

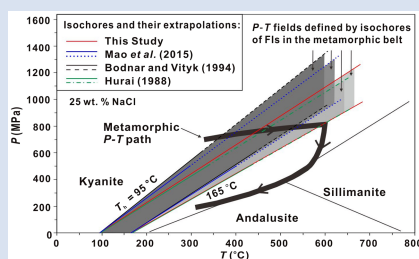
In situ determination of NaCl-H₂O isochores up to 900 °C and 1.2 GPa in a hydrothermal diamond-anvil cell

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Abstract



NaCl-H₂O is a typical binary system solution in geologic environments. However, its available *PVTX* properties (e.g., isochores) are primarily applicable in the pressure-temperature (*P-T*) range of <600 MPa and <700 °C. Here, we performed 53 experiments in a hydrothermal diamond-anvil cell (HDAC) to determine NaCl-H₂O isochores by using the newly defined α - β quartz *P-T* transition boundary as a pressure calibrant (Li and Chou, 2022). The refined isochores fitted with our data are expressed by: $P(\text{bar}) = A_1 + A_2 \times T(\text{°C})$ and

$$A_1 = 0.0061 + (0.2385 - a_1) \times T_h - (0.002855 + a_2) \times T_h^2 - (a_3 \times T_h + a_4 \times T_h^2) \times m$$

$$A_2 = a_1 + a_2 \times T_h + 9.888 \times 10^{-6} \times T_h^2 + (a_3 + a_4 \times T_h) \times m$$

where *m* is the NaCl molality (mole/kg H₂O), *T_h* (°C) is the liquid-vapour homogenisation (to the liquid phase) temperature, and *a*₁, *a*₂, *a*₃, and *a*₄ are constants (27.21, -0.05956, -0.3095, and 0.003232, respectively). The isochores have better applicability for the salinity range of 5–25 wt. % NaCl, 100 °C < *T_h* < 450 °C, and *P-T* range up to ~1.2 GPa and ~900 °C. Compared with previous data, these isochores are more precise above 600 MPa, and are particularly suitable for the geological applications involving saline fluids in the deep Earth.

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Introduction

NaCl-H₂O solutions exist widely in geologic environments. The properties of the pressure, volume, temperature, and composition (*PVTX*) of the binary system have been widely investigated to interpret the rock- and ore-forming conditions, and quantify mass transfer in many geological settings, such as subduction zones (Mantegazzi et al., 2013). Empirical and theoretical models have been published to describe the *PVTX* properties of NaCl-H₂O (e.g., Bodnar and Vityk, 1994). However, most *PVTX* models for NaCl-H₂O are applicable under low temperature (e.g., ≤700 °C) and pressure (e.g., ≤600 MPa) conditions (e.g., Bodnar and Sterner, 1987; Mao et al., 2015). This is mainly because these *PVTX* properties were derived from experimental results, such as those obtained with synthetic fluid inclusion (SFI) methods, using pressure vessels operated under relatively low *P-T* conditions (Gehrig, 1980; Zhang and Frantz, 1987; Bodnar and Vityk, 1994). Moreover, many *PVTX* models were built by using the equation of state (EoS) of H₂O suggested by Haar et al. (1984) (e.g., Driesner, 2007), which has been considered to have lower accuracy than other available models at pressures >600 MPa (e.g., Li and Chou, 2022). Consequently, most current available EoSs of H₂O-NaCl are only applicable to the upper crustal *P-T* conditions and thus unsuitable for describing geological processes in lower crustal conditions, such as those

involving the saline aqueous fluids released from subduction slabs where the pressure may become much higher than 600 MPa (e.g., Kawamoto et al., 2018).

In order to experimentally model such high *P-T* conditions, the hydrothermal diamond-anvil cell (HDAC; Bassett et al., 1993), is a good option. It can potentially yield a sample chamber with a constant volume during an experiment at pressures up to 2.5 GPa and temperatures from -190 °C to 1200 °C (Bassett et al., 1993), making it excellent to measure the *PVTX* properties or isochores of fluids under wide *P-T* conditions. By using the HDAC to measure the *PVTX* properties of a fluid, it is crucial to determine the pressure value at a set temperature in a homogenous fluid phase inside the sample chamber (Figure S-1a). Previously, pressure sensors based on shifts of Raman or fluorescence lines in some minerals or materials (e.g., quartz and ruby) were commonly used in HDAC experiments, despite their large associated uncertainties (Schmidt and Ziemann, 2000). By using these pressure sensors, Mantegazzi et al. (2013) used a diamond-anvil cell to determine the *PVTX* properties of NaCl-H₂O solutions at 0.5–4.5 GPa and ≤400 °C (extrapolated up to 800 °C).

To obtain NaCl-H₂O isochore data with high precision in a wide *PVTX* range through HDAC experiments, this study uses the α - β quartz phase transition *P-T* boundary as the pressure calibrant (Figure S-1a), as done by Shen et al. (1993).

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In HDAC experiments, the α - β quartz phase transition temperature (T_{tr}) can be measured by optical observation of interference fringes (Shen *et al.*, 1993) or the Raman shifts of the quartz 464 cm^{-1} band (Schmidt and Ziemann, 2000), with rigorous experimental conditions but large uncertainties (Li and Chou, 2022). Recently, Li and Chou (2022) found that the abrupt change in the Raman shift of the quartz 128 cm^{-1} band is much more sensitive and precise than that of the 464 cm^{-1} band during heating for the detection and measurement of the T_{tr} , particularly under high P conditions (Figure S-1b); a new α - β quartz P - T boundary with high precision was redefined by Li and Chou (2022). Moreover, a cooling system for HDAC was designed (Li *et al.*, 2020), which can be used to determine the true salinities of the loaded H_2O -NaCl solutions through ice melting temperatures (T_{ice} s). This prevents an erroneous assumption that the salinity of the prepared H_2O -NaCl solution is the true salinity of the loaded fluid, ignoring the effect of unavoidable evaporation of water and the corresponding increase in the salinity during loading (Li *et al.*, 2020). These new experimental procedures to measure the pressure and salinity can be applied to obtain fluid isochores with high precisions, especially at elevated temperatures and pressures. Therefore, here we loaded NaCl- H_2O solutions in an HDAC sample chamber together with a chip of natural quartz (Figure S-2) to measure NaCl- H_2O isochores with a Raman spectrometer.

Experimental Results

Experimental details are provided in the [Supplementary Information](#) (Experimental Methods). A total of 53 experiments with P - T range up to 1.2 GPa and 900 °C have been conducted with HDAC in this study. In each experiment, the T_{ice} , T_{tr} , and liquid-vapour homogenisation (to liquid phase) temperature (T_h) of the sample fluid were recorded (Table S-1). During heating, the Raman shifts of the 128 cm^{-1} Raman band of α -quartz in the sample chamber were collected in each experiment to determine T_{tr} s (Figure S-1b). Subsequently, the α - β quartz phase transition pressure (P_{tr}) was calculated from T_{tr} according to the refined α - β quartz P - T boundary of Li and Chou (2022).

Each isochore of NaCl- H_2O in our experiment is established through two P - T points; one is the P_h - T_h of the NaCl- H_2O system in the HDAC sample chamber, and the other is P_{tr} - T_{tr} described above (Figure S-1a). The P_h is the liquid-vapour homogenisation (to the liquid phase) pressure for the sample NaCl- H_2O fluid calculated from the measured T_h by using the equation of Bodnar (1983). All the P - T data of the NaCl- H_2O isochores are presented in Table S-1 and Figure 1.

Discussion

Isochores fitted with the experimental data. To facilitate the interpolation of our experimental results, we used the equation formats provided by Zhang and Frantz (1987) to fit the H_2O -NaCl isochores determined with (T_{tr} , P_h) and (T_{tr} , P_{tr}) listed in Table S-1. This is because their equations were built with the liquid-vapour homogenisation T and P and the corresponding entrapment P - T conditions of SFIs, which are similar to the data groups (T_{tr} , P_h and T_{tr} , P_{tr}) collected in this study (Table S-1). Moreover, their equations can accurately describe PVTX data from many experiments, as commented by Brown (1989). Accordingly, the H_2O -NaCl isochores of this study can be fitted by the following equation with $R^2 = 0.991$:

$$P \text{ (bar)} = A_1 + A_2 \times T \text{ (}^\circ\text{C)} \quad \text{Eq. 1}$$

where

$$A_1 = 0.0061 + (0.2385 - a_1) \times T_h - (0.002855 + a_2) \times T_h^2 - (a_3 \times T_h + a_4 \times T_h^2) \times m$$

and

$$A_2 = a_1 + a_2 \times T_h + 9.888 \times 10^{-6} \times T_h^2 + (a_3 + a_4 \times T_h) \times m$$

The constants A_1 and A_2 are functions of T_h (°C) of the fluid inclusions or NaCl- H_2O solution in the HDAC sample chamber and salinity (m , the NaCl molality in aqueous solution). The parameters a_1 , a_2 , a_3 , and a_4 are 27.21, -0.05956, -0.3095, and 0.003232, respectively. The average errors between the values calculated from Equation 1 and the experimental data for P_{tr} and isochore slopes are 3.7 % and 3.9 %, respectively (Table S-1). These fitting errors among NaCl- H_2O fluids with low to high salinities are consistent (Table S-1), and they are different from those of Zhang and Frantz (1987) which contain large errors for high density NaCl- H_2O solutions, as pointed out by Brown (1989), indicating a better fitting of Equation 1 in this study. Considering the P - T range of our experiments, Equation 1 is considered to have better applicability for solutions with salinity and T_h ranges of 5–21 wt. % NaCl and 100–450 °C, respectively.

Comparisons of experimental data with previous studies. The experimental data of this study are compared with those derived from Bodnar and Vityk (1994) and Mao *et al.* (2015) (Figure 1). The model of Bodnar and Vityk (1994), determined using the SFI method, is applicable at ≤ 600 MPa, and has been widely used to interpret the PVTX properties of geological fluids (*e.g.*, Sullivan *et al.*, 2022). Mao's model (Mao *et al.*, 2015), as a representative thermodynamic model, works up to 1000 °C and 500 MPa. The agreements, among the P_{tr} obtained from our experimental data and those from Bodnar and Vityk (1994) and Mao *et al.* (2015) below 600 MPa for the measured T_{tr} s (Figure 1), demonstrate the reliability of our experimental method and results. However, the deviations of P_{tr} values obtained in our experiments from those extrapolated from the previous isochores, particularly Bodnar and Vityk (1994), are evident above 600 MPa, and they increase with salinity (Figure 1).

The isochores of NaCl- H_2O solutions calculated with Equation 1 in this study were primarily compared with those derived from Hurai (1988), Bodnar and Vityk (1994), and Mao *et al.* (2015) in Figure 2. Note that the isochores of NaCl- H_2O fluids in Bodnar and Vityk (1994) derived from the SFI technology were approximated by connecting the P - T point at which the SFI was formed (P_f - T_f), and the P - T point defined by the observed liquid-vapour homogenisation (to liquid phase) T (P_h - T_h), assuming the volumes of the studied SFI at these two P - T points were the same (isochoric) (Bodnar, 1995). However, it was clearly shown in Figure 17.3 of Bodnar and Sterner (1987) that, even for the pure H_2O system, the volumes of the studied SFIs at these two P - T points were not expected to be the same for most of SFIs (Bodnar and Sterner, 1987; their Table 17.1). To clearly demonstrate their warning, their experimental results for SFIs trapped at 100 MPa and 300, 400, 500 and 600 °C were shown in Figure S-3 by adding the isochores based on the densities of pure H_2O at these two P - T points, which were derived from the well established EoS of H_2O IAPWS-95 (Wagner and Pruß, 2002). Therefore, the isochores obtained by SFI methods should strictly be called as iso- T_h lines, unless the SFI volumes are corrected. Conversely, our isochores were measured *in situ*, during which the volume of the HDAC sample chamber was kept constant. Accordingly, the isochores of this study with high salinities and low T_h s evidently deviate

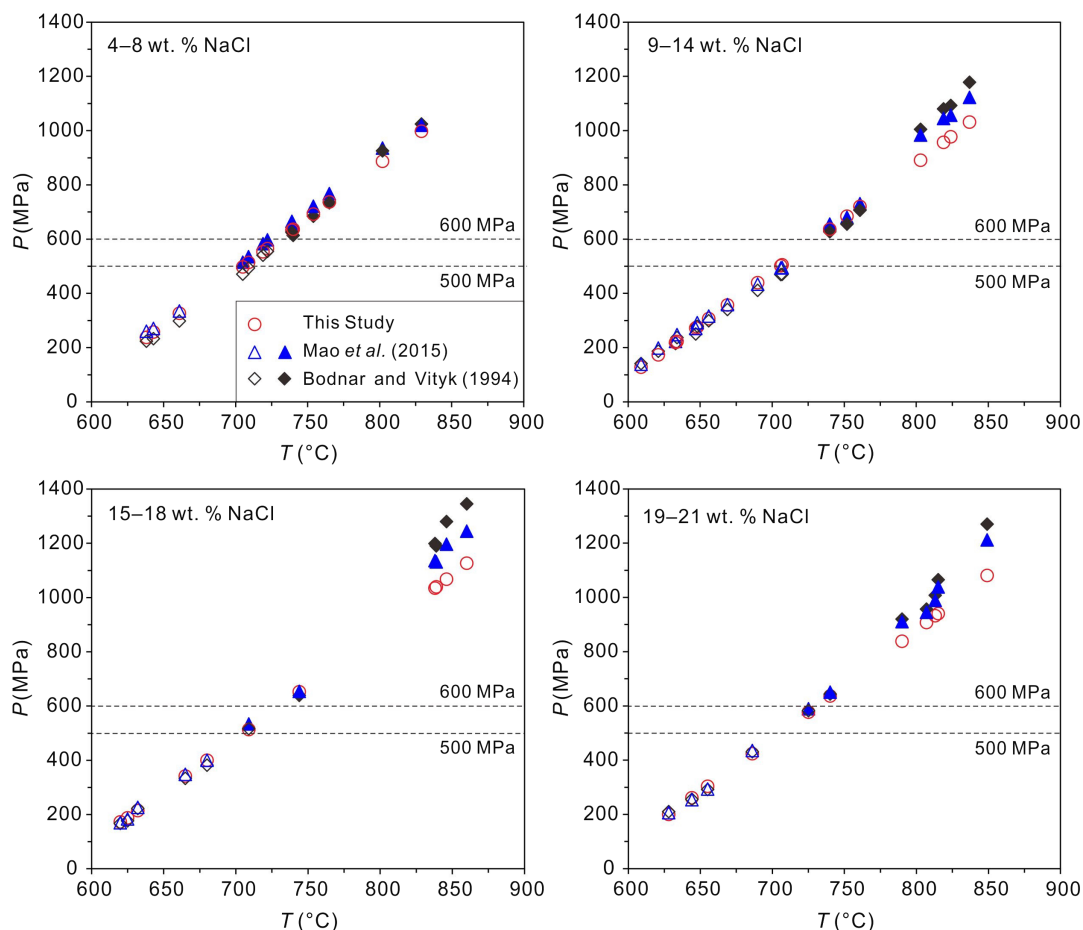


Figure 1 Comparisons of pressures at the measured α - β quartz phase transition temperatures (T_{tr} s). Plotted are the α - β quartz phase transition pressures (P_{tr} s), which were calculated from the equation of Li and Chou (2022). Other corresponding pressures were calculated from the isochores reported by Bodnar and Vityk (1994) and Mao *et al.* (2015) and shown by the open symbols below the 600 and 500 MPa isobars, respectively; the extrapolated pressures above the two isobars are shown by the solid symbols. All data are listed in Table S-1.

from those extrapolated from Bodnar and Vityk (1994) (Figure 2), consistent with the deviations of P_{tr} values above 600 MPa shown in Figure 1.

On the other hand, the isochores of Mao *et al.* (2015) were not only fitted with the accurate EoS of H_2O (IAPWS-95; Wagner and Pruß, 2002), but also calculated with the molar volume equation of the NaCl- H_2O PVTX model of Driesner (2007) that was developed with several thousand data points available from previous literature, including those derived from SFIs. This could cause the isochore data of Mao *et al.* (2015) to be closer to ours under low T_h and high salinity conditions, when compared with those of Bodnar and Vityk (1994) (Figures 1, 2c-f). Furthermore, the isochores of this study, particularly those with high salinities, agree excellently with those of Hurai (1988), which are shown in Figure 2 by the linear regression and extrapolated lines based on the data listed in Table S-2 and shown by the black dots in Figure 2. The data in Table S-2 were derived from the listed data of Hurai (1988), which summarized previously available data, especially those from Haas (1976) for vapour-saturated liquids, Hilbert (1979) for densities of solutions containing up to 25 wt. % NaCl at 20–40 MPa, 200–400 °C, and Gehrig (1980) for densities of solutions containing up to 20 wt. % NaCl at 10–300 MPa, 200–600 °C, covering 100–500 °C, ≤ 500 MPa and T_{hs} of 83–325 °C. The PVT data of Hilbert (1979) and Gehrig (1980) were collected with volume-calibrated pressure vessels under specified P - T conditions. Representative data of Gehrig (1980) for 20 wt. % NaCl solution are shown in

Figure S-4a as an example, to show these data were excellently presented by Hurai (1988). These features support the reliability of our isochores, implying that the isochores under our experimental conditions are approximately linear in P - T space and that the applicability of Equation 1 can be extended to the aqueous solutions containing 25 wt. % NaCl. Moreover, our isochores agree very well with those calculated from the density data reported by Pitzer *et al.* (1984) and Majer *et al.* (1988) within their rather limited applicable P - T areas (*i.e.* <100 or 40 MPa and <350 °C shown in Figure S-4b,c). However, deviations occur when extrapolating the isochores calculated from their data to higher P - T conditions, possibly due to the small curvature of their isochores, which are not suitable for linear extrapolations. Additionally, the PVTX models of Mantegazzi *et al.* (2013) and Fowler and Sherman (2020) are not considered for comparison here, as their isochores are only suitable for NaCl- H_2O solutions with densities (primarily >1.0 kg/cm³) much higher than those in this study.

Application of the isochores in the deep Earth setting. As discussed above, our isochores of the NaCl- H_2O solutions are more reliable under conditions above >600 MPa. Therefore, our isochores are expected to provide better applications in lower crustal conditions. For example, the isochores of fluid inclusions in metamorphic rocks are commonly used to determine the peak metamorphic conditions in the deep Earth setting such as a subduction zone (*e.g.*, Kawamoto *et al.*, 2018). The western Coast Mountains of British Columbia, Canada formed during terrane

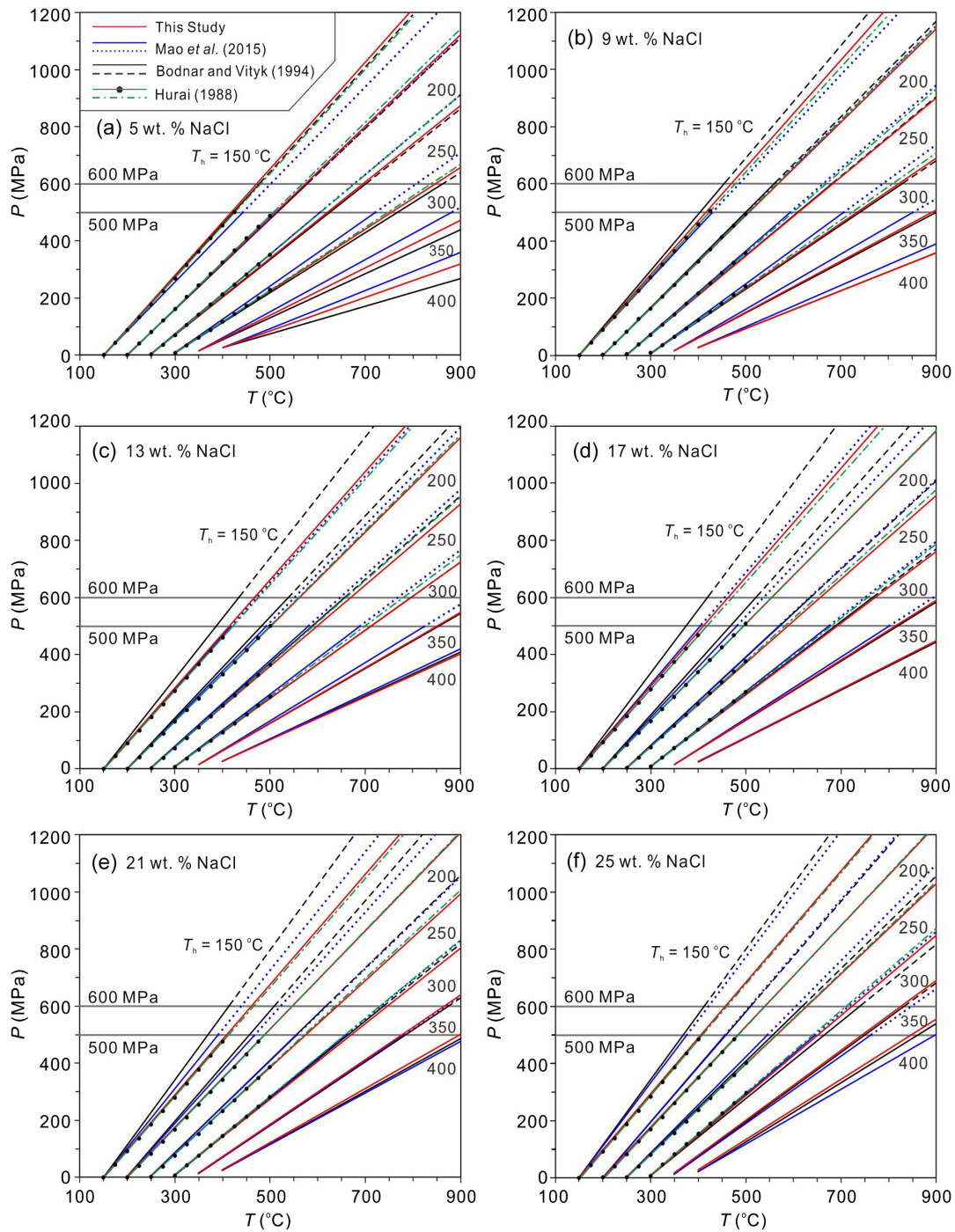


Figure 2 Comparisons of NaCl-H₂O isochores derived from our experimental data (red lines), those from [Bodnar and Vityk \(1994\)](#); black solid lines with dashed extrapolations above 600 MPa) and [Mao et al. \(2015\)](#); blue lines with dotted extrapolations above 500 MPa), and isochores (green lines with dash-dotted extrapolations above 500 MPa and 500 °C) linearly fitted with data from [Hurai \(1988\)](#); black circles). The homogenisation temperatures (T_h s) are marked.

accretion in the Jurassic and Cretaceous Periods ([Wolf et al., 2010](#)). The metamorphic framework in Prince Rupert of the Coast Mountains comprises schist, gneiss, and migmatite, displaying progressive regional metamorphism. In this area, the fluid inclusions in quartz contain 25 wt. % NaCl, and the observed homogenisation T_s (to liquid) were between 95 and 165 °C ([Hurai, 1989](#)). As shown in [Figure 3](#), the P - T field defined by the isochores of these fluid inclusions based on the models of [Bodnar and Vityk \(1994\)](#) and [Mao et al. \(2015\)](#) does not match the metamorphic P - T path derived from associated mineral assemblages ([Crawford et al., 1987](#)). However, the P - T field

derived from our isochore model matches perfectly well with the metamorphic P - T path.

The isochores of this study can also be used to infer the formation P - T conditions of melt inclusions (MIs) in plutonic rocks. A fluid subsystem inside MIs usually belongs to the NaCl-H₂O system, existing as a shrinkage bubble. Its isochores are commonly used to estimate the MI entrapment pressure ([Hurai et al., 2015](#)). By this method, the overestimation of the pressure values could be avoided, if our NaCl-H₂O isochores rather than those extrapolated from [Bodnar and Vityk \(1994\)](#) and [Mao et al. \(2015\)](#) were used.

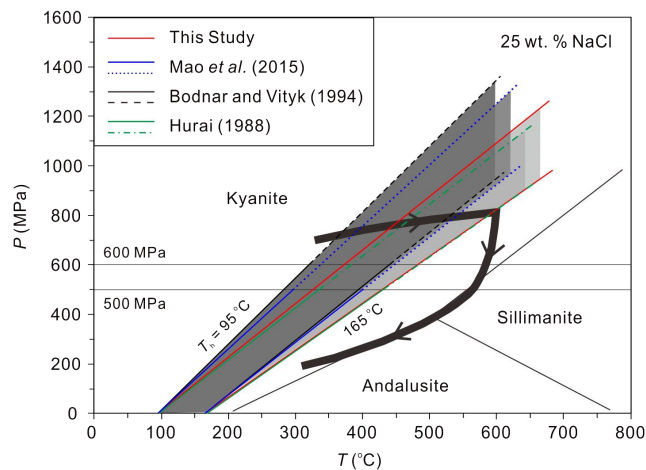


Figure 3 Application of NaCl-H₂O isochores obtained in this study for the determination of the peak metamorphic conditions in Prince Rupert of the Coast Mountain, British Columbia, Canada. Hurai (1989) reported that the fluid inclusions (FIs) in quartz in this metamorphic belt have salinities of ~25 wt. % NaCl with T_h s (to L) of 95–165 °C. The P - T fields (four shaded areas) defined by the FI isochores from Hurai (1988), Mao *et al.* (2015) and Bodnar and Vityk (1994), and this study are compared with the metamorphic P - T path (thick line with arrows) derived from associated mineral assemblages (Crawford *et al.*, 1987). The lines and symbols are the same as those in Figure 2.

Conclusions

The isochores of NaCl-H₂O solutions with salinities of up to 21 wt. % NaCl (applicable up to 25 wt. % NaCl), measured in the HDAC experiments by using the re-fitted α - β quartz P - T boundary of Li and Chou (2022), were extended to ~900 °C and ~1.2 GPa.

At pressures above 600 MPa, our isochores are considered to be reliable and accurate relative to previous ones and their extrapolations, particularly those derived from analyses of SFIs. Therefore, our isochores are more suitable to be applied for the interpretations of geological processes involving NaCl-H₂O fluids in the lower crust.

Our experiments also suggest a fast method for the accurate measurement of isochores of geologically important saline solutions with solutes of LiCl, NaCl, KCl, CaCl₂, *etc.*, and their mixtures by using HDAC and the newly calibrated α - β quartz P - T boundary of Li and Chou (2022) under wide P - T conditions.

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Additional Information

Supplementary Information accompanies this letter at <https://www.geochemicalperspectivesletters.org/article2429>.



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