

ORGANISATION INTERNATIONALE DE MÉTROLOGIE LÉGALE

INTERNATIONAL RECOMMENDATION

**Second Draft**

**Newtonian viscosity standard liquids for the calibration  
and verification of viscometers**

Subcommittee: TC17/SC5

Secretariat: Russia

**Participating countries:**

P.R.of China,  
Cuba,  
Poland,  
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Newtonian viscosity standard liquids for the calibration and verification of viscometers .....	3
Explanatory Note .....	3
1 Scope and field of application .....	4
2 Definitions .....	4
3 Units of measurement .....	4
4 Metrological requirements .....	5
5 Technical requirements for reference liquids.....	7
6 Storage.....	8
7 Viscosity gradation of reference liquids, preparation of mixtures .....	8
8 Metrological control for reference liquids.....	9
8.1 Long-term stability.....	9
8.2 Thermal stability.....	10
8.3 Newtonian flow behaviour .....	11
9 Calibration of Newtonian viscosity standard specimens .....	11
9.1 Measurement of kinematic viscosity.....	12
Standard viscometers .....	12
Calibration of reference VSS: .....	12
Calibration of working VSS: .....	12
Temperature measurement and devices for temperature control ...	12
Measurement.....	13
9.2 Calculation of kinematic viscosity.....	13
9.3 Measurement of density, calculation of dynamic viscosity .....	14
9.4 Determination of the temperature coefficient of viscosity .....	15
9.5 Measurement uncertainty.....	16
9.6 Calibration certificate.....	18
10 References .....	20
Appendix 1 .....	22
Appendix 2 .....	23
Appendix 3 .....	24
Appendix 4 .....	26
Appendix 5 .....	32

## NEWTONIAN VISCOSITY STANDARD LIQUIDS FOR THE CALIBRATION AND VERIFICATION OF VISCOMETERS

### Explanatory Note

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

*The First 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 Draft.*

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

## **1 SCOPE AND FIELD OF APPLICATION**

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"

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 17025.

## **2 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.

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

## **3 UNITS OF MEASUREMENT**

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

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

#### 4 METROLOGICAL REQUIREMENTS

The range of dynamic viscosity covered by Newtonian viscosity standard specimens ranges from about  $4 \cdot 10^{-1}$  to  $10^5$   $\text{mPa} \cdot \text{s}$  at  $20^\circ\text{C}$ . This corresponds to kinematic viscosities between about  $4 \cdot 10^{-1}$  and  $1.2 \cdot 10^5$   $\text{mm}^2/\text{s}$ . Standard specimens may be calibrated and used at various temperatures (usually between  $-40$  and  $+150^\circ\text{C}$ ) 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).

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

**Table 1**

Viscosity	$\varepsilon_v$ , %	$\varepsilon_v$ , %
	reference VSS	working VSS
$\nu \leq 50 \text{ mm}^2/\text{s}$	$\varepsilon_v \leq 5 \cdot 10^{-2}$ , %	$\varepsilon_v \leq 1 \cdot 10^{-1}$
$\nu > 50 \text{ mm}^2/\text{s}$	$\varepsilon_v \leq 1 \cdot 10^{-1}$	$\varepsilon_v \leq 2 \cdot 10^{-1}$

The density of standard specimens shall be determined with a relative uncertainty of  $\leq 2 \cdot 10^{-4}$  at each calibration temperature based on ISO 3838.

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  $0,005^\circ\text{C}$  for the reference VSS and shall be less than  $0,01^\circ\text{C}$  for the working VSS.

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

## 5 TECHNICAL REQUIREMENTS FOR REFERENCE LIQUIDS

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 several months)

b) thermal stability

(viscosity, irreversible during thermal stability testing)

c) Newtonian flow behaviour

d) good solubility in several cleaning agents

(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 Appendix 1

## **6 STORAGE**

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 proved 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.

## **7 VISCOSITY GRADATION OF REFERENCE LIQUIDS, PREPARATION OF MIXTURES**

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 Appendix 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 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 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 preparing 5 liters of mixture and 100 h for 50 liters of mixture.

## **8 METROLOGICAL CONTROL FOR REFERENCE LIQUIDS**

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 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  $4 \cdot 10^{-3}$

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) 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:

$T_{ref}$  - reference temperature (usually 20°C or 23 °C)

$T_{min}$ ;  $T_{max}$  - minimum and maximum temperature at which VSS of the liquid shall be calibrated (for example -40°C, +100°C)

$T_{o1}$ ,  $T_{o2}$  - minimum and maximum temperature to which a VSS may be exposed 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  $T_{ref}$ .
- Heat the viscometers to  $T_{max}$  for 1 hour with the capillary and the measuring bulb being filled with the liquid.
- Repeat the viscosity measurement at  $T_{ref}$ . The relative difference between the viscosities determined before and after heating shall not exceed  $4 \cdot 10^{-3} \text{ yr}^{-1}$ .
- Apply the same procedure (step a) to c).
- The viscosity change after cooling to  $T_{min}$  shall not exceed the value stated above.
- Measure the viscosity of a fresh sample at  $T_{max}/T_{min}$ , keep the viscometer at  $T_{max}/T_{min}$  for one hour with the capillary and measuring bulb being filled with the liquid. Repeat the viscosity determination at  $T_{max}/T_{min}$

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

- After viscosity determination at  $T_{ref}$ , two SSp containers (mostly brown glass bottles) are filled with the liquid and sealed. One container is stored at  $T_{o1}$ , the other at  $T_{o2}$  for one week. After storage, the viscosity at  $T_{ref}$  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  $4 \cdot 10^{-3} \text{ yr}^{-1}$ .

### 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 CALIBRATION OF NEWTONIAN VISCOSITY STANDARD SPECIMENS

## **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 and ISO 3105. Additional requirements and deviations from ISO 3104 and ISO 3105 are described below in order to meet the uncertainty requirements given in OIML-DI No. 17.

### **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 0.1% 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 and 3105.

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

$$\frac{t_{\max} - t_{\min}}{\bar{t}} \leq 1 \cdot 10^{-3}, \quad (1)$$

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  $0.7 \cdot 10^{-3}$

## **9.2 Calculation of kinematic viscosity**

For each viscometer the kinematic viscosity  $\nu$  is calculated according to eq. (2)

$$\nu = C \cdot \bar{t} \cdot \frac{g}{\Delta t_h \cdot g}, \quad (2)$$

with

$\Delta t_h$  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  $C > 0,1 \text{ mm}^2/\text{s}^2$  the kinetic energy correction may be neglected and equation 2 takes on the following form:

$$\nu = C \frac{g}{g'} \quad [3]$$

For viscometers whose constants  $C < 0,1 \text{ mm}^2/\text{s}^2$  the kinetic energy correction may be significant if the minimum 200-s slow time is not observed.

From the viscosity values  $\nu_1$  and  $\nu_2$  determined with the two standard viscometers the arithmetic mean value  $\bar{\nu}$  is calculated.

### **9.3 Measurement of density, calculation of dynamic viscosity**

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

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  $\eta$  is calculated from the kinematic viscosity and the density  $\rho$  determined at the same temperature according to

$$\eta = \nu \cdot \rho \quad (4)$$

#### **9.4 Determination of the temperature coefficient of viscosity**

The knowledge of the temperature coefficient of viscosity  $U_\nu$  defined as

$$U_\nu = -\frac{1}{\nu} \frac{d\nu}{dT} \quad (4)$$

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 SSp can adapt the quality of temperature control necessary to use the SSp within its uncertainty.  $U_\nu$  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 Appendix 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”. Convenient summaries can be found in [ ]. The coverage factor should be  $k=2$  corresponding to an interval of confidence of 95%. An example is given in Appendix 4.

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

According to OIML document D 17, 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 1)

	C	$u_c$	$U_v$	$u_v$	$u_{\text{cert}}$
	$\text{mm}^2/\text{s}^2$		$1/^\circ\text{C}$		
reference VSS	0.003	$1 \cdot 10^{-3}$	0.07	$1.24 \cdot 10^{-3}$	$2 \cdot 10^{-3}$
	50	$8 \cdot 10^{-3}$	0.10	$7.9 \cdot 10^{-3}$	$1 \cdot 10^{-2}$
working VSS	0.003	$2 \cdot 10^{-3}$	0.07	$2.4 \cdot 10^{-3}$	$3 \cdot 10^{-3}$
	50	$1 \cdot 10^{-2}$	0.10	$1 \cdot 10^{-2}$	$2 \cdot 10^{-2}$

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)
- address and name of the calibration laboratory
- example of calibration certificate is given in Appendix 5.

## 10 REFERENCES

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-  
stoppered pycnometers and graduated bicapillary pycnometer 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

## APPENDIX 1

Examples of liquids which are commonly used as reference liquids:

Type of liquid	viscosity
	mPa · s
chemically pure hydrocarbons (organic solvents)	0.3 to 1.5
mineral oils without polymer additives	1 to 1000
hydrogenated polyalphaolefin liquids	50 to 5000
polyisobutylenes	>5000

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.

## APPENDIX 2

### Examples of mixing equations

The kinematic viscosity  $\nu$  of a binary mixture of components with viscosities  $\nu_1$  and  $\nu_2$  can approximately be calculated using one of the following equations:

$$\ln \nu = \varphi_1 \cdot \ln \nu_1 + \varphi_2 \cdot \ln \nu_2$$

$$1/\nu = w_1 \cdot 1/\nu_1 + w_2 \cdot 1/\nu_2$$

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

The volume fractions  $\varphi_1$ ,  $\varphi_2$  denote the volume ratio in relation to the total volume of the mixture.

### APPENDIX 3

#### Example of determination of the temperature coefficient of viscosity

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

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

the temperature coefficient of viscosity  $U_\nu$  is given by

$$U_\nu = 2,303 \frac{\log \nu_1 - \log \nu_2}{T_2 - T_1} \frac{T_1 + C}{T_2 + C} \frac{T_2 + C}{T_1 + C}$$

$U_\nu$  is calculated in 1/°C if  $T$  is measured in °C and  $\nu_1, \nu_2, \nu_3$  in mm<sup>2</sup>/s.

$C$  is calculated according to

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

with

$$P = (\log \nu_1 - \log \nu_2) \cdot (T_3 - T_2)$$



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

$v_1$ ,  $v_2$ ,  $v_3$  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.

## APPENDIX 4

### 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  $x_i$  ( $i = 1, \dots, r$ ) the relative variance  $(S')^2$

is given by:

$$\sigma_{s_y}^2 \equiv \sum_{i=1}^r \sigma_{s_y}^2 \quad (1)$$

For repeatedly measured quantities  $s_{x_i}$  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:  $U = \frac{1}{2}(a_u - a_l)$  an estimate value for the relative variance of  $X_i$  is calculated from the relative uncertainty  $U$  according to:

$$\sigma_{s_{x_i}}^2 = \frac{1}{3} U^2 \quad (2)$$

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

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

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  $\nu$  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, $S_c$ , %	0,10
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 8.1 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

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

Uncertainty of temperature measurement: two contributions have to be considered, the calibration uncertainty of the thermometer which is taken as  $0,005^\circ\text{C}$  and the temperature gradient between the position of the thermometer and the viscometer in the thermostat which is  $0,005^\circ\text{C}$ . In the worst case both contributions have to be added resulting in an uncertainty of the sample temperature of  $0,01^\circ\text{C}$ . 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  $U_v = 0,07 \text{ } 1/^\circ\text{C}$ :

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

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:

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

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

This value has to be 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:

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

## APPENDIX 5

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,  °C	Viscosity  mm <sup>2</sup> /s   mPa · s  Kinematic   Dynamic		Density  g/sm <sup>3</sup>

**Uncertainties:**

**Adress:**

**Tel.**

**Fax:**