



Contributing to accurate high pressure viscosity measurements: Vibrating wire viscometer and falling body viscometer techniques



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ABSTRACT

Two new techniques for measuring viscosities at high pressure have been implemented at the TERMOCAL laboratory in order to obtain accurate values of thermophysical properties such as viscosity, especially at high pressures.

A vibrating-wire viscometer has been developed to accurately measure viscosities over the working ranges $T = (283.15 \text{ to } 423.15) \text{ K}$ and $p = (0.1 \text{ to } 140) \text{ MPa}$. The setup of the equipment includes calibration with toluene and its validation with n-dodecane.

A falling body viscometer able to measure viscosities at $T = (253.15 \text{ to } 523.15) \text{ K}$ and $p = (0.1 \text{ to } 140) \text{ MPa}$ is also presented in this work. Results of calibration with toluene and its verification with n-heptane and n-dodecane are reported.

The detailed uncertainty budgets for both techniques are included in this work. Moreover, the paper studies the compatibility of the results obtained using both techniques according to their corresponding uncertainties in order to obtain reliable data. New viscosity measurements of 1,2,4-trimethylbenzene and 2,2,4-trimethylpentane have been performed and included in the paper.

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1. Introduction

Most current techniques for measuring the viscosity of fluids require calibration with an appropriate reference fluid at the temperature and pressure measurement [1]. This imposes an upper limit on the achievable accuracy due to the lack of reference fluids, particularly at extreme pressures and temperatures. In fact, all viscosity measurements must be accredited in accordance with the viscosity of water at 20 °C under atmospheric pressure [2]. Yet, there is considerable controversy surrounding the value of the standard reference in these conditions, and there have been several new determinations of the property from the original measurement made in 1952 by Swindells *et al.* [3]. However, the viscosity value used as a reference has remained intact, despite its uncertainty [2].

Recent studies on viscosity revolve around two areas of great interest to researchers: developing techniques which can be used to determine viscosity over wide ranges of temperature, pressure and viscosity, and searching for standard liquids that can serve

as a reference to calibrate viscometers. Hence, our research group's interest in implementing two new viscometers which can work at high pressure based on different measurement principles.

2. Experimental section

2.1. Experimental techniques

2.1.1. Vibrating wire viscometer (VWV)

The first technique is a vibrating wire viscometer. Its measurement principle consists of a circular section wire of radius R , length L ($L \gg R$) and known density, tensioned and anchored at both ends [4]. It is surrounded by the fluid whose viscosity is being determined. The wire is oscillated on a plane perpendicular to its axis through an initial displacement in the initially stationary fluid. The equipment is used in forced mode, generating a disturbance and maintaining it in time. The resonance curve characteristics of the wire transverse oscillations are studied since they are determined by the viscosity and density of the fluid [5,6].

The Navier–Stokes equation allows viscosity to be calculated using the frequency and the damping of the wire oscillatory motion, both in vacuum and in the fluid of interest. The mathematical

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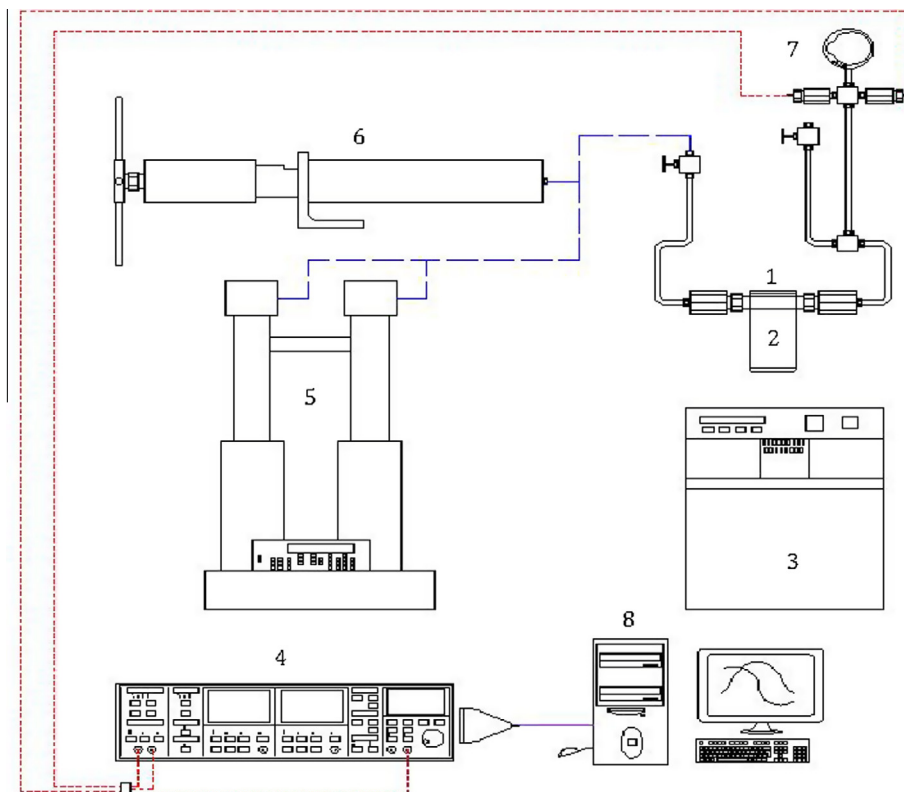


FIGURE 1. Schematic view of the technique. (1) Pressure vessel with the sensor inside; (2) magnet; (3) thermostatic bath; (4) lock-in amplifier; (5) syringe pumps; (6) pressurized cylinder; (7) digital manometer; (8) computer.

model imposes certain conditions which can be taken into account when designing the equipment, and there is a correction since the wire is not immersed in an infinite sample volume [6,7]. If the wire radius is measured accurately, no calibration liquid is necessary, such that it would be an absolute measuring method. The viscosity measurement range varies depending on the diameter of the wire used, such that the same equipment can operate in different ranges by simply changing the diameter, although it is still not possible to use it for high viscosities. In recent years, studies have been conducted aimed at increasing the viscosity range of these techniques [8,9]. Its main advantage is that it may be used to make absolute measurements or may be calibrated based on a small number of data.

The circulation of a constant sinusoidal current through the wire, combined with the constant magnetic field, produces the vibration of the wire. The electromotive force (EMF) generated through the vibrating wire can be measured with a lock-in amplifier in two stages, and is the sum of two complex terms V_1 and V_2 [10,11].

V_1 is the voltage due to the electrical impedance of the fixed wire and is expressed by the following equation:

$$V_1 = a + ib + icf, \tag{1}$$

where f is the frequency, i is the imaginary number and a, b, c are adjustable parameters determined by regression that account for the electrical impedance of the wire and absorb the offset used in the lock-in amplifier to ensure that the voltage signal is detected in the most sensitive range.

V_2 comes from the wire movement and is proportional to the speed of the wire. It is expressed by the following equation:

$$V_2 = \frac{i\Lambda f}{f_0 - (1 + \beta)f^2 + (\beta' + 2\Delta_0)f^2 i}, \tag{2}$$

where Λ is the amplitude, f is the driven frequency, f_0 is the resonance frequency in vacuum, Δ_0 is the logarithmic decrement of the wire in vacuum, $\beta = k \cdot \rho/\rho_s$ is the additional mass of the fluid and β' the damping due to the fluid viscosity ($\beta' = k' \cdot \rho/\rho_s$); k and k' are functions of $\Omega = (2\pi f \rho R^2)/\eta$. Here, ρ and η are the density and the viscosity of the fluid, respectively, and R and ρ_s are the radius and density of the wire.

Using the approximation $f_o^2 \approx (1 + \beta)f_r^2$ [12], viscosity can be expressed by equation (3):

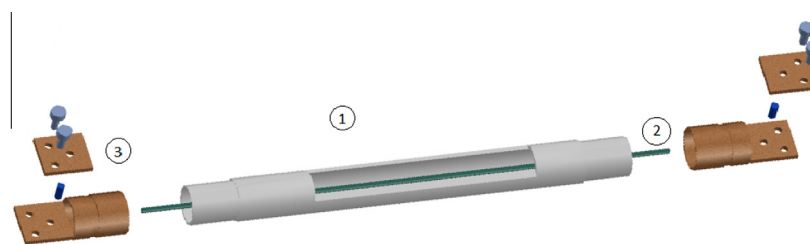


FIGURE 2. Vibrating-wire sensor. (1) Flow tube; (2) tungsten wire; (3) support terminal, clamping plate, alignment pin, and M2 screws.

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