Indirect and direct temperature calibration methodology of a rheometer using a Newtonian reference material

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Abstract. The purpose of the present communication is to compare the influence of temperature measurement and measuring geometries on viscosity measurements results of a Newtonian liquid when using a HAAKE MARS III rheometer.

1. Introduction

Viscosity is a measure of internal friction when a fluid is subjected to external forces and resists to flow due to internal friction [1]. The absolute viscosity, often called the dynamic or simple viscosity, of a fluid is the tangential force per unit area which is required to move one horizontal plane with respect to the other plane at unit velocity, when a unit distance is maintained between these two fluid planes. This absolute viscosity is a product of the kinematic viscosity and the fluid density [1]:

\[ \eta = \frac{\tau}{\dot{\gamma}} = \frac{F}{A \dot{\gamma}} = \frac{u}{\nu} \]

Newton’s Viscosity Law describes the state in which the viscosity is constant regardless of shear stress or shear rate. These are called Newtonian fluids. Many simple petroleum products, single grade, fully formulated automotive oils and industrial lubricants obey Newton’s Viscosity Law. Viscosity can be measured using viscosity measurement techniques. The most widely are using a capillary viscometer or a rotational viscometer or other viscometer systems [1].

Rheometers are mainly used to measure the viscosity of all type of liquids, i.e. Newtonian and non-Newtonian liquids. Rheometers are crucial in cosmetics, pharmaceuticals, food, polymers, petrochemicals industry. There they are used for quality control, for development of new formulations according to consumer preferences and to manufacturing requirements.

The temperature is one of the essential influencing quantities on measured viscosity [2]. A quantitative mismatch of viscosity measured by different rheometers or laboratories for identical samples is often due to errors in determining the true sample temperature. For example, a temperature difference of 1 K may yield a viscosity mismatch between 2% and 50%, depending on the type of sample and the reference temperature [3]. An accurate way of checking the true sample temperature is by placing a calibrated thermocouple or platinum resistance thermometer (PRT) directly in the sample (Figure 1) or by replacing the sample by, e.g., an aluminum disc, containing one or more properly mounted and calibrated thermocouple(s) [3]. Using such a setup, a plot of nominal (set) versus actual temperature is obtained. These data can be used for either correcting the experimental temperature data or for adjusting the necessary set temperatures of the rheometer. However, in other measuring geometries, such as concentric-cylinders (CC), the measurement gap does not withstand a reference thermometer. Other way of checking the temperature performance of the instrument is using a temperature calibration sample, i.e. a reference liquid which viscosity was determined

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with capillary viscometers. At the transition, the specified temperature of the temperature calibration sample allows a direct check of the temperature signal of the rheometer albeit for only one temperature.

2. Methods and Materials

2.1. Measuring Instruments
In this study was used a Searle type rheometer (Mars III, HAAKE Thermo Scientific). This rheometer has coupled a Peltier temperature module, that change temperature within the mid temperature range, from -60 °C for parallel plate as well as cone and plate measuring geometries or from -40 °C for coaxial cylinders up to 200 °C, using the indication of internal platinum resistance thermometers (PRT).

The temperature of the sample in the measuring gap was measured using a reference 100 Ohm PRT (MKT50, Anton Paar).

2.2. Materials
The rheometer temperature calibration was done using certified reference oil APN 26 (1-Decene based) from Paragon Scientific. The expanded uncertainty of this material can be considered negligible (below 0.2 %) comparing to rheometer’s measuring dispersion (above 1 %).

2.3. Temperature calibration methods
Two temperature calibration methods were used in the interval of [20, 60] ºC: a direct calibration method for rheometer with plate-plate (PP) geometry; and an indirect method with a concentric-cylinder (CC) geometry.

2.3.1. Direct calibration method
For checking the true sample temperature inside the measuring gap of the PP geometry a calibrated 100 Ohm PRT (MKT50, Anton Paar) was used in direct contact with a sample of APN 26 oil, as illustrated in Figure 1. The rheometer’s temperature measurement error was determined by comparison of its indication against the temperature measured by the reference 100 Ohm PRT located inside the sample in the measuring gap.

![Figure 1](image)

2.3.2. Indirect calibration method
The calibration of sample temperature of the rheometer (Mars III, HAAKE Thermo Scientific) when using a CC geometry was performed by indirect method by using a certified reference oil (APN26, Paragon Scientific), with a dynamic viscosity of 50.06 mPa·s at 20 °C, with the measurement program described on Table 1. Prior and after the calibration and adjustment the same certified reference oil (APN26, Paragon Scientific) was measured using a cone-plate (CP) geometry using the measurement program described on Table 1.

| Test t (°C) | Time for t stab. (s) | Number of measurements | Time of each step (s) | Shear rate (s⁻¹) |
|-------------|----------------------|------------------------|----------------------|-----------------|
| 20; 25; 40; 50; 60 | 1800                | 10                     | 60                   | 10              |
3. Results
In order to compare the relative viscosity errors before and after the temperature adjustment and also the difference between two types of geometry (CP and CC) using a rheometer (Mars III, HAAKE Thermo Scientific) a Newtonian reference oil (APN 26) was used in the temperature range between 20 °C and 60 °C. Viscosity measurements were performed with CP geometry before and after the adjustment of the rheometers’ internal PRTs. The CC geometry was only used after the temperature adjustment. The results are shown in Table 2.

Table 2. Viscosity relative error of the tested rheometer (Mars III, HAAKE Thermo Scientific): with the CP geometry before and after temperature adjustment and for CC geometry after adjustment, in the interval from [20, 60] °C, using certified reference oil (APN26, Paragon Scientific).

| Test t (°C) | Viscosity relative error (%) |
|-------------|-------------------------------|
|             | CP Before Adj. | CP After Adj. | CC After Adj. |
| 20          | 11%             | 5%             | 4%             |
| 25          | 5%              | 5%             | 1%             |
| 40          | -8%             | 7%             | -4%            |
| 50          | -16%            | 5%             | -1%            |
| 60          | -23%            | 11%            | 2%             |

Legend: Adj. – adjustment; Rheometer measuring geometries: CP – Cone-plate and CC – concentric cylinder.

The viscosity errors obtained for CP geometry before and after the temperature adjustment of rheometers’ internal PRTs are significantly different. For this measuring geometry it was observed that at 20 °C (without temperature adjustment) a relative viscosity error of 11 % and after temperature adjustment a 5 % error. This difference is even more accentuated for the temperature of 60 °C where the first case shows an error of -23 % and the second one an error of 11 % (Table 2).

4. Conclusions
The rheometer’s adjustment of its internal PRTs allowed a reduction of 2 to 12-fold of viscosity errors. Tests made with CC geometry obtained values closer to those indicated for the reference material with the measurements made in CP geometry after temperature adjustment.

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