Realization of National Institute for Standards (NIS - Egypt) Viscosity Scale in Wide Temperature Ranges

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Abstract

A new viscosity bathes has been entered the services in Thermal Metrology Laboratory-National Institute for Standards, NIS-Egypt in order to use in maintain and extend the national viscosity scale in wide temperature ranges, international comparison and routine calibration of viscometers. The medium of the bath should be homogenous enough in temperature so many thermal factors taken into account to estimate the temperature gradient, homogeneity, stability and thermal profile distribution with the related uncertainty to each parameter. The study carried out by two Standards Platinum Resistance Thermometer (SPRT) calibrated at fixed point according to ITS-90. A number of glass capillary viscometers have been selected and arranged into six groups. The selected viscometers and oils realize NIS viscosity scale in wide temperature ranges from -30 ^{o}C up to 100 ^{o}C.

Keywords: Viscosity, Viscometer, ITS-90, SPRT, Fixed Points, Uncertainty, Scale.

1. Introduction

Viscosity laboratory decided to extend the national viscosity scale in low temperature range down to -30^{o}C, new viscosity baths works with low temperature range using dry denatured ethanol is used as the bath medium because its property to absorb moisture from the atmosphere is removed from it. The oil bath model Koehler LKV4000 low temperature kinematic viscosity bath contains several upper holes to receive four viscometers at the same time; there is an additional small hole to insert the Standard Platinum Resistance Thermometers (SPRTs) to measure the temperature variation during the viscosity measurements. The cooling unit within the bath stabilize the bath temperature to desired setting within ± 0.02^{o}C.

The bath is filled with approximately 14L of ethanol as a medium to put inside it the viscometers in order to calibrate glass viscometer under test with references known oils or even known viscometers with unknown oils under test. The main target is realize the kinematic viscosity tests with glass capillary viscometers according to the ASTM D445 **(ASTM, 1992)** test method and related test specifications. The SPRTs SN 234, 247 has been calibrated at fixed point according to ITS-90 **(Preston-Thomas, 1990)**.

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2. Materials and Methods

Experimental arrangement

2.1. Thermal Profile Characterization of the bath

The bath filed with 14L of ethanol then the SPRTs have been inserted into the medium at different positions and levels. Five levels taken into the account from the top side down to the bottom separate equally from each other's, at each level the thermometer inserted in five positions as four at the corners and one at the center of the level as shown in Figure 1. So, twenty five position introduced into the study at each temperature set point, the range of interest is from -30 ^{o}C up to 10 ^{o}C. The measurements carried out at -30^{o}C, -20^{o}C, -10 ^{o}C and 10 ^{o}C. The two SPRT connected at the same time to resistance bridge model ASL F700 conjugated with standard resistor and the measuring system connected to PC working under LABVIEW environment.

Homogeneity is the main parameter should be studied to optimize and establish a suitable uniform medium realize the National Viscosity Scale. In order to find a closed value to the homogeneity as possible, thermal gradient taken into account and observed as a change of a temperature readings of a thermometer according to a change of its position inside a calibration bath **(Pornpatkul, 2012)**. Basic gradients that can be observed are vertical and horizontal gradient but sometimes more appropriate to define axial and a radial gradient. Axial gradient is determined as maximum temperature difference between two different positions in axial direction. The radial gradient is a maximum temperature difference between two different positions in a radial direction. So, three thermal factors were discussed as follows;

2.1.1. Thermal Profile Distribution

Consider that the temperature T_{Py}^{Lx} denotes to the temperature at level Lx and position Py, the two SPRT measure the temperature at different positions and levels as shown in figure 1, table1

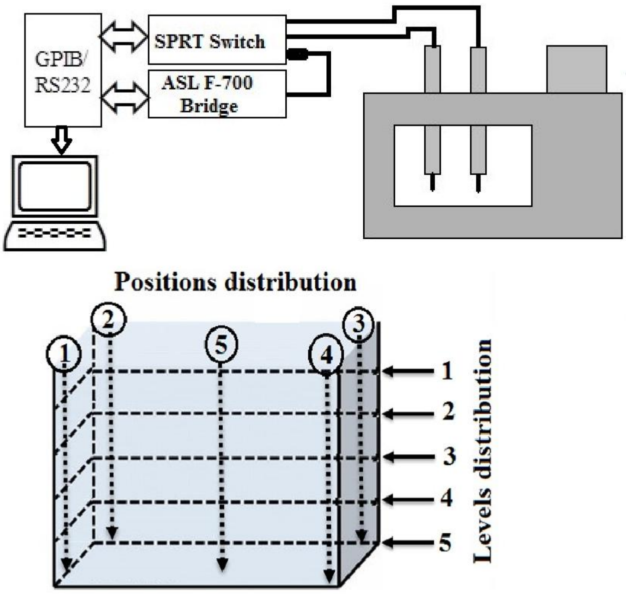

Figure 1. Schematic diagram of levels and position distribution

Table 1. Distribution of the temperature measurements using SPRT SN 234

L	**Temperature $\langle T_{Py}^{Lx} \rangle^*$ SPRT 234**					
	P	T	**-30.00**	**-20.00**	**-10.00**	**10.00**
1	1	$\langle T_{P1}^{L1} \rangle$	-30.00138	-20.02123	-10.02138	10.02811
	2	$\langle T_{P2}^{L1} \rangle$	-30.00149	-20.02127	-10.02154	10.02823
	3	$\langle T_{P3}^{L1} \rangle$	-30.00152	-20.02129	-10.02167	10.02841
	4	$\langle T_{P4}^{L1} \rangle$	-30.00144	-20.02139	-10.02172	10.02852
	5	$\langle T_{P5}^{L1} \rangle$	-30.00131	-20.02156	-10.02183	10.02857
	P	T	**-30.00**	**-20.00**	**-10.00**	**10.00**
2	1	$\langle T_{P1}^{L2} \rangle$	-30.00121	-20.02134	-10.02136	10.02822
	2	$\langle T_{P2}^{L2} \rangle$	-30.00124	-20.02156	-10.02176	10.02847
	3	$\langle T_{P3}^{L2} \rangle$	-30.00157	-20.02144	-10.02149	10.02833
	4	$\langle T_{P4}^{L2} \rangle$	-30.00156	-20.02165	-10.02159	10.028342
	5	$\langle T_{P5}^{L2} \rangle$	-30.00192	-20.02154	-10.02158	10.02897
	P	**T**	**-30.00**	**-20.00**	**-10.00**	**10.00**
3	1	$\langle T_{P1}^{L3} \rangle$	-30.00198	-20.02183	-10.02222	10.02865
	2	$\langle T_{P2}^{L3} \rangle$	-30.00204	-20.02194	-10.02234	10.02860
	3	$\langle T_{P3}^{L3} \rangle$	-30.00213	-20.02211	-10.02256	10.02880
	4	$\langle T_{P4}^{L3} \rangle$	-30.00209	-20.02256	-10.02277	10.02832
	5	$\langle T_{P5}^{L3} \rangle$	-30.00221	-20.02252	-10.02281	10.02854
	P	T	**-30.00**	**-20.00**	**-10.00**	**10.00**
4	1	$\langle T_{P1}^{L4} \rangle$	-30.00202	-20.02238	-10.02321	10.02910
	2	$\langle T_{P2}^{L4} \rangle$	-30.00218	-20.02241	-10.02333	10.02930
	3	$\langle T_{P3}^{L4} \rangle$	-30.00232	-20.02244	-10.02345	10.02949

	4	$\langle T_{P4}^{L4} \rangle$	-30.00241	-20.02253	-10.02377	10.02961
	5	$\langle T_{P5}^{L1} \rangle$	-30.00221	-20.02259	-10.02281	10.02983
5	P	T	**-30.00**	**-20.00**	**-10.00**	**10.00**
	1	$\langle T_{P1}^{L5} \rangle$	-30.00265	-20.02244	-10.02312	10.03017
	2	$\langle T_{P2}^{L5} \rangle$	-30.00273	-20.02257	-10.02319	10.03032
	3	$\langle T_{P3}^{L5} \rangle$	-30.00279	-20.02269	-10.02341	10.03048
	4	$\langle T_{P4}^{L5} \rangle$	-30.00281	-20.02274	-10.02369	10.03069
	5	$\langle T_{P5}^{L5} \rangle$	-30.00280	-20.02272	-10.02389	10.03083

**< > brackets indicates to the average*

2.1.2. Thermal Stability

The stability of the bath shows lower variation within the regulation specification of the bath. The fluctuation was monitored at different positions and levels as shown in figure 2 for SPRT SN-234 at setting point 20.0°C. The stability calculated at each level in the center point for continuous several hours (Ghazanfar, 2013).

$$T_{stability} = \frac{(\Delta t_{max} - \Delta t_{min})}{2} \tag{1}$$

During the progressive study of the performance of the Bath, it is found that the stability was better than ***0.015°C***.

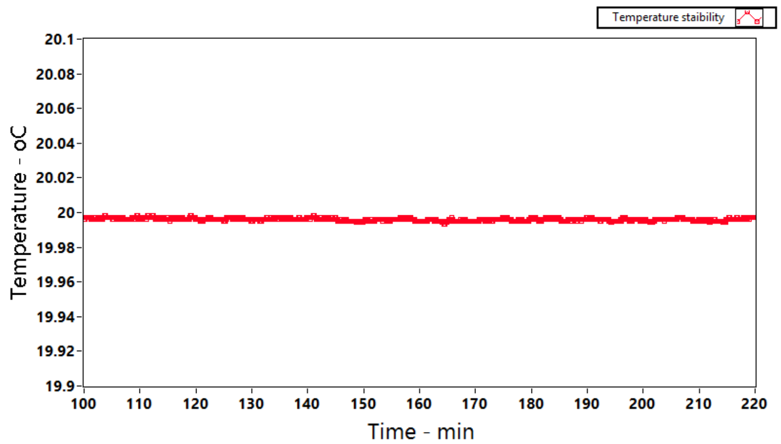

Figure 2. Temperature stability at the canter of the bath at 20.0 °C.

2.1.3. Thermal Gradient

The vertical gradient in a bath is termed “axial uniformity”. The horizontal gradient in a bath is termed “radial uniformity” **(EURAMET, 2011).**

2.1.3.1. Vertical Thermal Gradient

Figures from three to ten are shown the temperature gradient due to thermometer depths through five levels at different setting points for SPRTs SN 234 and SN 247.

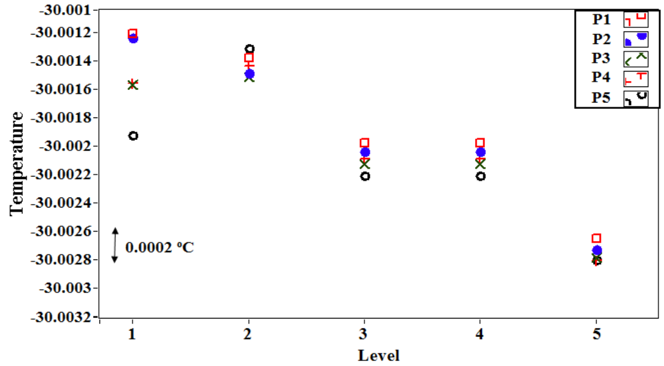

Figure 3. Thermal axial gradient at -30.0 ^{o}C for SPRT SN 234

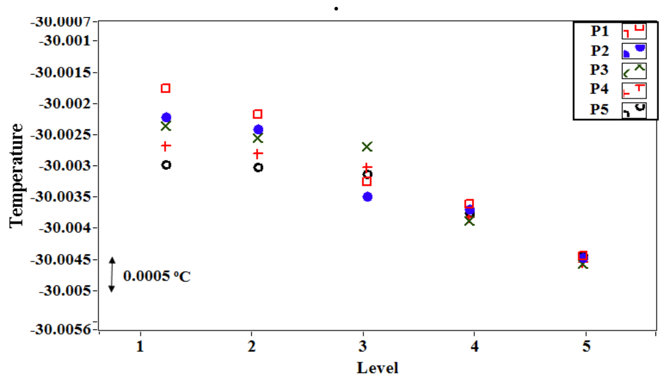

Figure 4. Thermal axial gradient at -30.0 ^{o}C for SPRT SN 247.

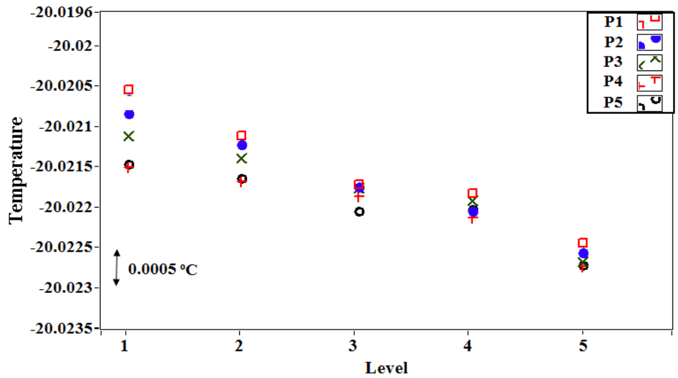

Figure 5. Thermal axial gradient at -20.0 oC for SPRT SN 234.

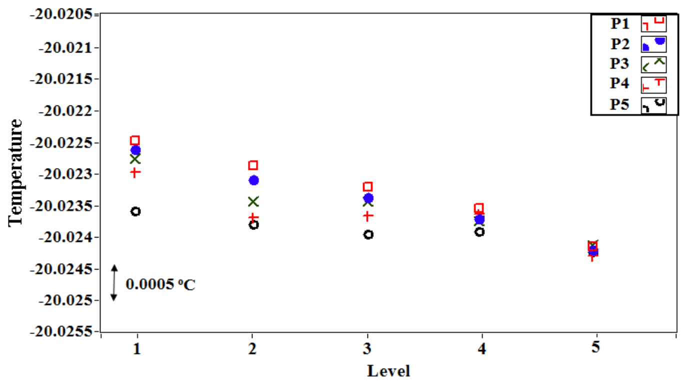

Figure 6. Thermal axial gradient at -20.0 ^{o}C for SPRT SN 247.

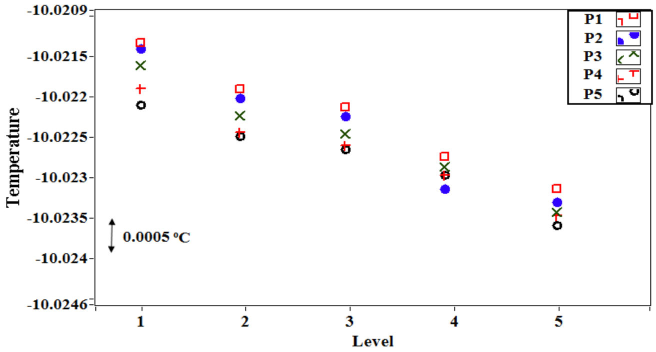

Figure 7. Thermal axial gradient at -10.0 °C for SPRT SN 234.

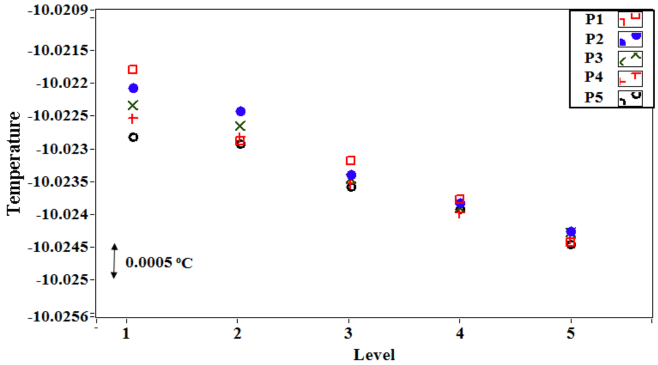

Figure 8. Thermal axial gradient at -10.0 °C for SPRT SN 247.

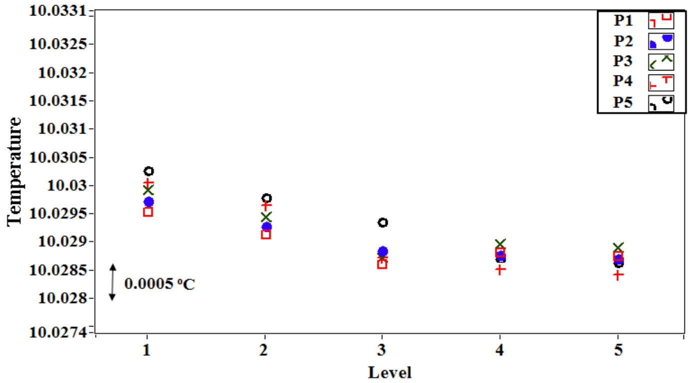

Figure 9. Thermal axial gradient at 10.0 °C for SPRT SN 234.

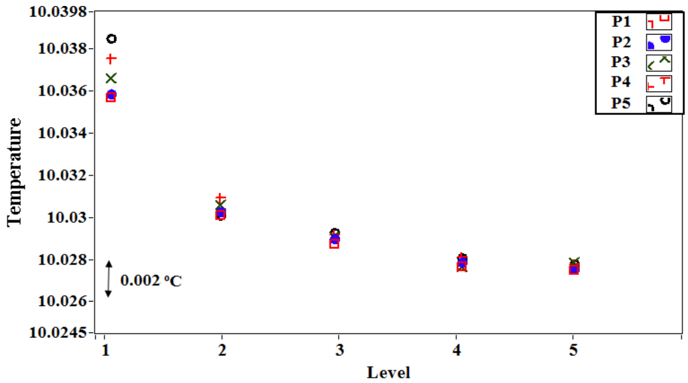

Figure 10. Thermal axial gradient at 10.0 °C for SPRT SN 247.

2.1.3.2. Horizontal Thermal Gradient

Figures from 11 to 18 show the temperature gradient due to thermometer positions at each level at different setting points for SPRTs SN 234 and SN 247.

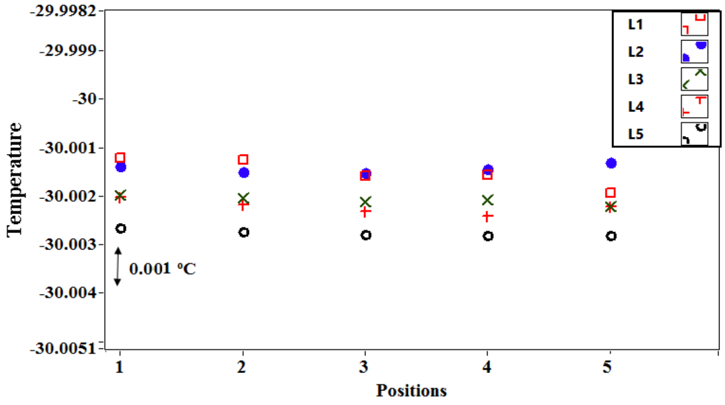

Figure 11. Thermal radial gradient at -30.0 ^{o}C for SPRT SN 234.

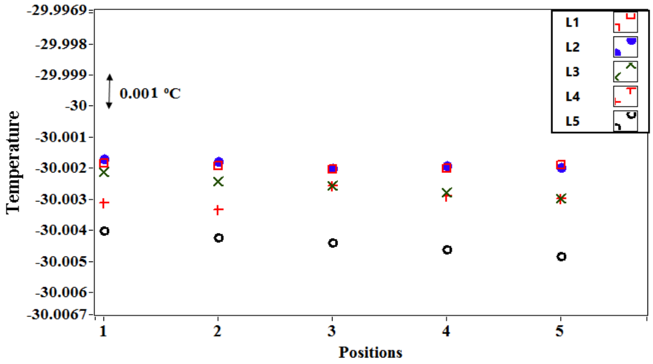

Figure 12. Thermal radial gradient at -30.0 ^{o}C for SPRT SN 247.

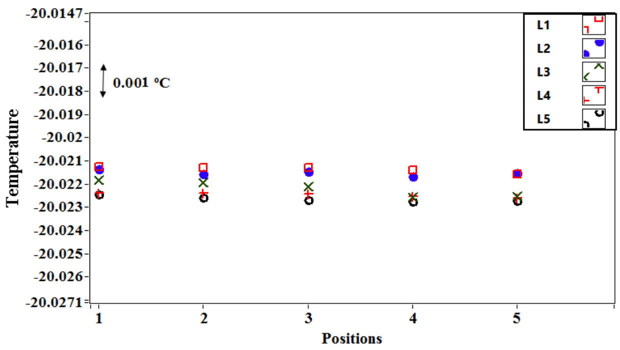

Figure 13. Thermal radial gradient at -20.0 ^{o}C for SPRT SN 234.

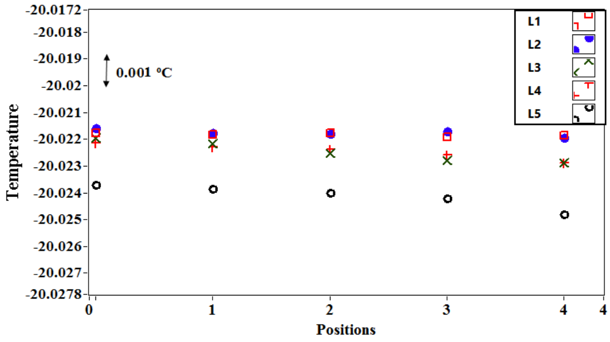

Figure 14. Thermal radial gradient at -20.0 ^{o}C for SPRT SN 247.

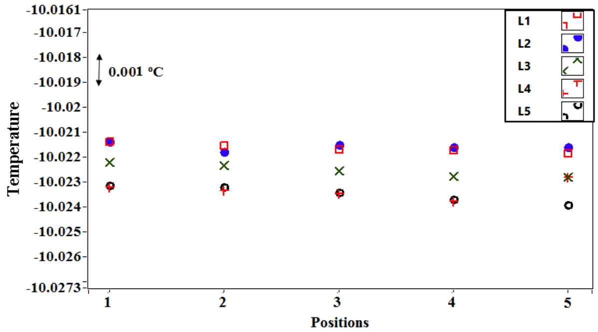

Figure 15. Thermal radial gradient at -10.0 ^{o}C for SPRT SN 234.

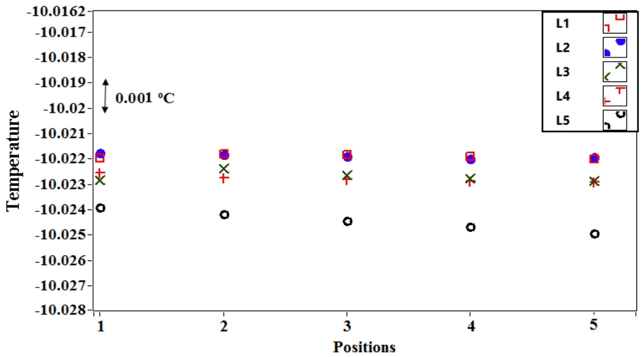

Figure 16. Thermal radial gradient at -10.0 ^{o}C for SPRT SN 247.

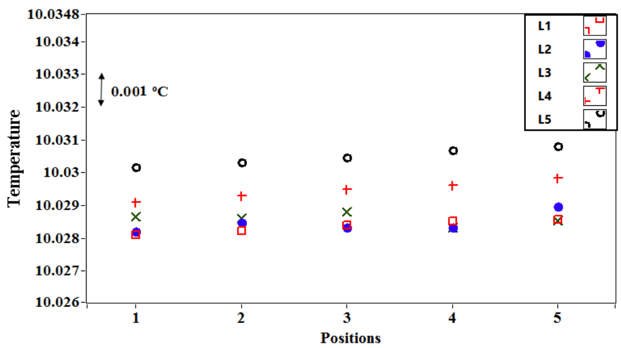

Figure 17. Thermal radial gradient at 10.0 ^{o}C for SPRT SN 234.

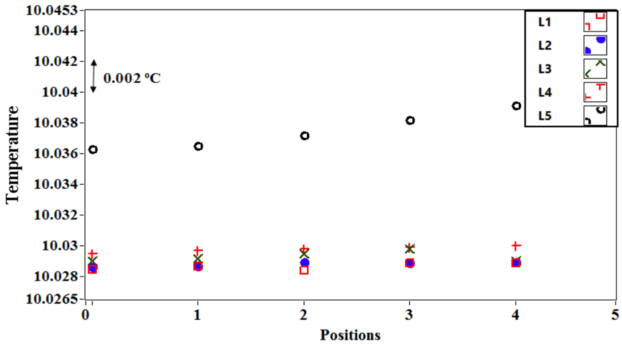

Figure 17. Thermal radial gradient at 10.0 ^{o}C for SPRT SN 247.

In order to calculated the thermal gradient which define from the following equation

$$\nabla T = \frac{\delta T}{\delta x} + \frac{\delta T}{\delta y} + \frac{\delta T}{\delta z} \quad (2)$$

The study was carried out on two dimensions vertical and horizontal; the axial thermal gradient and the radial thermal gradient is equivalent to **0.014 ^{o}C** and **0.012 ^{o}C** respectively.

2.2. Mounting the viscometers inside the bath

The bathes can accommodate several viscometers simultaneously, the viscometers was fixed to stainless steel holder then it was inserted into the bath. From the transparent window of the bath, the operator can determine precisely the flow time of the certain oil as shown in figure 18.

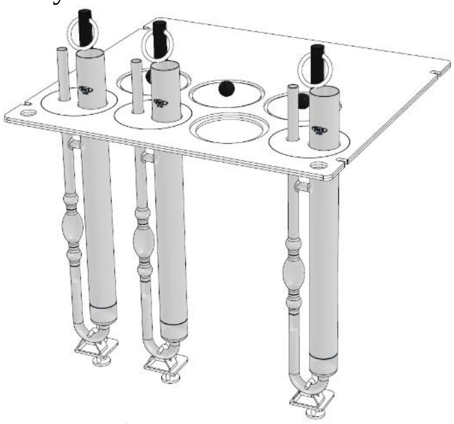

Figure 18. Mounting the viscometers into the bath.

3. Results and Discussion

Water is used to determine the viscometers constant of group 1, to determine the viscosity of a liquid, whose viscosity is higher than the water. The viscosity is obtained by the average of the two viscometers using the following equation **(A. Aliseda etal)**.

$$\nu = C.\ t \quad (3)$$

Where, (ν) is the kinematic viscosity values of the oil used, (C) is the instrumental constant, and (t) is the time for reproducible volume of the liquid to flow under gravity. A number of glass capillary viscometers have been selected and arranged in six groups table 2.

Table (2) shows the viscometers used for each group, together with their identification data.

Groups	Viscometers Type	Serial No.	Oils Used	Temperature
1	BS/U/SL/MV BS/U/SL/MV BS/U/SL/MV	6891 6890 6889	Water	20 ^{o}C & 25 ^{o}C
2	BS/U/SL/MV BS/U/SL/MV BS/U/SL/MV UBBELOHDE UBBELOHDE	6891 6890 6889 5965 36379	n-nonan	20 ^{o}C & 25 ^{o}C
3	UBBELOHDE UBBELOHDE UBBELOHDE UBBELOHDE UBBELOHDE	6695 5965 3266 6686 39125	171306	-20 ^{o}C
4	UBBELOHDE UBBELOHDE UBBELOHDE UBBELOHDE	5965 39125 2603 2602	131012	-20 ^{o}C
5	UBBELOHDE UBBELOHDE UBBELOHDE UBBELOHDE	6686 39125 5965 36379	170310	100 ^{o}C
6	UBBELOHDE UBBELOHDE	1392 790	05101	25 ^{o}C

For group one using master viscometers, double destilled water as a reference liquid table (3) gives the constants of each viscometer of this group. The constants were determined as an average value using double destilled water which has similar efflux time of the n-nonane oil at 20 ^{o}C and 25 ^{o}C).

Table (3): Results of the constants for the viscometers of group No. (1) with related uncertainties.

Viscometer Serial No.	Constant (C) mm^2/s^2	Uncertainty (UC) mm^2/s^2
6891	0.001655	±0.3%
6890	0.001992	±0.3%
6889	0.002088	±0.3%

* The uncertainties are referred to 95% confidence level and k=2

For group two using oil n-nonane **(Dullaert K, Mewis J)** as a transfer standard at 20 ^{o}C and 25 ^{o}C, the master viscometers in group one used to determine the viscosity of n-nonane then the viscosity of n-nonane used to determined the viscometers constant for the two remaining viscometers in this group, table (4).

Table (4): Results of the constants for the viscometers of group No. (1) with related uncertainties.

Viscometer No	n-nonane Vscossity mm^2/s^2 V=C.t	Average Flow Time s	Viscometer Constant C=V/T	Uncertainty U_C (mm^2/s^2)
36379	0.931432	564.024	0.001651	±0.3%
5965	0.931432	464.864	0.002004	±0.3%

For group three, using oil No (171306) ***(Standard Reference Material 717A)*** as a transfer standard at -20 ^oC the viscometers in group 3 with known constant which are coming from its calibration certificates used to determine the viscosity of the oil No (171306), table (5).

Table(5): Results of the viscosity of the oil No (171306) from viscometers constants

Viscometer No	Viscometer Constant C=V/T	Average Flow Time s	Oil No 171306 Vscossity mm^2/s^2 V=C.t
39125	0.005306	3616.54	19.18936124
6695	0.02993	640.91	19.18233653
3266	0.01092	1755.16	19.16638360
6686	0.01046	1830.15	19.14336900
Average Viscosity			19.17036259
Uncertainty			± 0.12%

For group four, using oil No (131012) as a transfer standard at -20 ^oC the viscometers in group 4 with known constant which are coming from its calibration certificates used to determine the viscosity of the oil No (131012), table (6).

Table (6): Results of the viscosity of the oil No (131012) from viscometers constants.

Viscometer No	Viscometer Constant C=V/T	Average Flow Time/ s	Oil No 131012 Vscossity mm^2/s^2 V=C.t
5965	0.002757	273.3867	0.753727
39125	0.005306	146.00	0.774676
2602	0.003512	211.36	0.742296
2603	0.003464	214.2567	0.742185
Average Viscosity			0.753221
Uncertainty			± 0.07%

For group five, using oil No (170310) as a transfer standard at 100 ^{o}C the viscometers in group 5 with known constant which are coming from its calibration certificates used to determine the viscosity of the oil No (170310), table 7.

Table (7): Results of the viscosity of the oil No (170310) from viscometers constants

Viscometer No	**Viscometer Constant C=V/T**	**Average Flow Time/ s**	**Oil No 171306 Vscossity mm^2/s^2 V=C.t**
6686	0.01046	177.473	1.856371
39125	0.005306	349.466	1.85427
5965	0.002757	672.713	1.854671
36379	0.001004	1848.395	1.855789
Average Viscosity			1.855275
Uncertainty			± 0.07%

For group six, using oil No (05101) **(Standard Reference Material 710A)** as a transfer standard at 25 ^{o}C the viscometers in group 6 with known constant which are coming from its calibration certificates used to determine the viscosity of the oil No (05101), table 8.

Table (8): The viscosity of oil No. (05101) at 25 ^{o}C.

Oil No.	**Viscometer No.**	**Viscosity (mm^2/s)**	***Uncertainty (mm^2/s)**
05101 @ 25^{o}C	1392	224961.55	
	790	224963.51	
Average		224962.53	
Uncertainty			0.25%

4. Conclusion

An intensive work was carried out on studying the new metrological viscosity bath to realize the national viscosity scale in wide temperature ranges. The results show that the bath worked with stability better than 0.015 ^{o}C. Thermal profile distribution achieved by two SPRTs calibrated at ITS-90 to calculate the thermal gradient homogenty. Thermal axial and radial gradient equivalent to 0.014 ^{o}C and 0.012 ^{o}C respectively. The selected groups of viscometers have been studied to determine the viscometer constant for unknown ones and known groups used to determine the viscosity of unknown oils by step-up-chain procedures.

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الملخص باللغة العربية

تحقيق المقياس القومي للزوجه للمرة الثالثة عند درجات حرارة مختلفة

ايمان محى الدين محمد [1]، مصطفى محمود مكاوي [1] ، هدى محمد أبوضره [2]

١. المعهد القومى للقياس والمعايره
٢. كلية البنات للاداب والعلوم والتربية – جامعه عين شمس

اللزوجة هي الخاصية الفيزيقية التي تحدد القوة التي يجب التغلب عليها عندما تستخدم السوائل بين سطحي معدنيين أو هي التي تتحكم في انسياب السوائل عند استخدامها في الطباعة والدهانات و الطلاء و غير ذلك.
تم تحقيق المقياس القومي للزوجة باستخدام المقاييس الزجاجية ذات الأنابيب الشعرية (الفسكوميترات) و السوائل العيارية ابتداء من الماء الذي تم تقطيرة مرتين و الذي تم الإتفاق عالميا علي أن تكون لزوجته عند درجة حرارة 20 0س و الضغط الجوي العادي (1.0035 مم2/ثانية) ثم تتابع الخطوات باستخدام السوائل النيوتينية حتي 200000 مم2/ثانية.

تمت دراسة للوسط الذي يوضع به الفسكوميترات وهو حمام كحول وحمام زيت. الدراسة شملت التوزيع الحراري بداخله في جميع الأماكن حيث تم تقسيم الحمام الى خمس مستويات أفقي وخمس مواضع رأسي ليكونوا 25 نقطه للدراسة. ومن التوزيع الحراري وجد ان الوسط متجانس رأسي وافقي بفروق طفيفة تصل الى 0.012 و 0.014 س^{o} على الترتيب ووجد أيضا ان مستوى الثبات للوسط يصل الى 0.015 س ^{o} على مدار عده ساعات من التشغيل المتواصل.

ومن النتائج تبين أن اللايقين في القراءات يتراوح ما بين 0.7 % بالنسبة للزيوت الخفيفة إلى 2.25% بالنسبة للزيوت الثقيلة.