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ORGANISATION INTERNATIONALE DE MÉTROLOGIE LÉGALE

INTERNATIONAL RECOMMENDATION

Third Commitee Draft of RecommendationSecond Draft

Newtonian viscosity standard liquids for the calibration and verification of viscometers

Subcommittee: TC17/SC5 Viscometry

Secretariat: Russia

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P.R.of China,
Cuba, Poland,
Russia, Slovakia,

USA,

Germany,

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Observing countries:

Austria, Egypt, Indonesia, Serbia, Bulgaria, Finland, Japan, Slovenia, Czech Republic, France, Romania, Spain

Liaisons:

ISO Central Secretariat

Explanatory Note

The International Recommendation "Newtonian Viscosity Standard Speciments for the Calibration and Verification of Viscometers" was developed with a view to harmonizing the procedure for preparation and certification of standard speciments of viscosity of liquids used for the metrological testing of instruments measuring the viscosity.

The ~~First~~ Second Draft of the Recommendation was commented by the USA, Japan, Poland, Bulgaria, Netherlands and Slovakia. For the most part verbal alterations were suggested. The summary of comments is attached to this ~~Second~~Third Draft.

The present ~~Second~~Third Draft has been worked out taking into account the received   remarks.

~~this this~~This recommendation specifies requirements to liquids for the preparation and calibration of Newtonian viscosity standard specimens (reference standard liquids), according to the OIML-DI No. 17 ~~,,Hierarchy scheme for instruments measuring the viscosity of liquids"~~[1].

Newtonian viscosity standard specimens are used for the calibration or verification of reference standard viscometers, working standard viscometers or ordinary viscometers and other viscosity measuring devices. For viscosity measurements of Newtonian liquids, instruments, calibrated or verified by means of these viscosity standard specimens are accepted as traceable to National Standards of Viscosity according to ISO/IEC 17025:2009 [2]~~.~~

The Recommendation uses the following general **terms** and **definitions**:

Newtonian reference liquids (RL) - are liquids in which the rate of shear is

proportional to the shearing stress. The constant ratio of the shearing stress to the rate of shear is the viscosity of the liquid[1].

Newtonian viscosity standard specimens (VSS) - are specimens of Newtonian reference liquids for which the dynamic viscosity and the kinematic viscosity have been determined traceable to National Standards and are documented in a calibration certificate.

Samples of the same RL may be qualified as reference and working VSS, depending on the order of standard viscometer used for calibration

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[1] Samples of the same RL may be qualified as reference and working VSS, depending on the order of standard viscometer used for calibration.

The SI unit of kinematic viscosity is m^2/s; for practical use, a submultiple (mm^2/s) is more convenient.

The SI unit of dynamic viscosity is Pa $\times$ s, for practical use, a submultiple (mPa $\times$ s) is more convenient.

The range of dynamic viscosity covered by Newtonian viscosity standard specimens ranges from about 4 $\times$ 10^{-1} mPa × s to 10^5 mPa $\times$ s at 20 °C. This corresponds to kinematic viscosities between about 4 $\times$ 10^{-1} mm2/s and 1.2 $\times$ 10^5 mm^2/s. Standard specimens may be calibrated and used at various temperatures (usually between

minus 40 °C and +150 °C) and atmospheric pressure.

Samples of the same RL may be qualified as reference and working VSS, depending on the class of standard viscometer used for calibration (see OIML-DI No.17).

From the viscosity values $\boldsymbol{v}_1$ and $\boldsymbol{v}_2$ determined with the two standard viscometers the arithmetic mean value is calculated.

The relative deviation $\varepsilon_v = \left| \dfrac{v_2 - v_1}{v} \right| \times 100\%$ for the arithmetic mean value v is shown in the Table 1.

Table 1

Viscosity, mm^2/s	The relative deviation for the reference VSS ε_v, %	The relative deviation for the working VSS ε_v, %
$v \leq 50$ mm^2/s	$\varepsilon_v \leq 5 \times 10^{-2}$ ~~$\varepsilon_v \leq 5 \cdot 10^{-2}$~~ ,%	$\varepsilon_v \leq 1 \times 10^{-1}$ ~~$\varepsilon_v \leq 1 \cdot 10^{-1}$~~
$v > 50$ mm^2/s		~~$\varepsilon_v \leq 2 \cdot 10^{-1}$~~

The density of standard specimens shall be determined with a relative uncertainty of ≤ 2 $\times$ 10^{-4} at each calibration temperature based on ISO 3838:2004 [4].

Thermostatic baths with automatic temperature control that allow the use of at least two viscometers at the same time. The temperature change during a measurement and temperature gradient between the positions of any thermometer and viscometer shall be less than 0.005°C for the reference VSS and shall be less than 0.01°C for the working VSS.

Time measuring devices that allow the determination of the flow time with a relative uncertainty of 0.02%.

Liquids which are used as reference liquids for viscosity measurements shall meet the following requirements:

a) long-term stability of the viscosity

(little or no change of viscosity when stored at ambient conditions over a period of 6 ~~several~~ months)

b) thermal stability

(viscosity, irreversible during thermal stability testing)

c) Newtonian flow behaviour

d) good solubility in several cleaning agents~~ms~~

(as to enable appropriate cleaning of instruments)

e) sufficiently transparent

(for the use in glass capillary viscometers)

f) viscosity-temperature coefficient as low as possible

(in order to reduce the requirements for temperature control)

g) stable composition

h) no sorption of water, inert with respect to air, glass and metal

i) no or low toxicity, high flash point

The fulfillment of requirements a) to c) has to be experimentally investigated for each liquid according to the specifications given in clause 7.

Requirements d) to i) may be checked by the use of information provided by the supplier of the liquid or by literature data.

Examples of liquids which meet these requirements and which are commonly used as reference liquids are listed in Annex~~ppendix~~ 1

With respect to dissolved gases, reference liquids shall be in equilibrium with air. Reference liquids shall be stored in closed containers at room temperature in the dark. Brown glass bottles or HDPE (high density polyethylene) containers have prooved to be suitable.

Note:

It is not recommended to keep reference liquids under inert gas atmosphere since the unavoidable exposure to air during the use of reference liquids may change the viscosity.

The number of reference liquids and the viscosity range covered depends on the industrial needs of each country.

If the viscosity gradation of the available base liquids is not sufficient, mixtures of suitable viscosity may be prepared from base liquids of neighbouring viscosity. The tests described in clause 8 have to be undertaken for each mixture, even if they have already been performed with the components.

The mixing ratio of both components necessary to achieve a certain viscosity value can approximately be calculated by the application of mixing-equations. Examples of such equations are given in Annexppendix 2. In addition, it is recommended to prepare one or several test mixtures of small quantities in order to achieve a good approximation of the expected viscosity value.

The facilities necessary for the preparation of the final mixture depend on the amount of liquid (usually between 5 l and 50 l) and the viscosity and involves routine laboratory equipment. The mixing device (stirrer, pump) shall not produce high shear rates in the liquid (avoid high-speed rotation and narrow gaps) in order to avoid degradation of liquids of high-molecular weight. Liquids of higher viscosity may be heated (usually up to 50 °C or 80 °C) in order to facilitate the mixing.

The time necessary to reach a complete and homogeneous mixture depends on the mixing facility, the viscosity and solubility properties of the components. The completeness of mixing can be checked by viscosity measurements on samples taken after different time of mixing. Typical mixing times may be between 24 h for prepearing 5 liters of mixture and 100 h for 50 liters of mixture.

For each batch of liquid to be used as reference liquid the tests described in clause 8.1 to 8.3 have to be performed.

8.1 Long-term stability

At least, four samples of the liquid are taken in intervals of approximately 3 months. The viscosity of each sample is determined at a fixed reference temperature (mostly 20 °C or 23 °C) with two viscometers of the same nominal viscometer constant following the procedure described in clause 9 8 .1. Since the viscosity changes to be measured are smaller than the uncertainty of an individual viscosity determination the same pair of viscometers and the same temperature measuring device shall always be used in order to eliminate the main uncertainty contributions of systematic uncertainty sources. From a plot of viscosity versus time the relative viscosity change within 6 months (ageing coefficient) is determined.

The ageing coefficient shall be less than 7×10^{-3} .4 • 10⁻³

If the liquid is to be used for reference viscosity standard specimens only, the ageing coefficient may be higher, but shall not exceed $4 \cdot 10^{-3.}$

Note :

Higher values of the ageing coefficient may be accepted if the total uncertainty (u in table

1 u_{cert} in Table 2) is kept within the specifications given in clause 9.5.

It may be necessary - especially in the case of mixtures - to monitor the ageing for a longer period since the ageing coefficient may decrease with time.

8.2 Thermal stability

The investigation of the thermal stability involves the following temperatures:

Tref -   reference temperature (usually 20 °C or 23 °C)

Tmin; Tmax - minimum and maximum temperature at which VSS of the liquid shall be calibrated (for example -40 °C, +100 °C)

~~*To1, To2*~~ ***To*** $_1$ **, *To*** $_2$ -   minimum and maximum temperature to which a VSS may be exposed$_{10}$ before use.

The following procedure is recommended:

a) Influence of short-term heating/cooling and duration of measurement

- The duration of measurements must be from 200 to 2000 s

- Fill two viscometers of the same nominal constant with a test portion of the liquid and measure the viscosity at *Tref.*

- Heat the viscometers to *Tmax* tor 1 h~~our~~ with the capillary and the measuring bulb being filled with the liquid.

- Repeat the viscosity measurement at *Tref.* The relative difference between the viscosities determined before and after heating shall not exceed 4 • $\underline{\times}$-10^{-3} ~~yr-1~~.

- Apply the same procedure (step a) to c).

- he viscosity change after cooling to *'Tmin-.* shall not exceed the value stated above.

- Measure the viscosity of a fresh sample at *Tmax/Tmin),* keep the viscometer at *Tmax/Tmin)* for ~~one hour~~<u>1 h</u> with the capillary and measuring bulb being filled with the liquid. Repeat the viscosity determination at *Tmax/Tmin)*

b) Influence of high and low temperature during delivery to the user

- After viscosity determination at *Tref,* two <u>VSS</u>~~SSp~~ containers (mostly brown glass bottles) are filled with the liquid and sealed. One container is stored at *To/,* the other at *To2* for one week. After storage, the viscosity at *Tref* is measured again using the same pair of viscometers and the same temperature measuring device.

- The change of viscosity before and after storage shall not exceed 4 $\underline{\times}$• 10^{-3} ~~yr-1~~.

8.3 **Newtonian flow behaviour**

Tests for Newtonian flow behaviour shall be performed at T_{min} using a viscometer that allows a variation of shear rate, for example pressure driven capillary devices or rotational viscometers.

9.1 Measurement of kinematic viscosity

The general procedure of viscosity determination and the instruments and other laboratory equipment to be used shall be in accordance with ISO 3104:1994 [5] and ISO 3105:1994 [6]. Additional requirements and deviations from ISO 3104:1994 [5] and ISO 3105:1994 [6] are described below in order to meet the uncertainty requirements given in OIML-DI No. 17[1].

Standard viscometers

Calibration of reference VSS:

Use two primary or secondary standard viscometers of approximately the same viscometer constant. The ($k=2$) uncertainty of viscometer constant, including the uncertainty of kinetic energy correction, must not exceed 0.03 1% at low and 0.8% at high values of the constant.

Calibration of working VSS:

Use two reference standard viscometers of approximately the same viscometer constant. The ($k=2$) uncertainty of viscometer constant, including the uncertainty of the kinetic energy correction, must not exceed 0.2 % at low and 1 % at high values of the constant.

Temperature measurement and devices for temperature control

Use suitable thermometers with a 0.005 °C scale interval. It may be necessary to apply corrections according to the calibration certificate. The depth of immersion shall be the same during calibration and use.

Measurement

The handling of viscometers such as filling and cleaning must be done in accordance with ISO 3104:1994 [5] and 3105:1994 [6].

When cleaning the standard viscometers with sulphochromic acid (saturated aqueous solution of $K_2Cr_2O_7$ mixed with H_2SO_4 in a ratio of about 1:1) the temperature should not exceed 30 °C and the cleaning time 2 h. This acid shall be used only for cleaning heavily contaminated viscometers.

With each viscometer a series of at least 5 flow-time measurements is carried out. The results shall meet the following condition

$$\left| \frac{t_{max} - t_{min}}{t} \right| \leq 1 \times 10^{-3}, \qquad \textbf{(1)}$$

where t_{min} and t_{max} are the shortest and longest flow time and $\bar{t}$ the arithmetic mean of the series.

If both viscosity determinations differ by more than the repeatability of the flow-time measurement, the uncertainty shall be increased accordingly. The relative difference shall not exceed 0.7×10^{-3}

9.2 Calculation of kinematic viscosity

For each viscometer the kinematic viscosity v is calculated according to eq. (2)

$$v = C \cdot (t - \Delta t_h) \cdot \frac{g'}{g} \, , \qquad (2)$$

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with

C – constant of the viscometer;

Δt_h kinetic energy correction ("Hagenbach"-correction),

g - acceleration due to gravity at the place of measurement

g' - acceleration due to gravity at the place of calibration of the standard viscometers (see calibration certificate of the viscometer).

For standard size capillary viscometers with constants C > 0.1 mm^2/s^2 the kinetic energy correction may be neglected and equation 2 takes on the following form:

$$v = C \cdot t \cdot \frac{g'}{g} \qquad\qquad 3)$$

For viscometers whose constants C < 0.1 mm^2/s^2 the kinetic energy correction may be significant if the minimum 200-s flow time is not observed.

From the viscosity values v_1 and v_2 determined with the two standard viscometers the arithmetic mean value $\bar{v}$ is calculated.

9.3 Measurement of density, calculation of dynamic viscosity

A suitable method for measurement of density is the use of two calibrated pyknometers of typically 25 ml or 50 ml of volume. Routine laboratory method that correct for air buoyancy can keep the relative uncertainty of mass determination below 2×10^{-5}.

An oscillation U-tube density meter use for measuring the density for a viscosity of less than 700 mPa × ÷ s.

For liquids that meet the stability requirements listed in clause 8, the density is not significantly changed by ageing. Therefore, it is necessary to determine the density of each batch of reference liquid only once.

The dynamic viscosity η is calculated from the kinematic viscosity and the density p determined at the same temperature according to

η = v× ÷ρ   (4)

9.4 Determination of the temperature coefficient of viscosity

The knowledge of the temperature coefficient of viscosity U_v defined as

$$U_v = - \frac{1}{v} \frac{dv}{v} <$$

is necessary at each calibration temperature T in order to calculate the uncertainty of viscosity determination. It shall also be stated in the calibration certificate, so that the user of the VSS ~~SSp~~ can adapt the quality of temperature control necessary to use the VSS ~~SSp~~ within its uncertainty. U_v shall be calculated from a 3 parameter equation describing the viscosity-temperature behaviour of the liquid. The parameters shall be determined from viscosity values at T_{max} T_{min} and a third temperature approximately in the middle of this interval.

Note :

A great number of viscosity-temperature equations are available. The use of the Vogel-equation for determining U_v is given as an example in Annexppendix 3.

Within the uncertainty of the temperature coefficient of viscosity, the change of U_v due to ageing can be neglected. Therefore, it is sufficient to determine the viscosity-temperature behaviour only once for each batch of reference liquid.

9.5 Measurement uncertainty

It is recommended that the uncertainty of the measurements be calculated according to the "Guide to the expression of uncertainty in measurement". Convinent summaries can be found in [ ]JCGM 100:2008 [7]. The coverage factor should be $k=2$ corresponding to an interval of confidence of 95 %. An example is given in Annexppendix 4.

The main contribution to the uncertainty arises from the uncertainty u_c of the viscometer constant C.

According to OIML document D 17OIML D 17 [1], the (k=2) uncertainty of the reference VSS must not exceed 0.2 % to 1 % (depending on the viscosity) and that of

the working VSS must not exceed 0,3 % to 2 % (depending on the viscosity). These limiting values are met by the requirements described in clause 4 and include a period of 6 months for the validity of the calibration. It also includes an estimated maximum contribution of up to 20 % of the total uncertainty in order to cover influences of minor importance like deviation from vertical alignment of the viscometer and uncertainty of the gravitational acceleration. Table 1 shows the resulting measurement uncertainties provided that the requirement of clause 4 are met.

Table 1

Uncertainty of viscosity measurement of reference and working VSSs at the limits of the viscosity range of this recommendation (see clause 4~~1~~)

	$C,$ mm^2/s^2	u_c	U_v $1/°C$	u_v	u_{cert}
Reference VSS	0.003	1×10^{-3}	0.07	1.2×10^{-3}	2×10^{-3}
	50	8×10^{-3}	0.10	7.9×10^{-3}	1×10^{-2}
Working VSS	0.003	2×10^{-3}	0.07	2.4×10^{-3}	3×10^{-3}
	50	1×10^{-2}	0.10	1.0×10^{-2}	2×10^{-2}

VSS	50	$8 \cdot 10^{-3}$	0.10	$7.9 \cdot 10^{-3}$	10^{-3} $1 \cdot 10^{-2}$
working VSS	0.003 50	$2 \cdot 10^{-3}$ $1 \cdot 10^{-2}$	0.07 0.10	$2.4 \cdot 10^{-3}$ $1 \cdot 10^{-2}$	$3 \cdot 10^{-3}$ $2 \cdot 10^{-2}$

C   viscometer constant

u_c relative uncertainty of the viscometer constant including uncertainty of the kinetic energy correction

U_v temperature coefficient of viscosity (typical values are used in the table)

u_v relative uncertainty of the viscosity determination using two standard viscometers, including an estimated maximum contribution of 20_%

from remaining sources of error

u_{cert} uncertainty valid for 6 months (including possible ageing) as should be stated in the certificate

9.6 Calibration certificate

For each VSS a calibration certificate shall be issued covering the following:

- type and purpose of the standard specimen (reference / working)

- identification number of the VSS

- dynamic and kinematic viscosity and temperature coefficient of viscosity at one or more temperatures

- uncertainty of the stated viscosity values including the possible ageing during the period of validity

  - date of certification and period of validity

- reference to the metrological basis of the viscosity determination (usually the viscosity of water as stated in ISO TR 3666[8])

  - address and name of the calibration laboratory

  - example of calibration certificate is given in Annexppendix 5.

1. OIML D 17

2. ISO 17025 (1999)

3. Hierarchy scheme for instruments measuring the viscosity of liquids

4. ISO 3104 (1994)   Petroleum products - Transparent and opaque liquids

   Determination of kinematic viscosity and calculation of dynamic viscosity

5.   ISO 3105 (1994) Glass capillary kinematic viscometers - Specifications and operating instructions

6.   ISO 3838 (1983) Crude petroleum and liquid or solid petroleum products - Determination of density or relative density - Capillary spoppered pyknometers and graduated bicapillary pyknometer methods

7. ISO TR 3666(1997): Viscosity of water

8. EA-4/02 (1999) Expression of the uncertainty of measurement in calibration. European Cooperation for Accreditation of Laboratories.

9. NIST 1297 (1994) Guidelines for evaluating and expressing the uncertainty of NIST measurement results, National Institute of Standards and Technology (USA).

10. ~~NBS Monograph 55, NBS Viscometer Calibrating Liquids and Capillary Tube Viscometers~~

11. ~~Journal of Research of the National Bureau of Standards, Vol.52, No.3, March 1954, Paper 2479~~

12. ~~ASTM Method of Test D 2162, Basic Calibration of Master Viscometers and Viscosity Oil Standards~~

1. OIML D 17 : 1987 Hierarchy scheme for instruments measuring the viscosity of liquids.

2. ISO/EIC 17025 : 2009 General requirements for the competence of testing and calibration laboratories.

3. OIML D 2 : 2007 Legal units of measurement.

4. ISO 3838 : 2004 Crude petroleum and liquid or solid petroleum products - Determination of density or relative density - Capillary-stoppered pyknometer and graduated bicapillary pyknometer methods.

5. ISO 3104 : 1994 Petroleum products - Transparent and opaque liquids - Determination of kinematic viscosity and calculation of dynamic viscosity.

6. ISO 3105 : 1994 Glass capillary kinematic viscometers - Specifications and operating instructions.

7. JCGM 100 :2008 Evaluation of measurement data — Guide to the expression of uncertainty in measurement.

8. ISO TR 3666 : 1997 Viscosity of water.

9. EA-4/02 (1999) Expression of the uncertainty of measurement in calibration.

European Cooperation for the Accreditation of Laboratories.

Examples of liquids which are commonly used as reference liquids:

Type of liquid	viscosity
chemically pure hydrocarbons (organic solvents)	0.3 to 1.5
mineral oils without polymer additives	1 to 1 000
hydrogenated polyalphaolefin liquids	50 to 5 000
polyisobutylenes	>5 000

Water should only be used for the calibration of primary standard viscometers because of the problems associated with the high surface tension. Its use is not recommended within the scope of this document.

The use of silicon oils in glass viscometers should be avoided unless the hydrophobian

properties are taken into account by special investigations.

Examples of mixing equations

The kinematic viscosity v of a binary mixture of components with viscosities v_1 and v_2 can approximately be calculated using one of the following equations:

$$\ln v = \varphi_1 \cdot \ln v_1 + \varphi_2 \cdot \ln v_2$$

$$1/v = w_1 \cdot 1/v_1 + w_2 \cdot v_2$$

The mass fractions w_1, w_2 denote the mass ratio of the components to the total mass of the mixture.

The volume fractions φ_1, φ_2 denote the volume ratio in relation to the total volume of the mixture.

Example of determination of the temperature coefficient of viscosity

Based on the Vogel-equation for the temperature dependence of the viscosity of liquids

$$v = A \cdot e^{B/(T+C)}$$

the temperature coefficient of viscosity U_v is given by

$$U_v = 2{,}303 \left| \frac{\log v_1 - \log v_2}{T_2 - T_1} \right|$$

U_v is calculated in 1/°C if T is measured in °C and v_1, v_2, v_3 in mm²/s.

C is calculated according to

$$C = \frac{P \cdot T_1 - Q \cdot T_3}{Q - P},$$

with

$P = (\log v_1 - \log v_2) \cdot (T_3 - T_2)$

$Q = (\log v_2 - \log v_3)(T_2 - T_1)$

v_1, v_2, v_3 are the kinematic viscosities at the temperatures T_1, T_2, T_3 and T is the temperature for which the temperature coefficient of viscosity is calculated. The Vogel-equation shall not be used for extrapolations.

Example for the calculation of the uncertainty of measurement for the viscosity of Newtonian viscosity standard specimens

The calculation of uncertainty of measurement is performed according to the method described in EA-4/02 , using the following relations:

For a measurement y depending on influence quantities x_i (i = 1,...r) the relative variance $(S')^2$

is given by:

$$\lessgtr s_y \Leftarrow \sum_{i=1}^{r} \lessgtr s_y <$$

For repeatedly measured quantities s_{xi} is the relative empirical standard

deviation. If for an influence quantity x_i, only an upper limit a_u and a lower limit a_l are known: $U = \dfrac{1}{2} \langle a_u - a_e \rangle$ an estimate value for the relative variance of X_i is calculated from the relative uncertainty U according to:

$$\langle S_{xi}^2 \rangle = \frac{1}{3} \langle U^2 \rangle \qquad (2)$$

Applying a coverage factor $k = 2$ the relative uncertainty of the measurement and is given by:

$$U_y = 2 \cdot S_y \qquad (3)$$

This means that in the case of a normal (Gaussian) distribution the-limits of uncertainty are for 95% level of confidence.

As an example, the viscosity of a reference VSS is calculated using the following assumptions:

- the viscosity v of the VSS is determined with two standard viscometers of constant C

- uncertainties arising from surface tension effects and end effects (kinetic energy correction) are included in the uncertainty of the viscometer constant.

The following contributions to the uncertainty are taken into account:

  - uncertainty of the viscometer constant

  - uncertainty of the sample temperature

  - uncertainty of flow-time determination

The influence of other parameters like:

  - drainage error

  - viscous heating

- deviations of vertical alignment of the viscometer

- uncertainty of the local value of acceleration due to gravity

is considered as negligible.

Since the viscosity determination is carried out using two viscometers of the same accuracy and since the viscosity of the VSS is calculated as the average of both measurements, the uncertainty calculated using eq. 3 has to be divided by a factor $\sqrt{2}$.

As an example, the viscosity of a reference VSS is calculated using the following assumptions, showing in Table 2:

Table 2

N	Contribution to the uncertainty	Value
1	The relative uncertainty of viscometer constant C_v in the calibration certificate, $S_{c,}$ %	<u>10</u>
2	The relative uncertainty of temperature control and measurement, S_t, %	0.04
3	The relative uncertainty of flow time measurement, S_{ft}, %	0.02
4	The relative uncertainty of the timing device S_w, %	0.01

According to the specifications of clause 9.58.1 the relative uncertainty of the viscometer constant is taken to be:

U'c = $1 \cdot 10^{-3}$

which leads by applying eq. (2) to

$(S'_c)^2 = 33 \cdot 10^{-8}$.

The relative uncertainty of the timing device (systematic uncertainty contribution):

$$U'_w = 2 \cdot 10^{-4}$$

$$(S'w)^2 = 1{,}3 \cdot 10^{-8}$$

Random uncertainty of the. flow-time measurement: the relative empirical standard deviation for $n = 5$ repeated flow-time measurement is assumed to be $3 \cdot 10^{-4}$. This standard deviation has to be multiplied by a factor $t = 1{,}4$ (for $k=2$ and $n = 5$). The corresponding relative variance is given by

$$\left(S'_R\right)^2 = \left|\frac{\left(3 \cdot 10^{-4} \cdot 1{,}4\right)^2}{5}\right| = 3{,}5 \cdot 10^{-8}$$

Uncertainty of temperature measurement: two contributions have to be considered, the calibration uncertainty of the thermometer which is taken as 0,005°C and the temperature gradient between the position of the thermometer and the viscometer in the thermostat which is 0,005°C. In the worst case both contributions have to be added resulting in an uncertainty of the sample temperature of 0,01°C. The corresponding contribution to the relative uncertainty of the viscosity U'_T is calculated by multiplying with temperature coefficient of

viscosity which is assumed to be $U_v = 0,07\ 1/°C$:

$U'_T = 0,07 \cdot 0,01 = 7 \cdot 10^{-4}$

The corresponding relative variance according to eq. (2) is:

$(S'_T)^2 = 16 \cdot 10^{-8}$

The relative variance of the viscosity measurement with one viscometer is given by:

$$S'^2_v <= S'^2_c << S'^2_w << S'^2_R << S'^2_T <= 53,8 \cdot 10^{-8}$$

$S'_v = 7,33 \cdot 10^{-4}$

This value has to me multiplied by $k = 2$ (95% level of confidence) and divided by $\sqrt{2}$ because two viscometers are used. In order to account for undetected systematic errors an additional contribution of 20% is taken into account thus the total relative uncertainty is:

U'_v $=$ $1,24 \cdot 10^{-3}$

Example for the calibration certificate for viscosity reference standard

CERTIFICATE OF CALIBRATION

Name of the calibration laboratory

Date of issue: Certificate number:

REFERENCE STANDARD OF VISCOSITY

Standard type: Lot No: Expiry Date:

Temperature, ^0C	Viscosity mm^2/s mPa $\cdot\times\cdot$ s	
	Kinematic	Dynamic

Uncertainties:

Adress:
Tel.

Fax: