

Maintenance of reference standards in the field of viscosity

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Abstract. Participation in the work of comparison in the field of viscosity, within the program conducted under the jurisdiction of ASTM (American Society for Testing and Materials), D-2 Committee, Subcommittee "Flow Properties," Newtonian Fluids) was necessary to ensure traceability of measuring unit of kinematic viscosity. Results of the comparison of the specialized participating laboratories on 4 continents, has proved annual capability of INM in the transmission unit of kinematic viscosity. Cannon Position Company in the US organizes co-operation program in the field of kinematic viscosity ASTM D 02.07. The company distributes standard substances Cannon viscosity participating laboratories and consolidate the results of the measurements. Physical-chemical laboratory has fully accepted the proposed schedule of the company Cannon. Final report of the comparison showed that in the year 2015 a number of 25 laboratories and institutes of metrology attended to the program.

1. Introduction

Since the setting up in 1938, by the man of science, inventor and Professor, Dr. Michael Cannon, firm Cannon Instrument Company had an international recognition in the field of viscosity.

The company reaches this year 79 years and has a collaboration with INM of 46 years.

Participation in the work of inter-comparison in the field of viscosity, within the framework of the program carried out under the responsibility of ASTM (D-Committee, Subcommittee 2 "Flow Properties," Newtonian fluids) has been necessary in order to ensure traceability of measuring unit of kinematic viscosity. Results of the comparison to the participating laboratories specialized on 4 continents, specialized laboratory annual capability of INM in the transmission unit of kinematic viscosity [1].

2. Presentation program ASTM

Cooperation program in the field of kinematic viscosity was discussed and adopted by the NIST (National Institute of Standards and Technology) formerly NBS (National Bureau of Standards)-Washington, in 1971. This program has been coordinated by the American society for Testing and materials (ASTM) D-02, Committee on methods of analysis in the field of petroleum, Subcommittee RASDVU (Properties, flow of Newtonian fluids). In this Position the U.S. Cannon Company uses to organize the cooperation program in the field of kinematic viscosity ASTM - D02.07.A [2].

The Cannon Company distributes standard substances to the participating laboratories and finally collect all the kinematic viscosity measurements.



2.1. Reference documents

Reference documents for participating laboratories were international standards ASTM D 445 and SR EN ISO 3104 „Petroleum products. Transparent and opaque liquids. Determination of kinematic viscosity and calculation dynamic viscosity” and ISO 3105 „Glass capillary Kinematic viscometers - Specifications and operating instruction.

2.2. The participants in the ASTM D 02.07.

In the year 2015 were invited 25 laboratories from all over the world, such as:

Cannon Instrument Company – State College

Central Office of Measures – Poland

Centro Nacional de Metrologia – CENAM (Mexico)

Chevron – Richmond, CA

Evonik Oil Additives USA – Horsham, PA

INMETRO, The Nat'l Inst. Of Metrology – Brazil

Instituto Nacional De Tecnologia Industrial (Quimica) – Argentina

Institute for Metrology (Romania)

Kenya Bureau of Standards – Kenya

Laboratorio Dr.Lantos – Argentina

Lacomel –Costa Rica

National Institute of Metrology – China

JX Nippon Oil Co. Central Technical Research - Japan

PETROCHINA Dalian Lube Oil R&D - China

Poulten Selfe and Lee Ltd. – England

Runningland Metrology & Testing (Shanghai) – China

Slovak Institut of Metrology – Slovakia

U.S. Army Primary Standards Laboratory – Repsol, SA- Spania

ZMK – Analytik – Germany

Laboratoriuo de Lubrificantes – Uruguay [3].

2.3. Reference materials

Reference materials have been delivered to the INM by the Cannon Company in April 2015 (Table1).

According to the Cannon Protocol the reporting of the results had to be done up to 1 June 2015. INM laboratory measurements proceeded in Aprilie and May 2015. The laboratory sent via fax and email the results of measurements at the end of May. The final report was received on June 22, 2015.

Table 1. Standard Materials and temperatures proposed to be measured in 2015 [3]

Code	Temperature °C
S6 (Mineral Oil)	20; 25; 40
N26 (Polialfaolefina)	40; 100
S200 (Polialfaolefina)	20; 25
S60 (Mineral Oil)	40;100
S600 (Polialfaolefina)	40
S8000 (Polibutena)	20; 25

3. Principle of Measurement

A common method for measurement the viscosity of the Newtonian fluids uses capillary extruded viscometers - Ubbelohde Type. Measurement of the viscosity of liquids shall be based on the measurement of flow time of a given volume of liquid, by a capillary tube with geometrical characteristics well established. The glass capillary viscometers, as Ubbelohde type, result in the formation of a column of liquid on which the atmospheric pressure acts on both from top to bottom (at

the top level) and bottom-up (at the lower level) until the two pressures of the opposite direction shall be cancelled. The lower level of liquid formed by the breaking of the liquid column due to the atmospheric pressure transmitted through the tube connection shall make the so-called "suspended level", as shown in Figure 1 [4].

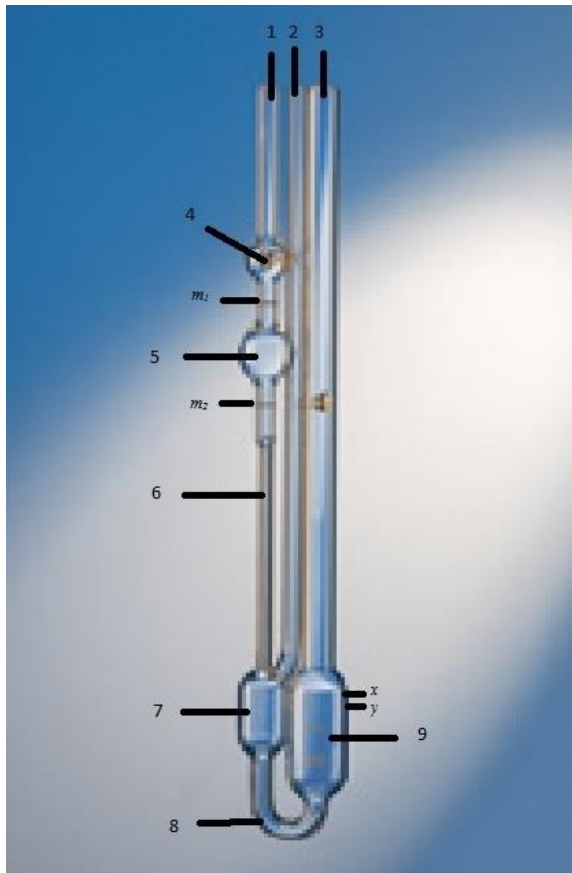


Figure 1. Capillary Viscometers with Suspended Level, Ubbelohde Type by comparison to the National Standard of Physikalisch Technische Bundesanstalt of Viscosity, PTB, Germany.[4]

1-capillary branch; 2-tube connection with the atmosphere; 3-branch with tank; 4-bubble of safety; 5-measuring Bull; 6-capillary; 7-suspended bubble level; 8-extension tubular fuel tank; 9-tank.

(On either side of the bubble of measurement shall be drawn two marks m_1 and m_2 , which defines the volume of liquid whose capillary leakage through it is measured. The capillary tube is open in the bubble suspended level 2, which communicates with the atmosphere and the tank 9, on which are marked levels x and y , which indicates the minimum level from the viscometer and the maximum one, accordingly.)

4. The principle of calibration

The kinematic viscosity is measured by using the Ubbelohde dedicated viscometer. The flow time t , of the liquid between the two marks of a standard viscometer is directly proportional to the kinematic viscosity ν [5]:

$$\nu = k \cdot t \quad (1)$$

where: ν - kinematics viscosity of the liquid, [mm^2/s];

k - constant of the glass capillary [mm^2/s^2];

t - average time flow of liquid between the capillary marks [s].

National standard of the unit of measure of viscosity mm^2/s kinematic is a secondary standard one and consists of two sets of capillaries viscometers Ubbelohde, manufactured by the company Schott Gerate which allow to determine the kinematic viscosity of Newtonian fluids in the range of about (0.5 - 100 000 mm^2/s). Maintain traceability to the SI was achieved by calibration of two sets of capillaries at the PTB-Germany.

5. Results presentation and conclusions

According to the Protocol of the program, each participant ASTM has assigned a call sign in the final report. National Institute of Metrology has #26, respectively.

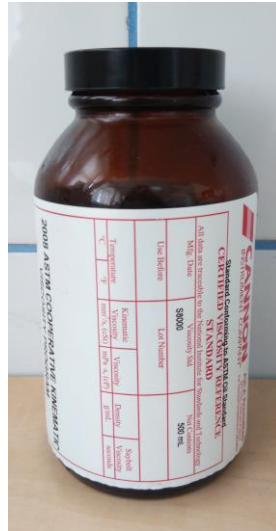


Figure 2. Reference Material Supplied to NIM by the CANNON Company, (S 8000)

In Table 2 are shown the measured reference materials (codification), temperature measurement, the value reported by the INM and the relative error of kinematic viscosity e_r versus the benchmark.

Relative error e_r represents the ratio between the absolute error of measurement (ΔX) and the real measured value of (X_e), % [6].

$$e_r = \Delta X / X_e \cdot 100 \quad (2)$$

Table 2. Measurements of Reference Materials [3]

Code(type) error e_r (%)	T (°C)	Value reported ν (mm ² /s)	Relative
S6 (Mineral Oil)	20	10,77	0,13
S6 (Mineral Oil)	25	9,082	0,03
S6 (Mineral Oil)	40	5,842	0,08
N26 (Polialfaolefina)	40	25,53	0,02
N26 (Polialfaolefina)	100	5,163	0,18
S200 (Polialfaolefina)	20	543,9	0,09
S200 (Polialfaolefina)	25	403,9	-0,02
S60 (Mineral Oil)	40	54,92	0,07
S60 (Mineral Oil)	100	7,726	0,23
S600 (Polialfaolefina)	40	520,2	0,16
S8000 (Polibutena)	20	35862	0,15
S8000 (Polibutena)	25	22839	0,14

In Table 2 there are presented all 166 stock viscosity determination for each sample/set temperature and error percentage compared to the average of all values of viscosity. Measurements of viscosity values with an error $> 1.36\%$ have been removed in the calculation of the average.

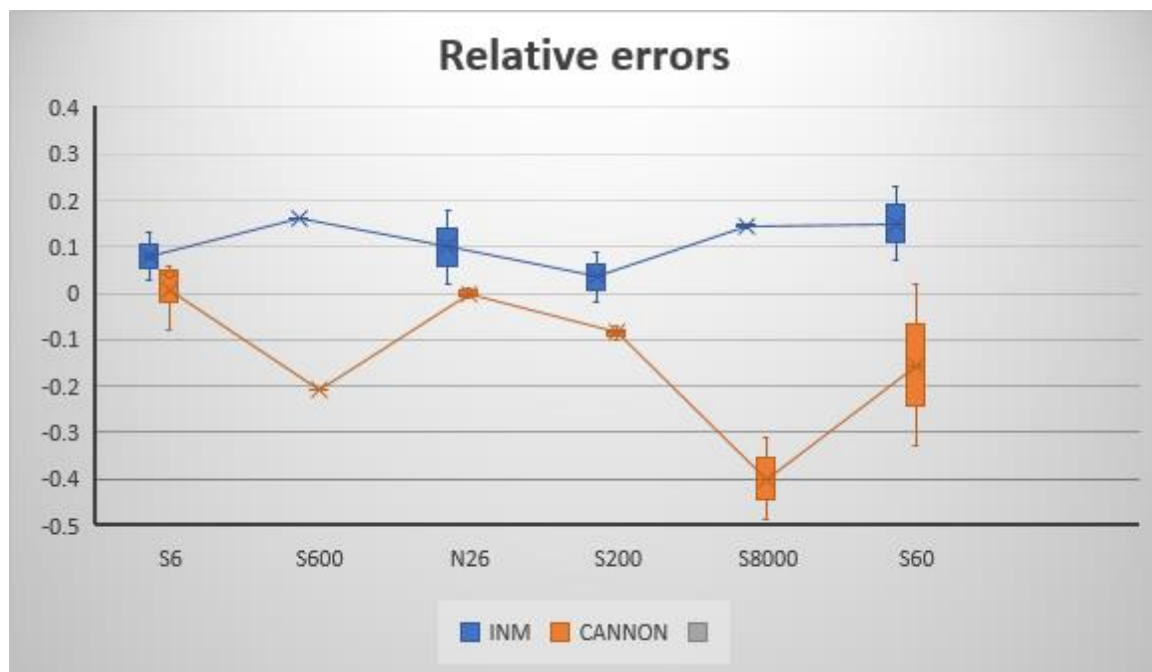
In Table 3, there are presented the following issues: the measured reference materials (codification), temperature measurement, the values reported by INM, the values reported by the Cannon Company, and e_r , relative error of kinematic reported viscosities versus the benchmarks.

Table 3: Reference Material Measurement by comparison with Cannon [3]

Code (type)	T (°C)	Value reported ν (mm ² /s)		Relative error e_r (%)	
		INM	Cannon	INM	Cannon
S6(Mineral Oil)	20	10,77	10,77	0,13	0,06
S6(Mineral Oil)	25	9,082	9,075	0,03	0,04
S6(Mineral Oil)	40	5,842	5,833	0,08	-0,08
N26(Polialfaolefina)	40	25,53	25,53	0,02	-0,01
N26(Polialfaolefina)	100	5,163	5,155	0,18	0,01
S200(Polialfaolefina)	20	543,9	543,0	0,09	-0,07
S200(Polialfaolefina)	25	403,9	403,6	-0,02	-0,10
S60(Mineral Oil)	40	54,92	54,70	0,07	-0,33
S60(Mineral Oil)	100	7,726	7,710	0,23	0,02
S600(Polialfaolefina)	40	520,2	518,2	0,16	-0,21
S8000(Polibutena)	20	35862	35696	0,15	-0,31
S8000(Polibutena)	25	22839	22695	0,14	-0,49

A good accordance between the averages of the values of viscosity determined by laboratory of INM vs. Cannon's finally resulted. ASTM comparison procedure is a good opportunity to prove the laboratory capability and, therefore, to maintain a good level of technical and professional competence of the personnel involved in the measurements of viscosity.

In 2015 the reported relative error is less than 0.20% out of reference amount for 73 % of results. The reported relative error is less than 0.30% out of reference amount for 80 % of results. INM reported 11 of 12 results with an average relative error of less than 0,20%, as it is shown in Figure 3.

**Figure 3.** Relative errors of Kinematic Viscosity e_r Reported: INM vs. CANNON

References

- [1] ***<https://www.cannoninstrument.com/en/catalog/node/viscosity-flash-point-standards>
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- [3] ***Report of the annual ASTM cooperative kinematic viscosity program, Spring 2015
- [4] Solomon M 1958 *Viscozimetre și elemente de teoria viscozității*, Tehnica, Bucuresti, Romania
- [5] Oiml R 1995 Second Preliminary Draft (2CD), Procedure for the Kinematic Viscosity Measurements by Means of Standard Viscometers
- [6] ***ISO/IEC Guide 98-3:2008 (JCGM/WG1/100) Preview Uncertainty of measurement Part 3: Guide to the expression of uncertainty in measurement (GUM:1995)