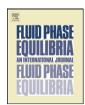
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New device for fast measuring surface tension, density and viscosity of liquids

Dequan Shi*, Guili Gao, Dayong Li, Jingwei Dong, Lihua Wang

Department of Material Science and Engineering, Harbin University of Science and Technology, No.4, Linyuan Road, Dongli District, Harbin 150040, PR China

ARTICLE INFO

Article history:
Received 10 June 2008
Received in revised form 28 August 2008
Accepted 30 August 2008
Available online 6 September 2008

Keywords: New device Fast measurement Surface tension Density Viscosity

ABSTRACT

A new device has been developed which entails sequentially fast measurements of surface tension, the density and the viscosity of liquids. In this new device, the surface tension is quickly calculated by the sum of the different contributions from the pressure difference in bubbles, the number of bubbles in 5 s and the capillary diameter. The measurement of the density and the viscosity are based on the Archimedes principle and the rotating cylinder method. The measurement range of the surface tension, the density and the viscosity is $17-75 \, \text{mN/m}$, $0-2.0 \times 10^3 \, \text{kg/m}^3$ and $10-10^5 \, \text{mPa} \, \text{s}$, respectively. At 295 K temperature, some experiments on the glycol and glycerol have been made, and the results show that the accuracy of measuring the surface tension, the density and the viscosity is below $\pm 1.0\%$, $\pm 1.0\%$ and $\pm 1.5\%$, respectively, and that the reproducibility is satisfactory.

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1. Introduction

Surface tension, density and viscosity are main physical parameters of liquids [1,2]. There has been a growing interest in fast measurement of surface tension, density and viscosity because they are critical parameters sensitive to material property changes caused by chemical reaction, solidification, gelation, and deposition of substances [3-6]. So, fast measurement of surface tension, density and viscosity can offer a powerful tool for monitoring the quality of liquids and processes involving a liquid environment. In production, if the surface tension were exactly measured, the process would be optimized in good time to assure the product quality [7]. The measurement of the density has been widely applied in the management of oil storage and the analysis of alcoholic drink, oil and beverages [8]. The viscosity is often used to appraise the quality and performance of automobile engine oil [9]. In study of mass transfer, it was desired to conveniently achieve a range of density, viscosity and surface tension, for which aqueous ethylene glycol and aqueous propane solutions at 298 K were chosen, and methanol and ethanol were also utilized [10]. Consequently, fast measurement of these three parameters is strongly recommended. Roach reported a new method to dynamically measure surface tension, density and viscosity of melts [11], and Vaisburd described a combined unit for measuring surface tension, density, and viscosity of oxide melts [12]. However, in Roach method, surface tension, density and viscosity were measured according to a modified Bernoulli formulation, and Vaisburd used the maximum bubble pressure method, which period is longer than 2 min, to measure these three parameters of oxide melts, therefore, they are not adequate for fast measurement and thus there is a need to develop a new device.

In present study, a sort of creative design has been put forward, and a new device which can fast measure the surface tension, the density and the viscosity of liquids has been constructed. Because the new device has high measuring accuracy and reproducibility, it can provide a tool for experimentally studying the property of liquids and comprehensively evaluating the quality of liquids.

2. Measurement principle

2.1. Principle of fast measuring surface tension

There have been some methods to measure surface tension of liquids [13], however, the maximum bubble pressure, the Wilhelmy plate and the drop shape methods are widely used [14–16]. As accuracy the Wilhelmy plate and the drop shape methods are, the measurement time is usually too long and the operation is too complicated, and what is important is that they cannot meet the structural requirement of device. In the maximum bubble pressure method, pressurized gas is blown in liquids through a capillary slowly, and the pressure in the bubble is monitored by a pressure measurement device. When the diameter of the bubble is as same as that of the capillary, the pressure in the bubble reaches the maximum value, and the surface tension can be calculated by the maximum pressure, the capillary diameter, the dipping depth

^{*} Corresponding author. Tel.: +86 451 86392558; fax: +86 451 86392558. E-mail address: shidequan2008@yahoo.com.cn (D. Shi).

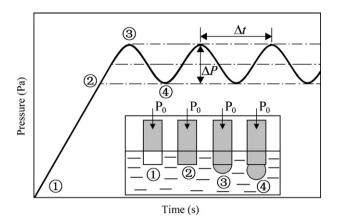


Fig. 1. Relationship between the pressure in the bubble and the time of blowing air.

of the capillary and the density of liquids. However, the dipping depth of the capillary must be fixed strictly and measured exactly and the speed of blowing bubbles must be very slow. To avoid these two disadvantages, an improved method for fast measuring surface tension has been developed.

In this new method, pressurized air is blown in liquids through a capillary and bubbles will generate and vanish continuously at the speed of 2–3 bubbles per second. The real-time pressure in the bubble is monitored by a pressure difference sensor (The model is HB-XYL and it is made by Huibang Science Technique Co., Ltd., Beijing. The measurement range is 0-1 kPa and the precision is $\pm 0.05\%$). The relationship between the pressure in the bubble and the time of blowing gas is shown in Fig. 1. The shape of the pressure curve is like a sinusoid on the condition that the speed of generating bubbles is stable. The number ①, ②, ③ and ④ denotes four states of the bubble, respectively, namely, not blowing gas, not generating the bubble, the bubble with maximal curvature radius and the bubble of escaping from the capillary. The direct factors to affect the horizontal position of the sinusoid are the dipping depth of the capillary and the density of liquids. During the course of blowing gas. according to the Laplace's formula [17], the shape of the sinusoid is mainly decided by the surface tension σ and the capillary diameter ϕ_x . When the blowing velocity is fixed, the greater the surface tension σ of liquids is, the bigger the amplitude ΔP is and the longer the period Δt is. Because the amplitude and the period of the sinusoid reflect the additional pressure caused by the surface tension and the speed of generating bubbles, respectively, they are taken as basic parameters calculating the surface tension. When the surface tension σ retains the same, the bigger the capillary diameter ϕ_x is, the bigger the amplitude ΔP is and the longer the period Δt is. Therefore, the capillary diameter is also considered as a parameter. To simplify calculation, the period Δt of the sinusoid is replaced by the number *N* of bubbles in a fixed time.

Because the capillary is disposable and their diameter differs, it is indispensable that the diameter be quickly measured. During the pressurized gas blowing in air through the capillary, the bigger the capillary diameter is, the lower the pressure in the capillary is. On the contrary, the smaller the capillary diameter is, the higher the pressure in the capillary is. Consequently, the pressure in the capillary before being dipped in the liquid can be used to express the actual value of the capillary diameter, and the real-time pressure can be automatically monitored by the pressure difference sensor.

Over 293–298 K temperature range, we have performed many experiments on various kinds of liquids, such as 95.5% methanol, 99.0% n-pentane, 75% acetic acid, surface tension test mixtures (which are mixed by 100% 2-ethoxyethanol, 99.8% formamide and pure distilled water according to different proportion and their

surface tensions range from 17 mN/m to 75 mN/m with 1 mN/m interval) and so on. The actual surface tension of liquids is measured by the KSV's SIGMA700. At the same time, three parameters calculating the surface tension are also fast measured by computer. Through regressive analysis, the surface tension of liquids can be calculated according to Eq. (1) [18,19]:

$$\sigma = 0.1182 \Delta P + 3.7428 N + 0.0158 \Delta \phi_x - 28.6387 (\text{mN/m})$$
 (1)

where ΔP is the amplitude of bubbles, N is the number of bubbles in 5 s, and $\Delta \phi_x$ is the difference between actual value ϕ_x and standard value ϕ_0 of the capillary diameter. To simplification, the pressure difference ΔP and the capillary diameter ϕ_x are the A/D conversion results of the corresponding pressure and thus they are dimensionless. So, it is only on the magnitude that the surface tension is equivalent to the sum of the different contributions from ΔP , N and $\Delta \phi_x$ in Eq. (1).

The experimental results lead to the conclusion that the measurement range of Eq. (1) is from 17 mN/m to 75 mN/m and it is applicable for the liquids at 293–298 K temperature range.

2.2. Principle of fast measuring density

When the buoyancy of the air is ignored, according to the Archimedes principle, the buoyancy caused by the liquid can be calculated by Eq. (2) if a solid is dipped in the liquid [20]:

$$F = g\rho V \tag{2}$$

where F is the buoyancy caused by the liquid, ρ is the density of the liquid, g is the acceleration of gravity ($g = 9.8 \text{ m/s}^2$), and V is the volume of the solid dipped in the liquid.

Because the volume of the solid dipped in the liquid can be measured in advance, the density of the liquid would be quickly calculated according to Eq. (2) if the buoyancy were exactly measured. To simplify the structure of the new device, a hollow cylinder, whose diameter and height are 65 mm and 60 mm, respectively, is entirely dipped in the measured liquid and a load sensor (The model of the sensor is BK-3A and it is made by Jinghuichuan Instrument Technology Co., Ltd., Beijing. According to the stress and strain theory, the force is measured and the precision is $\pm 0.1\%$. When working, it is always pulled.) is used to monitor the magnitude of the force. After the hollow cylinder is entirely dipped in the liquid, one force F_1 can be measured by the sensor. Also, before the hollow cylinder is dipped in the liquid, anthor force F_2 can be measured and it is equivalent with the weight of the cylinder. So, it is a constant and equals to 4.5 N. According to Eq. (2), the buoyancy F after the cylinder is dipped in the liquid is the difference between F_2 and F_1 . Therefore, when the diameter (D = 65 mm) and height (h = 60 mm) of the cylinder are introduced, the density ρ of the liquid can be calculated according to Eq. (3):

$$\rho = \frac{F_2 - F_1}{gV} = 512.5144(4.5 - F_1) \quad (kg/m^3)$$
 (3)

Assuming the density of the liquid ranges from 0 to 2.0×10^3 kg/m³, the range of the load sensor will be 0–4.9 N. So, the measurement range of the load sensor is determined to be 0–0.5 kg.

2.3. Principle of fast measuring viscosity

In this study, the rotating cylinder method [21], as shown in Fig. 2, is used to measure the viscosity of Newtonian fluids. Its principle is as follows. The measured liquid is filled in the space between the internal and external cylinder. When the external cylinder rotates with uniform speed under the drive of DC motor and the internal cylinder keeps motionless, the radial velocity gradient and the internal friction will occur inside the liquid. As a

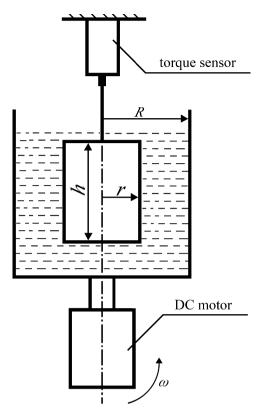


Fig. 2. The principle of the rotating cylinder method.

result, the measured liquid will bring the shear stress to bear on the internal cylinder. Therefore, if the shear stress is exactly measured, the viscosity of liquids can be fast calculated. To avoid the turbulent flow and the glide between the internal cylinder and the measured liquid, the rotational angular velocity and the diameter of the external cylinder are designated to be 3.14 rad/s and 95 mm, respectively. According to the rotating cylinder method, the relation between the viscosity and the rotary torque can be expressed as Eq. (4) [22]:

$$\eta = \frac{M}{4\pi h} \left(\frac{1}{r^2} - \frac{1}{R^2} \right) \frac{1}{\omega} = \frac{M}{4\pi h} \left(\frac{1}{r^2} - \frac{1}{R^2} \right) \frac{60}{2\pi n} \tag{4}$$

where η is the viscosity of the measured liquid, h is the height of internal cylinder, h = 60 mm, r is the radius of internal cylinder, r = 32.5 mm, R is the radius of external cylinder, R = 47.5 mm, and n is the rotational angular velocity, n = 30 rpm. So, the Eq. (4) can be simplified as the following equation:

$$\eta = 2.1268 \times 10^5 M \text{ (mPa s)}$$

where M is the rotary torque, N m. It is obvious that the accurate measurement of the rotary torque is the key factor to fast measure the viscosity of liquids. In this study, a static torque sensor (The model of the sensor is AKC-17A and it is made by China Academy of Aerospace Aerodynamics, Beijing. The measurement range is 0–0.05 N m and the precision is 0.1%) is used to monitor the rotary torque and the viscosity of measured Newtonian fluids ranges from 10 mPa s to $10^5 \text{ mPa} \text{ s}$.

3. Structure and working process of new device

The block diagram of the new device, shown in Fig. 3, is made up of the multi-functional detector, the viscosity measurement cell, the gas feeding cell, the control pannel and computer. Under the control of computer, the surface tension, the density, the viscosity

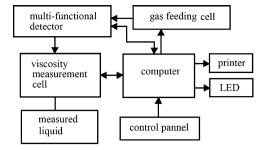


Fig. 3. Block diagram of new device for fast measuring surface tension, density and viscosity of liquids.

and the temperature of liquids can be fast measured by the multifunctional detector and the viscosity measurement cell. The gas feeding cell is mainly composed of the micro-air pump, the electromagnetic reversing valve and the floater flowmeter. Through the electromagnetic reversing valve and the floater flowmeter, pressurized air is blown in the capillary of the multi-functional detector. The capillary diameter and the pressure curve in bubbles can be automatically measured and thus the surface tension of liquids can be calculated according to Eq. (1). The computer carries out the functions of acquiring data, controlling the actions of the device, displaying and printing the results. The multi-functional detector, shown schematically in Fig. 4, is designed to realize fast measurement of the surface tension, the density, the viscosity and the tempereture of liquids. A ring-shaped gas chamber is used to buffer pressurized air in order to form the stable bubble. Pressurized air is blown in liquids through the capillary and the pressure in the capillary is monitored by the pressure difference sensor connected with the gas chamber. A negative temperature coefficient sensor (The model is MJSP503 and it is made by Shanghai Minjie Electronic Co., Ltd. The measurement range is 0-125 °C and the precision is $\pm 0.5\,^{\circ}\text{C}$) is used to a thermistor and to measure the temperature of liquids because it has the merits of good linearity, high precision and stability. The internal cylinder made of stainless steel is joined to the torque sensor and the load sensor by the connecting rod.

The amount of the liquid for a measurement is 150–180 mL. When different function key is operated, the device can fast measure single parameter, also multi-parameters of the surface tension, the density and the viscosity. The period of measuring multi-parameters is below 20 s, and the period of measuring single parameter is below 10 s. Consequently, the new device is adequate for fast measurement of the physical parameters of liquids.

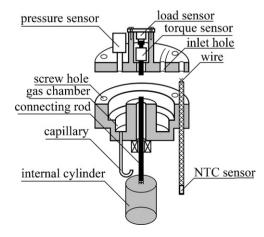


Fig. 4. Structure of multi-functional detector.

Table 1Experimental results of surface tension, density and viscosity of the glycol and glycerol

| Object | T(K) | $\sigma (\text{mN m}^{-1})$ | | D _i (%) | ρ (kg m ⁻³) | | D (%) | η (mPa s) | | D _i (%) |
|-----------|------|------------------------------|-------|--------------------|------------------------------|--------|-------|-----------|--------|--------------------|
| | | $\overline{x_i}$ | A_i | | $\overline{x_i}$ | A_i | | χ_i | A_i | |
| Glycol | 295 | 48.5 | 48.4 | 0.2 | 1122.5 | 1113.6 | 0.8 | 21.5 | 21.2 | 1.4 |
| | | 48.3 | 48.1 | 0.4 | 1121.2 | 1116.7 | 0.4 | 21.3 | 21.5 | -0.9 |
| | | 48.3 | 48.7 | -0.8 | 1121.0 | 1113.2 | 0.7 | 21.4 | 21.2 | 0.9 |
| | | 48.0 | 47.5 | 1.0 | 1122.6 | 1111.5 | 1.0 | 21.5 | 21.4 | 0.5 |
| | | 48.1 | 48.2 | -0.2 | 1120.3 | 1112.5 | 0.7 | 21.6 | 21.4 | 0.9 |
| | | 46.9 | 47.3 | -0.8 | 1120.5 | 1113.8 | 0.6 | 21.7 | 21.8 | -0.5 |
| | | 48.2 | 48.0 | 0.4 | 1119.6 | 1116.3 | 0.3 | 20.8 | 21.1 | -1.4 |
| | | 48.3 | 48.4 | -0.2 | 1118.2 | 1120.4 | -0.2 | 21.0 | 20.8 | 1.0 |
| | | 47.8 | 47.9 | -0.2 | 1119.8 | 1114.2 | 0.5 | 20.9 | 20.7 | 1.0 |
| | | 47.1 | 46.9 | | | | | | | |
| \bar{x} | - | 47.95 | 47.94 | -0.02 | 1120.7 | 1114.7 | 0.54 | 21.26 | 21.21 | 0.24 |
| CV (%) | - | 1.16 | 1.12 | - | 0.12 | 0.23 | - | 1.56 | 1.56 | - |
| Glycerol | 295 | 63.0 | 63.4 | -0.6 | 1268.8 | 1256.2 | 1.0 | 1503.2 | 1492.8 | 0.7 |
| | | 63.1 | 62.8 | 0.5 | 1267.1 | 1260.8 | 0.5 | 1499.5 | 1502.5 | -0.2 |
| | | 63.3 | 63.2 | 0.2 | 1268.3 | 1259.5 | 0.7 | 1508.8 | 1499.8 | 0.6 |
| | | 62.5 | 62.8 | -0.5 | 1269.1 | 1266.6 | 0.2 | 1498.7 | 1503.2 | -0.3 |
| | | 62.5 | 63.1 | -1.0 | 1266.7 | 1262.9 | 0.3 | 1492.3 | 1498.3 | -0.4 |
| | | 62.7 | 62.5 | 0.3 | 1269.5 | 1259.4 | 0.8 | 1497.1 | 1512.2 | -1.0 |
| | | 63.2 | 62.7 | 0.8 | 1266.5 | 1258.9 | 0.6 | 1504.9 | 1497.4 | 0.5 |
| | | 62.6 | 62.4 | 0.4 | 1260.0 | 1261.3 | -0.1 | 1499.2 | 1496.2 | 0.2 |
| | | 63.0 | 62.6 | 0.6 | 1268.4 | 1263.3 | 0.4 | 1492.1 | 1499.6 | -0.5 |
| | | 62.8 | 62.9 | -0.2 | 1269.2 | 1261.6 | 0.6 | 1499.0 | 1509.6 | -0.7 |
| \bar{x} | - | 62.87 | 62.84 | 0.02 | 1267.4 | 1261.0 | 0.5 | 1499.5 | 1501.2 | -0.12 |
| CV (%) | - | 0.46 | 0.50 | - | 0.26 | 0.23 | - | 0.35 | 0.40 | - |

Note: x_i is the measured value, A_i is the reference value, and D_i is the relative error between x_i and A_i .

Once the computer is launched, the capillary diameter will be automatically measured and stored. When the functional key is operated, the multi-functional detector will be driven forward, and the capillary, the internal cylinder and the temperature sensor will be dipped in the fixed depth of the liquid. Then, the corresponding parameter will be fast measured.

Supposing that the "multi-parameters" key is operated, the process of fast measuring the multi-parameters of liquids is as follows. After the internal cylinder is entirely dipped in liquids, the buoyancy caused by liquids is monitored by the load sensor and the data are automatically acquired and stored by computer. Then, the computer launches the micro-air pump, and the pressurized air is blown in the capillary through the electromagnetic reversing valve and the floater flowmeter. In the following 5 s, bubbles will form one after another. The real-time pressure in the capillary is monitored by computer. After a period of 5 s, the micro-air pump is closed. Then, the DC motor is started up and thus the external cylinder and liquids will rotate with constant angular velocity. The rotary torque caused by viscous force is monitored by the torque sensor. When the torque recorded by computer keeps stable, the DC motor will stop and the temperature of liquids is measured by the temperature sensor. After all data are obtained, the multi-functional detector will be lift. Simultaneously, computer will calculate the amplitude and the number of bubbles, and the surface tension, the density, the viscosity and the temperature of the liquid are fast calculated. In the end, all the results will be displayed on LED and printed by micro-printer. After all parameters are measured, the internal cylinder can be uninstalled and cleaned.

4. Experiments and discussions

To assess the accuracy and the reproducibility, the surface tension, the density and the viscosity of 99.0% glycol and 98.0% glycerol are measured by this new device at 295 K temperature. Under the same condition, the actual values A_i of these three parameters are also measured by the KSV's SIGMA700 (which is used to measure

the surface tension according to the Wilhelmy method), the Anton-Paar's DMA4500 densimeter (which is used to measure the density according to oscillating U-tube principle) and the NXS-11A viscosimeter (which is used to measure the viscosity according to the rotating cylinder method), respectively. The experimental results are shown in Table 1.

The average value $(\bar{x} = (1/n) \sum x_i)$ and the relative error $(D_i = ((x_i - A_i)/A_i) \times 100\%)$ are used to appraise the measurement accuracy, and their calculating results are shown in Table 1. As for the glycol, the average values of the surface tension, the density and the viscosity measured by the device are 47.95 mN/m, 1120.7 kg/m³ and 21.26 mPas, respectively. They agree very well with the reference values, which are $47.94 \,\mathrm{mN/m}$, $1114.7 \,\mathrm{kg/m^3}$ and $21.21 \,\mathrm{mPa}$ s. Their relative errors of single measurement are below $\pm 1.0\%$, $\pm 1.0\%$ and $\pm 1.4\%$, and the average relative errors are very small (about -0.02%, 0.54% and 0.24%, respectively). Similarly, for the glycerol, the average values of these three parameters measured by the device are 62.87 mN/m, 1267.4 kg/m³ and 1499.5 mPa s, which also agree very well with their reference values. The average relative errors are 0.02%, 0.50% and -0.12%. So, the accuracy of the new device is high. In Table 1, the average relative error of the density is big compared with that of the other parameters and it is always positive. When the density is measured, the volume of the connecting rod dipped in liquids is very small and the measurement error is below $\pm 1.0\%$, so it can be neglected and thus the measured value of the density will little higher than the actual value.

According to national standard GB4649-93, the surface tension, density and viscosity of 99.0% glycol and 98.0% glycerol at 295 K temperature are $48.5 \,\mathrm{mN/m}$ and $62.4 \,\mathrm{mN/m}$, $1112.6 \,\mathrm{kg/m^3}$ and $1261.2 \,\mathrm{kg/m^3}$, $21.0 \,\mathrm{mPa}\,\mathrm{s}$ and $1505 \,\mathrm{mPa}\,\mathrm{s}$. So, according to the results of Table 1, both the average values measured by the device and the average reference values agree with that provided by GB4549-93. For the glycol, the average relative errors of surface tension, density and viscosity between the measured values and the GB4549-93 values are -1.13%, 0.73% and 1.24%, however, the average relative errors between the reference values and the GB4549-93

values are -1.15%, 0.19%, and 1.00%. Also, for the glycerol, the former errors are 0.75%, 0.49%, and -0.37% while the latter errors are 0.71%, -0.02%, and -0.25%. Therefore, compared with the GB4549-93 values, they have high measurement precision and the results measured by other apparatus can be the reference values of this new device.

The coefficient of variation is introduced to appraise the reproducibility of the measurement results and it can be calculated according to Eq. (6) [23]:

$$CV(\%) = \frac{\sqrt{\left(\sum_{i=1}^{n} (x_i - \bar{x})^2\right)/(n-1)}}{\bar{x}} \times 100$$
 (6)

where CV is the coefficient of variation, \bar{x} is the average value, x_i (i = 1,..., n) is the value of single measurement. The coefficient of variation is calculated by Matlab and the results are also shown in Table 1. For the glycol, the CV of surface tension, density and viscosity measured by the device are 0.46%, 0.12%, and 1.56%, and the CV of the reference values are 1.12%, 0.23%, and 1.56%. So, the coefficient of variation is approached to each other. For the glycerol, the CV of the measured values is very close to that of the reference value, which is can be seen from Table 1. Therefore, it has been testified that the reproducibility of the new device is satisfactory compared with other apparatus. Therefore, this new device can provide a tool for studying the physical properties and evaluating the quality of the liquid.

5. Conclusions

In this work, a new device for sequentially fast measuring the surface tension, the density and the viscosity of liquids has been described. It is mainly composed of the multi-functional detector, the viscosity measurement unit, the gas feeding unit and computer. The surface tension is fast calculated according to the pressure difference in bubbles, the number of bubbles and the capillary diameter, and the empirical formula has obtained by many experiments on various kinds of liquids at 293–298 K temperature range. The density is measured according to the Archimedes prin-

ciple and and the viscosity of liquids is measued according to the rotating cylinder method. The measurement range of surface tension, density and viscosity is 17–75 mN/m, 0–2.0 \times 10^3 kg/m³ and $10–10^5$ mPa s, respectively. Experiments on the glycol and glycerol show that the accuracy of the surface tension, the density and the viscosity is $\pm 1.0\%$, $\pm 1.0\%$ and $\pm 1.4\%$ and small coefficient of variation proved the reproducibility is better than other devices.

Acknowledgements

The authors would like to acknowledge W.M. Wang, Q. Sun and G.W. Gao for useful discussions and experimental support. This work was supported by the Science and Technology Innovation Talents Foundation of Harbin (Grant No. 2006RFOXG048).

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