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Thermochimica Acta 373 (2001) 13–22

thermochimica
acta

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A new flow calorimeter using a eutectic molten salt as the temperature control medium

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Received 3 December 2000; received in revised form 17 January 2001; accepted 19 January 2001

Abstract

A new isothermal flow calorimeter for measuring heats of mixing of two fluid streams at high temperatures and pressures is described. The main advantages of the new calorimeter over earlier ones are simplification of the design making it easier to maintain the equipment, reduction of the temperature gradients so that baseline corrections are minimized, and ability to make reliable measurements above 350°C. Immersion of the reaction vessel and associated tubing in a eutectic salt bath and the use of a thermoelectric cooler made it possible to maintain the reaction zone at a constant temperature. Energy effects from 2.5 to 2000 mW can be measured to a precision of ±1.5% or ±0.5 mW, whichever is greater. The relatively high uncertainty is due to the difficulties of operating at temperatures and pressures near and above the critical point of water. The calorimeter was tested by measuring the heat of mixing of sodium chloride solutions and water at 300 (9.3 MPa), 350 (17.6 MPa), 370 (24.7 MPa), and 380°C (24.7 MPa). The results at 300 and 350°C were in good agreement with our earlier results [Thermochim. Acta 285 (1996) 11] and were within 10% of earlier results by Busey at al. [J. Chem. Thermodyn. 16 (1984) 343]. No data are available in the literature at 370 and 380°C. However, our results at these temperatures appear to be reasonable.

Keywords: Aqueous chemistry; Flow calorimeter; Heat of mixing; High temperature; High pressure

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1. Introduction

A versatile calorimeter is needed for the determination of thermodynamic data for a wide variety of chemical reactions at high temperatures and pressures. High temperature thermodynamic data are important in many scientific and industrial applications including geochemistry, environmental chemistry, chemical oceanography, hydrometallurgy, electric power plant operation, water desalination, gas processing, and the oxidation of chemical wastes in supercritical water.

Isothermal flow calorimetry is based on measuring the power required to maintain the reaction zone at a constant temperature during the course of a reaction. The isothermal condition is achieved in the calorimeter described here by adjusting the power output of a controlled heater to balance the rate of energy production arising from the chemical reaction plus the constant power removed by a thermoelectric device.

Over the last two decades, several generations of isothermal flow calorimeters constructed in our laboratory have been reported [1–8]. We have operated flow calorimeters constructed of stainless steel, Hastelloy-C, tantalum, nickel, platinum–iridium, and platinum–rhodium tubing. The availability of these

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0040-6031/01/$ – see front matter
PII: S0040-6031(01)00462-2

calorimeters has allowed us to study a variety of systems in the temperature range 15–350°C and pressure range 0.1–35 MPa. Thermodynamic data for ligand interaction with protons and metal ions in aqueous solutions [9–15], heats of mixing in binary and ternary systems involving organic compounds and carbon dioxide [16–21], and vapor–liquid equilibrium data [22,23] have been determined by isothermal flow calorimetry at high temperatures and high pressures. Measurements above 125°C were carried out in calorimeters consisting of concentric cans, each held at a different temperature. This arrangement led to significant temperature gradients that increased markedly with temperature. Although, they increased with temperature, baseline corrections gave results that were accurate to a few percentage points up to 300°C. Above 350°C the results were so uncertain that it became apparent that a new design was required in order to obtain meaningful results.

This paper describes a new isothermal flow calorimeter for measuring heats of mixing of two fluids (liquid, gas, or supercritical fluid) at high temperatures and pressures. The new calorimeter retains all the desirable operating characteristics and advantages of the previous calorimeters [7,8] while offering several significant improvements. Use of a salt bath instead of concentric cans and a thermoelectric cooler instead of nickel bolts as the heat-leak path as in a previous calorimeter [8] results in minimizing the temperature gradient; improving temperature control of the reaction zone; and simplifying calorimeter construction, operation, maintenance, and repair. This new calorimeter has been used to produce precise results at 370 and 380°C [24–27]. Tubing thicker than that used earlier [7,8] was employed in order to allow operation at temperatures above 350°C and pressure to 50 MPa. The performance of the calorimeter has been tested (pressures given in parentheses) by measuring heats of dilution of aqueous solutions of sodium chloride at 300 (9.3 MPa), 350 (17.6 MPa), 370 (24.7 MPa), and 380°C (24.7 MPa) and the results at the two lower temperatures were compared with those reported earlier by us and others.

Flow calorimeters which have been operated at temperatures above 300°C have been built by workers at Oak Ridge National Laboratory [28] and at Laboratoire de Thermodynamique et Génie Chimique, Université Blaise Pascal [29,30]. The calorimeter used by the Oak Ridge Group and one of the calorimeters developed at Blaise Pascal [30] are heat conduction calorimeters built by Setaram and modified by these two groups for operation at high temperatures. The Oak Ridge instrument has been used to measure heats up to 400°C. The Blaise Pascal instrument has been used to measure heats up to 300°C. The heat generated by a reaction in these calorimeters is sensed by a platinum/platinum alloy thermopile. Each calorimeter employs concentric tubing for heat exchange. The temperature of the reaction zone is controlled by placing it in an air bath, which is located in a temperature controlled furnace. This has the design advantage of not requiring any active heat removal.

The Blaise Pascal group has constructed a second flow-mixing calorimeter in combination with a vibrating tube densimeter and has operated it up to 300°C and 20 MPa. They report that their measurements are accurate to better than 5%. This latter calorimeter is a heat compensation calorimeter based on the same principles as that of our earlier calorimeters. It has at least three improvements: first, replacement of many of the concentric cans with a vacuum can; second, placement of the mixing coils and calibration heater on the inside of the inner vessel instead of on the outside; and third, combining the calorimeter with a vibrating tube densimeter so simultaneous heat and density measurements can be made.

The calorimeter that is reported in the present work is also a heat compensation calorimeter. This new design allowed us to use the same heat measurement electronics used in earlier versions, but with the better temperature control afforded by the liquid bath.

2. Overall description of calorimeter

The calorimeter described here is a modification of the isothermal calorimeters constructed by us for use at high pressures and low (below 125°C) temperatures [7] and at high temperatures and high pressures [8]. A schematic diagram showing the main components of the calorimeter is given in Fig. 1. These components consist of the reaction vessel which contains the nickel isothermal cylinder, the high temperature thermoelectric device, and the mixing and equilibration tubing; the salt bath and contents; the fluid delivery and pressure control systems consisting of high pressure

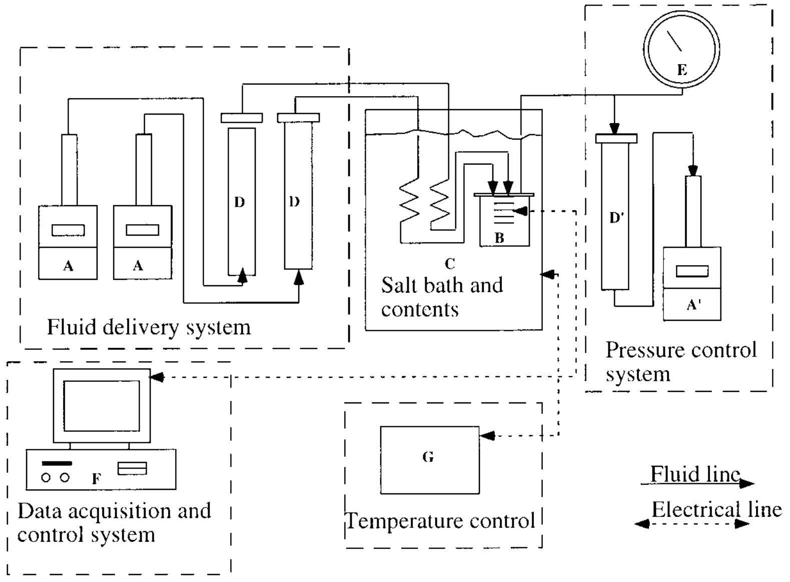

Fig. 1. Schematic of main components of calorimeter. A: delivery pumps, A′: pump run in reverse to maintain pressure, B: reaction vessel (Fig. 2), C: salt bath and contents (Fig. 3) to provide constant temperature environment, D: pushers containing reactant fluids (Fig. 6), D′: pusher to receive waste solution, E: pressure gage, F: computer, G: interface, H: temperature controller for salt bath, and I: temperature controller that compensates and accounts for the heat of reaction by maintaining the isothermal cylinder at a constant temperature.

delivery pumps and pushers; the temperature control system which consists of a temperature controller, a digital thermometer, and an isothermal control unit; and a data acquisition system that contains a 386 SX IBM-compatible computer and an interface (Intelligent DVM, DeltaQuest). The primary differences between the bath and reaction vessel described here and those described earlier [7] are the use of a high temperature thermoelectric device as a cooler, the use of a high temperature thermistor for temperature control, the use of a eutectic salt mixture as bath liquid capable of use at high temperature, and the use of platinum wires as the control and calibration heaters.

A reaction is initiated by starting the pumps and expelling the reactants at a constant rate from the pushers through the equilibration tubing on the isothermal cylinder. A thermopile removes energy from the isothermal cylinder at a constant rate and discharges it to the surrounding salt bath. A control heater compensates for the energy of the reaction and the cooling effect of the thermoelectric device and maintains the cylinder at a constant temperature. The differences in the rates of energy supplied by the heater before, during, and after the reaction are a direct measure of the energy of the reaction. The frequency of electrical pulses (0–10^5 pulse s^{-1}) supplied to the heater is recorded on the computer. Energy effects due to heats of mixing can be measured in the range 2.5–2000 mW with a precision of ±1.5% or ±0.5 mW, whichever is greater. The method is applicable to either endothermic or exothermic reactions. Heat capacity measurements are not required for isothermal calorimeters and heat conduction calorimeters. Isothermal conditions exist in the operation of the calorimeter described here as the fluids enter and leave the reaction zone at the same temperature and the temperature difference between the reaction zone and the surroundings is the same during both the electrical calibration and experimental runs.

3. Reaction vessel

The reaction vessel (Fig. 2) consists of a stainless steel container enclosing the isothermal cylinder and equilibration tubing. The thermoelectric device is in

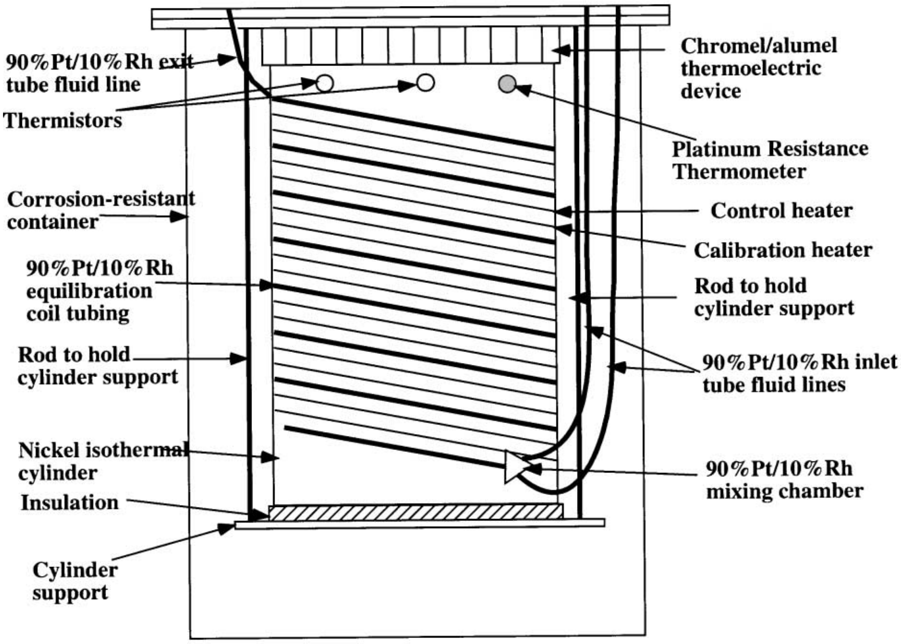

Fig. 2. Reaction vessel and contents.

contact with the top of the stainless steel container to facilitate transfer of heat from the cooler to the salt bath. The stainless steel container is fitted with a copper O-ring seal and the lid is attached by stainless steel bolts and nuts. The copper O-ring must be replaced after several months of operation as it corrodes. The unit is suspended 25 cm below the mount situated on the top of the salt bath by 3 stainless steel support rods (two are shown in Fig. 3). The isothermal cylinder is constructed of nickel as nickel is more resistant to corrosion and has a higher thermal conductivity than stainless steel. The nickel isothermal

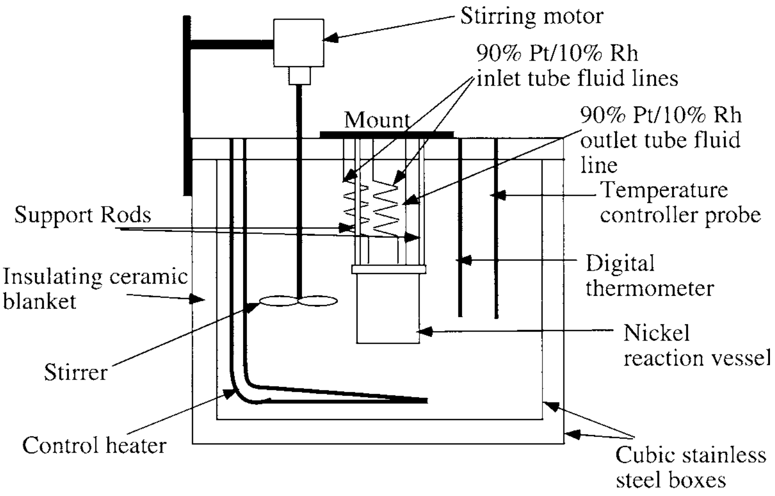

Fig. 3. Salt bath and contents.

cylinder is 4.29 cm o.d., 8.2 cm high, with a wall thickness of 0.32 cm. The top of the cylinder is closed and is 0.64 cm thick. Four 0.16 cm diameter holes were drilled through the side near the top to accommodate three control thermistors and a platinum resistance thermometer. The cylinder has one 11-turn spiral groove machined into its side, accommodating the equilibration coil which is constructed of 150 cm of 2.38 mm o.d., 0.79 mm wall thickness 90% platinum–10% rhodium tubing (Engelhard, NJ). The calibration and control heaters each consist of approximately 150 cm of platinum–13% rhodium wire (0.203 mm o.d., Omega wire no. SP13RH-008) in high temperature glass sleeving (Omega Engineering Inc., Stamford, CT), coiled around the outside of the isothermal cylinder.

The thermoelectric device, shown in Fig. 4, is constructed of alternately welded 5 mm chromel wires (o.d. 0.821 mm, Omega wire no. SPCH-032) and 5 mm alumel wires (o.d. 0.821 mm, Omega wire no. SPCH-032) (Omega Engineering Inc., Stamford, CT) with 100 joints at both the top and bottom cast into ceramic adhesive (Cotronic 903 HP, Cotronics Corp., Brooklyn, NY).

The tubes containing the reactants join in a triangular shaped mixing chamber (made of platinum–10% rhodium) as shown in Fig. 5. The two inlet tubes and the exit tube from the mixing chamber were connected to the mixing chamber by gold brazing. Before entering the reaction vessel, the reactants are equilibrated to the bath temperature in two 2 m lengths of platinum–10% rhodium tubing. In order to obtain continuous tubing, the available 1.22 m platinum–rhodium pieces were connected by gold brazing using 0.794 mm o.d., 0.198 mm wall thickness platinum–10% rhodium tubing as an inner sleeve. The sleeve length was about 1.5 cm for each union.

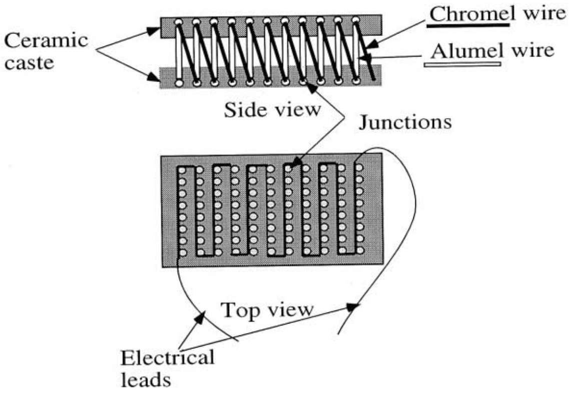

Fig. 4. Chromel/alumel thermoelectric device.

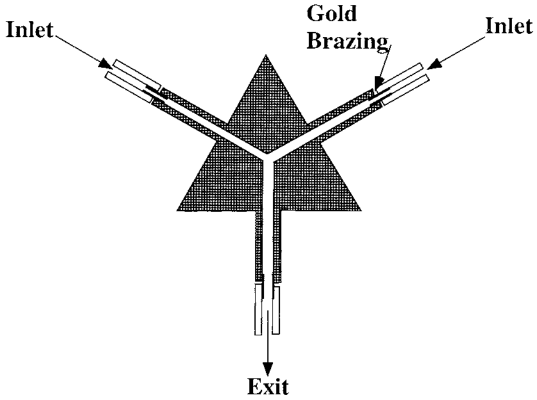

Fig. 5. Triangular mixing chamber.

The isothermal cylinder (Fig. 2) was monitored and controlled continuously at a constant temperature using the output of a wheatstone bridge, one leg of which is one of the thermistors (Thermometrics Inc., Edison, NJ), attached into the holes drilled near the top of the cylinder (Fig. 2) with CC high temperature cement (Omega Engineering Inc., Stamford, CT). To cover the wide range of operating temperatures, three thermistors with resistances of 1.0, 1.5 and 2.0 MΩ, respectively, at 125°C, were used). The use of high temperature thermistors allows the use of our present electronics. This arrangement works well even though there is some drift of the response with time. One 100 Ω platinum resistance thermometer (Omega Engineering Inc., Stamford, CT) at 0°C, cemented into a hole drilled near the top of the cylinder, was used to measure the temperature of the cylinder. The control heater and the appropriate thermistor, depending on the temperature range of interest, were connected to a Hart Scientific 3704 (American Fork, UT) isothermal control unit. The temperature of the isothermal cylinder is controlled with a precision of ±0.0005°C. The uncertainty in the temperature control of the isothermal cylinder is a result of the fluctuations in the temperature bath, which are due to the bath being operated at temperatures much higher than ambient.

4. Salt bath

The salt bath (Fig. 3) was made of two cubic stainless steel boxes, a temperature controller probe, a Hart Scientific (American Fork, UT) 1006 digital thermometer, a stirrer and a 500 W copper immersion control heater (Fisher Scientific, Santa Clara, CA). Ceramic blanket material (Cotronics Corp., Brooklyn, NY) was placed in the space between the two boxes for insulation. A mixture of sodium nitrite, sodium nitrate, and potassium nitrate (40, 7, and 53 wt.%, respectively) was put in the salt bath and heated to the molten state. This mixture was chosen because it forms a eutectic melt at 150°C, is stable, and has a low vapor pressure at the temperatures of interest. The thermistor used in the temperature controller probe is the same type as that used for the isothermal cylinder in the reaction vessel. The bath temperature is set with the digital thermometer and controlled with a precision of ±0.005°C using a Tronac (Orem, UT) PTC-41 temperature controller, connected to the heater and thermistors.

5. Fluid delivery and pressure control

The fluid delivery and pressure control system used are the same as those described previously [8]. Fig. 1 shows a schematic diagram of the fluid delivery system which consists of two Isco 260D syringe pumps (Lincoln, NE) and two high-pressure pushers. The specially designed pushers, made of stainless steel (7.8 cm o.d., 5.2 cm i.d., and 109.5 cm in length) with opening on the top and bottom (Fig. 6), were used to prevent corrosion of the pumps by the electrolyte solutions. Solutions under study were contained in Teflon bags inside the pushers and were expelled into the tubes leading to the mixing chamber in the calorimeter by pressure transmitted by distilled water from the pumps. The pressure control system (Fig. 1) consists of an Isco 260D syringe pump running in the constant pressure mode and a waste solution container which is of the same construction as the pushers used for fluid delivery. The flow rates of the pumps were calibrated gravimetrically using distilled water and the accuracy was found to be better than ±0.5%. The pressure stability attained with the pressure control system is ±0.01 MPa.

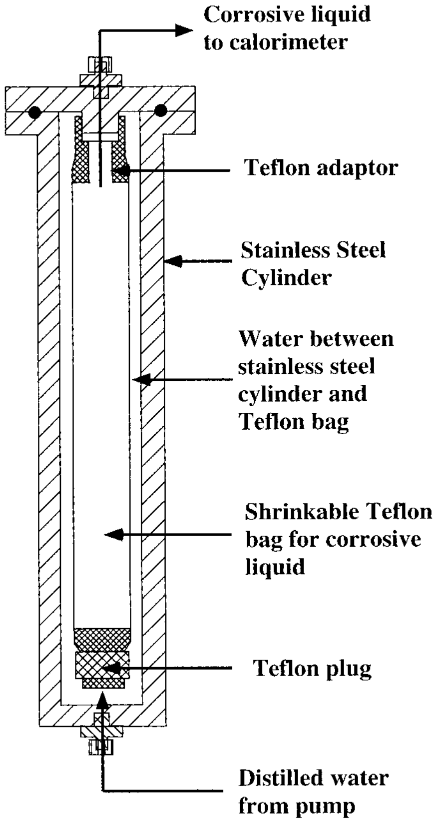

Fig. 6. Schematic of pusher.

6. Procedure

The reaction vessel is immersed in the salt bath set at the desired temperature and left there at least overnight to assure that its temperature reaches that of the salt bath. The isothermal control unit is adjusted to maintain the vessel at the bath temperature. The pumps are filled with distilled water which is pumped into the pushers to expel the reactants into the calorimeter. The measurements are made in a steady state, fixed composition mode. The experimental determination consists of running one pump at the total flow rate to determine a baseline heater pulse rate or the number of pulses required to balance the heat removal and maintain the cylinder isothermal when no reaction occurs. Next, both pumps are run at their respective flow rate and after an equilibration period, the heater pulse rate is determined for the mixing solutions. The difference in the heater pulse rates before and during reaction is a measure of the heat of reaction. Electrical calibration is performed by adding power to the calibration heater from a dc power source. The amount of electrical power is obtained by measuring the potential drop across the calibration heater and across a 100 Ω standard resistor in series with the heater.

7. Performance

The performance of the calorimeter was tested by measuring heats of dilution of aqueous sodium chloride at 300 (9.3 MPa), 350 (17.6 MPa), 370 (24.7 MPa), and 380°C (24.7 MPa). The solutions used in the measurements were prepared by dissolving appropriate amounts of sodium chloride (Aldrich ACS reagent, >99%) in distilled water, deionized using a fixed-bed cation exchange column. Measurements were made at previously used concentrations and temperatures [8]. The results showed that the calorimeter was capable of producing good calorimetric data over a wide range of temperature, pressure, flow rate, and concentration conditions. The results for the dilution of 0.5000m $NaCl_{(aq)}$ are listed in Table 1 together with the results obtained earlier [8,28], and values calculated using equations reported in the literature [31,32]. The results for dilution of 0.1002 and 0.3005m $NaCl_{(aq)}$ at 370°C (24.7 MPa) and at 380°C (24.7 MPa) measured on the new calorimeter [26] are listed in Table 2. Values from the present study are the arithmetic means of at least two experimental runs with standard deviation σ_{n-1} of n experiments of these means. The results of this study are in excellent agreement with values obtained previously by us [8]. Table 1 shows that, although, $\Delta_{dil}H$ values of this study are more negative than those obtained by Busey et al. [28], both studies are in reasonable agreement especially at high molality. However, the difference between results of the present study and those of Busey is greater at 350 than at 300°C.

Table 1
Heats of dilution ($\Delta_{dil}H$) of 0.5000m $NaCl_{(aq)}$ at 300 (9.3 MPa) and 350°C (17.6 MPa)

m_f (mol kg^{-1})	$\Delta_{dil}H$ (kJ mol^{-1})				
	This study[a]	Previous study[b]	Busey et al.[c]	Pitzer et al.[d]	Archer[e]
300°C (9.3 MPa)					
0.4496	−1.26 ± 0.05[f]	−1.19	−1.13	−1.26	−1.34
0.3993	−2.57 ± 0.05	−2.44	−2.38	−2.61	−2.79
03490	−3.99 ± 0.03	−3.85	−3.73	−4.10	−4.40
0.2989	−5.6 ± 0.1	−5.42	−5.21	−5.74	−6.17
0.2489	−7.50 ± 0.06	−7.21	−6.90	−7.59	−8.18
0.1989	−9.64 ± 0.04	−9.36	−8.87	−9.71	−10.50
0.1490	−12.13 ± 0.02	−12.08	−11.24	−12.23	−13.25
0.0993	−15.3 ± 0.1	−15.64	−14.16	−15.39	−16.70
0.0496	−19.78 ± 0.06	−20.41		−19.84	−21.55
350°C (17.6 MPa)					
0.4496	−3.10 ± 0.05[f]	−2.98	−3.0		
0.3993	−6.92 ± 0.06	−7.04	−6.7		
0.3490	−10.8 ± 0.1	−10.95	−10.7		
0.2989	−15.6 ± 0.2	−15.78	−14.9		
0.2488	−21.3 ± 0.2	−21.11	−19.4		
0.1989	−27.9 ± 0.2	−27.81	−24.8		
0.1490	−36.0 ± 0.4	−36.03	−31.9		
0.0993	−46.2 ± 0.4	−46.36	−41.6		

[a] This study.
[b] [8].
[c] Calculated from the L_ϕ (apparent relative molar enthalpy) values in Table 5 and the pressure coefficients for L_ϕ in Table 7 of [28].
[d] Calculated using the Pitzer ion-interaction model of Pitzer et al. in [29].
[e] Calculated using the modified Pitzer ion-interaction model of Archer in [30].
[f] $n = 3$.

Table 2
Heats of dilution ($\Delta_{dil}H$) of 0.1002m and 0.3005m $NaCl_{(aq)}$ at 370 (24.7 MPa) and 380°C (24.7 MPa)[a]

0.1002m Initially		0.3005m Initially	
m_f (mol kg^{-1})	$\Delta_{dil}H$ (kJ mol^{-1})	m_f (mol kg^{-1})	$\Delta_{dil}H$ (kJ mol^{-1})
370°C (24.7 MPa)			
0.0902	−4.05 ± 0.12[b]	0.2703	−3.07 ± 0.14[c]
0.0801	−8.74 ± 0.09	0.2402	−7.29 ± 0.22
0.0701	−13.89 ± 0.09	0.2101	−12.18 ± 0.33
0.0601	−19.55 ± 0.13	0.1800	−17.90 ± 0.37
0.0500	−26.68 ± 0.11	0.1499	−25.00 ± 0.36
0.0400	−34.55 ± 0.21	0.1199	−32.39 ± 00.63
0.0300	−44.99 ± 0.34	0.0898	−41.35 ± 0.11
0.0200	−59.27 ± 0.49	0.0599	−51.01 ± 0.65
0.0100	−80.68 ± 0.58	0.0299	−64.27 ± 1.86
380°C (24.7 MPa)			
0.0902	−13.42 ± 0.38[c]	0.2703	−10.04 ± 0.29[c]
0.0801	−28.05 ± 0.45	0.2402	−23.27 ± 0.41
0.0701	−47.74 ± 0.97	0.2101	−38.38 ± 0.17
0.0601	−70.25 ± 1.15	0.1800	−55.91 ± 0.59
0.0500	−98.83 ± 1.05	0.1499	−78.07 ± 0.41
0.0400	−126.97 ± 0.93	0.1199	−103.56 ± 0.86
0.0300	−162.39 ± 1.84	0.0898	−136.41 ± 1.89
0.0200	−207.84 ± 2.37	0.0599	−175.81 ± 1.31
0.0100	−264.29 ± 16.9	0.0299	−236.31 ± 0.32

[a] [26].
[b] $n = 3$.
[c] $n = 2$.

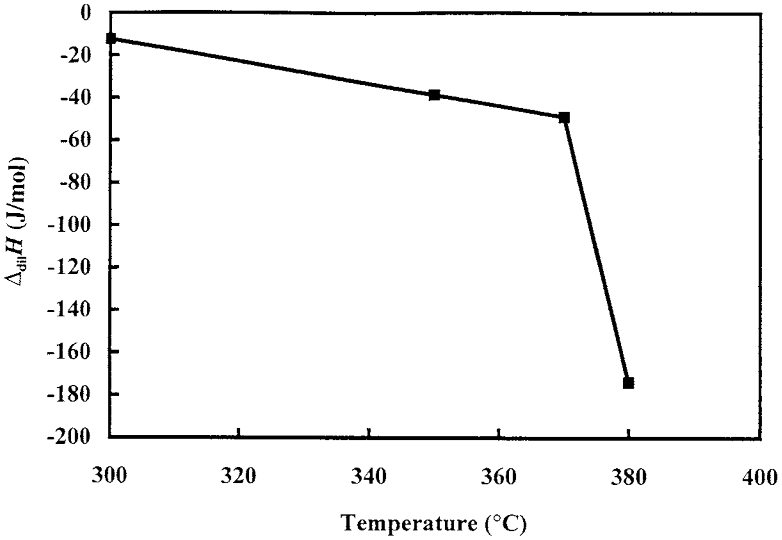

Fig. 7. Heats of dilution in going from 0.3m to 0.05m NaCl as a function of temperature. The pressures are 9.3 MPa at 300°C, 17.6 MPa at 350°C, 24.7 MPa at 370 and 380°C.

Table 1 also gives heat of dilution values for NaCl calculated from the models of Pitzer et al. [31] and Archer et al. [32] at 300°C (9.3 MPa). Both models used the calorimetric data of Busey et al. [28] up to 300°C together with other related thermodynamic data to obtain fitting parameters. The model of Pitzer et al. [31] is valid to 300°C while that of Archer et al. [32] extends to 325°C. It is seen in Table 1 that $\Delta_{\mathrm{dil}}H$ values obtained in this study are in good agreement with those using either the Pitzer or Archer model. The small differences between our heat data and those predicted by these models are probably due to the fact that neither of these models accounts for ion association. The $\Delta_{\mathrm{dil}}H$ values for dilution of a 0.3 molal to a 0.05m NaCl solution obtained at 300 (9.3 MPa), 350 (17.6 MPa), 370 (24.7 MPa) and 380°C (24.7 MPa) by interpolation of the data shown in Tables 1 and 2 are shown in Fig. 7. The trend with temperature to 370°C is the one expected. The $-\Delta_{\mathrm{dil}}H$ values increase as temperature increases. The sharp increase in the $-\Delta_{\mathrm{dil}}H$ value in going from 370 to 380°C is a result of the solution changing from a liquid to a highly compressible fluid as the critical point of the solution is approached as temperature increases. This increased compressibility results in a marked increase in $-\Delta_{\mathrm{dil}}H$ as has been predicted [33] and as has been shown for heats of mixing where one or both of the components are near their critical points [33].

8. Conclusions

In summary, the following changes were made to our earlier calorimeter: incorporation of thicker tubing, replacement of concentric cans by a eutectic salt bath, and incorporation of a thermoelectric device for heat removal. These changes resulted in the following improvements. First, use of a salt bath resulted in the near-elimination of temperature gradients making baseline corrections negligible at high temperatures. Second, use of thicker tubing made successful operation of the calorimeter at higher temperature and pressure combinations possible. Third, elimination of the concentric cans and the associated temperature controllers resulted in improved speed and ease of operation, and simplified maintenance.

Few high temperature (>300°C) calorimetric data exist. The heat of dilution of NaCl is a good candidate as a test system for calorimeter calibration. Unfortunately, the only two studies above 300°C [8,28] differ by about 10%. It would be desirable for other laboratories to make independent measurements at temperatures above 300°C in order to establish a value for this important quantity.

Acknowledgements

This material is based upon work supported by the US Army Research Office under Grant no. DAAG55-97-1-01129.

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