COOMET.M.V-K1 key intercomparison of liquid viscosity measurements

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with contribution by:

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1. INTRODUCTION

The measurement of viscosity is very important for many industrial fields (petroleum products, polymers, food, pharmaceutics and others).
The need for traceability impels an increasing number of national accredited laboratories to provide viscometer calibrations.
The organization of the first COOMET key comparison on viscosity was planned at the 9th Meeting of COOMET TC 1.6 "Mass and related quantities" that took place on 21 – 23 April, 2004 at the Slovak Metrological Institute, Bratislava.

National standards of viscosity are presented by a set of capillary viscometers with overlapping measuring ranges covering the range of kinematic viscosities from about 0,3 mm^2/s to above 100 000 mm^2/s. The viscometer types that are used for this purpose are suspended-level (Ubbelohde) viscometers.

D.I Mendeleyev VNIIM, (RF) agreed to be the pilot laboratory for the comparison.

This comparison was carried out according to the rules set forth by the "Mutual recognition of national measurement standards and of measurement certificates issued by national metrological institutes" (MRA) and was registered as a regional key comparison.

The aim of this first COOMET key comparison (CCM.V-S1) was to determine the degrees of equivalence between individual NMIs and to compare them with the reference values (KCRV) obtained in the first key comparison CCM (CCM.V-K1) that was conducted in 2002.

Three samples of Newtonian liquids with nominal kinematic viscosities of 30 mm^2/s at 20°C, 100 mm^2/s at 20°C, 1000 mm^2/s at 20°C, were used.

2 First regional key comparison of liquid viscosity measurements (COOMET Project 333)

The following laboratories participated in this key comparison:

- BelGIM, (Belorussian State Institute for Metrology, Republic of Belarus);
- NCM, (National Centre of Metrology, Bulgaria);
- Ukrmetrteststandart, (Ukrainian Metrological Test Standart, Ukraine);
- VNIIM, (D.I. Mendeleyev Institute for Metrology, Russian Federation);

  The measurements were carried out on samples of three standard liquids provided by VNIIM as the pilot laboratory.
  Details of the comparison procedure are specified in the Technical Protocol.

Technical Protocol for COOMET 333 key comparison

Purpose of this document

The purpose of this document is to provide the participating laboratories with instructions for the handling of the liquids and to report on the measurement results, the measuring procedure and the apparatus. It is important that all instructions given in this document be followed. This will ensure that the measurement data are obtained under comparable conditions and are presented in the same format. Any deviation from the instructions has to be reported to the Pilot Laboratory.

- Measurement program for kinematic viscosity:

  Standard liquid REV 20 at 20^0C, approximate kinematic viscosity: 30 mm^2/s,
  density: 0, 86627 g/sm^3;

  Standard liquid REV 100 at 20^0C, approximate kinematic viscosity: 100 mm^2/s,
  density: 0,87287 g/sm^3;

  Standard liquid REV 1000 at 20^0C, approximate kinematic viscosity:

1000 mm^2/s, density: 0,89050 g/sm^3;

- All standard liquids are the mix of mineral oils. For all liquids, the long-term stability of the kinematic viscosity is better than 0,1% over six months.
- The standard liquids are packed into the 250 ml bottles made of dark glass.
- The uncertainty of the viscosity data is stated to be relative to the uncertainty for water, which means that the uncertainty of the water value
- [1] is not taken into account.

Timetable:

December 2004 (pilot laboratory):	Mailing of standard liquids, data sheets, and technical report to the participants
March 2005 (all participants):	Start of the comparison
June 2005 (all participants):	Completion of measurements
July 2005 (all participants):	Submission of results to the pilot laboratory
September 2005 (pilot laboratory):	Submission of draft report to the participants
March 2006 (all participants):	COOMET meeting at VNIIFTRI, Moscow, discussion of results
December 2006 (pilot laboratory):	Submission of the final report to the participants
October 2007 (all participants):	COOMET meeting at BelGIM, Minsk, discussion of the Draft B

3. Results of the key comparison

The aim of the comparison was to determine the degree of equivalence of the national standards of Belarus, Ukraine and Bulgaria against the standards of the national metrological institutes (NMI) that took part in the CCM.V-K1 [2] comparison.

To achieve this goal the following tasks were carried out:

- Transformation of initial data of comparison and evaluation of their uncertainty
- Calculation of degrees of equivalence and corresponding uncertainties;
- Determination of the best uncertainty of measurements reported to the participants;

Methods of measurements used by the participants in the comparison

All participants used two suspended-level (Ubbelohde) viscometers with close calibration constants, as national standards.

The working equation is given by:

$$\gamma = K \cdot \tau, \quad \text{where}$$

γ-kinematic viscosity, K-viscometer constant, τ – flow time, c

The uncertainty is calculated according to the international Guide [3] and are specified for k=2.

The relative overall uncertainty in the measurement of the kinematic viscosity (U_v), calculated by each participant is:

$$U_\gamma = 2 \cdot \sqrt{(S_k)^2 + (S_{\vartheta\gamma})^2 + \frac{1}{2}\left[(S_T)^2 + (S_t)^2\right]}$$

S_k- relative uncertainty constant K, (from certificate of calibration);

S_t –relative uncertainty of the time device;

$S_{\vartheta\gamma}$ - relative uncertainty of thermometer, gradient of temperature in the thermostat bath and temperature coefficient of liquid viscosity;

S_T - relative uncertainty of flow time measurements;

Results of the comparison measurements are shown in Table 1.

Table 1: Results of the CCM.V-K1 Comparison.

Participants		Liquid "30"	Liquid"100"	Liquid"100"
VNIIM	ν, mm^2/s	28,557	91,681	1018,2
	10^3 u_ν	2,0	2,4	2,6
BELGIM	ν, mm^2/s	28,545	91,482	1019,0
	10^3 u_ν	4,2	5,4	2,0
UKRMETR	ν, mm^2/s	28,539	91,474	1016,1
	10^3 u_ν	2,4	2,2	2,4
NCM	ν, mm^2/s	28,529	91,723	1020,6
	10^3 u_ν	2,0	3,2	3,2

10^3 u_ν –overall relative measurement uncertainty, obtained by each participant (κ=2);

The measurement results for standard liquid REV 30 at 20^0C, standard liquid REV 100 at 20^0C and standard liquid REV 1000 are given in Table 1.

The data from Table 1 are also plotted in Fig.1, Fig.2 and Fig.3 in the Appendix 1.

4. Procedure for linking of COOMET KC and CIPM KC

The linking of the results of the comparisons was fulfilled across the results of measurements obtained in the CIPM and COOMET comparisons, respectively.

As the nominal values of viscosity obtained in RMO and CIPM comparisons were different, so the basic criterion at the choice of pair values obtained by VNIIM in the both comparisons were used as identical measurement sets. So the results for Liquid “30” were linked to Liquid “10” (Liquid A), those for Liquid “100” were linked to Liquid “400”(Liquid B2), those for Liquid “1000” to Liquid “1300” (Liquid B1). For the evaluation of data the procedure specified in the Recommendation of the COOMET for evaluation of key comparison data was followed.

The data of the CCM.V-K1 [2] key comparison for three liquid samples - Liquids A, B1, B2 (key significance) χ_{ref}, the data obtained by the linking institute (VNIIM) χ^*, and the respective relate uncertainty are shown in Table 2.

Table 2

	Liquid «10 / 20°C» (Liquid A)	**Liquid «400 / 100°C» (Liquid B2)**	**Liquid «1300 / 20°C» (Liquid B1)**
x_{ref}, $u_{rel}(x_{ref})\times 10^3$	9.6519, 1.28	394.075, 0.9	1285.57, 0.9
x^*, $u_{rel}(x^*)\times 10^3$	9,6558, 1.0	394.010, 1.2	1286,90 1.3

Multiplication correction *c* is used for calculating transformed data.

$$c = \frac{x^*}{\widetilde{x}^*}, \quad u_{rel}^2(c) = 2(1-\rho)u_{rel}^2(\widetilde{x}^*),$$

Where $\widetilde{x}^*$ — results of measurements obtained by VNIIM in the COOMET KC; $\rho = 0.76$ — correlation coefficient for the results of measurements made at VNIIM calculated from the analysis of the measurement uncertainties budget.
The calculation results of the coefficients of transformed data are shown in the Tables A1-1, A1-2, A1-3.

The transformed data of comparison and respective uncertainties are calculated by:

$$\widetilde{x}_i' = c\,\widetilde{x}_i, \quad u_{rel}^2(\widetilde{x}_i') = u_{rel}^2(\widetilde{x}_i) + u_{rel}^2(c) = u_{rel}^2(\widetilde{x}_i) + 2(1-\rho)u_{rel}^2(x^*).$$

The degrees of equivalence and respective uncertainties are calculated by:

$$d_i = \widetilde{x}_i' - x_{ref}, \quad u^2(d_i) = c^2u^2(\widetilde{x}_i) + u^2(x_{ref}) + 2(1-\rho)u^2(x^*)\left(1 - \frac{u^2(x_{ref})}{u^2(x^*)}\right).$$

For the convenience of comparing the results obtained in COOMET key comparisons with those of the CIPM KC the degrees of equivalence are also presented as a ratio with respect to the reference value of the key comparison:

$$\Delta_i = \frac{d_i}{x_{ref}}, \quad U(\Delta_i) = \frac{2u(d_i)}{x_{ref}}.$$

An objective confirmation of the uncertainties claimed by the participants is the conformance to the inequation

$$|\Delta_i| \le U(\Delta_i).$$

The results of the evaluation of data obtained for all liquids used in the comparison are tabulated in Tables A1-1, A1-2, A1-3 (Apendix A1).

Figs, A2-1 to A2- 3 (Apendix A2) show the results of CCM.V-K1 comparison and the transformed results of the CCM.V-S1 comparison. Green highlighted are the results obtained by the linking laboratory (VNIIM). The transformed results of the participants in the CCM.V-S1 comparison are blue colored. The results are presented as deviations from the KCRV of CCM.V-K1 and as the claimed standard deviations of the measurement results. It is to be pointed out that standard uncertainties are shown and not extended ones, therefore some intervals do not

cross. As mentioned above, all laboratories have confirmed the claimed measurement uncertainties.

5. References

1 ISO TR 3666: Viscosity of Water (1998)

2. CCM.V-K1 Intercomparison, Gunther Klingenberg and Harro Bauer PTB

3. Guide to the Expression of Uncertainty in Measurement? Corrected and reprinted 1995, International Organization of Standardization (Geneva, Switzerland)

4 “The Evaluation of Key Comparison Data”, M.G. Cox, Metrologia, 2002, **39,** 589-595

Table A1-1. Liquid «30»

Laboratory	Initial data CCM.V K-1		Transformation data			Degree of equivalence			
	$\tilde{x}_i$	$u_{rel}(\tilde{x}_i)$, $\times 10^3$	c, $u_{rel}(c)\times 10^3$	$\tilde{x}_i'$	$u_{rel}(\tilde{x}_i')$, $\times 10^3$	d_i	$u(d_i)$	$\Delta_i \times 10^3$	$U(\Delta_i)\times 10^3$
BELGIM	28.545	2.1	0.33812, 0.7	9.6517	2.2	-0.0002	0.0297	0.0	6. 2
UKRMETR	28.539	1.2		9.6497	1.4	-0.0022	0.0246	-0.2	5.1
NCM	28.529	1.0		9.6463	1.2	-0.0056	0.0237	-0.6	4.9
VNIIM	$\tilde{x}^* = 28.557$	$u_{rel}(\tilde{x}^*)\times 10^3 = 1.0$							

Table A1-2 Liquid «100»

Laboratory	Initial data CCM.V K-1		Transformation data			Degree of equivalence			
	$\tilde{x}_i$	$u_{rel}(\tilde{x}_i)$, $\times 10^3$	c, $u_{rel}(c)\times 10^3$	$\tilde{x}_i'$	$u_{rel}(\tilde{x}_i')$, $\times 10^3$	d_i	$u(d_i)$	$\Delta_i \times 10^3$	$U(\Delta_i)\times 10^3$
BELGIM	91.482	2.7	4.29762, 0.8	393.155	2.8	-0.920	1.095	-2.3	5.6
UKRMETR	91.474	1.1		393.121	1.4	-0.954	0.508	-2.4	2.6
NCM	91.723	1.6		394.191	1.8	0.116	0.685	0.3	3.5
VNIIM	$\tilde{x}^* = 91.681$	$u_{rel}(\tilde{x}^*)\times 10^3 = 1.2$							

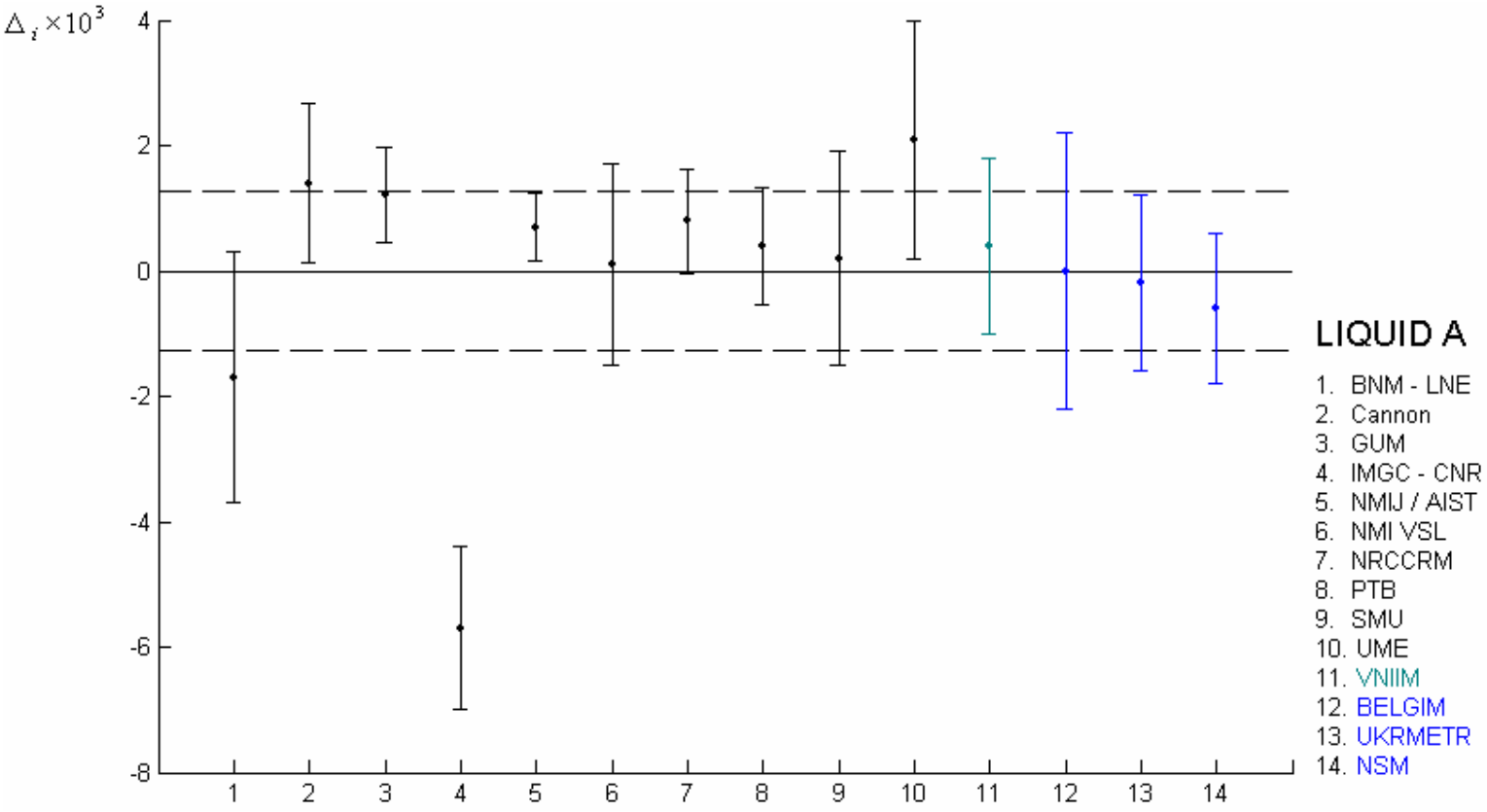
Table A1-3. Liquid «1000»

Laboratory	Initial data CCM.V K-1		Transformation data			Degree of equivalence			
	$\tilde{x}_i$	$u_{rel}(\tilde{x}_i)$, $\times 10^3$	c, $u_{rel}(c)\times 10^3$	$\tilde{x}_i'$	$u_{rel}(\tilde{x}_i')$, $\times 10^3$	d_i	$u(d_i)$	$\Delta_i \times 10^3$	$U(\Delta_i)\times 10^3$
BELGIM	1019.0	1.0	1.26390, 0.9	1287.91	1.3	2.34	1.79	1.8	2.8
UKRMETR	1016.4	1.2		1284.63	1.5	-0.94	1.98	-0.7	3.1
NCM	1020.6	1.6		1289.94	1.8	4.37	2.41	3.4	3.7
VNIIM	$\tilde{x}^* = 1018.2$	$u_{rel}(\tilde{x}^*)\times 10^3 = 1.3$							

На рисунках 1 – 3 приведены результаты сличений CCM.V-K1 и трансформированные результаты CCM**.**V-S1. Зеленым цветом выделены результаты ВНИИМ им.Д.И.Мендлеева, который выступает в качестве связующего института. Синим цветом представлены трансформированные результаты участников CCM**.**V-S1 сличений. Результаты представлены как отклонения от опорного значения ключевых сличений CCM.V-K1 и заявленные стандартные неопределенности результатов измерений. Пунктирной горизонтальной линией изображена стандартная неопределенность опорного значения. Особо отметим, что приведены стандартные неопределенности, а не расширенные, поэтому некоторые интервалы не пересекаются. Как уже было показано выше, все институты подтвердили заявленные неопределенности измерений.

Appendix A 2

Fig 2-1.



Figz 2-2.

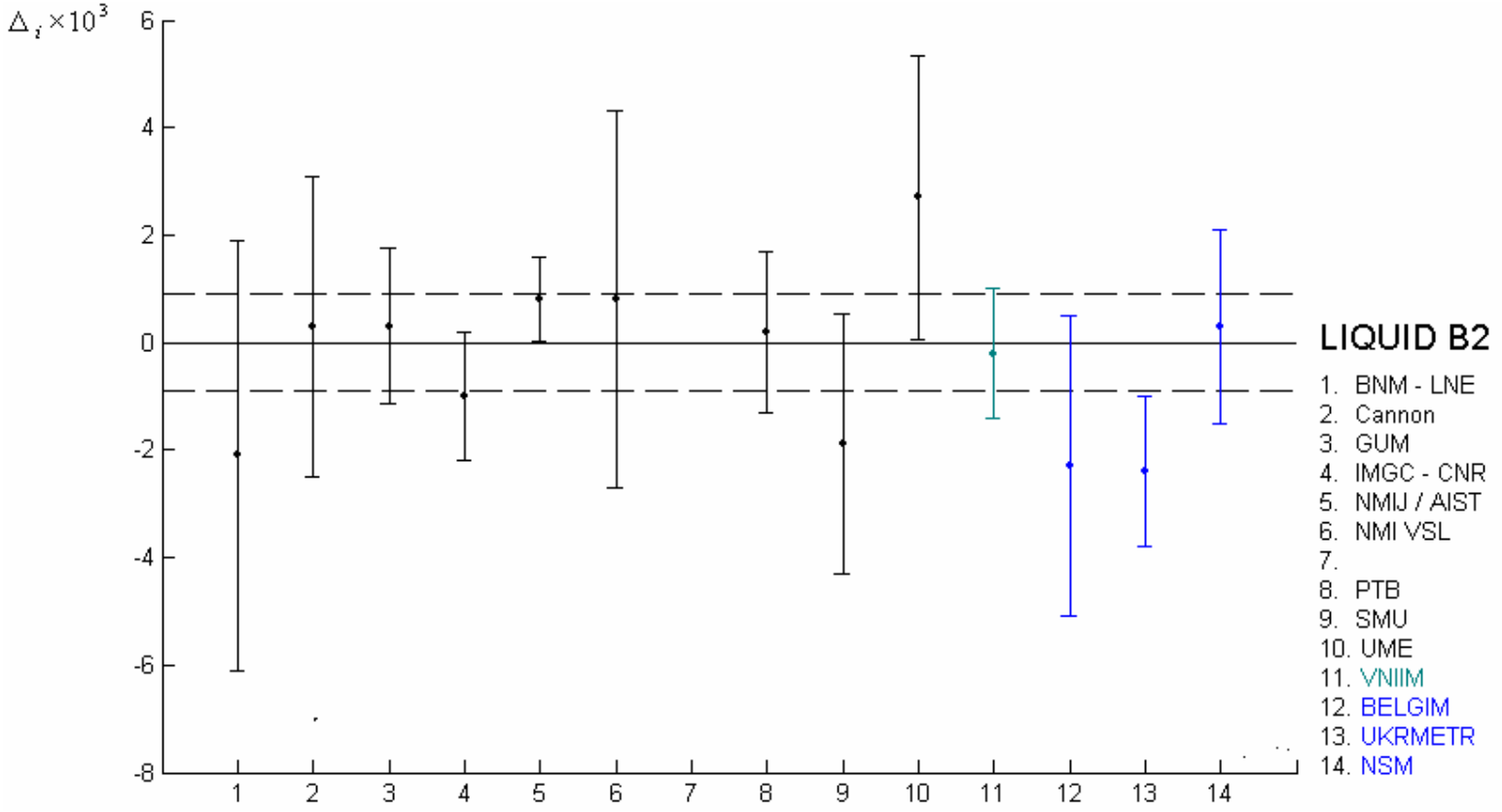
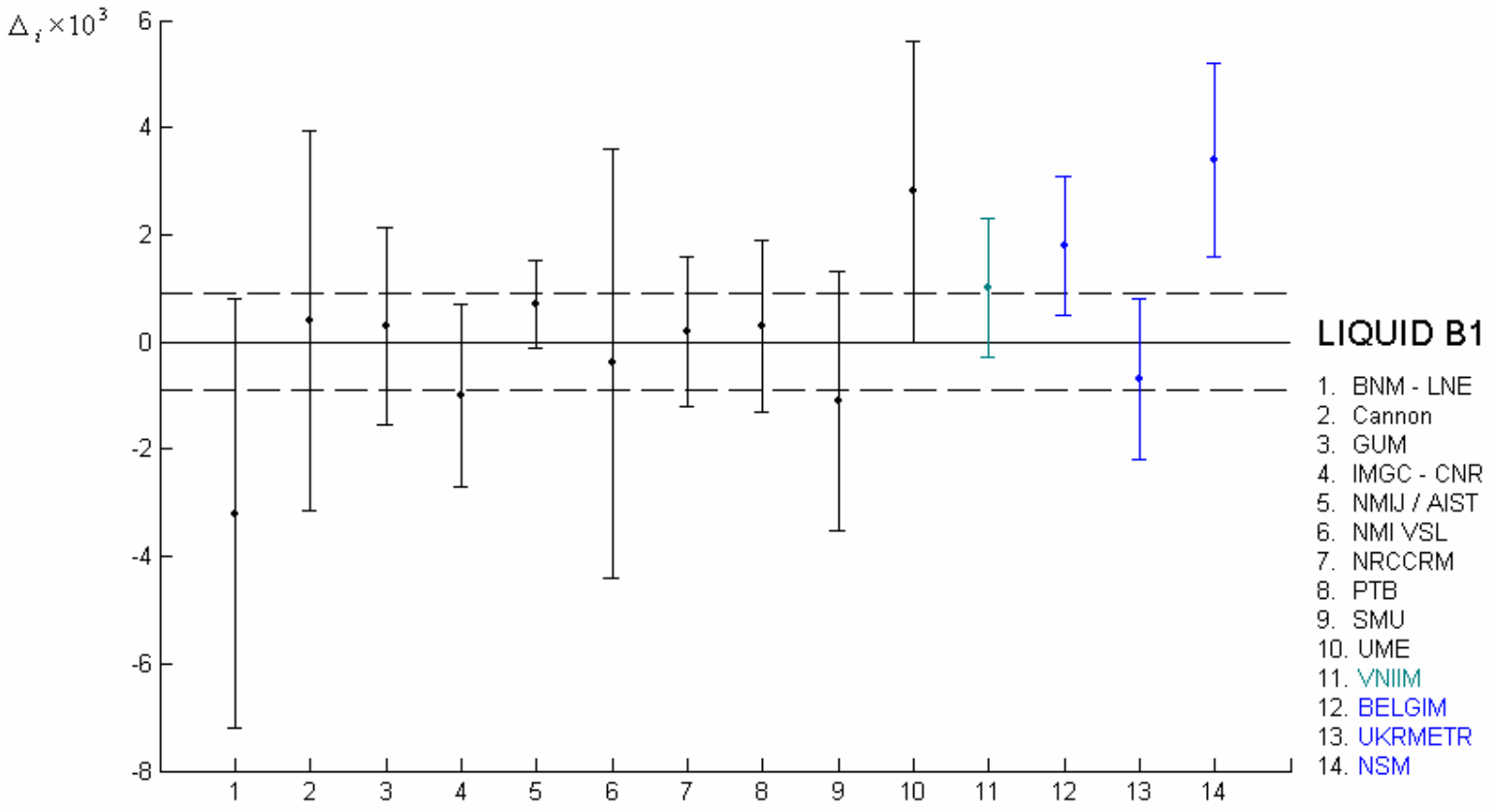


Fig 2-3.


$\Delta_i \times 10^3$
6
4
2
0
-2
-4
-6
-8
1
2
3
4
5
6
7
8
9
10
11
12
13
14
LIQUID B1
1. BNM - LNE
2. Cannon
3. GUM
4. IMGC - CNR
5. NMIJ / AIST
6. NMI VSL
7. NRCCRM
8. PTB
9. SMU
10. UME
11. VNIIM
12. BELGIM
13. UKRMETR
14. NSM