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The Italian primary kinematic viscosity standard: The viscosity scale

S. Lorefice[a,*], F. Saba[a,b]

[a] Istituto Nazionale di Ricerca Metrologica – INRIM, Strada delle Cacce, 91, 10135 Torino, Italy
[b] Politecnico di Torino – Dipartimento Energia, Corso Duca degli Abruzzi, 24, 10129 Torino, Italy

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ABSTRACT

The base for all viscosity measurements is the internationally accepted viscosity of double-distilled water at 20 °C. Using this water and liquids of ever greater viscosity, INRiM derives by a step-up procedure the constants for its master "glass capillary" Ubbelohde viscometers, and these are used in their turn to determine the viscosity of undetermined liquids and reference materials in the kinematic viscosities range from about 0.4 mm^2/s to above 700,000 mm^2/s with temperatures from 10 °C to 150 °C. From the results obtained on recalibration of the INRiM viscometers and the comparisons made with other independent laboratories, it is concluded that the present scale is realized with an uncertainty of about 0.2% at low viscosities rising to about 0.8% at high viscosities. This enable INRiM to provide traceability to Italian viscosity measurements as well as calibrating various types of viscometers for the highest measurement accuracy i.e. glass capillary, cups, rotational. This paper addresses the relevant procedure on the realisation of the Italian kinematic viscosity scale (viscosity standard) which was conducted over a number of years. The different sources of error in the viscometer calibration, mainly due to the contribution by the kinetic energy of the fluid stream, surface tension variations and, finally, buoyancy are considered. Special emphasis is also given on the uncertainty evaluation and in the international activities which provide evidence of the present measurement capabilities of the Italian laboratory and ensures smaller measurement uncertainties in the future.

1. Introduction

An increasing number of National Metrology Institutes (NMIs) and accredited calibration laboratories provide viscometer calibrations and supply calibrated reference liquids in a wide viscosity range. Metrological traceability is a key technical requirement of the ISO/IEC 17025:2005 standard, applied to calibration and testing laboratories that intend to prove their technical competence [1].

Viscosity measurements are important in many fields of both industry and research. Viscosity is a technological quantity which concerns the flow of matter. It is a physical quantity related to the transport properties. The viscosity measurements find great importance both in rheology through the studies of flow of matter e.g., mainly liquids but also soft solids or solids under conditions in which they flow rather than deform. Also in tribology through the mechanisms of friction, lubrication, and wear of interacting surfaces that are in relative motion.

The wide range of viscosity values of the more widely used fluid products, the role of working conditions (temperature, pressure, shear stress, time, time, etc.), the different flow behaviour (Newtonian and non-Newtonian), and the different ways in which viscosity can be measured and expressed, made it difficult to achieve a complete harmonization of the measuring methods and to realize a suitable standard for each working condition in the viscosity field. In order to overcome these drawbacks, each device, method and procedure for viscosity measurement should be traceable to the accepted internationally viscosity value of double distilled water at 20 °C [2,3].

The hierarchy scheme of viscosity instrumentation recommended by the Organization Internationale de Metrologie Legale (OIML) [4] can meet this requirements. The recommended scheme establishes a documented unbroken chain of calibrations which starting from the recognized international reference standards, the water viscosity value at 20 °C, allowing us traceable and reliable viscosity measurement results with the expected uncertainty values [5]. The complete viscosity traceability chain in Italy is shown in Fig. 1, which also includes international key comparisons that are organized among NMIs. This is fundamental for the highest agreement of viscosity among NMIs, and also provides evidence of the present measurement capabilities of the INRiM and ensures smaller measurement uncertainties in the future.

The objective of this paper is to outline the realization of the Italian primary viscosity standard, named the viscosity scale, considering the different sources of error in the viscometer calibration, mainly due to the contribution by the kinetic energy of the fluid stream, surface

* Corresponding author.
E-mail address: s.lorefice@inrim.it (S. Lorefice).

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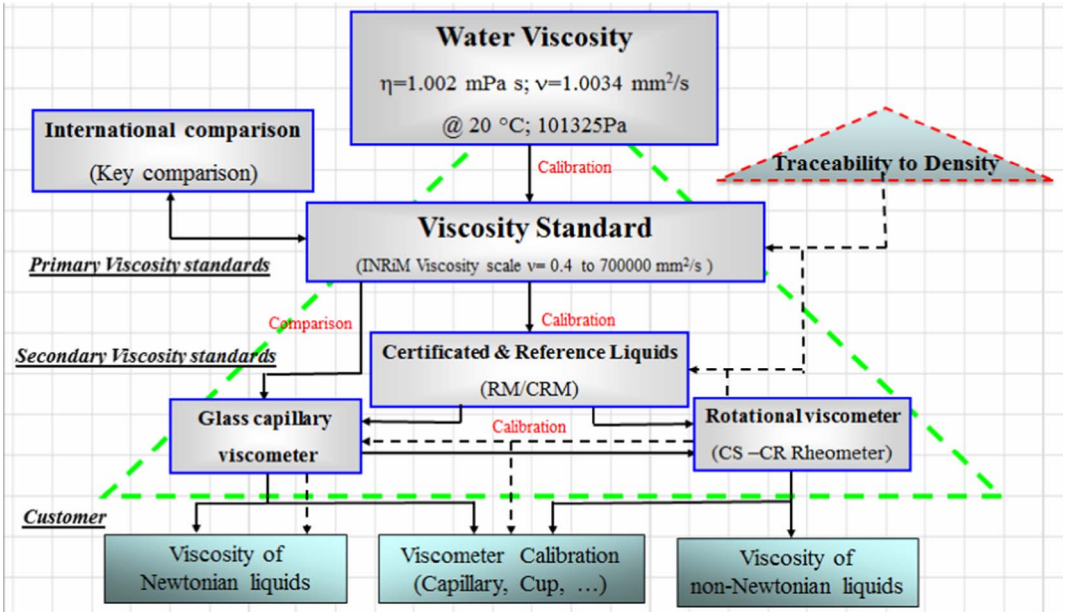

Fig. 1. The Italian hierarchy regarding viscosity measurements.

tension variations and, finally, buoyancy. Special emphasis is also given to the uncertainty evaluation and in the international activities which have provided evidence of the present measurement capabilities of the Italian Metrological Institute INRiM.

2. The viscosity scale of INRiM

The INRiM viscosity standard, namely viscosity scale for Newtonian liquids, that is, liquids showing a rate of shear proportional to the applied shear stress, is an independent standard based on viscosity of water at 20 °C, with overlapping measuring ranges covering the range of kinematic viscosities from about 0.4 mm^2/s to above 700,000 mm^2/s which has been implemented over many years. At the present time, it consists of sixteen groups, each one made of two glass capillary "master" viscometers of the Ubbelohde type having 400 mm long capillaries. Each viscometer, belonging to the same group, has same nominal instrumental constant and it is identified by the number of capillary followed with the same Italian alphabet letter and the number 1 or 2, respectively. The step-up procedure starting with freshly bi-distilled water at 20 °C (ν = 1.0034 mm^2/s) in the first step to calibrate the two viscometers having the smallest capillary bore (Viscometers Nos. 0.A1 and 0.A2, respectively having each one nominal instrumental constant K = 0.001 mm^2/s^2). Liquids having higher viscosity are used to calibrate the second set of viscometers, having a larger capillary bore. This second set is then used to determine the viscosity of the next standard viscosity liquids in the scale. This last liquids are used to calibrate the third set of viscometers, and so on until all the viscometers belonging to the Viscosity Scale have been calibrated as depicted in Fig. 2. The kinematic viscosity range of the set of reference liquids and temperature used to calibrate the identified pairs of master viscometers by according the step-up procedure for establishing the Italian Viscosity Scale are shown in Table 1. The Table also shows for each identified pairs of viscometers their diameter capillary and the nominal instrumental constant. Water is widely used as reference fluid to calibrate the viscometers of 1st and 2nd group; the kinematic viscosity value of water at 20 °C (ν = 1.0034 mm^2/s), whose uncertainty is by convention assumed to be zero, is also taken into account. The use of water as a viscometric basis has the advantages that the viscosity value can be easily and precisely reproduced by double distillation in a fused silica apparatus. There are, however, two disadvantages, which are associated with the low viscosity and the high surface tension of water. This requires separate determination of the kinetic energy correction and the surface tension correction for each viscometer, and this is the major part of the calibration work.

3. Experimental

3.1. Experimental facilities

3.1.1. Viscometers

The two primary viscometers for basic calibration with water and the fifteen groups of two "master" glass long-capillary suspended-level viscometers Ubbelohde type with nominal instrumental constants: K = 0.003/0.005/0.01/0.03/0.05/0.1/0.3/0.5/1/3/5/10/30/50/100 mm^2/s^2, respectively. The internal diameter of each capillary has been manufactured in accordance with the German standard DIN 51562-1:1999 [7], 400 mm length and 5.7 ml measuring volume.

The viscometers were always fixed in the same way to a frame mounted on the wall of the laboratory. That makes the contribution to uncertainty due to vertical alignment the lowest by using the same viscometer.

3.1.2. Temperature control and measurement

Under usual working conditions, the INRiM laboratory operated at room temperature of 20 °C ± 0.5 °C.

A stirred water thermostat of 70 L in volume (Tamson – visibility bath) linked to an external cooling unit was employed for long-capillary viscometers. Its performance had previously been assessed in the range between 20 C and 60 C. Temperature gradient was found to be less than 5 mK in the whole measurement zone both as thermal stability and as uniformity of the bath [8,9], Fig. 3.

Temperature measurements are usually performed by using two Pt100 platinum resistance thermometers and an AC Bridge (ASL F17).

The uncertainty due to the temperature measuring apparatus is 10 mK.

3.1.3. Flow-time measurement and vertical alignment

Flow-time measurements is usually performed by visual observation of the meniscus passing two ring marks at the viscometer using a multiple electronic stopwatch (six individual start/stop timers) with displays to 0.01 s, accurate to within 0.0001%. An automatic viscometer reader for flow times measuring was tested, too, Fig. 4. The