



Measurements of isobaric heat capacities for HFE-7000 and HFE-7100 at different temperatures and pressures



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ARTICLE INFO

Article history:

Received 2 March 2016

Received in revised form

8 June 2016

Accepted 10 June 2016

Available online 14 June 2016

Keywords:

Flow calorimeter

HFE-7000

HFE-7100

Heat capacity

ABSTRACT

This paper presents the heat capacities of HFE-7000 and HFE-7100 by a flow calorimeter. Ethanol has been used as a reference fluid to validate the flow calorimeter at different pressures and temperatures. With the flow calorimeter, the isobaric heat capacity of HFE-7000 (1-methoxyheptafluoropropane) and HFE-7100 (methoxy-nonafluorobutane) were measured in the temperature range from (255–294) K and from (255–323) K, respectively, and pressures up to 15 MPa. Through measuring the heat capacity of ethanol, the combined expanded uncertainty with a level of confidence of 0.95 ($k = 2$) of the measurement was estimated to less than $\pm 2\%$. The measured heat capacity values of HFE-7000 and HFE-7100 were correlated by the Garg equation. The calculated values showed good agreement with the experimental values.

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1. Introduction

At present, the stratospheric role of chlorinated compound as ozone depletion agents is well-known. A schedule has been proposed by the Montreal Protocol with its subsequent amendments to obsolete the substances that deplete the ozone layer, which lead to a continuous research to replace the traditional chlorinated compound with environmentally friendly alternatives [1]. Hydrofluoroethers (HFEs) as an environmentally acceptable alternative were first introduced in 1996 [2,3]. Hydrofluoroethers (HFEs) have been applied in this sense in industry such as paints, adhesives, cleaning of electronic components, refrigeration, and carrier fluids for lubricant deposition, because of their near zero ozone depletion, relatively low global warming potential and short atmospheric lifetimes.

Heat capacity is a thermophysical property of the liquid, the measurement of heat capacity is an important experimental study for establishing reliable thermodynamic equations of state [4–6]. The heat capacity of a substance is also a measure of the amount of energy required to produce a given temperature change within a unit quantity of that substance. It is used in engineering calculations that relate to the manner in which a given system may react to thermal stresses [7–11]. In view of an environmental friendliness,

the research on the heat capacity of Hydrofluoroethers (HFEs) draws attention from the public in the field of environment. According to the product catalogue of 3M Company, HFE-7000 (1-methoxyheptafluoropropane, CAS Registry Number: 375-03-1) is known as nonflammable and low global warming potential (GWP). HFE-7100 is not a pure compound but a mixture: “HFE-7100 fluid ($C_4F_9OCH_3$) consists of two inseparable isomers with essentially identical properties. These are $CF_3CF_2CF_2CF_2OCH_3$ (nonafluoro-*n*-butylmethylether, CAS No. 163702-07-6) and $(CF_3)_2CF_2CF_2OCH_3$ (nonafluoro-*iso*-butylmethylether, CAS No. 163702-08-7).”

In this work, a new adiabatic flow calorimeter was validated by measuring the isobaric heat capacity of ethanol at different temperatures and pressures. The heat capacity data of HFE-7000 and HFE-7100 are studied by different pressures and different temperatures. With the flow calorimeter, the heat capacity data of HFE-7000 was measured between (253–294) K with pressures up to 15 MPa, while the heat capacity data of HFE-7100 was measured between (253–323) K with pressures up to 15 MPa. The experimental results were correlated to a Garg equation. The equation could represent the present experimental data well.

2. Experimental section

2.1. Samples

The ethanol was provided by the China National Pharmaceutical Group, with a purity higher than 99.8%. The water content of the

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ethanol was below 0.05%. The results of Components were as shown in Table 1. The sample of ethanol was used without further purification. The sample of HFE-7000 was provided by 3M Company with the mass purity better than 99.5%, and used without further purification. The sample of HFE-7100 was also provided by 3M Company with the mass purity better than 99.8%, HFE-7100 fluid (C₄F₉OCH₃) consisted of two inseparable isomers with essentially identical properties. The composition of HFE-7100 was determined by FNMR spectrum. The mass ratio of CF₃CF₂CF₂CF₂OCH₃ (nonafluoro-*n*-butylmethylether, CAS No. 163702-07-6) and (CF₃)₂CF₂OCH₃ (nonafluoro-*iso*-butylmethylether, CAS No. 163702-08-7) was 0.452:0.548. The specifications of chemical samples were summarized in Table 2.

2.2. Experimental method

The principle of measurement has been described in the literature [12]. In a flow calorimeter, in order to determine the isobaric specific heat capacity, constant mass flow rate at constant pressure is heated by a constant heat flux from heater. The temperature increment, between the inlet and the outlet liquid temperatures, can be related with heat capacity by the flowing relation.

$$c_p = Q/(m\Delta T) \quad (1)$$

Where Q means heat flux, ΔT means the temperature increment between the inlet and the outlet liquid temperatures, and c_p describes the heat capacity of the liquid.

In the experimental measurement, there always exists some heat loss from sample fluid to the surroundings. To avoid the effect of this heat loss, we actually measured the apparent heat capacity, which is shown by the following relation.

$$c_{p(app)} = \frac{Q}{m\Delta T} = \frac{Q_1 + Q_L}{m\Delta T} = c_p + \frac{Q_L}{m\Delta T} \quad (2)$$

Where Q means the power input of the heater, Q_1 means heat flux of the liquid absorbed $Q_1 = Q - Q_L$, Q_L means the heat loss and $c_{p(app)}$ describes the apparent heat capacity. An integral mean value of the real heat capacity, c_p , may be obtained by extrapolating $c_{p(app)}$ to $1/m \rightarrow 0$. The required suppression of heat losses has been attained by following methods. Convective molecular heat losses are eliminated by placing the calorimeter in an evacuated chamber. The radiative heat losses are minimized by radiation shields [13].

However, there is also a small temperature difference between the thermometers of the calorimeter when the heater is switched off ($Q = 0$). Considering the small errors of the two thermometers, a heater-on experiment with a temperature rise of ΔT_h and a heater-off experiment with a temperature rise of ΔT_b , are carried out under other conditions unchanged. The relationship $\Delta T = \Delta T_h - \Delta T_b$ can then be used in equation (2).

Table 1
List of components in ethanol from the China national pharmaceutical group.

Components	Mass fraction %
Ethanol	>99.8
H ₂ O	<0.05
CH ₃ COOH	<0.03
NH ₃	<0.02
CH ₃ OH	<0.1
Non-volatile mater	<0.01

2.3. Apparatus

The apparatus had been described in the detail by literature [12], where it was described in detail. It was designed for measurement at pressures up to 20 MPa and the temperature from (243–393) K. As shown in Fig. 1, the main components of the apparatus were flow calorimeter, closed circulation system and stable liquid. The apparatus were suitable for the measurement of liquid, however, it required that the liquid should not contain particles and the viscosity should be relatively small, the viscosity range was from (0.3–5)cP.

Flow calorimeter was based on measuring the power to keep the fluid the temperature increment constant [14,15]. The flow calorimeter was designed in the vacuum can. The flow calorimeter consists of three parts: two secondary standard 100Ω platinum resistance thermometers and a microheater. The microheater section was located between the two 100Ω platinum resistance thermometers. Two platinum resistance thermometers were inserted into copper bars to indirectly detect the inlet and outlet temperatures of the fluid. Rough evaluation showed that the indirect measurement of temperatures results in an error of less than 0.001%, so the measured temperatures needed no calibration [16]. This section was protected from heat leaks by keeping high vacuum circumstance and using the radiation shield.

The closed circulation system was designed by stainless steel tube, which was immersed in a bath to preheat the fluid and the temperature was automatically controlled. The uncertainty of the bath temperature remained constant to ± 20 mK.

The stable liquid flowed through calorimeter by a precision isocratic pump with dual floating pistons and the liquid through the cell was controlled at a constant flow rate. The isocratic pump was supplied by Beijing star technology development limited company. The flow range of isocratic pump was from (0.01–9.99) ml/min and the pressure up to 40 MPa.

In addition, the pressure was controlled by the back valve. The mass collected was determined by weighing and the interval of sample collecting time.

In the word, the steady pump, digital balance, the flow calorimeter and the thermostated bath were controlled, and the data of the flow rate, temperature and voltage were also measured by automated software.

2.4. Assessment of uncertainties

The method of measurement uncertainty determination described in Ref. [17] was adopted. According to the Guide to the Expression of Uncertainty in Measurements (GUM) [18], $u(Y)$ can be obtained from the square root of its variance, $u^2(Y)$, expressed by

$$u^2(Y) = \sum_{i=1}^n \sum_{j=1}^n \frac{\partial f}{\partial x_i} \frac{\partial f}{\partial x_j} u(x_i, x_j) \quad (3)$$

where $\partial f/\partial x$ is sensitivity coefficient, $u(x_i, x_j)$ is the covariance of x_i, x_j and $u(x_i, x_i) = u^2(x_i)$.

The evaluation of expanded uncertainty of a measurement result is always an approximate evaluation. Expanded uncertainty $U(k)$ is evaluated by the approximate method and is equal to

$$U(k) = ku(c) \quad (4)$$

Where $u(c)$ is described as standard uncertainty.

The experimental uncertainties were predicted according to the ISO recommendation (1993) with the coverage factor $k = 2$. The standard coverage factor $k = 2$ can yield an expanded uncertainty

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