

Can the vapour phase be neglected to estimate bulk salinity of halite bearing aqueous fluid inclusions?

Research Article

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Abstract: The bulk salinity cannot be directly obtained from the dissolution temperatures of halite in highly saline fluid inclusions that contain solid, liquid, and vapour at room temperature. At least two of the following independent parameters must be determined to estimate the bulk composition and density of these inclusions: 1. dissolution temperature of halite in the presence of vapour; 2. total homogenization temperature of liquid and vapour; and 3. volume fraction of the vapour phase. A new V^m - x diagram for phase stabilities in the H_2O - $NaCl$ system has been constructed to obtain these bulk fluid properties from inclusions that homogenize liquid and vapour phase at higher temperatures than dissolution of halite.

Keywords: fluid inclusions • H_2O - $NaCl$ system • bulk salinity • V^m - x properties

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1. Introduction

The salinity of aqueous fluid inclusions can be determined by microthermometry, mainly by cooling the samples to low temperatures and measuring dissolution temperatures of ice and salt-hydrates [1]. At higher temperatures, the dissolution temperature of salt also can be used to estimate salinities [2, 3]. The most common types of salt in natural fluid inclusions are $NaCl$ and the phase changes in the binary H_2O - $NaCl$ system are well known [4] and references therein. The relationship between temperature (T), pressure (p), molar volume (V^m) and composition (x) in this system can be thermodynamically modelled [5] or it can be modelled with purely empirical equations, which

usually include only a part of these intensive variables [2, 6], e.g. T (dissolution temperature) and x (salinity) (Figure 1). Phase assemblages at selected temperatures and pressures can be determined by using thermodynamic models, which may predict the properties of coexisting phases at constant total volume, e.g. the presence of a liquid and vapour phase in a fluid inclusion.

The dissolution temperature of ice is often used to calculate the bulk salinity of fluid inclusions [7] from the "melting" curve. "Melting" is in principle not the correct terminology in binary systems because it refers to the properties of pure chemical species, e.g. the melting temperature of ice (system H_2O) is $0^\circ C$ at 0.1 MPa. The range of ice dissolution temperatures in the H_2O - $NaCl$ system is between $-21.2^\circ C$ and $0^\circ C$ at pressures below 0.1 MPa (Figure 1). A prerequisite for the application of this method is the presence of a vapour phase in the inclusions during the entire dissolution path. Ice may dissolve

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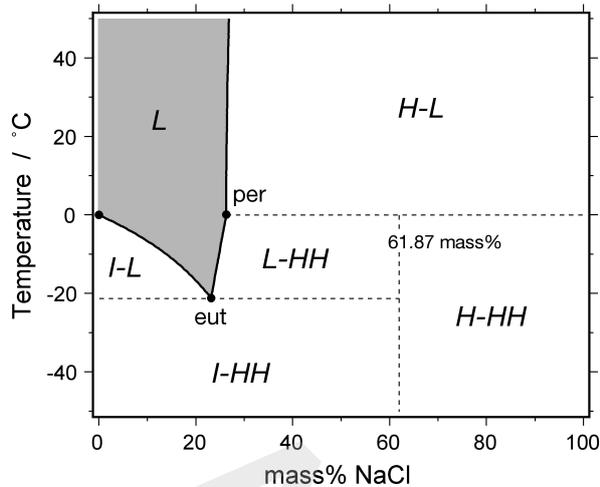


Figure 1. T - w diagram of the H_2O - NaCl system, at low variable pressures, in the presence of a vapour phase; generally used to estimate salinities from final dissolution temperatures of ice, hydrohalite and halite in fluid inclusions. The shaded area contains a homogeneous liquid phase (L), two-phase fields are indicated with H - L (halite + liquid), H - HH (halite + hydrohalite), I - HH (ice + hydrohalite), HH - L (hydrohalite + liquid), and I - L (ice + liquid). The eutectic point is illustrated at 23.2 mass% NaCl and -21.2 °C (eut), the peritectic point at 26.3 mass% NaCl and $+0.1$ °C (per). The composition of hydrohalite is indicated with 61.87 mass% NaCl .

metastably at higher temperatures (even at positive degrees Celsius) in inclusions that do not contain a vapour phase. The properties of the vapour phase can be completely neglected in bulk salinity calculations because it contains only minor amounts of H_2O molecules and even less NaCl molecules. In fluid inclusions with higher salinity, i.e. above eutectic compositions (23.2 mass % NaCl), hydrohalite is the last phase to dissolve and bulk salinities can also be directly calculated from the final dissolution temperature [2]. Hydrohalite dissolution takes place in the temperature range of -21.2 to $+0.1$ °C (Figure 1). Final dissolution temperatures above $+0.1$ °C are occasionally observed, and represent metastable dissolution processes, that are mainly caused by the delayed transformation of hydrohalite into halite at the peritectic point. The properties of the vapour phase can again be neglected to estimate bulk salinities.

Fluid inclusions with high bulk salinity (> 26.3 mass % NaCl) may contain a salt crystal in addition to a vapour and liquid phase at room temperature. The generally accepted method to determine bulk salinities of these inclusions is to measure the dissolution temperature of halite in the presence of a vapour phase [2], and absence of a vapour phase [3]. This study is restricted to the former phase change behaviour. There are a number of limita-

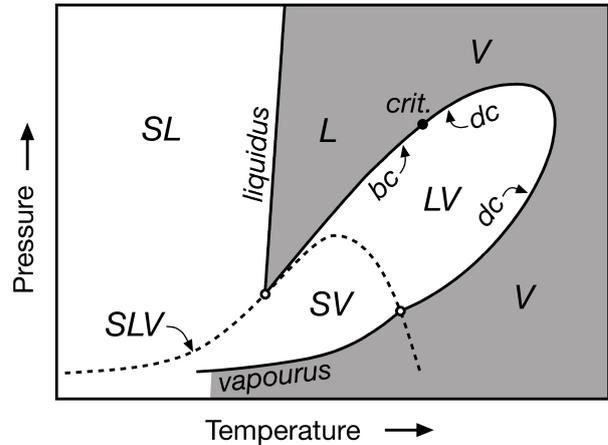


Figure 2. A schematic p - T phase diagram of the binary H_2O - NaCl system, for a specific composition (isopleth). Phases are abbreviated with L for liquid, V for vapour, and S for solid. The shaded area contains a homogeneous fluid mixture; two-phase fields are illustrated with SL , LV and SV . The dashed curve (SLV) illustrates the stability of three phases. bc is the bubble point curve, dc is the dew point curve and $crit$ is the critical point of this fluid mixture.

tions to this method because the properties of the vapour phase cannot be neglected at these higher temperatures. It may contain considerable amounts of H_2O , and, consequently, bulk salinities are substantially lower than estimated directly from the dissolution temperature of halite. This study presents the extent to which the vapour phase may affect bulk salinity estimations, and the effect is modelled by combining the equation of state for the H_2O - NaCl system according to Anderko and Pitzer [5] and the purely empirical modelling of the three phase curve (i.e. the phase assemblage solid + liquid + vapour) [4].

2. Topology of the binary H_2O - NaCl system at high temperatures and pressures

The H_2O - NaCl fluid system has been investigated in a variety of papers e.g. [8–10]. A p - T phase diagram of this system contains a variety of elements that define the stability of phase assemblages (Figure 2). The limits of a homogeneous mixture, either liquid-like (high density) or vapour-like (low density) are defined by the liquidus, bubble point curve, critical point, dew point curve and vapourus. The positions of these curves are variable, dependent on bulk composition. Figure 2 shows three fields of two coexisting phases: liquid + vapour (LV), solid + liquid (SL), and solid + vapour (SV). The SLV curve illustrates

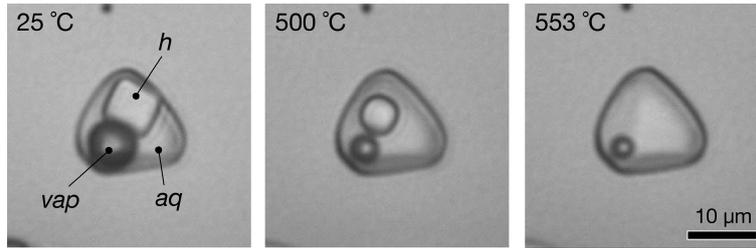


Figure 3. Heating sequence (25 °C → 500 °C → 553 °C) of synthetic H₂O-NaCl-rich fluid inclusion in natural quartz. At 25 °C the inclusion contains a halite crystal (*h*), vapour (*vap*) and a brine (*aq*). Complete dissolution of the halite crystal occurs at 553 °C. The homogenization of liquid and vapour phase occurs at a temperature > 600 °C.

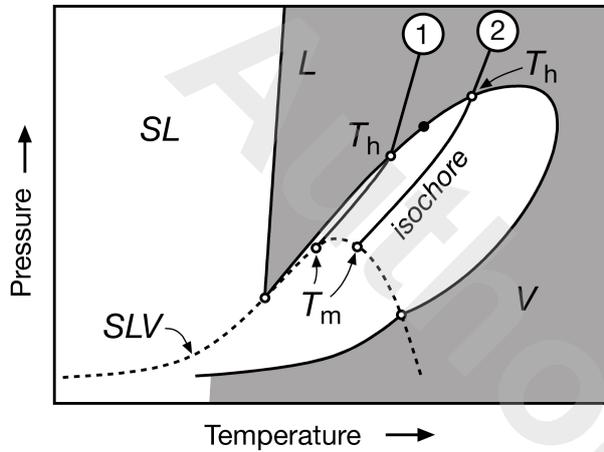


Figure 4. Schematic p - T diagram of the binary H₂O-NaCl system, similar to Figure 1. Isochores are illustrated for a high-density fluid (1) that homogenize in the liquid phase, and for a lower density fluid (2) that homogenizes in the vapour phase at higher temperatures and pressures. The isochores have different intersection points with the SLV curve, and, therefore, different dissolution temperatures of halite, although they have the same bulk salinity.

the coexistence of three phases, i.e. solid+liquid+vapour. The dissolution of halite in the presence of only a liquid phase occurs at the liquidus curve, which is nearly vertical in a p - T diagram. The intersection of the liquidus and the SLV curve illustrates dissolution of halite in the presence of both a vapour and liquid phase.

Fluid inclusions that contain a halite crystal, liquid and vapour phase are located on the SLV curve. Upon heating, and before homogenization and complete dissolution, the fluid inclusion pressure is defined by the SLV curve. Several homogenization-dissolution sequences can take place in these inclusions: 1. $SLV \rightarrow SL \rightarrow L$ (i.e. homogenization of liquid and vapour, followed by dissolution of halite); 2. $SLV \rightarrow LV \rightarrow L$ and 3. $SLV \rightarrow LV \rightarrow V$. This study is restricted to the sequences 2 and 3 (Figure 3), i.e. dissolution of halite followed by homogenization of liquid

and vapour. During heating after dissolution of halite, the p - T conditions move into the LV field along an isochore that is specific for the bulk density of the inclusion (Figure 4). The intersection of this isochore and the SLV curve marks the dissolution temperature of halite (T^m), which is variable according to the bulk density at a constant bulk composition (compare isochore 1 and 2 in Figure 4). In other words, inclusions with the same bulk composition display higher T^m at lower bulk densities (i.e. higher bulk molar volumes). Differences in density, subsequently, are characterized by a range of total homogenization temperatures (T^h).

Thermodynamic modelling of isochores within the LV field, and the properties of the SLV curve can be used to calculate bulk fluid properties of inclusions, by measuring both the dissolution temperature of halite and subsequently the homogenization temperature of the liquid and vapour phases. The volume fraction of the vapour phase is directly related to bulk densities and therefore also can be used to determine bulk fluid properties in combination with dissolution temperatures.

3. Modelling of phase equilibria

3.1. Two-phase field: liquid-vapour equilibrium

Isochores within the LV field can be modelled with an equation of state for the H₂O-NaCl system according to Anderko and Pitzer [5]. This equation of state was originally designed to reproduce liquid-vapour equilibria (bubble point curve and dew point curve, see Figure 2) and isochores of homogeneous mixtures between 300 and 500 °C. It was suggested that this equation is valid up to 900 °C and 500 MPa. Anderko and Pitzer [5] used a Helmholtz energy function according to theoretical considerations [11, 12] that are defined by the intensive variables of tem-

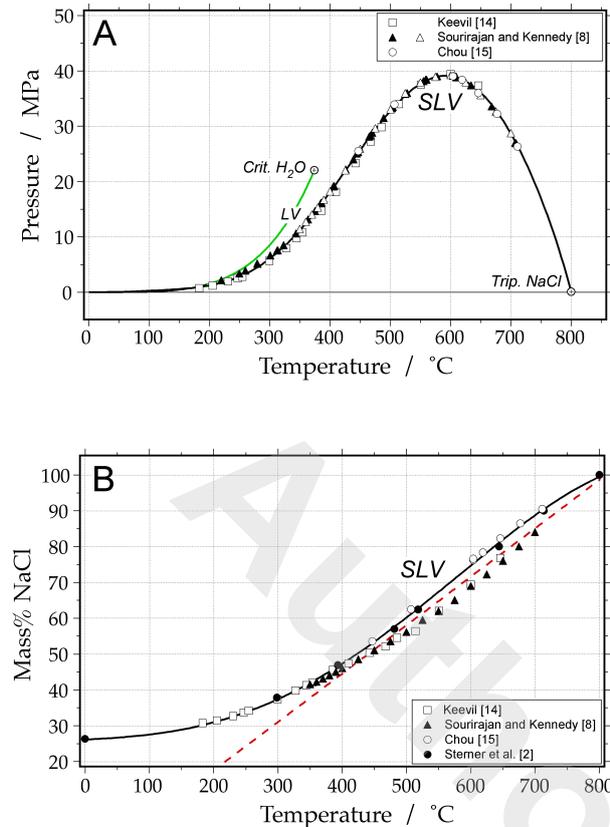


Figure 5. A. p - T diagram of the SLV curve. The green curve illustrates the position of the LV curve of pure H_2O with its critical point at 374 °C and 22 MPa for comparison. The solid curve is the empirical best fit [4] between 0 and 800 °C . B. w - T diagram (NaCl mass fraction, in %) of the SLV curve. The empirical best-fit curve (solid line) [4] only is based on the experimental data from Chou [15] and Sterner et al. [2]. The red dashed line is modelled according to Anderko and Pitzer [5].

perature (T), molar volume (V^m) and the amount of substance fraction (x). The pressure (p) is calculated from the partial derivative of the Helmholtz energy function towards volume at constant temperature, according to standard thermodynamic functions e.g. [13]. This equation can be directly applied to fluid inclusion because it gives a relationship between variables that directly describe the most important properties of fluid inclusions, and that can be obtained by microthermometry.

3.2. Three-phase curve (SLV)

The SLV curve represents all possible halite dissolution temperatures in the presence of a vapour phase. The p - T conditions of this curve were investigated in a number of studies [2, 8, 14, 15] (Figure 5A). Experimental T - w studies

display variable results (Figure 5B); Chou [15] illustrating that the estimated NaCl mass fractions in previous work [8, 14] were underestimated. In this study, both pressure and NaCl mass fraction are modelled as a function only of temperature by purely empirical equations [4], in the range of 0 °C to 800 °C .

The NaCl mass fraction (w) and amount of substance fraction (x) at selected p - T conditions on the SLV curve alternatively can be calculated with the Anderko and Pitzer equation of state [5] (dashed red line in Figure 5B). The obtained SLV curve is consistent with experimental data above 350 °C .

3.3. Volume fractions

The measurement of total homogenization temperatures technically is restricted depending on the type of heating stage. Temperatures over 600 °C cannot be measured with standard stages (e.g. Linkam MDS 600). Alternatively, volume fraction estimates of the vapour phase (φ^{vap}) [16] at halite dissolution temperatures can be used instead to provide a sufficient number of parameters, which allow the calculation of bulk fluid properties.

4. V^m - x diagram

The principle objective of fluid inclusion research is to determine the bulk composition and bulk molar volume (or density) of the fluid that is trapped in a host crystal. Isochore construction, trapping conditions, and other interpretations mainly depend on these parameters. The advantage of a V^m - x diagram is that the axes define these desired bulk properties of fluid inclusions. Within this diagram, lines of equal dissolution temperature, equal homogenization temperature ($LV \rightarrow L$, or $LV \rightarrow V$), and equal volume fraction of the vapour phase can be constructed from thermodynamic models or directly interpolated from experimental data. The measurement of only two of these variables is sufficient to obtain an intersection point that defines bulk composition (e.g. salinity) and bulk molar volume (or density).

4.1. Examples of V^m - x diagrams for fluid inclusion studies

The application of V^m - x diagrams in fluid inclusion studies is limited due to the complexity of equations (purely empirical and thermodynamical) to calculate bulk fluid properties and the lack of computer programs to facilitate

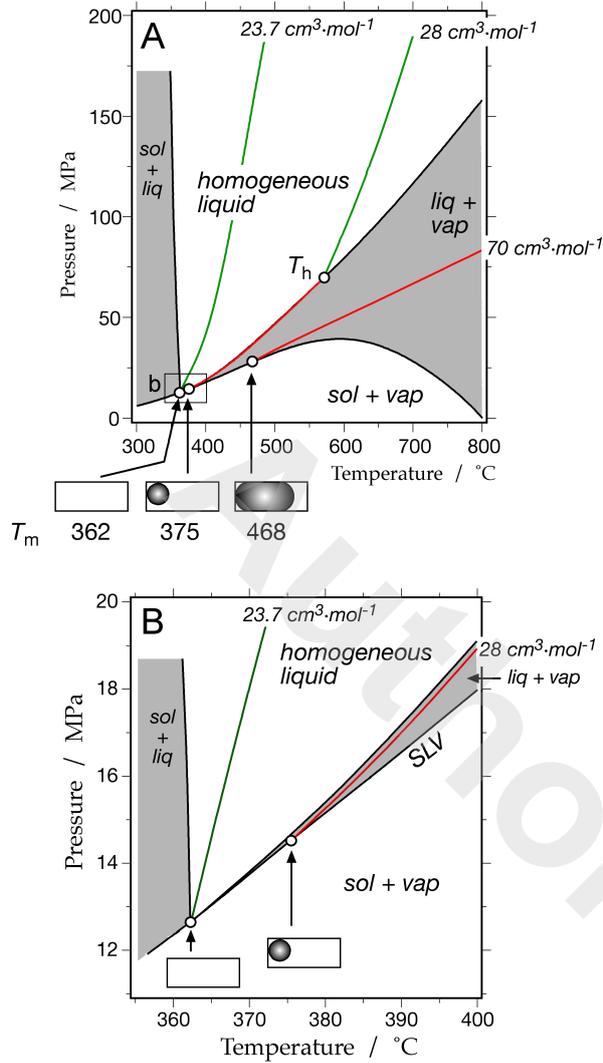


Figure 6. A. p - T diagram of a 40 mass % NaCl solution [4, 5]. Three schematic fluid inclusions are illustrated with different bulk V^m values, and, therefore, different halite dissolution temperatures (T^m in °C). The red lines are isochores within the LV field; green lines are isochores of homogeneous fluid mixtures, above homogenization temperatures. B. Enlargement of part of diagram in Figure 5A, to illustrate that the $28 \text{ cm}^3 \cdot \text{mol}^{-1}$ isochore is positioned within the LV field.

this method. Moreover, the reliability of V^m - x diagrams depends on the availability of accurate equations of state for specific binary fluid mixtures. These equations must give a full description of the relationship between the intensive variables p , T , V^m and x , which exclude purely empirical models.

Accurate V^m - x diagrams only are available for the binary systems CO_2 - CH_4 [18], CO_2 - N_2 [18], and H_2O - CO_2 [19]. The first two systems are defined by a modified

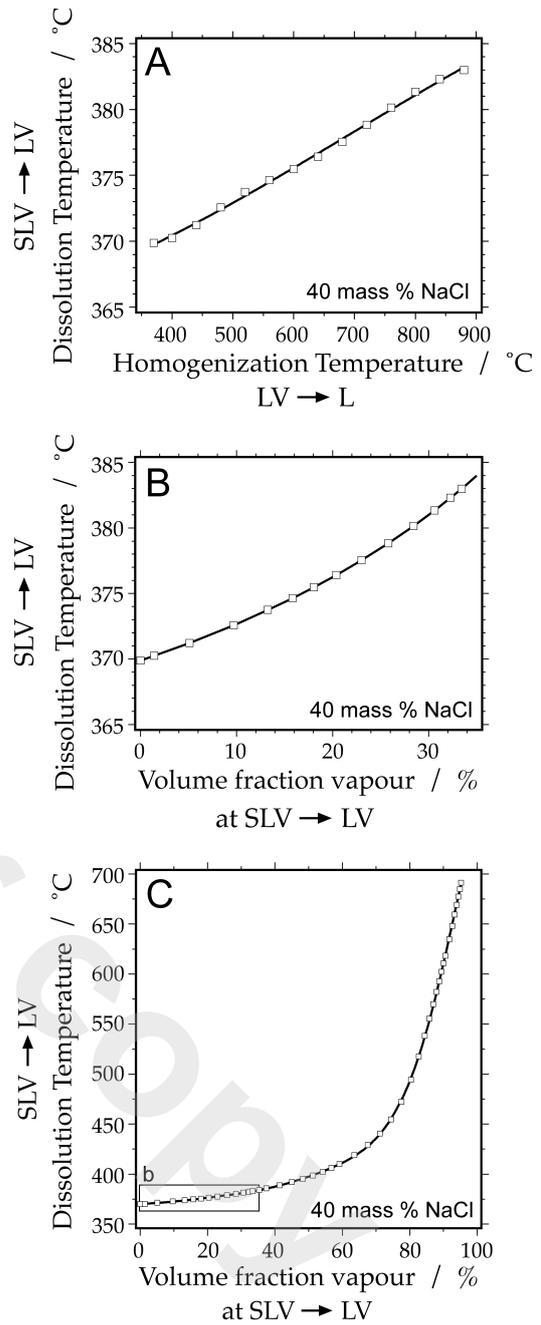


Figure 7. A. T^m - T^h diagram for a 40 mass % NaCl solution. The open squares are calculated data according to the combination of liquid vapour equilibria [4] and SLV curve properties [4]. B. T^m - φ^{vap} diagram for a 40 mass % NaCl solution in the same T^m range as in A. C. T^m - φ^{vap} diagram for a 40 mass % NaCl solution with the complete variation in φ^{vap} (from 0 to 100 %) and corresponding T^m values.

Soave-Redlich-Kwong equation of state [18] that is combined with an empirical equation of the fugacity of solid CO_2 . Fluid inclusions containing a mixture of CO_2 - CH_4 or CO_2 - N_2 reveal a depressed dissolution temperature of CO_2 . The bulk fluid properties of these inclusions can be obtained by combining this temperature with the homogenization temperature of the CO_2 -rich liquid and CH_4 -rich (or N_2 -rich) vapour phases. Both temperatures can be plotted in a V^m - x diagram to calculate bulk composition and bulk molar volume of the inclusion [17, 18]. The V^m - x diagram of the system H_2O - CO_2 is constructed directly from experimental data [19]. It contains smoothed curves of the total homogenization of an H_2O -rich liquid phase and CO_2 -rich vapour phase, in addition to thermodynamically calculated curves [20] of the partial homogenization temperatures of CO_2 phases (L and V). Measurement of both parameters defines the bulk fluid properties of the inclusion. In addition, a combination of the partial homogenization temperature of CO_2 phases and a volume fraction estimation of the H_2O -rich phase also are sufficient to determine the bulk composition and molar volume of fluid inclusions.

4.2. The binary H_2O - NaCl

The same previously described principles are valid for binary H_2O - NaCl mixtures: at least two parameters must be obtained to calculate bulk fluid properties. The measurement of just one parameter, e.g. the final ice dissolution temperature, may provide the bulk salinity of fluid inclusions, but the bulk molar volume (or density) cannot be obtained from this temperature. At higher temperatures, dissolution temperatures of halite are not even sufficient to estimate bulk salinities. In addition, a volume fraction estimation of the vapour phase or a total homogenization temperature provides the extra information to determine bulk densities and salinities. The obtained measurements must be treated with an adequate equation of state [5] and purely empirical equations for the SLV curve [4] to obtain reliable bulk properties of the fluid.

A system with 40 mass % NaCl and 60 mass % H_2O is selected to illustrate phase changes that take place in fluid inclusions in a heating experiment. At room temperature, a fluid inclusion with this composition contains three phases: solid, liquid, and vapour (c.f. Figure 3 at 25 °C). Different temperatures of homogenization and dissolution are obtained for this inclusion, dependent on its bulk molar volume. An inclusion with a bulk molar volume of $23.7 \text{ cm}^3 \cdot \text{mol}^{-1}$ displays equal T^m and T^h (phase change $SLV \rightarrow L$) at 362 °C (Figure 6). An inclusion with higher molar volume ($28 \text{ cm}^3 \cdot \text{mol}^{-1}$) has T^m of 375 °C. At this temperature, the 16.5 vol% vapour phase has a V^m of

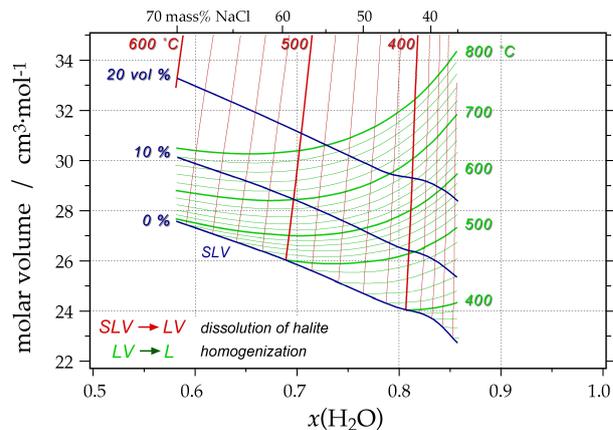


Figure 8. V^m - x diagram of the binary H_2O - NaCl system with isotherms for equal T^h values (green lines), isotherms for equal T^m values (red lines), and lines of equal vapour phase volume fraction (in %) at the dissolution temperature of halite (blue lines). The SLV curve is located at $T^h = T^m$ intersection points (vol% = 0)

$258.2 \text{ cm}^3 \cdot \text{mol}^{-1}$. The vapour phase remains present up to total homogenization at 572 °C ($LV \rightarrow L$). The inclusion with a relatively low density ($70 \text{ cm}^3 \cdot \text{mol}^{-1}$) displays halite dissolution at 468 °C, in the presence of a 77.3 vol % vapour phase, with a V^m of $144.7 \text{ cm}^3 \cdot \text{mol}^{-1}$. The total homogenization of this inclusion exceeds the limits of the Anderko and Pitzer equation of state [5], and exceeds the maximum temperatures normally reached by heating stages. The bulk properties of this inclusion only can be obtained by the measurement of a second independent parameter, i.e. ϕ^{vap} at the dissolution temperature of halite. The systematic increase of T^m with increasing T^h for a 40 NaCl mass % fluid mixture is illustrated in Figure 7A. A similar effect is illustrated in Figure 7B for the increase with volume fraction of the vapour phase. The T^m value for a constant bulk composition is strongly affected for ϕ^{vap} values above 60 % (Figure 7C). The dissolution temperature may rise up to about 700 °C in vapour-rich fluid inclusions.

The result of these calculations also can be displayed in a V^m - x diagram of the binary H_2O - NaCl system for a variety of compositions (Figure 8). The use of this diagram can be illustrated with an example: a fluid inclusion with $T^m(SLV \rightarrow LV) = 500 \text{ °C}$ and $T^h(LV \rightarrow L) = 800 \text{ °C}$. The bulk V^m and $x(\text{H}_2\text{O})$ of this fluid inclusion are $30.44 \text{ cm}^3 \cdot \text{mol}^{-1}$ and 0.7013 (corresponding to 58.05 mass% NaCl), respectively. This inclusion displays an 18 vol % vapour phase at T^m . A fluid inclusion with simultaneous halite dissolution and LV homogenization at 500 °C (at the SLV curve) has V^m and $x(\text{H}_2\text{O})$ values of $26.06 \text{ cm}^3 \cdot \text{mol}^{-1}$ and 0.6887 (corresponding to 59.45 mass

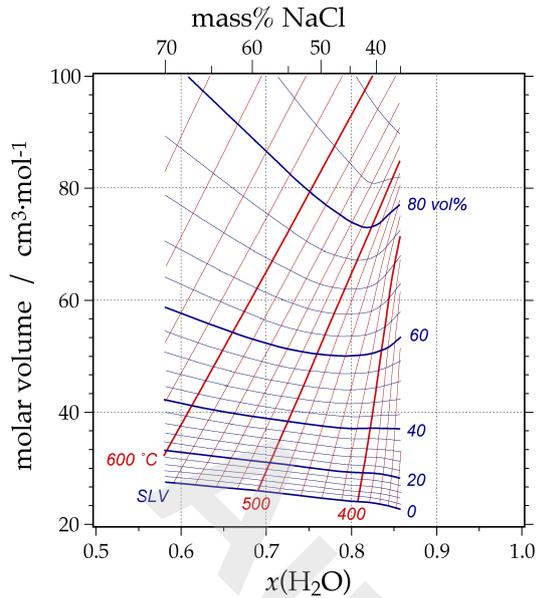


Figure 9. V^m - x diagram of the binary H_2O - NaCl system with only the isotherms for equal T^m values (red lines), and the lines of equal vapour phase volume fraction (blue lines, in %) at the dissolution temperature of halite.

% NaCl), respectively. Figure 8 is restricted to total homogenizations to the liquid phase. Total homogenizations in the vapour phase occur at much higher temperatures, which often exceed the limits of heating stages. This figure can be extended to higher V^m values by using the lines of equal volume fractions of the vapour to estimate the properties of low bulk-density fluid inclusions (Figure 9). For example, a T^m of 500 °C corresponds to a range in bulk salinity of 35.29 to 59.54 mass % NaCl, dependent on the volume fraction (range of 0 to 85 %), whereas the V^m varies between 26.06 and 84.79 $\text{cm}^3\cdot\text{mol}^{-1}$.

The V^m - x diagrams presented in Figure 8 and 9 can be applied manually or by using software that extracts data points from scanned graphs (e.g. "DataThief" [21]). The calculation procedures that are used to design the V^m - x diagrams are embedded in the software package "FLUIDS" [21] as part of the Anderko and Pitzer equation of state, and will be available for fluid inclusion research.

5. Conclusions

Salinity calculations of fluid inclusions that are based only on dissolution temperature of halite, in the presence of a vapour phase (SLV curve), can be highly overestimated, if the properties of the vapour phase are neglected. The here presented V^m - x diagram of the system H_2O -

NaCl is an ideal tool for obtaining bulk fluid properties (salinity and density) by using two independent parameters that can be selected from 1. dissolution temperature of halite (SLV \rightarrow LV); 2. total homogenisation temperature (LV \rightarrow L, or LV \rightarrow V); and 3. volume fraction of the vapour phase.

This diagram was constructed with an accurate equation of state based on thermodynamic modelling [5] for liquid-vapour equilibria above 300 °C, and purely empirical modelling of the SLV curve [4] in the range 0 to 800 °C.

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