ARTICLE

DOI: 10.1590/2317-488920192018090

BJGEO
Brazilian Journal of Geology

Calibration of high-temperature furnace assemblies for experiments between 200 and 600 MPa with end-loaded piston-cylinder apparatuses

Silvio Roberto Farias Vlach[1]*, Andrés Fabián Salazar-Naranjo[1], Johan Santiago Torres-Corredor[1], Paulo Rafael de Carvalho[1], Guilherme Mallmann[2]

Abstract

We present pressure calibration results for piston-cylinder ¾" and 1" high temperature furnace assemblies in the 200–600 MPa range aiming to expand its applicability to simulate upper crustal conditions. The furnace assemblies were made up by crushable MgO, graphite heater, pyrex glass spacer, and NaCl sleeve. Twenty-one experiments were carried out over a range of pressures (200–600 MPa) and temperatures (870–945°C) to determine the liquidus curve of NaCl. The obtained curve slope fits that given by Siewert et al. (1998), but offsets by ~ -55 MPa, implying the need of a constant P upward correction in the 300–600 MPa and ca. 300-400 MPa pressure ranges for the 3/4" and 1" assemblies, respectively. However, if the NaCl melting points (918–920°C) obtained by Bohlen (1984) at 500 MPa are used as reference, no pressure correction is needed. Larger offsets (to -120 MPa) were obtained under pressure conditions around and lower than 300 MPa with the 1" assembly. Our results confirm the applicability of Bristol-type end-loaded piston-cylinders coupled with appropriate furnace assemblies in assessing relatively low-pressure conditions (down to ~ 200 MPa, ~ 7 km depth) found in the Earth's upper crust, where important magmatic, hydrothermal and metamorphic processes occur.

KEYWORDS: End-loaded piston-cylinder apparatus; low-pressure calibration; NaCl melting method; experimental petrology and geochemistry.

INTRODUCTION

The piston-cylinder press is an apparatus developed by Boyd & England (1960) to simulate P-T conditions in the Earth's interior. Its original design allowed high-temperature experiments in the pressure range between 0.5 and 4.0 GPa (5–40 Kbar), covering lower crust down to upper mantle conditions. The apparatus has been the work-horse of experimental petrology laboratories worldwide in studies of phase equilibria, melting and crystallization (*e.g.*, Holloway & Wood 1988, Young 2003). Due to issues with internal friction and experimental assembly (solid media) reliability, however, the piston-cylinder press has rarely been used to simulate pressure conditions lower than 500 MPa, and both the internally or externally heated pressure vessels have been preferred for (see Holloway & Wood 1988, for descriptions and applicability of the available experimental devices).

Nevertheless, there are some inherent advantages in using a piston-cylinder apparatus over internally/externally heated pressure vessels, such as faster heating and cooling rates, easier maintenance and superior safety. Thus, some recent efforts have been made to develop and optimize piston-cylinder furnace assemblies for low-pressure environments. For instance, Baker (2004) calibrated a ¾" (19.1 mm) crushable alumina-pyrex glass-NaCl assembly in the 400–500 MPa range. This assembly was later modified by Moore *et al.* (2008), who replaced crushable alumina by crushable MgO and used both massive and powdered pyrex glass to carry out experiments down to 300 MPa. Masotta *et al.* (2012) designed new ¾" and 1" (19–25 mm) furnace assemblies using similar solid media components to expand the pressure range accessible with the non-end loaded QUICKpress (Depths of the Earth Co, USA) piston-cylinder apparatus down to 150 MPa. Given the great interest in the development of low-pressure (200–400 MPa) petrologic process simulations, as well as mineral synthesis, we work on the calibration of the available end-loaded piston-cylinder apparatus for such conditions.

Several methods based on well-constrained natural or synthetic phase relations and/or melting reactions can be used to calibrate high-temperature piston-cylinder assemblies at medium- to high-pressure conditions (*e.g.*, Boettcher & Wyllie 1968, Johannes *et al.* 1971, McDade *et al.* 2002). At relatively low-pressures (*ca.* ≤ 700 MPa) and temperatures up to 1,100°C, the most commonly used methods are based on the progressive increase of the melting temperature of halide salts (most commonly NaCl), and/or of the H_2O solubility in natural or synthetic glasses (*e.g.*, Bohlen 1984, Baker 2004, Moore *et al.* 2008). The melting methods have the advantage of not requiring chemical

[1]Universidade de São Paulo – São Paulo (SP), Brazil.
E-mails: srfvlach@usp.br, andres.salazar@usp.br, ssantiago_93@hotmail.com, paulo.rafael.carvalho@usp.br

[2]Australian National University – Canberra, Australia.
E-mail: guil.mallmann@anu.edu.au

*Corresponding author

analysis of run products, but the melting reaction needs to be bracketed by a large number of experiments.

In this contribution, we report a pressure calibration using the NaCl melting method for ¾" and 1" furnace assemblies carried out with the 200 tons Bristol-type end-loaded piston-cylinder apparatus of the Experimental Petrology and Geochemistry Laboratory at the GeoAnalitica core facility, University of São Paulo (see also Mallmann *et al.* 2014). Our results demonstrate the feasibility of high temperature experiments at pressures as low as 200 MPa with uncertainties arguably in the order of 25 MPa, therefore allowing simulation of geochemical and petrological natural processes, as well as to perform mineral synthesis at upper crustal conditions.

MATERIALS AND METHODS

Pressure calibration experiments were performed with an end-loaded Bristol-type piston-cylinder apparatus (Fig. 1) using two different experimental assemblies. For the pressure range between 400 and 600 MPa, a ¾" (19.1 mm) pressure plate was used with a furnace assembly composed of crushable MgO, graphite, pyrex (B-silicate) glass and NaCl (Fig. 2). This furnace assembly is almost frictionless (McDade *et al.* 2002), particularly if powdered pyrex glass is used at the internal bottom of the graphite cylinder, below the MgO rod, as done. For the 300–400 MPa pressure range, a variant of the furnace assembly designed by Moore *et al.* (2008) was employed with both ¾" and 1" (25 mm) pressure plates (Fig. 2), while experiments in the 200–300 MPa pressure range were performed solely with the new 1" low-T (LT) assembly and pressure plate.

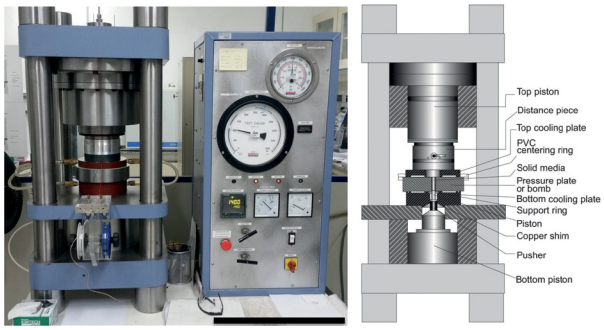

Figure 1. The Bristol-type end-loaded piston-cylinder (200 tons) in operation at the Experimental Petrology and Geochemistry Lab from the GeoAnalitica core facility, University of São Paulo (left), with the corresponding technical drawn identifying its main core components (right). Black bar in the left image measures 48 cm.

1: pressure plates; 2: steel plugs; 3: pyrex glass insulators; 4: NaCl sleeves and graphite lids; 5: pyrex glass spacers; 6: graphite heaters and lids; 7: upper and lower crushable MgO rods; 8: weld-closed Au sample capsule; 9: thermocouple probe insulator; 10: Pb foil.

Figure 2. The ¾" (from Bristol, left) and the new 1" (from Depths of the Earth Co, right) pressure plates and the assembly cell components used in our experiments. Note the Tungsten Carbide core in the ¾" plate, designed for medium- to high-pressure (500–1,500 MPa) conditions. See also Figure 3.

The pressure plate for the 1" experiments and furnace assembly components for all experiments (Fig. 2) were provided by the Depths of the Earth Co (USA), except for the NaCl sleeves, which were pressed in-house to *ca.* 1,500 psi (pounds per square inch) from reagent-grade NaCl powder, previously dried at 110°C by 24 h, using a salt cell die and a 25 ton Carver press. The pressed sleeves were recrystallized at 450°C for 4 h in a box furnace under environmental atmosphere in preparation for the low-pressure experiments.

The nominal hydraulic (oil) pressure in the Bristol piston-cylinder press is measured with an EN 839-1 precision gauge (SM Gauge Co, UK) whose minor subdivision is 20 psi (≈ 0.14 MPa, *cf.* Fig. 1), which gives a reading precision close to 10 psi. According to the press manufacturer, the minimum recommended reliable oil pressure reading should be about 600 psi. The temperature is routinely set up and monitored using a B-type thermocouple ($Pt_{94}Rh_{06}$-$Pt_{70}Rh_{30}$) probe controlled by a 2404 model Eurotherm PID device.

In all experiments reported in this study, the end-load pressure applied to the press, the temperature of the pressure plate refrigerating water flow and the room temperature were maintained at 4,200 ± 200 psi, 20 ± 1°C and 21 ± 2°C, respectively.

As a first step, we carried out a temperature calibration using the melting point of Au (1,064°C at atmospheric pressure). Standard ¾" furnace assemblies were prepared, substituting however the conventional noble metal sample capsules (see Figs. 2 and 3) for alumina rods with equivalent dimensions (3 mm diameter, 7 mm length), with *ca.* 1 mm diameter and 1 mm height centralized cavities in their lower and upper ends, partially filled in with sub-millimetric Au (99.99%) chips and covered with 3 mm diameter thin disks made of alumina. This arrangement allows setting a minimum nominal pressure (» 250 psi) on the experimental assembly to warrant good electric flow, needed for heating the graphite furnace, without transmitting such pressure to the Au chips. In the final mounted assembly cells, the alumina rods locate exactly as represented for sample capsules in Figure 3.

In each experiment, temperature was increased automatically at a rate of 5°C/min to 1,040°C and then manually at 0.5°C/min to the final desired value. The experiment was held under these conditions for 30 min, followed by quenching to room temperature at a cooling rate of ~ 30°C/sec. The recovered charges were then inspected under a stereomicroscope for melting evidence (Fig. 4A).

Pressure calibrations were carried using the NaCl *liquidus* temperature curve as a function of pressure given by Siewert *et al.* (1998), based on experimental data and their

best adjustment to the Simon's equation (Simon 1953). Our experimental procedures were similar to those described by Baker (2004) and Masotta *et al.* (2012).

Dried (110°C, 24 h) reagent-grade NaCl powder and a small (0.3–0.5 mm diameter) Pt sphere were placed inside a 3 mm diameter Au tube welded at the base. The Pt sphere was placed near the top, and the whole assembly dried for 2h before weld-closed. The capsule was then loaded into the previously dried mounted furnace assembly components. All capsules were mounted following the *trash-can* type design. In the assemblies, the Au capsules were armored by a thin layer of dry pyrophyllite powder (*cf.* Fig. 3).

The final assemblies were externally covered with a thin Pb foil and mounted in ¾" or 1" pressure plates as the case, the plates internal assembly-containing walls being previously lubricated with PTFE-based dry spray. A two-bore alumina tubing was used as electric insulator between thermocouple wires; the free space between wires and insulator walls was filled in with MgO powder, by using a lab vacuum machine. This procedure helps preventing thermocouple wires/insulator failure (Moore *et al.* 2008).

In all runs the assembly was pressurized up to 10% above the selected nominal pressure at room temperature. The temperature was automatically increased up to 750°C and then manually and slowly increased up to the final desired value. Pressure was constantly adjusted (typically upwards) during heating to maintain the desired nominal pressure of the experiment constant. Thus, the adopted approach is somewhat similar to the "hot piston-in" technique (*e.g.*, Johannes *et al.* 1971). After equilibration for ~30 min at the chosen pressure and temperature, the experiment was quenched by cutting off the power supply. The recovered experimental charges were slowly sanded longitudinally with conventional 300 grit sand papers and continuously examined under a binocular microscope until the position of the Pt sphere could be identified. NaCl melting is confirmed by the movement of the Pt sphere (which is much denser than NaCl liquid) from the top to the bottom of the capsule (Fig. 4B).

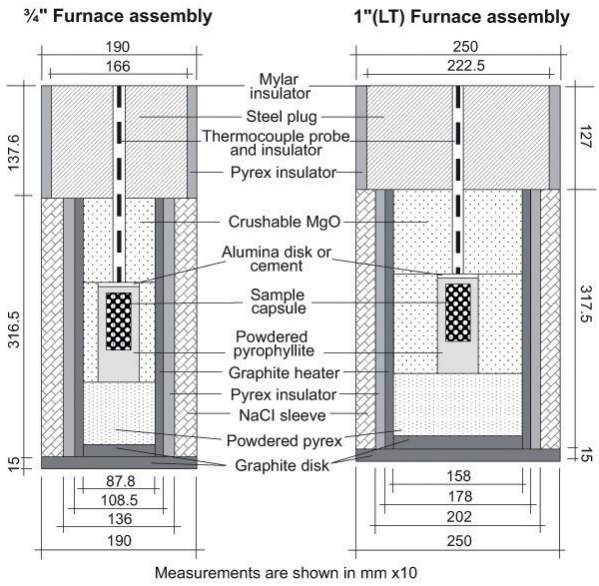

Figure 3. Schematic cross sections of the ¾" (left) and 1" (Low Temperature, right) piston-cylinder furnace assemblies (solid media) used in the present study. According to Moore *et al.* (2008) and Masotta *et al.* (2012), respectively, with minor modifications. See actual constituents in Figure 2.

RESULTS

Temperature calibration

Temperature was calibrated using the melting point of Au (1,064°C at atmospheric pressure). Four experiments were carried out at temperatures of 1,061°C, 1,064°C (x2) and 1,067°C. Melting was confirmed in run products at 1,067°C (complete, *cf.* Fig. 4A) and 1,064°C (moderate to complete), while no Au melting was evident in the 1,061°C experiment. These results suggest that temperature readings deviations are < 2–3°C under the applied experimental condition.

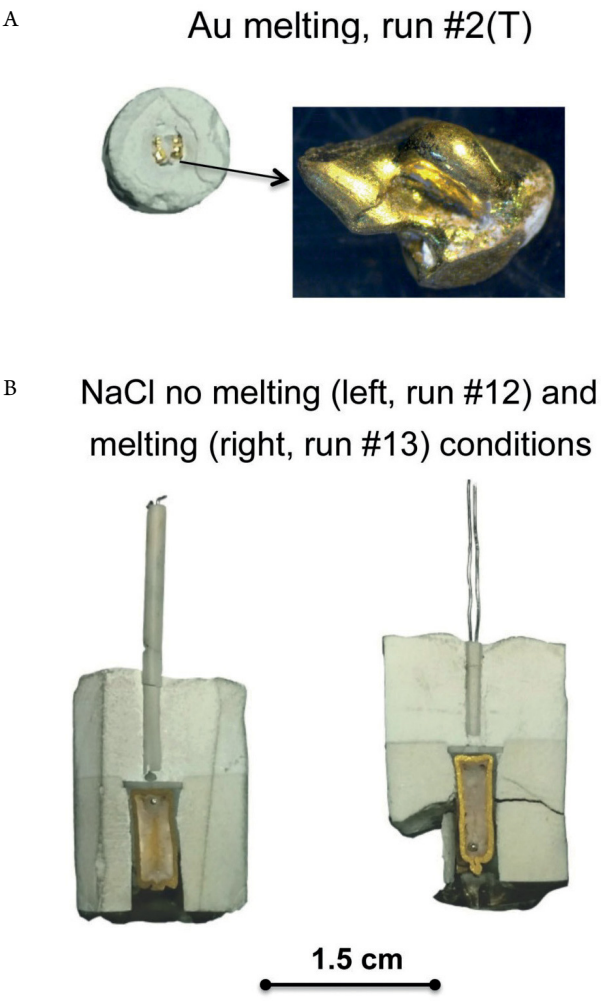

Figure 4. (A) Top end of the alumina rod with melted Au and stereomicroscope optical image illustrating the melting of the inserted Au chips in one of the experiments carried out under 1,064°C and atmospheric pressure. (B) Photographs showing products obtained through the NaCl melting experiments after partial sanding of the cell assembly. Note the positions of the Pt spheres in the top (left) and bottom (right) of the NaCl filled in Au capsules, demonstrating no melting and melting situations, respectively. Thermocouple wires, welded at their lower-ends, and respective two-bore alumina insulators point to the assembly top.

While temperature gradients can be relatively large in these types of assemblies, due to different solid media constituents, their geometry, thermocouple position, as well as specific P-T conditions (*e.g.*, Watson *et al.* 2002, Schilling & Wunder 2004), in the experiments reported herein Au melting occurred at both ends of the alumina rod, which were spaced by *ca.* 5mm. As this distance is similar to the internal height of the noble metal capsule in real experiments, the results point to no significant temperature gradient in well-centered experimental capsules with 3 mm external diameter and up to 6 mm external length at low pressures using our furnace assembly arrangements.

Pressure calibration

Twenty-one runs were carried out for pressure calibration. Their results (NaCl melting and no-melting conditions at the given P and T values) are summarized in Table 1 and compared with the NaCl melting curve of Siewert *et al.* (1998) within the range of interest in Figure 5. We did not carry out experiments below the nominal pressure of 230 MPa due to limitations imposed by the piston-cylinder manufacturer. Of note, the results obtained around 400 MPa with both the ¾" and 1" assemblies are in strong agreement.

Table 1. Experimental products (NaCl melted and no melted conditions) obtained for the experimented temperatures and pressures. P_{Nom} = nominal pressure, $P_{Siewert}$ = expected melting pressure according the Siewert *et al.* (1998) liquidus curve for the given temperature (T). Runs #1 to #4 are the NaCl melting values obtained by Bohlen (1984), using non end-loaded piston-cylinder (PCB), internal-heated vessel (IHB) and low-temperature assembly (LT).

Run#	T °(C)	P_{Nom} (MPa)	$P_{Siewert}$ (MPa)	Melt	No melt	Furnace assembly
1	915	495.0	526.5		X	¾"
2	925	500.0	578.5	X		¾"
3	945	605.0	686.5	X		¾"
4	895	400.0	425.5		X	¾"
5	935	602.0	631.5		X	¾"
6	905	400.0	475.5	X		¾"
7	890	402.0	401.0		X	¾"
8	890	315.0	401.0	X		¾"
9	880	320.0	353.0		X	¾"
10	905	390.0	475.5	X		1" (LT)
11	900	340.0	450.5	X		1" (LT)
12	895	403.0	425.5		X	1" (LT)
13	903	390.0	465.5	X		1" (LT)
14	875	295.0	329.5		X	1" (LT)
15	885	338.0	377.0		X	1" (LT)
16	885	230.0	377.0	X		1" (LT)
17	885	275.0	401.0	X		1" (LT)
18	895	301.0	425.5	X		1" (LT)
19	865	230.0	283.5		X	1" (LT)
20	870	230.0	305.5		X	1" (LT)
21	875	230.0	329.5		X	1" (LT)
#1	915	500.0	526.5		X	PCB
#2	920	500.0	450.5	X		PCB
#3	918	500.0	542.0		X	IHB
#4	922	500.0	562.5	X		IHB

We also plotted in Figure 5 the maximum errors envelope, computed from the deviations for the NaCl melting temperature under atmosphere conditions and the fitting parameters of the Simon's equation as given by Siewert *et al.* (1998), which consider a ± 5°C temperature deviation in each of their individual measurements. The average NaCl melting point values of Bohlen (1984) at 500 MPa, 920°C and 917.5°C (*cf.* Tab. 1), obtained with internal-heated vessel and non end-loaded piston-cylinder press apparatuses respectively, were also plotted in the diagram. These values are significantly higher than the temperature (910°C) given by the model of Siewert *et al.* (1998), but still within the estimated error envelope.

Our results at 500°C strongly match those from Bohlen (1984). However, when they are compared with the curve modeled by Siewert *et al.* (1998), it can be observed that, for a given pressure gauge reading, the obtained NaCl melting temperatures are always *higher than* those predicted, although, except for the low-pressure range investigated with the 1' assembly, within the error envelope. The best fit of our data has almost a same slope down to *ca.* 300 MPa. This implies a higher pressure over the sample for all investigated cases, an intriguing finding, as it is opposite to the conventional situations, in which actual pressures over samples are expected to be *lower* than nominal pressures (*e.g.*, McDade *et al.* 2002, Masotta *et al.* 2012). The reasons for that are not well understood; most probably they are related to inherent instrumental features (*e.g.*, gauge calibration by the manufacturer) and to the available reference NaCl melting values, although solid media characteristics, NaCl purity grade and the method chosen for incrementing the pressure ("piston-in" *vs.* "piston-out", *cf.* discussion in Johannes *et al.* 1971 and McDade *et al.* 2002) might also play some role.

Taking the midpoint between our melting and non melting temperatures (Tab. 1) as representative of the NaCl melting

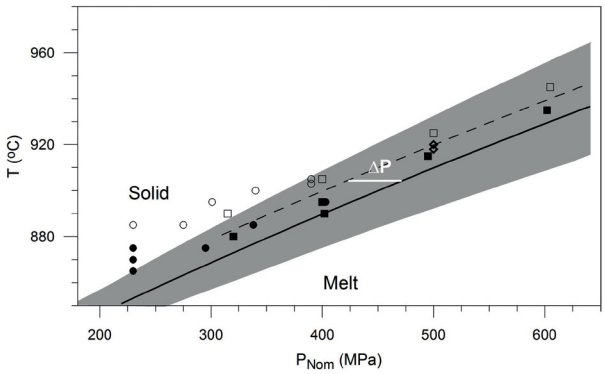

Figure 5. Temperature vs. nominal pressure (P_{Nom}) diagram, illustrating the NaCl melting curve of Siewert et al. (1998) in heavy black and its maximum error envelope (gray area). Open diamonds indicate NaCl melting points obtained by Bohlen (1984) at 500 MPa with piston-cylinder and internal-heated vessel apparatuses. Squares and circles symbolize ¾" and 1" experiments, open and filled symbols in each case indicate melt and no melt results, respectively. Dashed curve correspond to the Siewert et al. (1998) melting curve fitted according to the Simon's equation, considering our ¾" results in the 300–600 MPa pressure range. White bar illustrates the almost constant pressure difference between these melting curves for a given temperature. See text for discussion.

point at a given nominal pressure and their departure from the Siewert *et al.* (1998) melting curve for the whole examined 200–600 MPa pressure range, the best fit relating real and nominal pressure values is given by the second order polynomial (Eq. 1):

$$P_{Siewert} = 328\ (\pm 46) - 0.19\ (\pm 0.23) * P_{Nom} + 0.0012\ (\pm 0.0003) * P^2_{Nom} \quad (1)$$

in which:
$P_{Siewert}$ and P_{Nom} are the pressures calculated through the reference melting curve of Siewert *et al.* (1998) and gauge readings (nominal), respectively, for a given temperature (Fig. 6).

Pressure deviations in this diagram were considered taking a deviation of ± 25 MPa for $P_{Siewert}$, as graphically estimated from the temperature measurement deviations (± 5°C) adopted by these authors, and 0.1 MPa for our P_{Nom} readings, a value almost negligible. Albeit for the high determination coefficient for this fit ($r^2 = 0.99$), which arises from the relatively large individual uncertainties on $P_{Siewert}$ of our individual data points, better linear fits are obtained when two independent datasets are considered separately, as follows:

- results for the ¾ ± cell in the 300–600 MPa pressure range are best fitted by Equation 2:

$$P_{Siewert}\ (MPa) = 1.00\ (\pm 0.03) * P_{Nom}\ (MPa) + 54\ (\pm 13) \quad (2)$$

- results for the 1" cell in the *ca.* 200–400 pressure range are best adjusted by Equation 3:

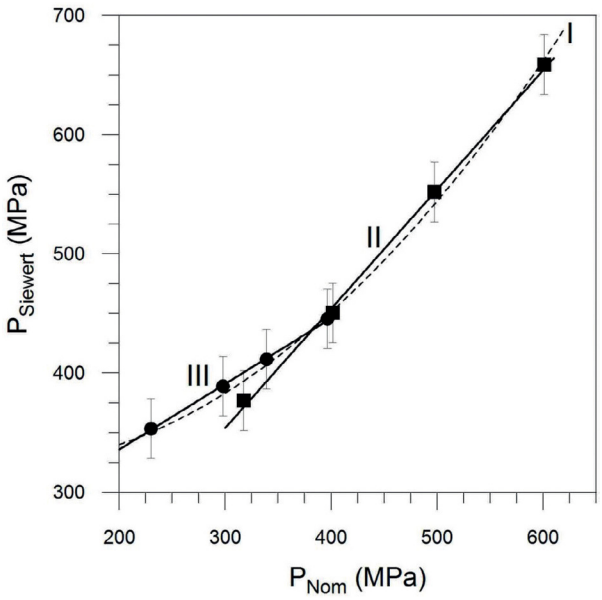

Figure 6. Comparison between the nominal, gauge reading, pressure (P_{Nom}) and that predicted by the calibration of Siewert et al. (1998), $P_{Siewert}$. P_{Nom} was taken at the middle point between the (P_{Nom}, T) experimental results. The best fit of all results for ¾" and 1" assemblies (I, dashed line) is given by $P_{Siewert} = 328\ (\pm 46) - 0.19\ (\pm 0.23) * P_{Nom} + 0.0012\ (\pm 0.003) * (P_{Nom})^2$, $r^2 = 0.99$. More accurate fits (solid lines, II and III) are obtained considering the results for each furnace assembly, as II (¾"): $P_{Siewert} = 1.00\ (\pm 0.01) * P_{Nom} + 54\ (\pm 13)$, $r^2 = 1.00$, and III (1"): $P_{Siewert} = 0.55\ (\pm 0.01) * P_{Nom} + 226\ (\pm 4)$, $r^2 = 1.00$. See text.

$$P_{Siewert}\ (MPa) = 0.55\ (\pm 0.01) * P_{Nom}\ (MPa) + 226\ (\pm 4) \quad (3)$$

Both these fits give determination coefficients close to 1.00. These results indicate that the ¾" assembly may be used at pressures down to 300 MPa with a pressure correction of *ca.* +55 MPa in the 300–600 MPa pressure range, as compared with the reference melting curve. Importantly, however, if we consider our results around 500 MPa and reference them to the NaCl melting points of Bohlen (1984), the emerging conclusion is that no pressure correction is needed. In the case of the 1" assembly, a somewhat similar correction in relation to the melting curve may be applied over the 300–400 MPa range without introducing significant errors, however a high correction (almost the double) must be applied in the lowest-pressure experimented range, between 200 and 300 MPa.

It is somewhat difficult to address precisely error estimates for the obtained pressure corrections. As pointed out previously, the reading errors are about 10 psi in our pressure gauge and the estimated precision in temperature readings about ± 2–3°C. As mentioned above, Siewert *et al.* (1998) estimate a ± 5°C deviation in their temperature measurements, while a somewhat minor deviation may be roughly inferred from the data presented by Bohlen (1984) for 500 MPa. If we consider ± 5°C temperature deviations as the main error source, a reference figure of about ± 25 MPa, as suggested by Masotta *et al.* (2002), for the corrected pressure uncertainties appears to be reasonable.

Comparisons with previous calibrations

Our results with the ¾" assembly are in strong agreement with those reported by Bohlen (1984) at 500 MPa and Baker (2004) at 400–500 MPa, based on the water solubility method in albite glasses and obtained with non end-loaded piston-cylinder presses. In fact, as shown in Figure 5, the best adjust of the Simon equation in the 300–600 MPa range suggests no significant correction needs if referenced to the NaCl melting points of Bohlen (1984), especially if his higher value, obtained with an internal-heated vessel and usually taken as more precise, is considered. On the other hand, an upward almost constant correction of about 55 MPa is needed if the NaCl melting curve of Siewert *et al.* (1998) is considered and it should be applied all over the pressure range experimented with the ¾" solid media. A similar conclusion was found by Moore *et al.* (2008) based on the water solubility in rhyolite glass and the empirical H_2O solubility model of Liu *et al.* (2005). The authors found a linear (1:1) correlation between predicted and measured H_2O contents within the 300–500 MPa range, pointing also to no pressure correction needs.

In the low-pressure range experimented with the new 1" LT furnace assembly (200–400 MPa), however, our results do not conform with those obtained by Masotta *et al.* (2012), which applied both the NaCl melting and the H_2O solubility in albite and rhyolite glass methods, based on the experimental data of Behrens *et al.* (2001) and Moore *et al.* (1998). The authors found the following equation, valid for the 150–500 MPa range (Eq. 4):

$$P_{Cr} (\pm 25) = -0.115*P_{Nom} + 78.23 \quad (4)$$

in which:

P_{Cr} = the needed P correction, all values given in MPa.

The major difference in our case is that nominal, gauge-reading, pressures are lower than actual pressure over the sample, as pointed out and discussed above.

FINAL REMARKS AND RECOMMENDATIONS

As emphasized by Masotta *et al.* (2012) among others, the NaCl melting calibration method may be effectively used for low pressure calibrations in the 800–1,100°C range of piston-cylinder apparatuses. In fact, Baker (2004) showed that actual pressure values obtained by this method strongly agree with independent calibrations obtained from H_2O solubility in albite and in silicic glasses. However, the available NaCl melting point data around 500 MPa reveal discrepancies (up to 8–10°C) and more accurate NaCl melting temperatures on the pressure range of interest would be desirable to allow further correction optimization.

In conclusion, relatively low pressure experiments, down to 200 MPa (*ca.* 7 km depth), and temperatures up to *ca.* 1,100°C may be routinely carried out with the Bristol piston-cylinder press using both the 1" low- and high-T assemblies and pressure plates currently available in the Experimental Petrology and Geochemistry Lab at GeoAnalítica, Universidade de São Paulo (USP). This expands the working pressure range of the facility to a significant extent, allowing experimental studies under conditions relevant to magmatic, hydrothermal and metamorphic processes in relatively shallow crust.

As a final remark, future studies applying the present calibrations should mention the reference NaCl melting temperature (*i.e.*, Bohlen 1984 or Siewert *et al.* 1998) that is being used. Importantly, we recommend that procedures for experimental cell preparation, compression, and heating described in the present study should be followed.

ACKNOWLEDGEMENTS

The authors thank Dr. Tracy Paul (Depths of the Earth Co) for help and suggestions, and FAPESP for financial support, which allowed commissioning of equipment (Grant 2010/05512-1 to G. Mallmann) and acquisition of consumables (Grant 2012/06082-6 to E. Ruberti) for the Laboratory of Experimental Petrology and Geochemistry at GeoAnalítica-USP. S. Vlach also acknowledges CNPq (Grant 309557/2014-1). A. Salazar-Naranjo benefits from a CNPq MsC Scholarship; J. Torres-Corredor and P. Carvalho from CAPES MsC Scholarships (Finance Code 001). Comments and suggestions of two anonymous reviewers were much appreciated.

ARTICLE INFORMATION

Manuscript ID: 20180090. Received on: 08/10/2018. Approved on: 02/22/2019.

S. V. designed the experimental set-ups and wrote the first draft of the manuscript. S. V., A. S.-N., J. T.-C. and P. C. performed the experiments. S. V. and A. S.-N. drew/obtained diagrams and pictures. G. M. gave suggestions on the experimental procedures and reviewed the manuscript.

Competing interests: The authors declare no competing interests.

REFERENCES

Baker D.R. 2004. Piston-cylinder calibration at 400 to 500 MPa: A comparison of using water solubility in albite melt and NaCl melting. *American Mineralogist*, **89**(10):1553-1556. https://doi.org/10.2138/am-2004-1026

Behrens H., Meyer M., Holtz F., Benne D., Nowak M. 2001. The effect of alkali ionic radius, temperature, and pressure on the solubility of water in $MAlSiO_4$ melts (M=Li,Na,K,Rb). *Chemical Geology*, **174**(1-3):275-289. http://dx.doi.org/10.1016%2FS0009-2541(00)00320-X

Boettcher A.L., Wyllie P.J. 1968. The quartz-coesite transition measured in the presence of a silicate liquid and calibration of piston-cylinder apparatus. *Contributions to Mineralogy and Petrology*, **17**(3):224-232. https://doi.org/10.1007/BF00388945

Bohlen S.R. 1984. Equilibria for precise pressure calibration and a frictionless furnace assembly for the piston-cylinder apparatus. *Neues Jahrbuch für Mineralogie Monatshefte*, **9**:404-412.

Boyd J.R., England J.L. 1960. Apparatus for phase-equilibrium measurements at pressures up to 50 kilobars and temperatures up to 1750°C. *Journal of Geophysical Research*, **65**(2):741-748. https://doi.org/10.1029/JZ065i002p00741

Holloway J.R., Wood B.J. 1988. *Simulating the Earth: experimental geochemistry*. Boston: Unwin Hyman Ltd., 196 p.

Johannes W., Bell P.M., Mao H.K., Boettcher A.L, Chipman D.W., Hays J.F., Newton R.C., Seifert F. 1971. An interlaboratory comparison of piston-cylinder pressure calibration using the albite-breakdown reaction. *Contributions to Mineralogy and Petrology*, **32**(1):24-38. https://doi.org/10.1007/BF00372231

Liu Y., Zhang Y., Behrens H. 2005. Solubility of H_2O in rhyolite melts at low pressures and a new empirical model for mixed H_2O-CO_2 solubility in rhyolitic glasses. *Journal of Volcanology and Geothermal Research*, **143**(1-3):219-235. http://dx.doi.org/10.1016/j.jvolgeores.2004.09.019

Mallmann G., Fonseca R.O.C., Silva A.B. 2014. An experimental study of the partitioning of trace elements between rutile and silicate melt as a function of oxygen fugacity. *Anais da Academia Brasileira de Ciências*, **86**(4):1609-1629. http://dx.doi.org/10.1590/0001-3765201420140014

Masotta M., Freda C., Paul T.A., Moore G.M., Gaeta P.S., Scarlato P., Troll V.R. 2012. Low pressure experiments in piston cylinder apparatus: Calibration of newly designed 25 mm furnace assemblies to P=150 MPa. *Chemical Geology*, **312-313**:74-79. http://dx.doi.org/10.1016/j.chemgeo.2012.04.011

McDade P., Wood B.J., Westrenen W.V., Brooker R., Gudmundsson G., Soulard H., Najorka J., Blundy J. 2002. Pressure corrections for a selection of piston-cylinder cell assemblies. *Mineralogical Magazine*, **66**(6):1021-1028. https://doi.org/10.1180/0026461026660074

Moore G., Roggensack K., Klonowski S. 2008. A low-pressure-high-temperature technique for the piston-cylinder. *American Mineralogist*, **93**(1):48-52. https://doi.org/10.2138/am.2008.2618

Moore G., Vennemann T., Carmichael I.S.E. 1998. An empirical model for the solutility of H_2O in magmas to 3 kbars. *American Mineralogist*, **83**(1):36-42. https://doi.org/10.2138/am-1998-0103

Schilling F., Wunder B. 2004. Temperature distribution in piston-cylinder assemblies: Numerical simulations and laboratory experiments. *European Journal of Mineralogy*, **16**(1):7-14. http://dx.doi.org/10.1127/0935-1221/2004/0016-0007

Siewert R., Büttner H., Rosenhauer M. 1998. Experimental investigation of thermodynamic melting properties in the system NaCl-KCl at pressures up to 7000 bar. *Neues Jahrbuch für Mineralogie Monatshefte*, **172**(2-3):259-278. http://dx.doi.org/10.1127/njma/172/1998/259

Simon F. 1953. The melting of iron at high pressures. *Nature*, **172**:746-747. https://doi.org/10.1038/172746a0

Watson E.B., Wark D.A., Price J.D., Van Orman J.A. 2002. Mapping the thermal structure of solid-media pressure assemblies. *Contributions to Mineralogy and Petrology*, **142**(6):640-652. http://dx.doi.org/10.1007/s00410-001-0327-4

Young D.A. 2003. *Mind over magma:* the story of igneous petrology. Princeton: Princeton University Press, 686 p.