

Formation constant of the double salt $\text{CsCl}\cdot 2\text{NaCl}\cdot 2\text{H}_2\text{O}(\text{cr})$

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Abstract: In the ternary system $\text{CsCl} - \text{NaCl} - \text{H}_2\text{O}$, at a temperature of 298.15 K, a double salt with the stoichiometric formula $\text{CsCl}\cdot 2\text{NaCl}\cdot 2\text{H}_2\text{O}(\text{cr})$ is known to be formed. This double salt and the anhydrous $\text{CsCl}(\text{cr})$ are the end-members of a solid solution. For the pure double salt, the solubility constant was determined. The obtained value was applied to calculate the solubility diagram also of the quaternary system $\text{CsCl} - \text{NaCl} - \text{KCl} - \text{H}_2\text{O}$ and the quaternary-reciprocal system $\text{Cs}^+, \text{Na}^+ || \text{Cl}^-, \text{SO}_4^{2-} - \text{H}_2\text{O}$. The solubility constant together with a solid solution between $\text{CsCl}\cdot 2\text{NaCl}\cdot 2\text{H}_2\text{O}(\text{cr})$ and $\text{CsCl}(\text{cr})$ were implemented in THEREDA, which extends the applicability of the existing cesium dataset.

1 Introduction

The knowledge of the solubility of cesium salts is of particular interest for a repository for radioactive waste – especially in the salt host rock – but also for industrial processes *i.e.* the production of refined chemicals (Cs compounds). In the system $\text{CsCl} - \text{NaCl} - \text{H}_2\text{O}$, in addition to $\text{CsCl}(\text{cr})$ and $\text{NaCl}(\text{cr})$ (halite), a double salt with the stoichiometric composition $\text{CsCl}\cdot 2\text{NaCl}\cdot 2\text{H}_2\text{O}(\text{cr})$ is known, which is also an end-member of a solid solution with $\text{CsCl}(\text{cr})$ [PLY/TUL1957], [CHO/ROM1983], [ARK/KAS1984]. So far, no solubility constant is known for this double salt.

Due to the very high solubility of the pure cesium salts (*e.g.* $\text{CsCl}(\text{cr})$, $\text{Cs}_2\text{SO}_4(\text{cr})$), the Pitzer ion-interaction model is necessary for a description of the solubility behavior [PIT1991]. Up to now, quite a number of publications are containing a

Pitzer model for systems including cesium chloride and sodium chloride [PIT1991], de Lima et al. [DEL/PIT1983], Rard et al. [RAR/MIL1982], Holmes et al. [HOL/MES1990], Neck et al. [NEC/KOE1998], El Guendouzi et al. [ELG/BEN2004], Scharge et al. [SCH/MUN2012], [SCH/MUN2013]. None of them contain the double salt $\text{CsCl}\cdot 2\text{NaCl}\cdot 2\text{H}_2\text{O}(\text{cr})$.

Solubility studies in the ternary system $\text{CsCl} - \text{NaCl} - \text{H}_2\text{O}$ have been conducted over the last seven decades. The first work was carried out by Plyushchev et al. [PLY/TUL1957]. The authors describe a solid solution present in the system, but do not mention any double salt. In addition, their solubility data show the largest scattering. Arkhipov et al. first described the solubility of the pure salts and the invariant point and later of the entire system [ARK/KAS1970], [ARK/KAS1984]. Balarew et al. [BAL/KET1975] also investigated the solubility in this ternary system. Again, no double salt was reported in these papers. The

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double salt, $\text{CsCl}\cdot 2\text{NaCl}\cdot 2\text{H}_2\text{O}$, was first described by Chou et al. [CHO/LEE1983], [CHO/ROM1983]. Its structure was solved in 1984 by Evans et al. [EVA/KON1984]. The latest work on solubility determinations in this system was performed by Gao et al. [GAO/LI2017]. Apart from a somewhat wider scatter of solubility data in the solid solution range (< 4 mol NaCl per kg H_2O), the solubility data from the various publications are in good agreement (the data of all the authors mentioned are presented in section 3).

In the $\text{CsCl} - \text{KCl} - \text{H}_2\text{O}$ system, for which a very limited solid solution formation was also described [ARK/KAS1984], the existing solubility data can be described with the pure phases $\text{CsCl}(\text{cr})$ and $\text{KCl}(\text{cr})$ (sylvite), see Figure 1. In contrast, the implementation of a solid solution series is necessary for a correct description of the solubility data in the $\text{CsCl} - \text{NaCl} - \text{H}_2\text{O}$ system, see Figure 2.

2 Adjustment of formation constant for $\text{CsCl}\cdot 2\text{NaCl}\cdot 2\text{H}_2\text{O}(\text{cr})$

In the available literature, the solubility data are given as mass percentages. All datasets were recalculated to molal scale (moles salt per kg H_2O).

Table 1 Solution composition of the invariant point $\text{CsCl}\cdot 2\text{NaCl}\cdot 2\text{H}_2\text{O}(\text{cr}) + \text{NaCl}(\text{cr})$.

NaCl [mol/kg H_2O]	CsCl [mol/kg H_2O]	Phase information as given in reference	Reference
4.299	6.012	$\text{NaCl} + \text{Cs}(\text{Na})\text{Cl}$	[ARK/KAS1984]
4.428	5.728	$\text{NaCl}(\text{cr}) + \text{CsCl}\cdot 2\text{NaCl}\cdot 2\text{H}_2\text{O}(\text{cr})$	[CHO/ROM1983]
4.451	5.487	$\text{NaCl}(\text{cr}) + \text{CsCl}\cdot 2\text{NaCl}\cdot 2\text{H}_2\text{O}(\text{cr})$	[CHO/ROM1983]
4.557	5.236	$\text{NaCl}(\text{cr}) + \text{CsCl}\cdot 2\text{NaCl}\cdot 2\text{H}_2\text{O}(\text{cr})$	[CHO/ROM1983]
4.300	5.424	$\text{NaCl}(\text{cr}) + \text{CsCl}\cdot 2\text{NaCl}\cdot 2\text{H}_2\text{O}(\text{cr})$	[GAO/LI2017]
4.467	5.487	$\text{NaCl}(\text{cr}) + \text{solid solution}$	[PLY/TUL1957]
4.441	5.562	Mean of the above	
4.386*	5.582*	$\text{NaCl}(\text{cr}) + \text{CsCl}\cdot 2\text{NaCl}\cdot 2\text{H}_2\text{O}(\text{cr})$	This work

* Calculated using obtained $\text{CsCl}\cdot 2\text{NaCl}\cdot 2\text{H}_2\text{O}(\text{cr})$ solubility product and an ideal solid solution with the end members $\text{CsCl}(\text{cr})$ and $\text{CsCl}\cdot 2\text{NaCl}\cdot 2\text{H}_2\text{O}(\text{cr})$

For the solubility constant fitting of the double salt, the solution compositions of the invariant point $\text{CsCl}\cdot 2\text{NaCl}\cdot 2\text{H}_2\text{O}(\text{cr}) + \text{NaCl}(\text{cr})$ available in the literature were used, see Table 1.

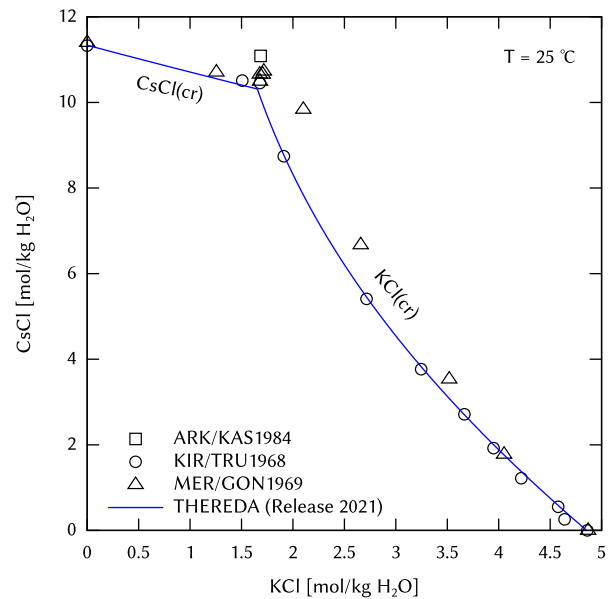
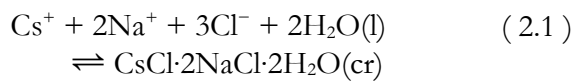


Figure 1 Solubility diagram of the ternary system $\text{CsCl} - \text{KCl} - \text{H}_2\text{O}$, Symbols: experimental solubility data from literature, Lines: calculated using the THEREDA dataset (Release 2021).

The solubility constant ($\log K_S^0$) was determined using the geochemical software PHREEQC (version 3.7) [PAR/APE2013] coupled with the parameter estimation software UCODE_2014 [POE/HIL2014] which uses a minimization of sum of squared residuals approach. The experimentally determined solution compositions of the invariant point $\text{CsCl}\cdot 2\text{NaCl}\cdot 2\text{H}_2\text{O}(\text{cr}) + \text{NaCl}(\text{cr})$ available in the literature (Table 1) were used for this fit. In the fit, an ideal solid solution between $\text{CsCl}\cdot 2\text{NaCl}\cdot 2\text{H}_2\text{O}(\text{cr})$ and $\text{CsCl}(\text{cr})$ was included in the calculation. To meet the THEREDA requirements, the formation reaction of the double salt according (2.1) and the corresponding formation constant was calculated from the solubility constant.



From the obtained reaction constant, the free molar Gibbs energy of the reaction ($\Delta_r G^0$) was calculated using equation (2.2), followed by the molar Gibbs free energy of formation ($\Delta_f G^0$) using equation (2.3) as well as the formation data ($\Delta_f H^0$ & S^0) for Na^+ [GUI/FAN2003], Cs^+ [GRE/FUG1992], Cl^- [GUI/FAN2003] and $\text{H}_2\text{O}(\text{l})$ [LEM/BER2013].

$$\Delta_r G^0 = -RT \ln K^0 \quad (2.2)$$

$$\Delta_f G_i^0 = \Delta_r G_i^0 - \sum v_i \Delta_f G_j^0 \quad (2.3)$$

The results are given in Table 2.

Table 2 Formation constants for $\text{CsCl}\cdot 2\text{NaCl}\cdot 2\text{H}_2\text{O}(\text{cr})$.

Data Type	Value
$\log K^0(2.1)$	$-4.288 \pm 0.014^*$
$\Delta_r G_m^0$	$24\,476 \pm 79^{**} \text{ J/mol}$
$\Delta_f G_m^0$	$-1\,658\,820 \pm 1\,416^{**} \text{ J/mol}$

* The stated uncertainty refers to the standard deviation (2σ).

** Uncertainty calculated from $\log K$'s uncertainty and the elements formation data uncertainty.

3 Results and Discussion

The implementation of the double salt $\text{CsCl}\cdot 2\text{NaCl}\cdot 2\text{H}_2\text{O}(\text{cr})$ with its determined formation constant together with a solid solution between $\text{CsCl}(\text{cr})$ and $\text{CsCl}\cdot 2\text{NaCl}\cdot 2\text{H}_2\text{O}(\text{cr})$ significantly improves the modeling of the ternary system $\text{CsCl} - \text{NaCl} - \text{H}_2\text{O}$ compared to the experimental data as can be seen in Figure 2.

Only in the region of the solid solution, there is a slight deviation between the experimental data and the calculated results.

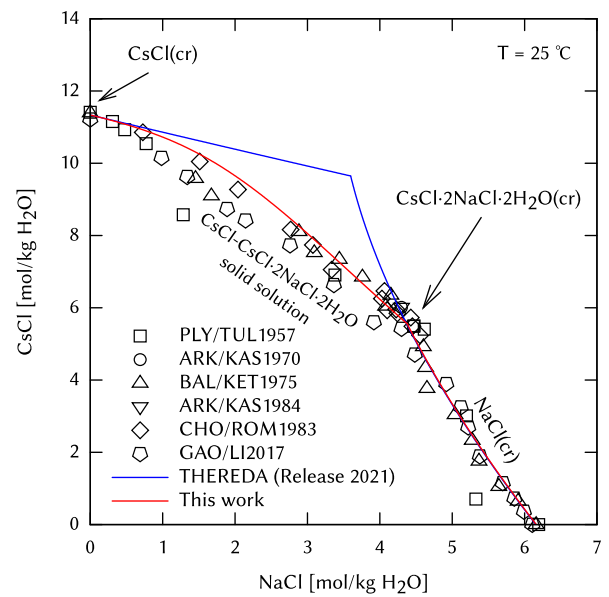


Figure 2 Solubility diagram of the ternary system $\text{CsCl} - \text{NaCl} - \text{H}_2\text{O}$. Symbols: experimental solubility data from literature, Lines: calculation results using the THEREDA dataset (Release 2021) without (blue) and with double salt and an ideal solid solution from this work (red).

3.1 The quaternary system $\text{CsCl} - \text{NaCl} - \text{KCl} - \text{H}_2\text{O}$

In order to test the effects of the implementation of the double salt and the ideal solid solution, the experimentally determined solubility data from [ARK/KAS1984] for the quaternary system $\text{CsCl} - \text{NaCl} - \text{KCl} - \text{H}_2\text{O}$ were recalculated. The results are shown in Figure 3.

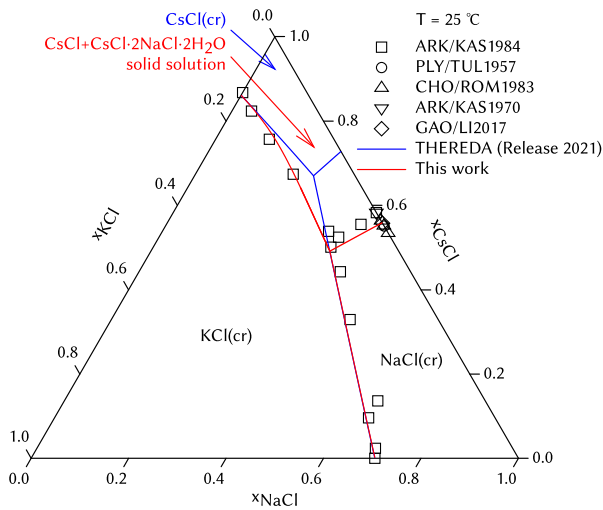


Figure 3 Solubility diagram of the quaternary system CsCl – NaCl – KCl – H₂O. Symbols: experimental solubility data from literature, Lines: Calculations using the THEREDA dataset (Release 2021) without (blue) and with double salt and an ideal solid solution from this work (red).

The modelling result shows a very good agreement with the experimental data of the dominant solid phases. No further adjustment of the Pitzer coefficients or other solubility constants of the solid phases occurring in this system were necessary.

Since the diagram in Figure 3 only represents phase areas as a function of the relative ion concentrations, but not the single ion concentrations, the values of the invariant point NaCl(cr)-KCl(cr)-CsCl·2NaCl·2H₂O(cr) are additionally listed in Table 3.

Table 3 Comparison between experimental [ARK/KAS1984] and calculated concentrations at the invariant point NaCl(cr)-KCl(cr)-CsCl·2NaCl·2H₂O(cr).

	Experimentally determined concentration	Calculated concentration
Cs ⁺	5.387 mol/kg H ₂ O	5.215 mol/kg H ₂ O
Na ⁺	3.920 mol/kg H ₂ O	3.896 mol/kg H ₂ O
K ⁺	1.445 mol/kg H ₂ O	1.498 mol/kg H ₂ O

3.2 The quaternary-reciprocal system Cs⁺, Na⁺ || Cl⁻, SO₄²⁻ – H₂O

Also in the quaternary-reciprocal system Cs⁺, Na⁺ || Cl⁻, SO₄²⁻ – H₂O the modeling improves by including the double salt and the ideal solid solution. In particular, the chloride-rich part of the diagram is reproduced better, see Figure 4. However, the few experimental datasets for the quaternary-reciprocal system from Poletaev et al. [POL/PLY1974] and Shi et al. [SHI/CHE2020] are not consistent and highly scattered.

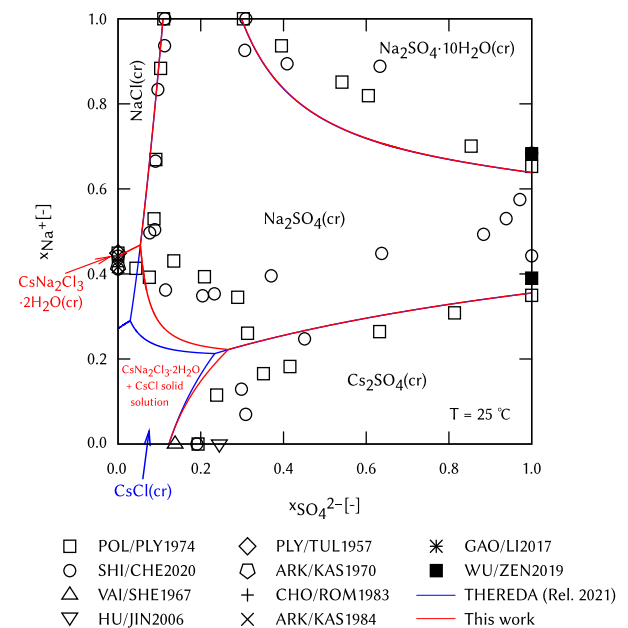


Figure 4 Solubility diagram of the quaternary-reciprocal system Cs⁺, Na⁺ || Cl⁻, SO₄²⁻ – H₂O, Symbols: experimental solubility data from literature, Lines: calculated using THEREDA dataset (Release 2021) without (blue) and with double salt and an ideal solid solution from this work (red).

A more perfect description of the quaternary-reciprocal system *e.g.*, for the phase boundary between Na₂SO₄·10H₂O(cr) (mirabilite) and Na₂SO₄(cr) (thenardite) could be achieved with a new optimization of the mixing parameters including the quaternary solubility data. However, this is not intended in the present stage of the development of the database.

4 Summary

The presented extension of the available cesium Pitzer data set by the $\text{CsCl}\cdot 2\text{NaCl}\cdot 2\text{H}_2\text{O}(\text{cr})$ double salt and an ideal solid solution of it with $\text{CsCl}(\text{cr})$ in the system $\text{CsCl} - \text{NaCl} - \text{H}_2\text{O}$ significantly improves the results of solubility calculations according the experimental data from literature.

It could be demonstrated that the extension is also an improvement for the modeling for higher systems – the quaternary system $\text{CsCl} - \text{NaCl} - \text{KCl} - \text{H}_2\text{O}$ as well as the quaternary-reciprocal system $\text{Cs}^+, \text{Na}^+ || \text{Cl}^-, \text{SO}_4^{2-} - \text{H}_2\text{O}$ – with respect to the occurring solid phases and their solubilities.

It must be emphasized that the determined solubility constant of the double salt as well as the Gibbs enthalpies calculated from it are valid only as long as the calculation is performed with an ideal solid solution between $\text{CsCl}(\text{cr})$ and $\text{CsCl}\cdot 2\text{NaCl}\cdot 2\text{H}_2\text{O}(\text{cr})$.

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