CALIBRATION OF AN ABSOLUTE VISCOMETER AT
LOW TEMPERATURE

S. LOREFICE
e-mail: S.Lorefice@imgc.to.cnr.it

SUMMARY
This paper is a report about a practical method used at the IMGC-CNR to characterise absolute viscometers at ambient
and also low temperatures by means of additive-free mineral Newtonian oils having a dynamic viscosity (traceable to
the Italian national standard scale) in the range 1 mPa s to 10000 mPa s.

Keywords: Viscosity, Viscometer, Standard, Traceability

1 INTRODUCTION
According to the quality management requirements, the
knowledge of all fluid properties must be traceable to
national or international standards, which necessarily
involves the calibration of instruments and the statement
of their measurement uncertainty. As regards viscosity,
there are many commercially available instruments and
apparatuses for the measurement of this quantity,
embodying different measuring principles.

The rotational principle finds a wide application in
research and industry [1]. Viscometers which are able to
work on such principle, called rotational viscometers,
can characterise the structure and flow properties of soft
solids and of several structured fluids (emulsions,
suspensions, and gels) as well as the temperature at
which an oil becomes unsuitable for lubrication of a
mechanical device such as an engine [2]. They are used
to measure dynamic or absolute viscosity, with a selec-
ted velocity gradient, using differently shaped rotors.

Among the different models, the design advantages and
disadvantages are pointed out from their features as:
range of fluids that can be measured, versatility in
general use, capability to perform rheological studies,
ease of treatment of data, and cost.

Unfortunately, only viscometers intended for R&D
applications seem to be apt to be calibrated in terms of
their typical features such as: speed, geometry and
sensitivity [3]. The calibration of users’ viscometers for
general purpose is usually carried out by comparison
against Newtonian oils, whose viscosities are known at
the usual testing temperatures, that range from 20 °C to
25 °C.

However, the calibration of most instruments is
temperature dependent, therefore it is necessary to
classify them in the whole prospective range of
working temperatures [4, 5]. An investigation aimed at
this goal has been carried out by the Istituto di
Metrologia “G. Colonnetti” (IMGC) of the Italian
National Research Council (CNR).

The paper presents the results of this exercise, the
method and the procedure used for characterising such
viscometers by comparison with Newtonian oils of
known viscosity both at ambient temperature and at
temperatures in the range –9 °C to 0 °C.

2 THEORY
A rotating solid body (spindle), immersed in a
homogeneous and isotropic fluid, experiences a viscous
drag or retarding force related to the fluid viscosity, $\eta$.

The general relation for Newtonian fluid, between the
shear stress $S$ and the shear rate $\dot{\gamma}$

$$S = \eta \dot{\gamma}$$

becomes

$$\frac{M}{4\pi h} \left( \frac{R_b^2 - R_c^2}{R_b^2 R_c^2} \right) = \eta \Omega$$

if we consider a system of two coaxial-cylinders,
consisting of a spindle of radius $R_b$, immersed in a
Newtonian fluid to a height $h$, which is caused to rotate
inside a cup of radius $R_c$ with an angular velocity $\Omega$
against an opposing torque $M$ under the following
assumptions:

- the fluid is incompressible;
- the streamlines of flow are circles on a horizontal
  plane perpendicular to the axis of rotation;
- there is no relative motion between the surface of the
  rotating body and the fluid in close contact with the
  body, i.e., no slippage;
- the system is iso-thermal.

Therefore the viscosity $\eta$ can be expressed as:

$$\eta = K \frac{M}{\Omega}$$

where $K$ is an instrumental constant, which takes into
account the geometry of the measuring system.

In the present investigation, a DV-II+ digital viscometer
by Brookfield has been used. It is a instrument suitable
for quality supervision of the rotational type, supplied
with a set of four different spindles: LV1, LV2, LV3 and
LV4, beside an additional coaxial-cylinder
accessory, the “UL Adaptor”. In fact, an appropriate
combination of a specific spindle and a suitable rotation
speed allows making measurements in a wide range of
viscosities.

The instrument’s principle consists in measuring the
torque necessary to overcome the viscous resistance of
the fluid against a spindle, which rotates with constant
The spindle is driven by a synchronous motor with eighteen programmed speeds.

A built-in microprocessor calculates the values of viscosity on the basis of the measured torque, the shear rate (speed) and the used spindle. The instrument is also provided with a RTD thermometer, so that the fluid temperature can be measured.

The screen of the DV-II+ displays the following data:

- the viscosity, measured in centipoise, “cP” units (1 cP = 1 mPa s);
- the torque, computed in “dyne cm”, but displayed as a “% of full scale”
- the shear stress, whose value is given only for the accessory UL Adaptator, in “dyne/cm²” units (1 dyne/cm² = 0,1 N/m²);
- the shear rate, given only for the accessory UL Adaptator, in units of reciprocal seconds (1/s);
- the temperature of fluid, in °C;
- the spindle rotation speed, in “rounds per minute” or “RPM”;
- the code for the selected spindle in use.

Further information can be displayed during operation. Measurement data are preceded by a question mark when the viscometer operates at spindle speeds that produce a torque lower than 10 % of the range (under-range message). When the torque exceeds 100 % (over-range) an error message “EEEE” is displayed.

3 REFERENCE STANDARD

As already mentioned, additive-free mineral oils with known viscosity in the range 1 mPa s to 10000 mPa s are used at the IMGC as reference measuring standards, able to link up the user’s instruments with the primary viscosity standard (figure 1).

The viscosity values for each oil have been determined by using comparative measurements with the more suitable reference glass capillary viscometers, at the five nominal temperatures of –10 °C, 0 °C, 5 °C, 20 °C and 25 °C. The instrumental constants of such reference viscometers were directly traceable to the IMGC viscosity scale. In order to achieve the best accuracy, in addition to control of the temperature within 5 mK at 20 °C and the careful measurement of the efflux time, different sources of error in the viscometer calibration have been considered [6].

By using capillary viscometers the so-called kinematic viscosity \( \nu \) is observed, therefore it was necessary to carry out measurements of the density \( \rho \) at the above five temperatures, in order to determine the corresponding dynamic viscosities. The relationship between these quantities is recalled below:

\[
\eta = \nu \rho \tag{4}
\]

Oil density was determined by means of a pycnometer.

4 CALIBRATION AND TESTING

The variables that can affect the viscometer measurements may be related to the used instrument, or to the test fluid. Variables related to test fluid are related to its rheological properties, while instrument variables include viscometer design, the spindle geometry system used, the sample volume, the spindle speed (or shear rate). First of all, however, it is necessary to ensure that viscosity measurements are made under laminar flow conditions, because turbulence produces a false (too high) viscometer reading, directly related to the degree of turbulence in the fluid.

![Figure 1. The IMGC hierarchy scheme as regards viscosity in Italy.](image-url)
The calibration consists in the determination of the correction factor $F$ for each spindle in its whole viscosity range, by changing its rotational speed. The $F$ factor is defined as the ratio between the viscosity value of the standard liquid ($\eta_{\text{std}}$) and the average of the viscometer readings ($\eta_{\text{rdg}}$)

$$F = \frac{\eta_{\text{std}}}{\eta_{\text{rdg}}}$$

at the test temperature. The use of more Newtonian standards does not change the correction factor, when the temperature of the fluid remains the same.

If it is required to use the viscometer at a different temperature, it is necessary to determine a new correction factor.

A temperature coefficient of the correction factor $\alpha_F$ can be determined at the two extremes of operating temperature:

$$\alpha_F = \frac{F_{\text{ref}} - F_t}{T_{\text{ref}} - T_t}$$

where $F_{\text{ref}}$ is the correction factor determined at the reference temperature $T_{\text{ref}}$ and $F_t$ is the new correction factor determined at the extreme temperature $T_t$. That allows to obtain viscosity measurement at a different temperature $T$ (within the range):

$$\eta(T) = \eta_{\text{rdg}} F_{\text{ref}} \left(1 + \alpha_F \left(T_{\text{ref}} - T\right)\right)$$

The set up used at the IMGC for the calibration of the rotational viscometer is shown in Figure 2.

Figure 2. The IMGC experimental apparatus for rotational viscometers calibration.

The calibration procedure has been carried out separately for each measuring system (spindle), in accordance with the following conditions:

- **Preparation**: at least two standard oils have been used, having Newtonian behaviour, with viscosity values in the range defined by the selected spindle and speeds.
- **Calibration**: the viscosity of the first standard has been measured by means of the selected spindle at different constant speeds. The viscosity measurements have been repeated with the second standard under the same operating conditions, and particularly at the same fluid temperature.
- **Evaluation**: only the recorded viscometer readings that yielded a viscosity value between 10 % and 100 % of full viscosity range (torque/scale) have been retained for evaluation.
- **Computation**: a calibration table has been produced for the select spindle, giving the correction factor at the reference temperature as well as the temperature coefficient, the same procedure having been repeated at a different temperature.

The results obtained from the instrument calibration related to the “UL Adaptaor” spindle are shown in the Table 1, just as an example.

In each row of the table, referring to the selected spindle speed, are presented: the shear stress and the shear rate displayed by the instrument, the testing temperature and the average of viscosity readings, which is obtained from three cycles up and down, with its standard deviation.

The table also gives the reference viscosity value of the standard, which is determined at the testing temperature; the correction factor $F$, the full scale viscosity range $f.s.$, which is calculated under instruction of the instrument’s operation manual and the relative uncertainty $U_{f.s.}$ to be referred to full-scale values of viscosity.

An addition to the table shows: the average of correction factors $F_{\text{ref}}$ at the reference temperature of 22 °C, the average of temperature coefficient $\alpha$, and their uncertainties. The calibration uncertainties take into account different contributions due to:

- uncertainty of viscosity standard oils;
- reading repeatability;
- influence of temperature
The uncertainties are given as expanded uncertainty with a coverage factor $k = 2$ (95 %), in agreement with the EA-4/02 guide suggestions.

## 5 CONCLUSIONS

The procedure used at the IMGC – CNR for characterising rotational viscometers for general purpose has been presented. The method, which is based on a comparison with reference Newtonian oils at the usual range of ambient temperatures, has been also applied in the temperature range –9 °C to 0 °C.

The results obtained from the instrument calibration have been shown, relating to a coaxial-cylinder measuring system.

## 6 REFERENCES


