Reference standard liquids (Newtonian viscosity standard for the calibration and verification of viscometers)

Liquides de référence (étalon de viscosité newtonien pour l’étalonnage et la vérification des viscosimètres)
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Foreword

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This publication – reference OIML D 33, edition 2019 (E) – was developed by Project Group 1 of OIML Technical Subcommittee TC 17/SC 5 Viscosimetry. It was approved for final publication by the International Committee of Legal Metrology at its 54th meeting in 2019 and will be submitted to the International Conference on Legal Metrology in 2020 for formal sanction.

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Reference standard liquids
(Newtonian viscosity standard for the calibration and verification of viscometers)

1 Scope and field of application

This International Document specifies requirements for liquids used for the preparation and calibration of Newtonian viscosity standard specimens (reference standard liquids), according to OIML D 17 [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 in accordance with national standards of viscosity according to ISO/IEC 17025:2017 [2].

2 Terms and definitions

This Document uses the following general terms and definitions.

2.1 Newtonian reference liquids (RL)
liquid in which the shear rate is proportional to the shear stress during a viscous flow

Note: The ratio of the shear stress to the shear rate is the dynamic viscosity of the liquid, according to Newton’s Law of Viscosity. The kinematic viscosity is the ratio of the dynamic viscosity to the density of the liquid.

2.2 Newtonian viscosity standard specimen (VSS)
Newtonian liquid sufficiently homogeneous and stable with respect to viscosity (dynamic and kinematic) which has been determined traceable to international or national measurement standards, by a metrologically valid procedure, and is documented in a calibration certificate together with the associated uncertainty, and a statement of metrological traceability

3 Units of measurement

The SI unit of kinematic viscosity (ν) is m²·s⁻¹. For practical use, a submultiple (mm²·s⁻¹) is more convenient. The SI unit of dynamic viscosity (η) is Pa·s. For practical use, a submultiple (mPa·s) is more convenient [3].
4 Metrological requirements

The range of dynamic viscosity covered by Newtonian viscosity standard specimens ranges from about $4 \times 10^{-1}$ mPa·s to $10^5$ mPa·s at 20 °C. This corresponds to kinematic viscosities between about $4 \times 10^{-1}$ mm²·s⁻¹ and $1 \times 10^5$ mm²·s⁻¹. Standard specimens may be calibrated and used at various temperatures (usually between −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 D 17 [1]).

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

The relative deviation (1) shall meet the requirements shown in Table 1.

$$\epsilon_\nu = \frac{|\nu_2 - \nu_1|}{\nu} \times 100 \% \quad (1)$$

<table>
<thead>
<tr>
<th>Viscosity/ mm²·s⁻¹</th>
<th>Relative deviation for the reference VSS %</th>
<th>Relative deviation for the working VSS %</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\nu \leq 50$</td>
<td>$\epsilon_\nu \leq 0.05$</td>
<td>$\epsilon_\nu \leq 0.1$</td>
</tr>
<tr>
<td>$\nu &gt; 50$</td>
<td>$\epsilon_\nu \leq 0.1$</td>
<td>$\epsilon_\nu \leq 0.2$</td>
</tr>
</tbody>
</table>

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

It is important to use at least two viscometers at the same time in thermostatic baths with automatic temperature control. For measurements between the temperatures 15 °C and 100 °C, the temperature change during a measurement shall be less than 0.005 °C for the reference VSS and shall be less than 0.01 °C for the working VSS. For bath temperatures between 15 °C and 100 °C, the temperature gradient between the positions of any thermometer and viscometer shall be less than 0.005 °C for the reference VSS and 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 % shall be used.

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 in viscosity when stored at ambient conditions for 6 months (see 8.1));

b) thermal stability (viscosity reversible during thermal stability testing (see 8.2));

c) Newtonian flow behaviour;

d) good solubility in at least one common laboratory cleaning agent (to enable appropriate cleaning of instruments);
e) sufficiently transparent (for use in glass capillary viscometers);

f) viscosity-temperature coefficient as low as possible to reduce the requirements for temperature control;

g) no absorption of water, inert with respect to air, glass and metal;

h) no or low toxicity, high flash point above temperature of intended use where possible.

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

Requirements d) to h) 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 A.

6 Storage

Reference liquids shall be in equilibrium with air as dissolved gas, and 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: If a reference liquid is stored under an inert gas atmosphere, it should be open and exposed to air for one day before using it.

7 Viscosity gradation of reference liquids, preparation of mixtures

The number of reference liquids and the viscosity range covered depend 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 carried out 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 be calculated approximately by the application of mixing equations. Examples of such equations are given in Annex B. 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 involve routine laboratory equipment. The mixing device (stirrer, pump) shall not produce high shear rates in the liquid (high-speed rotation and narrow gaps shall be avoided) 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, but not above the highest temperature of intended use) in order to facilitate the mixing.

The time necessary to reach a 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 of samples taken after different durations of mixing. Typical mixing times may be between 24 h for preparing 5 L of mixture and 100 h for 50 L of mixture.
8 Metrological control for reference liquids

For each batch of liquid to be used as the reference liquid, the tests described in 8.1 to 8.3 shall 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 (generally 20 °C or 23 °C) with two viscometers of the same nominal viscometer constant following the procedure described in 9.1. 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 0.007 during 3 months.

If the liquid is to be used for reference viscosity standard specimens only, the ageing coefficient shall not exceed 0.004.

Note: Higher values of the ageing coefficient may be accepted if the total uncertainty (\( \mu_{\text{cert}} \) in Table 2) is kept within the specifications given in 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 determination of the thermal stability involves the following temperatures:

- \( T_{\text{ref}} \): reference temperature (usually 20 °C or 23 °C);
- \( T_{\text{min}}, T_{\text{max}} \): minimum and maximum temperatures at which the VSS of the liquid shall be calibrated (for example –40 °C, +100 °C); and
- \( T_{\text{O1}}, T_{\text{O2}} \): minimum and maximum temperature to which a VSS may be exposed during storage and delivery before use.

The following procedure is recommended to identify the effects below:

1) Influence of short-term heating/cooling and duration of measurement:
   a) The duration of measurements must be 200 s to 2 000 s.
   b) Fill two viscometers of the same nominal constant with a test portion of the liquid and measure the viscosity at \( T_{\text{ref}} \).
   c) Put the viscometer in a thermostatic bath at \( T_{\text{max}} \) for 1 h with the capillary and the measuring bulb being filled with the liquid.
   d) Repeat the viscosity measurement at \( T_{\text{ref}} \). The relative difference between the viscosities determined before and after heating should not exceed 0.004.
   e) Repeat the same procedure (steps a) to c)) at \( T_{\text{min}} \).
   f) The viscosity change after cooling to \( T_{\text{min}} \) should not exceed the value stated above.
   g) Measure the viscosity of a fresh sample at \( T_{\text{max}} (T_{\text{min}}) \), keep the viscometer at \( T_{\text{max}} (T_{\text{min}}) \) for 1 h with the capillary and measuring bulb being filled with the liquid. Repeat the viscosity determination at \( T_{\text{max}} (T_{\text{min}}) \).
2) Influence of high and low temperatures during delivery to the user:
   a) After viscosity determination at $T_{\text{ref}}$, two VSS containers (generally 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 $T_{\text{ref}}$ is measured again using the same pair of viscometers and the same temperature measuring device.
   b) The relative change of viscosity before and after storage shall not exceed 0.004.

8.3 Newtonian flow behaviour
Tests for Newtonian flow behaviour shall be performed within the VSS’s expected shear range of usage. This shall be performed at $T_{\text{min}}$ using a viscometer, with adequate accuracy, that allows the variation of the shear rate, for example a four-bulb dilute solution glass capillary viscometer, a pressure-driven capillary device or a rotational viscometer.

9 Test method and model test report: calibration of the Newtonian viscosity standard

9.1 Measurement of kinematic viscosity

9.1.1 Standard viscometers

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

9.1.1.2 Calibration of the working VSS
Use two reference standard viscometers of approximately the same viscometer constant.

The expanded uncertainty ($k = 2$) of the viscometer constant, including the uncertainty of the kinetic energy correction, must not exceed 0.2 % at the low value and 1 % at the high value of the constant.

9.1.1.3 Temperature measurement and devices for temperature control
Suitable thermometers shall be used. The temperature measurement expanded uncertainty ($k = 2$) shall not exceed 0.005 °C. It may be necessary to apply corrections according to the calibration certificate for the thermometers. The depth of immersion shall be the same during calibration and use.

9.1.1.4 Measurement
The handling of viscometers such as filling and cleaning must be done in accordance with ISO 3104:1994 [5] and ISO 3105:1994 [6].
When cleaning standard viscometers with sulfochromic acid (saturated aqueous solution of K₂Cr₂O₇ mixed with H₂SO₄ 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.

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

\[
\frac{t_{\text{max}} - t_{\text{min}}}{\bar{t}} \leq 0.001
\]  

(2)

Where \( t_{\text{min}} \) and \( t_{\text{max}} \) are the shortest and the longest flow times and \( \bar{t} \) is the arithmetic mean of the series.

The relative repeatability uncertainty of the flow-time measurement shall not exceed \( 0.7 \times 10^{-3} \).

### 9.2 Calculation of the kinematic viscosity

For each viscometer, the kinematic viscosity \( \nu \) is calculated according to eq. (3)

\[
\nu = C(\bar{t} - \Delta t_h) \frac{g}{g'}
\]  

(3)

where

- \( C \) constant of the viscometer;
- \( \Delta t_h \) 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 viscometer (see calibration certificate of the viscometer).

For capillary viscometers whose constant \( C > 0.1 \, \text{mm}^2 \cdot \text{s}^{-2} \) the kinetic energy correction may be neglected and eq. (3) takes on the following form:

\[
\nu = C\bar{t} \frac{g}{g'}
\]  

(4)

For viscometers whose constant \( C < 0.1 \, \text{mm}^2 \cdot \text{s}^{-2} \) the kinetic energy correction may be significant if the minimum 200 s flow 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 pyknometers of typically 25 ml or 50 ml of volume. A routine laboratory method that corrects for air buoyancy can keep the relative uncertainty of the mass determination below \( 2 \times 10^{-5} \).

An oscillation \( U \)-tube density meter can be used for measuring the density (also for liquids with viscosity values above 700 mPa·s, when the viscosity correction is taken into account) ISO 15212 [7].

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

The dynamic viscosity \( \eta \) is calculated from the kinematic viscosity \( \nu \) and the density \( \rho \) determined at the same temperature according to:

\[
\eta = \nu \times \rho
\]  

(5)
9.4 Determination of the temperature coefficient of viscosity

It is necessary to know the temperature coefficient of viscosity $A$ defined as:

$$ A = -\frac{1}{\nu} \frac{d\nu}{dT} $$

(6)

At each calibration, temperature $T$ is necessary in order to calculate the uncertainty of the viscosity determined value. The uncertainty shall also be stated in the calibration certificate, so that the user of the VSS can adapt the quality of the temperature control necessary to use the VSS within its uncertainty. $A$ 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 $A$ is given as an example in Annex C.

Within the uncertainty of the temperature coefficient of viscosity, the change in $A$ 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 JCGM 100:2008 [8]. The coverage factor should be $k = 2$ corresponding to a level of confidence of 95 %. An example is given in Annex D.

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

According to OIML D 17 [1], the expanded uncertainty ($k = 2$) 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. They also include an estimated maximum contribution of up to 20 % of the total uncertainty in order to cover influences of minor importance such as deviation from the vertical alignment of the viscometer and the uncertainty of the gravitational acceleration. Table 2 shows the resulting measurement uncertainties provided that the requirements of clause 4 are met.

*Note:* Limiting values that are met by the requirements described in clause 4 could be extended based on the previous long term stability evaluation of the particular material type.

Table 2 - Uncertainty of viscosity measurement of reference and working VSSs at the limits of the viscosity range of this Document (see clause 4)

<table>
<thead>
<tr>
<th></th>
<th>$C$/ mm$^2$·s$^{-2}$</th>
<th>$u_c$</th>
<th>$A$/ °C$^{-1}$</th>
<th>$u_v$</th>
<th>$u_{cert}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reference VSS</td>
<td>0.003</td>
<td>0.001</td>
<td>0.07</td>
<td>0.0012</td>
<td>0.002</td>
</tr>
<tr>
<td></td>
<td>50</td>
<td>0.008</td>
<td>0.10</td>
<td>0.0079</td>
<td>0.01</td>
</tr>
<tr>
<td>Working VSS</td>
<td>0.003</td>
<td>0.002</td>
<td>0.07</td>
<td>0.0024</td>
<td>0.003</td>
</tr>
<tr>
<td></td>
<td>50</td>
<td>0.01</td>
<td>0.10</td>
<td>0.001</td>
<td>0.02</td>
</tr>
</tbody>
</table>
where

\[ C \] constant of the viscometer;
\[ u_c \] relative uncertainty of the viscometer constant including the uncertainty of the kinetic energy correction;
\[ A \] temperature coefficient of the 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 the remaining sources of error;
\[ u_{\text{cert}} \] 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 the viscosity at one or more temperatures;
- density (if dynamic viscosity was calculated);
- uncertainty of the stated viscosity values including the possible ageing during the period of validity;
- date of certification and the period of validity if the customer agrees;
- reference to the metrological basis of the viscosity determination (usually the viscosity of water as stated in ISO TR 3666:1998 [9]);
- name and address of the calibration laboratory.

An example of a calibration certificate is given in Annex E.

Note: Additional requirements on calibration certificates can be found in chapter 7.8 of ISO/IEC 17025:2017 [2].
10 References


[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.*


Annex A
Examples of liquids which are commonly used as reference liquids
(Mandatory)

<table>
<thead>
<tr>
<th>Type of liquid</th>
<th>Viscosity mPa·s</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chemically pure hydrocarbons (organic solvents)</td>
<td>0.3 to 1.5</td>
</tr>
<tr>
<td>Mineral oils without polymer additives</td>
<td>1 to 1 000</td>
</tr>
<tr>
<td>Hydrogenated polyalphaolefin liquids</td>
<td>50 to 5 000</td>
</tr>
<tr>
<td>Polyisobutlenes</td>
<td>&gt; 5 000</td>
</tr>
</tbody>
</table>

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 hydrophobic properties are taken into account by special investigations.
Annex B
Examples of mixing equations
(Mandatory)

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

\[
\ln \nu = \varphi_1 \ln \nu_1 + \varphi_2 \ln \nu_2 \tag{B.1}
\]

\[
\frac{1}{\nu} = w_1 \frac{1}{\nu_1} + w_2 \frac{1}{\nu_2} \tag{B.2}
\]

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

The volume fractions $\varphi_1$ and $\varphi_2$ denote the volume ratio of the components in the total volume of the mixture.
Annex C

Example of determination of the temperature coefficient of viscosity

(Mandatory)

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

\[ \nu = A e^{\frac{B}{(T+C)}} \]  

(C.1)

the temperature coefficient of viscosity \( A \) is given by:

\[ A = 2.303 \frac{(\log \nu_1 - \log \nu_2) (T_1 + C) \times (T_2 + C)}{(T_2 - T_1) (T + C)^2} \]  

(C.2)

\( A \) is calculated in \( 1/°C \) if \( T \) is measured in °C and \( \nu_1, \nu_2 \) and \( \nu_3 \) in mm²/s.

\( C \) is calculated according to:

\[ C = \frac{P T_3 - Q T_3}{Q - P} \]  

(C.3)

with

\[ P = (\log \nu_1 - \log \nu_2) (T_3 - T_2) \]  

(C.4)

\[ Q = (\log \nu_2 - \log \nu_3) (T_2 - T_1) \]  

(C.5)

\( \nu_1, \nu_2 \) and \( \nu_3 \) 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.
Annex D
Example for the calculation of the uncertainty of measurement for the viscosity of Newtonian viscosity standard
(Mandatory)

The uncertainty of measurement calculation is performed according to the method described in EA-4/02 [10], using the following relations:

For a measurement \( y \) depending on influence quantities \( x_i (i = 1, \ldots, r) \) the relative variance \( \left( S'_y \right)^2 \) is given by:

\[
(S'_y)^2 = \sum_{i=1}^{r} (S'_x)^2
\]

(D.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:

\[
u = \frac{1}{2} (a_u - a_l)
\]

(D.2)

an estimate value for the relative variance of \( x_i \) is calculated from the relative uncertainty \( u \) according to:

\[
(S'_{x_i})^2 = \frac{1}{3} (u')^2
\]

(D.3)

Applying a coverage factor \( k = 2 \) the relative expanded uncertainty of the measurement is:

\[
U'_y = 2S'_y
\]

(D.4)

This means that in the case of a normal (Gaussian) distribution the limits of uncertainty are for a 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 such as:

- 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. D.4 has to be divided by a factor \( \sqrt{2} \).
According to the specifications in 9.5 the relative uncertainty of the viscometer constant is taken to be:

\[ u'_c = 1 \times 10^{-3} \]  

which leads by applying eq. (D.3) to:

\[ (S'_c)^2 = 33 \times 10^{-8}. \]  

As an example, the viscosity of a reference VSS is calculated using the following assumptions, shown in Table 3:

Table 3 – Example uncertainty of the viscosity of a reference VSS

<table>
<thead>
<tr>
<th>N</th>
<th>Contribution to the uncertainty</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>The relative uncertainty of viscometer constant ( C_v ) in the calibration certificate, ( S_{C_v} ), %</td>
<td>0.06</td>
</tr>
<tr>
<td>2</td>
<td>The relative uncertainty of temperature control and measurement, ( S_{T} ), %</td>
<td>0.04</td>
</tr>
<tr>
<td>3</td>
<td>The relative uncertainty of flow time measurement, ( S_{R} ), %</td>
<td>0.02</td>
</tr>
<tr>
<td>4</td>
<td>The relative uncertainty of the timing device ( S_{n} ), %</td>
<td>0.01</td>
</tr>
</tbody>
</table>

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

\[ u'_w = 2 \times 10^{-4} \]  

\[ (S'_w)^2 = 1.3 \times 10^{-8} \]  

Random uncertainty of the flow-time measurement: In this example, the relative empirical standard deviation for \( n = 5 \) repeated flow-time measurements is assumed to be \( 3 \times 10^{-4} \). This standard deviation has to be multiplied by a factor \( t = 1.4 \) (for \( n = 5 \)). The corresponding relative variance is given by

\[ (S'_R)^2 = \frac{(3\times10^{-4}\times1.4)^2}{5} = 3.5 \times 10^{-8} \]  

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 difference 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 by the temperature coefficient of viscosity which is assumed to be \( u_v = 0.07 \, ^\circ C^{-1} \):

\[ u'_T = 0.07 \times \sqrt{(0.005^2 + 0.005^2)} = 5 \times 10^{-4} \]  

The corresponding relative variance according to eq. (D.3) is:

\[ (S'_T)^2 = 8 \times 10^{-8} \]  

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

\[ (S'_\nu)^2 = (S'_c)^2 + (S'_w)^2 + (S'_R)^2 + (S'_T)^2 = 6.77 \times 10^{-4} \]  

This value has to be multiplied by \( k = 2 \) (95 % level of confidence) and divided by \( \sqrt{2} \) because two viscometers are used. Thus the total relative uncertainty is:

\[ U'_\nu = 1.14 \times 10^{-3} \]
Annex E
Example for the calibration certificate for viscosity reference standard
(Mandatory)

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
Kinematic: mm²·s⁻¹
Dynamic: mPa·s

Temperature coefficient of viscosity, °C⁻¹
Density: g·cm⁻³

Uncertainties:

Address:
Tel.:
Fax: