the vapor phase in significant amounts (Simonson et al., 1994). Based on these reported preliminary results from ongoing research in this laboratory, for an approximate seawater salinity (3% NaCl) at 325°C saturated steam should contain about 50 ppm sodium, while at higher temperature and salinity (350°C, 10% NaCl) the expected sodium concentration in steam increases to about 200 ppm. Sodium is not generally observed in steam condensate at the wellhead from the high-temperature reservoir at concentrations even approaching these levels (Mark Walters, personal communication, 1994). Probably the least likely explanation for these observations is the assumption of minimal NaCl in the condensed phase within the reservoir: this explanation would require significant flushing of the original marine fluids with waters of very low salinity, and appears to be inconsistent with the available fluid inclusion data. Dilution of the high-temperature steam with steam from the 'normal' reservoir is almost certainly occurring in at least some wells in the Northwest Geysers. For this dilution to be the sole mechanism for sodium reduction, the ratio of 'normal' to 'high-temperature' steam produced would have to be greater than 100 to 1. While this may be possible, this mechanism would also dilute significantly the chloride in steam, and probably the noncondensible gases as well.

Steam produced at the Northwest Geysers is significantly superheated. Thus a possible mechanism for the production of chloride-bearing steam with low sodium levels from a brine originally high in sodium chloride is the deposition of halite from undersaturated (superheated) steam which is cooled and depressurized by moving up through the well. Due to the higher sublimation pressure of ammonium chloride as compared with sodium chloride, this mechanism could effectively deplete NaCl in steam while leaving both NH_4Cl and HCl concentrations relatively unaffected. The quantitative aspects of this possible mechanism, including comprehensive calculations of the concentrations of all chlorides in steam (NaCl, NH_4Cl, and HCl) in equilibrium at high temperatures with acidic, mixed (NaCl + NH_4Cl) brines and the solubilities of solid NaCl and NH_4Cl in superheated steam as functions of temperature and pressure, are being addressed in detail in this program in order to assess the plausibility of this proposed mechanism. It seems likely that the overall process leading to the observed steam compositions are a combination of mechanisms, probably including both steam mixing (dilution) and solid salt deposition. Other considerations listed above, including details of the brine compositions in equilibrium with steam containing additional noncondensible gases (H_2S and CO_2) and the effect of adsorption on the partitioning behavior of various solutes in brines, will be addressed in both further experimental and modeling studies.

It seems clear from the trends with temperature of the partitioning equilibrium constants shown Figure 1 that desuperheating of steam containing HCl and NH_4Cl should be an effective mechanism for the reduction of chloride in steam. The partitioning constant at saturation pressure for HCl decreases by about two orders of magnitude on changing temperature from 350 to 250°C, while that for NH_4Cl is lower by about three log units. Thus the formation of a small amount of liquid in equilibrium with steam at lower temperature will be very effective in removing both NH_4Cl and HCl from steam. In carrying out such a process care should be taken to avoid detrimental effects on system components (e.g., well casing) due to the formation of an acidic, high chloride brine at high temperatures, and some neutralizing agent may be required to protect these components. Significant steam enthalpy will be lost in this process, and the cost of mitigation may be high. However, the injection of a smaller amount of neutralizer (e.g., NaOH) at high concentrations may not be effective in removing the chloride, as a significant portion of the total chloride should be present as ammonium chloride and hence less prone to be removed from steam as NaCl on caustic neutralization.

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