## Improvement unity of measurements of the viscosity liquid medium in the ranges of temperature minus 40 °C to minus 5 °C and 100 °C to 150 °C

A. Neklyudova, A. A. Demyanov D. I. Mendeleyev Institute for Metrology, Russia, St.Petersburg Phone: 8(812)323-96-05, A.A.Tsurko@vniim.ru

### Annotation

The problem of unity measurement of viscosity of liquid medium in the range of temperature minus 40 °C to minus 5 °C and 100 °C to 150 °C has been considered.

Metrological characteristics of standard samples of viscosity of liquid on domestic production have been given.

The aim of the work has been designated and the tasks aiming to solve the lack of tools to verify and calibrate viscosity measurement instruments in the range of temperature mentioned have been formulated.

The results of development, as well as research samples of viscosity of liquid certified in the range of temperature minus 35 °C to minus 5 °C and 100 °C to 150 °C have been presented.

#### Stating a general problem and its relation to important scientific or practical tasks

Ensuring uniformity of measurement of viscosity of liquid is one of the most important conditions for socio-economic development of a country and for a progress in science and technology. It provides the basis for fair trade principles both on domestic and international markets, which is especially important in the context of tough requirements to the accuracy of assessing quality and statistics on the number of natural energy sources.

In view of the fact that a large number of measurement instruments allowing to determine viscosity of liquid in the range of temperature minus 40 °C to 150 °C are used in oil extraction and refinery under state regulation to ensure uniformity of measurement, data on measurement instruments have to go through primary, periodic, extraordinary, inspection or verification expert [1].

In return, verification of measurement instruments of viscosity implies the use of such means of verification as standard samples of viscosity of liquid.

Used in the calibration standard samples applied in verification have to be included into the state registry of standard samples of the Russian Federation, to have established metrological characteristics and to be suitable for use within the predetermined range of temperature.

There currently exist standard samples of viscosity of liquid in Russia, which have been certified in the range of viscosity 1,3 mPa·s (mm<sup>2</sup>/s) to  $1,0\cdot10^5$  mPa·s (mm<sup>2</sup>/s) and in the range of temperature 20° C to 100 °C, with a relative expanded uncertainty with a coverage factor *k*=2,  $Uo\eta(v)$ :

• 0,2 % – in the range of viscosity 1,3 mPa s ( $mm^2/s$ ) to 30 000,0 mPa s ( $mm^2/s$ );

• 0,3 % – in the range of viscosity 30 000,0 mPa $\cdot$ s (mm<sup>2</sup>/s) to 1,0 $\cdot$ 10<sup>5</sup> mPa $\cdot$ s (mm<sup>2</sup>/s).

Standard samples of viscosity of liquid certified in range of temperature minus 40 °C to 20 °C and 100 °C to 150 °C are not produced in the Russian Federation [2].

Hence, activities in the field with improvement of unity of measurement of liquid viscosity in the temperature minus 40 °C to 20 °C and 100 °C to 150 °C are still claimed to be topical issue.

# Analysis of recent research and publications, providing solutions to the problem and relied upon by the author. Addressing the problem, which this work is dedicated

A large number of works devoted to the measurement of viscosity in a wide range of temperature have been published at different times such Russian scientists as M. V. Lomonosov, D. I. Mendeleev, N. P. Petrov, F. N. Shvedov, Soviet scientists as P. A. Rebinder, M. P. Volarovich, G. V. Vinogradov and others.

The team of VNIIM scientists for many years have been engaged into research work in the area of viscometry, but it was only 2015 in the laboratory of state standards of measurement of liquid density and viscosity, when a reference of equipment to reproduce a unit of kinematic viscosity in the range of temperature minus 40 °C to 150 °C was developed and investigated. Therefore, it formed the basis for the possibility to carry out research on viscosity of liquid media in the range of temperature mentioned.

## Setting the aim and formulating tasks of the work

**The aim** of this work should be development and research on standard samples of viscosity of liquid, certified in the range of temperature minus 35 °C to minus 5° C and 100° C to 150 °C.

The following objectives to achieve the aim have been formulated:

- define technical and metrological requirements to the samples of viscosity of fluid, certified in the range of temperature minus 35 °C to minus 5 °C and 100 °C to 150 °C;

- identify materials which the samples will be manufactured from;

- identify a set of equipment for research and validation of the developed samples;

- conduct a study aimed to establish metrological characteristics of the developed samples;

- work out technical documentation needed for tests to approve a type of the developed samples.

Presentation of the material in the research with explanation of the scientific results obtained

Technical and metrological requirements to the developed samples of viscosity of fluid, certified in the range of temperature minus 35 °C to minus 5 °C and 100 °C to 150 °C have been determined by analysis of modern measurement instruments of viscosity and described in accordance with [3, 4] technical specification and technical conditions.

Taking into consideration the terms of technical specifications of the requirements given, the analysis of existing mineral and synthetic oils having Newtonian flow regime has been done. Further, seven types of oils and some of their mixtures have been selected on nominal values of viscosity at various temperatures, and, finally, their actual values of kinematic and dynamic viscosity reported in chart 1 have been determined.

	Average measured values of dynamic and kinematic							
	viscosity							
The name oil / mixture	mPa·s							
	Temperature measurements, °C							
	-5	-15	-25	-30	-35	150		
Synthetic oil 40	1470	4300	13780					
Synthetic oil 60	2055	4997	16686					
Synthetic oil 30	614			8172	15625			
Synthetic oil 30A			3563	7220				
Semi-synthetic oil 40	727			6565				
Mixture of mineral oils 10		_			-	22,1		
Mixture of mineral oils 30						42,6		
Mixture of mineral oils 60						62,3		
The relative expanded uncertainty of the measured values of viscosity of the oils, with the								

Chart 1. The determined results of the actual values of viscosity.

Coverage factor k=2,  $Uo\eta(v)=\pm(0,2...2,0)$  %. After analyzing the measured values of viscosity of the oils under study, it has been decided to create five samples of viscosity:

• two samples certified in the range of temperature below 0 °C: REV-10000-1 and REV-20000-1 based on synthetic oils;

• three samples, certified in the range of temperature above 100 °C: REV-10000, REV-30000 and REV-60000 based on mixtures of mineral oils.

Establishment of metrological characteristics of the developed samples in the range of temperature 100 °C to 150 °C has been carried out on GET 17-96, the reference of equipment, developed while implementing the measures to improve it.

Establishment of metrological characteristics of the samples in the range of temperature minus 35 °C to minus 5 °C has been carried out on the state standard of the units of dynamic and kinematic viscosity of the 2<sup>nd</sup> discharge ranging 0,2 to 20000,0 mPa⋅s (mm2/s) № 2302-0229 (3.1.ZZB.0229.2016).

The documentation to be included into the registry of standard samples of fluid viscosity of the Russian Federation in the range of temperature minus 35 °C to minus 5 °C and 100 °C to 150 °C has been worked out in accordance with requirements [3, 6, 7, 8].

The study of the metrological characteristics of samples REV-10000-1 and REV-20000-1 has been carried out to the full extent provided for normative documentation.

The study of the metrological characteristics of samples REV-10000, REV-30000 and REV-60000 has been carried out upon a reduced program, as these samples have been already included into the state registry of standard samples of the Russian Federation in the range of temperature 20 °C to 100 °C [3].

In the course of the study the following metrological characteristics of the developed samples have been determined: certified values of viscosity, relative expanded uncertainty of the certified values; also homogeneity both inside a sample unit and between instances in a batch, short and long term instability have been investigated.

Charts 2-6 present the metrological characteristics of the developed samples determined in the study.

		The name of the certified				Certified value of physical quantity					
		characteristics, the designation of				at a temperature, °C					
		al quar	l quantities			(-35,00±0,02)		),00±0,02)			
REV-10000-1 Dynamic viscos Kinematic visco		osity, m	sity, mPa∙s		1	15625		8172,2			
		osity, n	sity, mm²/s		1	17676		9284,0			
Relativ	Relative expanded uncertainty (U) wi			th the coverage		2			1,5		
factor <i>k</i> =2, %				_			2		1,5		
Chart 3. Metrological characteristics of sample REV-20000-1.											
	The	e name of the certified	k	Certified value of physical quantity at a							
Index	charad	cteristics, the designat	tion	n temperature, °C							
	of un	units of physical quantities			(-25,00±0,02)		(-15,00±0,02)		,00±0,02)		
REV-	Dyr	namic viscosity, mPa∙s	S	16	6686,0		4997,3		2054,7		
20000- 1	Kine	ematic viscosity, mm <sup>2</sup> /	s	18807,0			5675,3		2351,6		
Relativ	Relative expanded uncertainty (U) with			th 10							
the coverage factor $k=2, \%$				± 1,2			± 0,5				
Chart 4. M	Metrolog	gical characteristics of									
	The n	ame of the certified Certified value of physical quantity at a temperature,						perature,			
Index	cha	aracteristics, the	D°								
Index	desi	gnation of units of		∆t=±0,01°C		;	∆t=±0,02°C				
	ph	ysical quantities	20,0	0	40,00	50,00	100,00	125,00	150,00		
REV-	Dynar	nic viscosity, mPa·s	9386	6,0	1823,0	918,1	77,02	-	-		
10000	Kinem	atic viscosity, mm <sup>2</sup> /s	1050	0,0	2066,0	1047,0	90,73	40,89	22,07		
Relative	Relative expanded uncertainty (U)			± 0,2							
with th	ne cove	rage factor <i>k</i> =2, %			± 0,2			± 0,30			
Chart 5. I	Metrolo	gical characteristics of	f sampl								
		name of the certified		Certified value of physical quantity at a							
Index		haracteristics, the				temperature, °C					
		signation of units of		∆t=±0,01°0			L	∆t=±0,02°	-		
	р	hysical quantities	20,0	00	40,00	50,00	100,00	125,00	150,00		
REV-		amic viscosity, mPa·s	2828	30,0		2273,0	175,70	-	-		
30000	Kiner	natic viscosity, mm <sup>2</sup> /s	3159	31590,0 5874,0		2586,0	206,33	89,04	42,57		
Relative expanded uncertainty ( <i>U</i> ) with the coverage factor <i>k</i> =2, %				± 0,2			± 0,30				

Chart 2. Metrological characteristics of sample REV-10000-1.

Chart 6. Metrological characteristics of the sample REV-60000.

Index	The name of the certified	Certified value of physical quantity at a temperature,							
	characteristics, the	D°							
	designation of units of	Δ	t=±0,01°C	Δt=±0,02°C					
	physical quantities	20,00	40,00	50,00	100,00	125,00	150,00		
РЭВ-	Dynamic viscosity, mPa·s	50550,0	9133,0	3622,0	279,91	-	-		
60000	Kinematic viscosity, mm <sup>2</sup> /s	56430,0	10320,0	4120,0	328,11	129,61	62,31		
Relative expanded uncertainty (U)		± 0,2			± 0,30				
with the coverage factor <i>k</i> =2, %									

Conclusions according to the research and perspectives of further research in this direction conclusions on the research and perspectives on further research in this area

The developed and investigated samples of viscosity of fluid, certified in the range of temperature minus 40 °C to minus 5 °C and 100 °C to 150 °C allow to solve the lack of tools to verify and calibrate viscosity measurement instruments in the range of temperature mentioned, and could be also applied in tests to approve a type, in metrological certification of viscosity measurement techniques and errors control of measurement techniques in the course of their application.

The developed samples could be applied in comparison and calibration of measurement standards as liquid-comparators.

In the future we are planning to conduct studies aimed at defining dependence of dynamic viscosity of the developed samples on high shear rates, and in the case of sustainable results to justify the use of the samples for calibration of minirotory viscometers and simulators for cold cranking of the engine.

## List of sources used

1. The Russian Federation. Laws. On ensuring the uniformity of measurements [Text]: Feder. the law: [adopted by the State. Duma 11.06.2008 G.: approve. SAFECOM Federation 18.06.2008]. – [5th ed.]. - M.: OS-89, [2008]. – 31, [1] p.; 21 cm. – (Current law). – ISBN 978-5-9957-0006-7.

2. Official website of Rosstandart. Federal information Fund for ensuring the uniformity of measurement. Standard specimens of approved types [Electronic resource]. – Date of access 14.11.2016 – URL: http://www.fundmetrology.ru/default.aspx .

3. GOST 8.315-97 GSI. Standard samples of composition and properties of substances and materials. The main provisions (with amendments No. 1) [Text]. – Instead of GOST 8.315-91;]. 1998-07-01. Minsk: Interstate. Council for standardization, Metrology and certification, Moscow, STANDARTINFORM publ, COP. 2008. - 25 p.

4. GOST 2.114-95 ESKD. Technical specifications (with changes № 1, 2, as amended). [Text]. - 1996-07-01. Minsk: Interstate. Council for standardization, Metrology and certification, Moscow, STANDARTINFORM publ, COP. 2008. - 15 p.

5. GOST ISO/IEC 17025-2009 General requirements for the competence of testing and calibration laboratories [Text]. – 2012-01-01. Minsk: Interstate. Council for standardization, Metrology and certification, Moscow, STANDARTINFORM publ, COP. 2012. - 28 p

6. GOST R 8.694-2010 (ISO Guide 35:2006) GSI. Standard samples of materials (substances). General statistical principles of determination of metrological characteristics [Text]. – 2012-07-01. M.: STANDARTINFORM, COP. 2012. – 69 p.

7. MI 3300-2010, GSEI. Recommendations for the preparation, execution and review of materials testing of standard samples for the purposes of type approval. Characteristics [Text]. – 2010-12-11. Yekaterinburg.: FGUP "UNIIM". 2012. – 40 p.

8. GOST 8.025-96 ICG. National verification scheme for measuring instruments of viscosity of the fluid. [Text]. – 2012-01-01. Minsk: Interstate. Council for standardization, Metrology and certification; M.: IPK Publishing house of standards, COP. 1997. – 8 p.