American Mineralogist, Volume 67, pages 175–179, 1982

Temperature calibration of a new rapid quench vessel

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Abstract

A 61 cm long rapid quench vessel of new design has been calibrated for internal temperature gradients at elevated temperature and pressure with a water pressure medium. Calibrations were done over 6.8 cm of the hot end of the vessel at angles of inclination (measured from the horizontal) of −10°, −5°, −2.5°, 0°, +2.5°, +5°, and +10° at temperatures of 300, 450, 600, and 750°C and 2 kbar = P_{tot}. Results show a water pressure medium is satisfactory at all temperatures if the hot end is raised +5°.

A similar set of measurements were performed on a standard 30.5 cm vessel with a filler rod. The results show the temperature gradient to be essentially inclination independent from −10° to +10° although the temperature correction is inclination dependent.

Introduction

Temperature gradients within cold seal pressure vessels are of constant concern to experimental petrologists. Boettcher and Kerrick (1971) have shown that a short (20.3 cm) pressure vessel with a 17 cm bore will have acceptable gradients if care is taken in orientation of the vessel and a filler rod is used. The rapid quench pressure vessel suggested by Greenwood (verbal communication) and developed by Wellman (1970) is much more sensitive to orientation effects and pressure media. This vessel consists of two rod vessels connected by high pressure cone seals with a small filler rod or none at all. One rod vessel is heated and the other cooled. The combination is much longer than the standard rod vessel. The sample temperature is dropped quickly at the end of an experimental run by tipping the vessel so that the sample capsule slides down the open bore into the water-cooled end.

Rudert *et al.* (1976) observed in the calibration of their double rod quench vessel that water was an unacceptable pressure medium when the vessel was horizontal because large (>10°C) gradients were observed within the capsule region.

Ludington (1978) showed gradients in the sample region could be diminished if the hot end of the vessel were tilted upward. The present study was initiated to determine the temperature gradient in a rapid quench vessel of different design from the one used by Rudert *et al.* (1976) and Ludington (1978).

Experimental procedure

Figure 1 shows the tipping vessel used in the calibrations. It consisted of a single rod of Rene 41 with a length of 61 cm (24″) a bore depth of 59 cm, a diameter of 3.2 cm (1.25″) and a bore diameter of 0.64 cm (0.25″). The vessel was heat treated for maximum strength after the method of Luth and Tuttle (1963). The thermocouple placed inside the vessel was inserted in a reentrant dummy charge capsule filled with quartz. The capsule was 3.8 cm long with a 2.9 cm bore depth and 0.16 cm bore diameter. A 1.3 cm filler rod with a 0.16 cm bore diameter for the thermocouple was positioned just above (to the right in Figure 1) the charge capsule to damp out some of the convection in the pressurizing medium. Capsule and filler rod fitted smoothly inside the vessel. The vessel was placed in a clamshell furnace (Lindberg Model 55031) with the well for the external thermocouple at 6 o'clock. Temperature control was achieved with a Love Instruments Model 71 solid state potentiometric controller. The opposite end of the vessel was cooled by water flowing through 0.9 cm copper tubing wrapped around approximately 11 cm of the vessel just above the cone seal. Metal foil between the vessel and cooling coil improved the thermal contact. Coolant temperature and ambient temperature were monitored and held constant at about 10 and 21°C, respectively. Coolant flow was constant at 1 l/min.

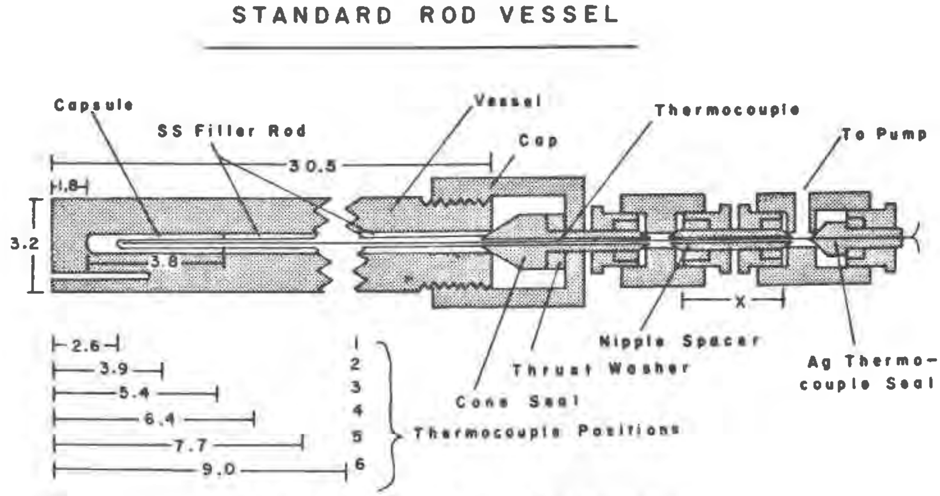

Fig. 1. Rapid quench vessel. Units are in cm.

Measurements were made with the vessel at inclinations of −10°, −5°, −2.5°, 0°, +2.5°, +5° and +10° from the horizontal. At positive angles the hot end of the vessel was raised above the cold end. For each angle, temperatures were measured at distances from the bottom of the bore of 0.8, 2.2, 3.5, 4.7, 5.7, and 6.8 cm. The thermocouple position was adjusted by means of successively longer spacers attached to the sealing cone (see distance x in Fig. 1). Calibrations were done at nominal temperatures of 300°C, 450°C, 600°C, and 750°C. A total of 336 measurments (7 angles × 6 spacers × 4 temperatures × 2 thermocouples) plus a number of rechecking measuements were made. All measurements were made at 2 kbar $P_{H_2O} = P_{tot}$. For each spacer the requisite 56 measurements were made before changing the spacer.

Thermocouples (type K), manufactured by Ther-

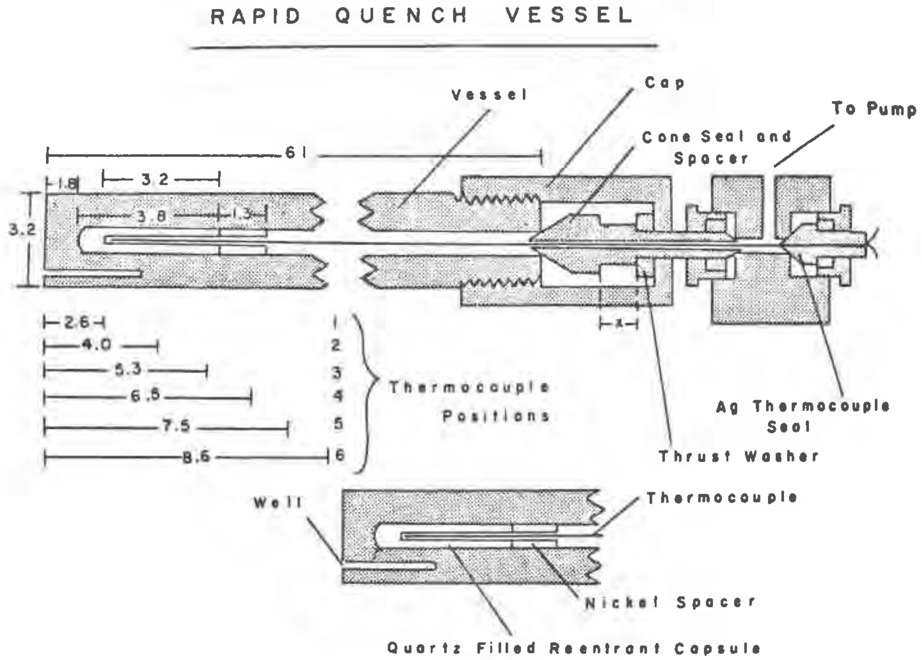

Fig. 2. Standard rod vessel. Units are in cm.

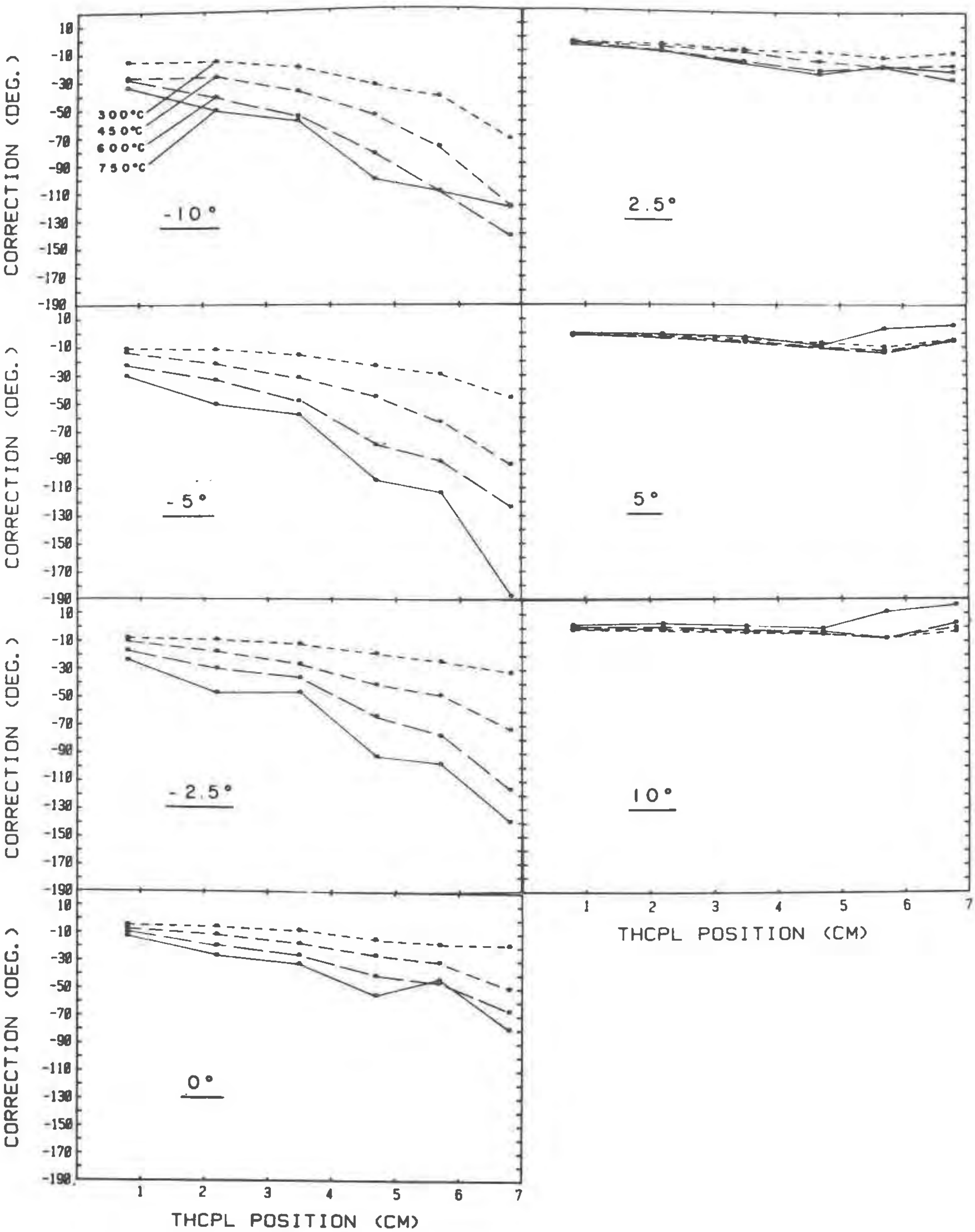

Fig. 3. Temperature differentials (°C) for the 61 cm (24″) rapid quench vessel at the angles of inclination indicated. Note: At +10°, the hot end of the vessel is raised 10° from the horizontal.

moelectric, were calibrated throughout the range of experimentation using the following melting point standards:

Tin	231.9°C
Lead	327.4°C
Antimony	630.5°C

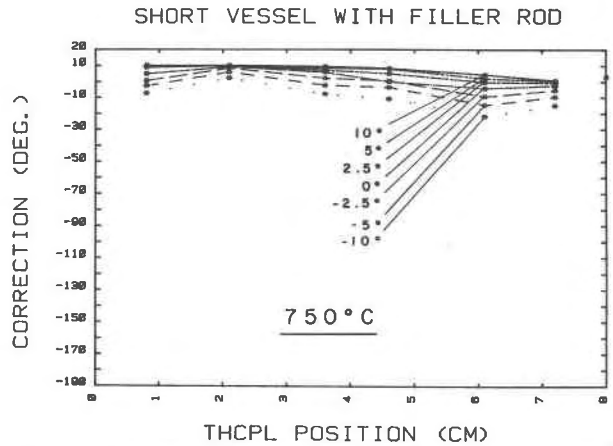

Fig. 4. Temperature differentials for the 30.5 cm (12″) rod vessel with full length filler rod at a nominal temperature of 750°C for the seven angles of inclination.

Sodium Chloride	801.0°C
Silver	960.8°C

Melting point standards were sealed in evacuated reentrant silica tubes to prevent oxidation. Latent heat of melting was sufficient in all cases to give good calibration. Reproducability was ±0.1 to 0.2°C for Pb, Zn, NaCl, and Ag over six measurements. Sb yielded somewhat poorer values with variations of 0.5° to 1.0°C over six measurements. The silver calibration standard was measured under an argon atmosphere to eliminate oxidation of the sheathing material used on the type K thermocouples. Temperature was measured with a Hewlett-Packard Digital voltmeter.

As a companion experiment, a cold seal pressure vessel 30.5 cm long (12″) 3.2 cm (1.25″) in diameter, with a 0.64 cm (0.25″) bore was also calibrated (Fig. 2). Its bore was filled by the capsule and filter rod. It was calibrated at a nominal temperature of 750°C at the same angles as above with the thermocouple positions of 0.8, 2.1, 3.6, 4.6, 6.1, and 7.2 cm from the bottom of the bore (see distance x in Fig. 2).

Observations and discussion

Figure 3 illustrates the correction which must be added to the well thermocouple at the indicated bore position for each of the seven angles of inclination. As the angle increases from −10° to +10°, corrections become smaller for all temperatures. At −10° and high temperature (750°C), convection made accurate temperature measurement impossible because temperatures oscillate over a range of ±10°C.

At negative angles (the hot end of the vessel down), temperature differentials of 70 to 180°C are noted throughout the investigated temperature range. The situation is still unfavorable at 0° inclination. A slight positive angle (2.5°) improves gradients to 15 to 25°C over 6.8 cm. At +10°, the differential decreases to about 15°C for temperatures up to 600°C and to about 20°C for a nominal temperature of 750°C over 6.8 cm. The conclusion is that disastrous gradients are present at lower angles and improve as the inclination angle is increased at all temperatures. Irregularities are observed at thermocouple positions greater than 5 cm for +5° and +10°. The reproduceable smooth rise in this portion of the calibration for 750°C relative to the other curves with increasing angle of inclination indicates a convective change between 600°C and 750°C in the open portion of the bore beyond 5.1 cm.

Below +5° differentials across even short capsules become large for all temperatures. For instance, if ±10°C were the maximum allowable differentials across a capsule: at +10° and +5°, a capsule at 750°C could have a maximum length of 4.7 cm; at +2.5°, a capsule at 750°C could have a maximum length of 3–4 cm; 0°, 2.5°, 5°, and 10° show no acceptable capsule lengths.

From another point of view, a capsule 3.5 cm long at 750°C will experience differentials of 2°C at +10°, 2°C at +5°, 10°C at +2.5°, 17°C at 0°, 20°C at −2.5°, 23°C at −5°, and 25°C at −10°. The authors would expect these general trends to apply in other systems with similar design, but with different absolute values of correction.

For comparison, the results for a shorter rod vessel (30.5 cm) with a full length filler rod are shown in Figure 4. Measurements were made only at a nominal temperature of 750°C. Orientation, although important, has much less effect than that observed for the rapid quench vessel. At 0° to +10° the differential is only about 10°C over the 7.2 cm examined. The capsule is 3.8 cm long and it will experience about a 10°C differential at all positive angles. The gradient increases to only about 10–15°C at an inclination of −10°, although the temperature correction is larger at negative angles.

Conclusions

Differentials of 10°C or less over 4.7 cm are observed in the rapid quench vessel used in this experiment with a water pressure medium at 2 kbar if the vessel is inclined at least +5° from the horizontal. From the work of Ludington (1978) and Rudert *et al.* (1976) one should expect gradients to become slightly greater at lower pressure. Gradients for 30.5 cm vessels with a full length filler rod are inclination independent for capsule lengths of 3–4 cm. Temperature corrections, however, are inclination dependent.

Acknowledgments

The authors would like to thank John Frantz for his assistance in setting up this calibration and for providing several of the spacers that were used and the Office of Basic Energy Sciences, Department of Energy, under whose auspices this work was done.

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Manuscript received, February 16, 1981;
accepted for publication, June 5, 1981.