SIM COMPARISON OF VISCOSITY STANDARDS
(SIM.VI.S1)

FINAL REPORT

Participating Laboratories: INTI[1] .....Argentina
INMETRO[2].....Brazil
CENAM[3] ..... Mexico
LATU[4] .....Uruguay

Guest Laboratory: PTB[5] .....Germany

1. Introduction

This comparison was conducted in 1997 under the framework of activities promoted by the Interamerican Metrology System SIM. The object of the comparison was to determine any significant differences in viscosity measurements that might exist among the National Measurement Institutes within the Americas, as well as to achieve agreement regarding the uniformity of measurement procedures and in the calculation of uncertainties, as it is established in internationally accepted viscosity standards.

This comparison was coordinated by CENAM, who provided the samples of standard fluids to the participants.

2. Procedure and implementation

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The selection of liquid samples was made considering industrial demands for viscosity measurements. Samples were part of batches produced in 1995, which have been monitored for viscosity stability. Complete records prior to their separation date from CENAM are available.[3] The samples were sent to the participating laboratories via special courier services through the Organization of American States (OAS). No shipping problems were reported.

The laboratories performed measurements at 20°C and 25°C using their standard viscometers. Their results included the most probable viscosity value and its expanded uncertainty ($k = 2$) in accordance with ISO *Guide* [1]. These results were sent to CENAM for compilation.

In November 1997, a meeting was held in INTI, Argentina, to discuss the results of the comparison. Representatives from CENAM, INTI and LATU attended.

3. Results

The participating laboratories performed the viscosity measurements using Ubbelohde capillary viscometers. An exception was LATU who used Cannon Fenske viscometers [2].

The reported results are listed in the tables 1 and 2 shown in figures 1 through 4. The reference values were calculated as the arithmetic mean of the values obtained by each laboratory. Uncertainties were determined by each laboratory using methods accepted for this purpose, in accordance with the ISO *Guide* [1]. It is worth noting that the viscosity value reported by INTI for liquid 710-10 at 20°C appeared questionable due to its large deviation from all other results. It was decided not to include this measurement result in the determination of the average viscosity value. Further investigation found that the viscometer used by INTI for this test was probably contaminated with a silicone-based liquid.

As per suggestion from PTB, a mechanism to compare all results was the so-called “normalized error” technique. This technique requires the calculation of the normalized error as

$$En_{lab} = \frac{u_{lab} - u_{ref}}{\sqrt{U^2(ref) + U^2(lab)}} \qquad (1)$$

where *U(ref)* represents the expanded uncertainty of the reference value, calculated from the standard deviation of the mean,

$$U(ref) = 2 \cdot \frac{s}{\sqrt{n}} = 2 \cdot \frac{\sqrt{\frac{\sum_{i=1}^{n} (u_{labi} - u_{ref})^2}{n-1}}}{\sqrt{n}} \qquad (2)$$

in the previous formula, u_{ref} is calculated as the arithmetic mean of the values reported from the participants. The coverage factor $k=2$ was used to obtain the expanded uncertainty to a level of approximately 95%.

$$u_{ref} = \frac{1}{n}\sum_{i=1}^{n} u_{labi} \qquad (3)$$

Tables 1 and 2 show the values of the normalized error for the participants. The proposed comparability criterion is **|En| ≤ 1**.[6]

LABORATORY	**Standard Liquid 710-10**					
	20 ºC			25 ºC		
	u mm²/s	U %	|En|	*u* mm²/s	U %	|En|
INTI	179,38	0,4	**4,7**			
INMETRO	175,96	0,19	0,04	134,07	0,19	0,12
CENAM	176,14	0,26	0,35	134,18	0,23	0,43
LATU	175,96	0,43	0,02	133,95	0,35	0,18
PTB	175,84	0,13	0,52	133,95	0,13	0,42
Reference value	175,975	0,07		134,038	0,08	

Table 1. Results of the comparison, standard liquid 710-10. The reference value was calculated as the arithmetic mean of the reported results

LABORATORY	**Standard Liquid 710-16**					
	20 ºC			25 ºC		
	u mm²/s	U %	|En|	*u* mm²/s	U %	|En|
INTI	4 582,7	0,50	0,58			
INMETRO	4 566,5	0,21	0,17	3 196,3	0,21	0,12
CENAM	4 570,0	0,26	0,10	3 198,2	0,26	0,31
LATU	4 565,0	0,44	0,17	3 197,0	0,43	0,12
PTB	4 558,9	0,17	0,88	3 189,9	0,17	0,83
Reference value	4 568,62	0,17		3 195,35	0,12	

Table 2. Results of the comparison, standard liquid 710-16. The reference value was calculated as the arithmetic mean of the reported results

[6] The normalized error criterion was used although there is correlation between some of the participants, in the sense that CENAM, INMETRO and INTI viscosity measurements are traceable to PTB standards. The measurements results from LATU are traceable to CANNON standards in the USA.

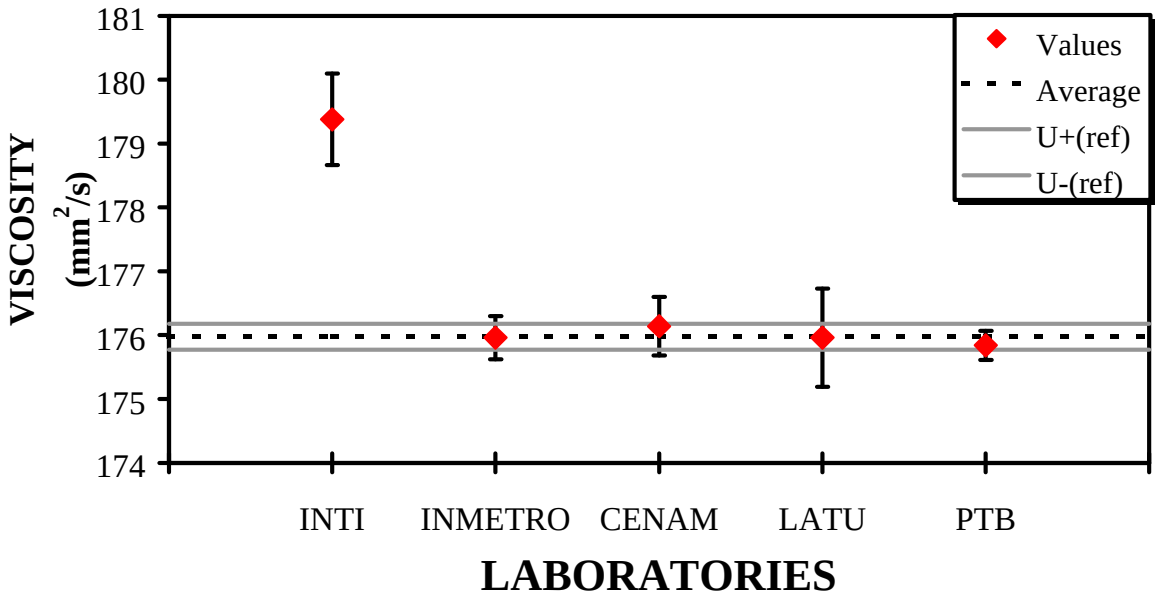

Figure 1. Results of viscosity measurements of liquid 710-10 at 20 °C. Shown are the viscosity measurement and their uncertainties. The limits represent the expanded uncertainty of the reference value (average viscosity value), calculated according to the corresponding degrees of freedom.

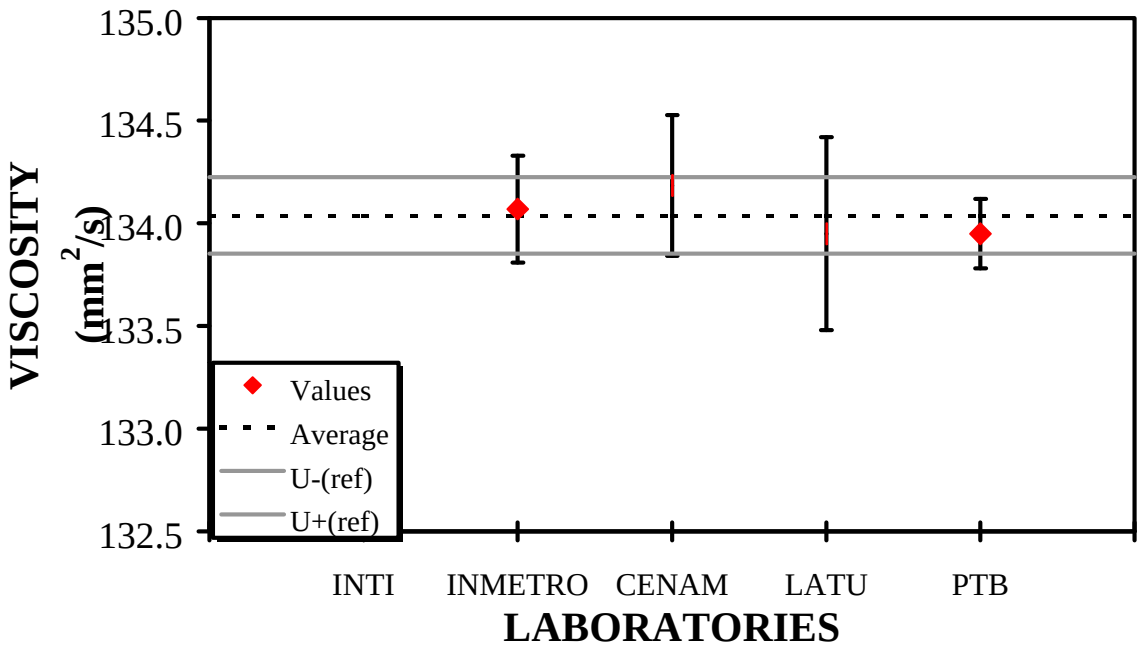

Figure 2. Results of viscosity measurements of liquid 710-10 at 25 °C. Shown are the viscosity measurement and their uncertainties. The limits represent the expanded uncertainty of the reference value (average viscosity value), calculated according to the corresponding degrees of freedom.

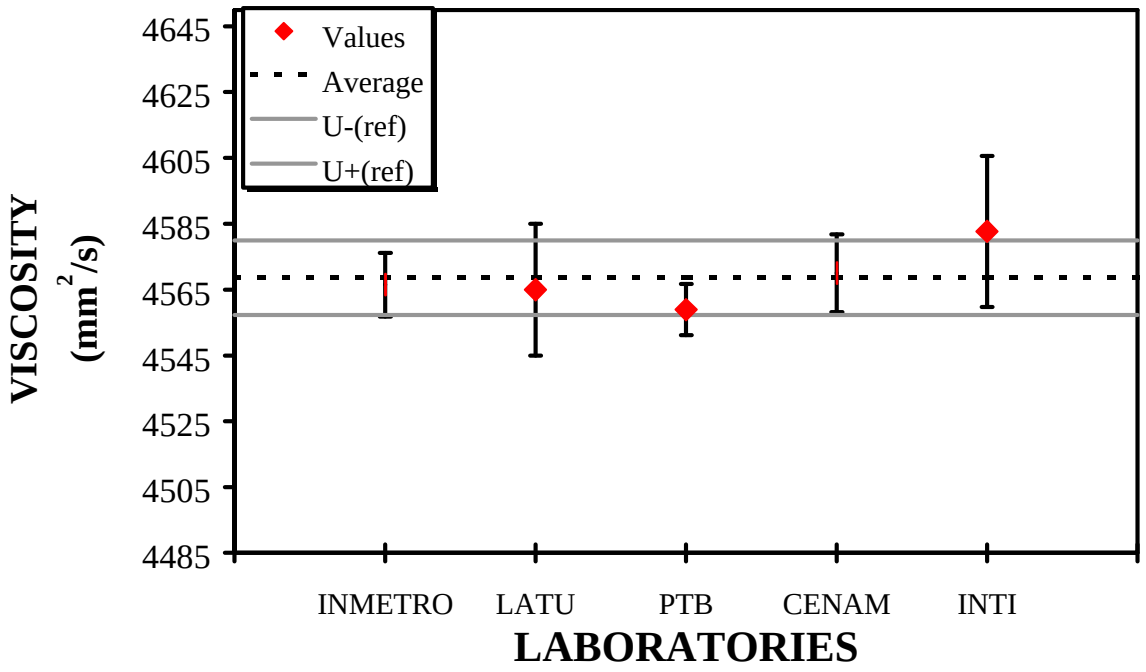

Figure 3. Results of viscosity measurements of liquid 710-16 at 20 °C. Shown are the viscosity measurement and their uncertainties. The limits represent the expanded uncertainty of the reference value (average viscosity value), calculated according to the corresponding degrees of freedom.

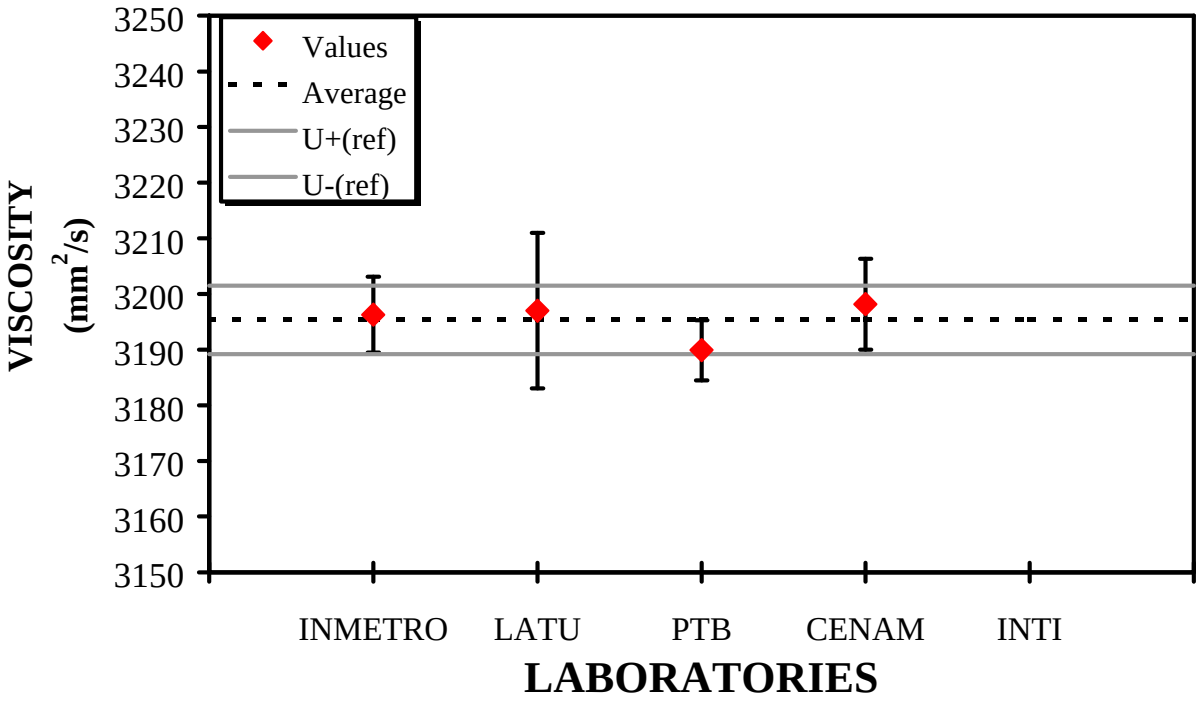

Figure 4. Results of viscosity measurements of liquid 710-16 at 25 °C. Shown are the viscosity measurement and their uncertainties. The limits represent the expanded uncertainty of the reference value (average viscosity value), calculated according to the corresponding degrees of freedom.

4. Conclusions

- This comparison shows good agreement among the kinematic viscosity measurements performed by the participating laboratories in spite of the use of capillary viscometers of different designs.
- As can be seen from tables 1 and 2, only the $|E_n|$ value from INTI, standard liquid 710-10 at 20 ºC, exceeds the comparability criterion. All the other results do no deviate significantly.
- According to the reported results it was concluded that no significant differences exist among measurement uncertainties of the participating laboratories. In part, this was expected due to the similarity of the equipment used. The representative from INMETRO was unable to attend the discussion meeting in INTI, making it impossible to evaluate the sources for uncertainty estimates from INMETRO, which appeared to be understated at that time. Afterwards INMETRO sent its revised uncertainty values to the pilot Laboratory, values that has been included in this report.
- The results show the equivalency of the SIM laboratories that took part in this metrological exercise. Further, the results of the SIM laboratories are in agreement with those from PTB (a leading EUROMET laboratory).

5 Acknowledgments

The participation and technical advice of PTB are greatly acknowledged. This report was edited by Pedro I. Espina.[7]

6. References

[1] ISO, *Guide to the Expression of Uncertainty in Measurement* (International Organization for Standardization, Geneva, Switzerland, 1993). This *Guide* was prepared by ISO Technical Advisory Group 4 (TAG 4), Working Group 3 (WG 3). ISO/TAG 4 has as its sponsors the BIPM, IEC, IFCC (International Federation of Clinical Chemistry), ISO, IUPAC (International Union of Pure and Applied Chemistry), IUPAP (International Union of Pure and Applied Physics), and OIML. Although the individual members of WG 3 were nominated by the BIPM, IEC, ISO, or OIML, the *Guide* is published by ISO in the name of all seven organizations.

[2] ISO 3105: 1994(E) *Glass capillary kinematic viscometers – Specifications and operating instructions*, Second Edition 1994-12-01 (International Organization for Standardization, Geneva, Switzerland, 1994).

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