# Visco handbook

THEORY AND APPLICATION OF VISCOMETRY
WITH GLASS CAPILLARY VISCOMETERS

SI Analytics
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# Welcome to SI Analytics!

We express our core competence, namely the production of analytical instruments, with our company name SI Analytics. SI also stands for the main products of our company: sensors and instruments.

As part of the history of SCHOTT® AG, SI Analytics has nearly 80 years experience in glass technology and in the development of analytical equipment. As always, our products are manufactured in Mainz with a high level of innovation and quality. Our electrodes, titrators and capillary viscometers will continue to be the right tools in any location where expertise in analytical measurement technology is required.

In 2011 SI Analytics became part of the listed company Xylem Inc., headquartered in Rye Brook / N.Y., USA. Xylem is a leading international provider of water solutions.

We are pleased to introduce you to the Visco Handbook!

It replaces the previous brochure "Theory and Practice of Capillary Viscometry". This has been updated, redesigned and restructured. The information content of some areas such as polymer analysis was increased and specially themed areas such as hydrodynamic principles were moved into their own appendices so as not to deter the reader by too much theory.

The focus was placed on the practical and general information needed by each user of capillary viscometry. Both laboratory experiences and policies of the relevant standards are considered here. We thus hope to provide you with a faithful companion for everyday laboratory work, one that you can use profitably.

We of SI Analytics are pleased to work with you successfully in the future.

SI Analytics GmbH

Dr. Robert Reining
CEO

# CONTENTS

## CHAPTER 1

### Viscosity - Rheology

## CHAPTER 2

### Fundamentals of capillary viscometry

## CHAPTER 3

### Measurement of the flow time

## CHAPTER 4

### Method for determining viscosity

## CHAPTER 5

## CHAPTER 6

### Handling of capillary viscometers

## CHAPTER 7

### Sources of error and special corrections

## CHAPTER 8

### Special application

### Appendix A

### Symbols and units used

### Bibliography

### Standards

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# CHAPTER 1

## VISCOSITY - RHEOLOGY

### 1.1 Introduction

The subject of this Visco Handbook is viscometry with glass capillary viscometers.

Viscometry deals with the determination of viscosity and is a branch of rheology, which is scientifically concerned with the flow and deformation behavior of materials. In rheology, substances, especially viscoelastic ("semi-solid"), are examined, which are classified as being between a fluid and a solid, based on their mechanical properties.

With glass capillary viscometers, only the viscosity of samples with ideal flow behavior (Newtonian liquids) is measured. This section briefly explains the difference between ideal and non-ideal flow properties. Additional information about rheology may be found in [1,19].

Viscosity characterizes the internal friction of liquids and gases. If a fluid medium is located between two plane-parallel plates, it will require some amount of force to displace the upper plate. The liquid starts to flow inside the gap. A layered flow builds up (Fig. 1).

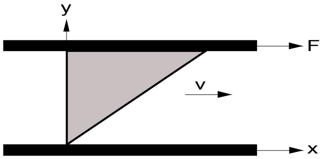


*Fig. 1 Basic shearing model in the case of laminar, stationary flow*

The fluid particles which are directly adjacent to the plates are firmly bonded to the surface by adhesion forces. In this process the fluid layer neighboring the plate being displaced adopts the velocity of the plate. All neighboring layers stay more and more behind with the increasing distance to the plate being moved. The cause for this phenomenon can be found in cohesion forces which counter-act the reciprocal dislocation of the individual layers.

The shear stress $\tau$ refers to the quotient of force $F$ and the boundary surface $A$ of the liquid:

$$\tau = \frac{F}{A} \tag{1.1}$$

The gratient of velocity, the shear rate $\dot{\gamma}$ is the differential quotient:

$$\dot{\gamma} = \frac{dv}{dy} \tag{1.2}$$

According to Newton's Viscosity Law, the shear stress $\tau$ is proportional to the shear rate $\dot{\gamma}$:

$$\tau = \eta \cdot \dot{\gamma} \tag{1.3}$$

The proportionality factor $\eta$ (pronounced "eta") is referred to as dynamic viscosity.

The unit of viscosity is Pa·s, wherein low-viscosity samples are usually specified in mPa·s and thus the former unit cP (centipoise) numerically corresponds to:

$$\eta = \frac{\tau}{\dot{\gamma}} \left[ \mathrm{Ns/m^2} \right] = \left[ \mathrm{Pa \cdot s} \right] \tag{1.4}$$

The relationship between dynamic viscosity $\eta$ and density $\rho$ is referred to as kinematic viscosity $\nu$ (pronounced "ny"):

$$\nu = \frac{\eta}{\rho} = \left[ \mathrm{m^2 / s} \right] \tag{1.5}$$

For reasons of convenience, the unit of $\mathrm{mm^2/s}$ is used, which then corresponds numerically to the former unit. cSt (centistokes).

In Newtonian fluids, the viscosity remains constant with a change of the shear rate. As a consequence of the differences in size, shape, and interaction between the molecules $\eta$ may change within very wide limits.

Examples:

| | |
|---|---|
| n-pentane | 0.230 mPas (20 °C) |
| Water | 1.002 mPas (20 °C) |
| Propanetriol<br>(Glycerol) | 1480 mPas (20 °C) |

## Temperature dependence of viscosity

During flow, liquid molecules slide against one another under the expenditure of energy (activation energy). The proportion of molecules that have this energy depends on the temperature and is described by the Boltzmann distribution.

This leads to the relationship:

$$\eta = k \cdot e^{-\frac{E_{visk}}{RT}} \qquad (1.6)$$

| | |
|---|---|
| $k$ | *Proportionality factor* |
| $E_{visk}$ | *Activation energy of the viscous fluid* |
| $R$ | *Gas constant* |
| $T$ | *Absolute temperature* |

After that, $\eta$ strongly decreases for liquids with increasing temperature. As a rule of thumb, one can say that the higher the absolute values of the viscosity and the lower the temperature, the greater is the decrease.

This effect is of great practical importance, for example, in lubrication technology, as will be shown later.

In addition to the temperature, the pressure also has an effect on the viscosity: An increase in pressure generally leads to increased viscosity. The effect does not appear so much in everyday life since only pressure increases of 10 to 100 bar (or even higher) lead to a significant increase in viscosity of liquids. The pressure dependence of the viscosity therefore plays no role in viscometry with glass capillary viscometers, which is discussed in this Visco handbook.

## Viscosity of mixtures/solutions

When liquids are mixed, the viscosity can be approximately calculated from the viscosities of the individual components according to a logarithmic mixing rule.

Example of two components:

$$\ln \eta_{mix} = w_1 \ln \eta_1 + w_2 \ln \eta_2 \qquad (1.7)$$

$w_1$, $w_2$: weight fractions of the components 1,2

This rule only applies to mixtures of similar components, and does not generally apply for aqueous solutions. For a better description of reality, equations must be selected with adjustable parameters [2].

The viscosity of the solutions of solid substances is often higher than that of the pure solvent. The specification is usually made as a relative or specific viscosity (see Chapter 8).

A particular behavior can be observed with the dependence of concentration of viscosity of electrolyte solutions.

If the liquid layers are moving at different velocities, the deformation of the ion cloud will cause the occurrence of additional inter-ionic interacting forces which will affect friction between the individual layers.

H. Falkenhagen has derived the limiting law of viscosity from the theory of inter-ionic interactions for highly dilute electrolyte solutions:

$$\eta_c = \eta_0 + K\sqrt{c} \qquad (1.8)$$

| | |
|---|---|
| $\eta_c$ | *Viscosity for the ion concentration c* |
| $\eta_0$ | *Viscosity of the pure solvent at the same temperature* |
| $K$ | *Constant which depends on the following variables:*<br>*- Temperature*<br>*- Dielectric constant of the solvent*<br>*- Ion valences*<br>*- Ion mobilities* |

## 1.2 Non-Newtonian flow behavior

### Shear thickening (dilatancy)

Shear viscosity increases with rising shear rate (work hardening: Fig. 2, Curve b).

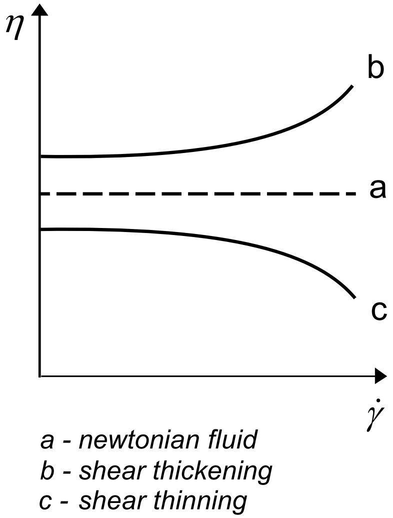


*Fig. 2  Viskosity curves of fluids*

### Shear thinning (pseudoplasticity)

At large shear rates, $\eta$ decreases with the shear rate (Fig. 2, Curve c). At small shear rates, substances usually have Newtonian behavior.

Examples:
- Lacquer/varnish
- Thermoplastics
- Polymer melts
- Adhesives
- Additives
- Emulsions
- Suspensions

### Yielding point (plasticity)

The liquids only begin to flow at a minimum shear strain. Below this yielding point, the substance behaves as a solid.

Examples:
- Paints, dispensions, cream
- Food (mayonnaise)
- Toothpaste
- Vaseline

In addition to these shear rate-dependent effects, shear time dependent flow behavior is observed in some non-Newtonian substances.

$$\tau = f(\dot{\gamma}, t) \tag{1.9}$$

The shear viscosity is thus influenced by the duration of the shear (see Fig. 3).

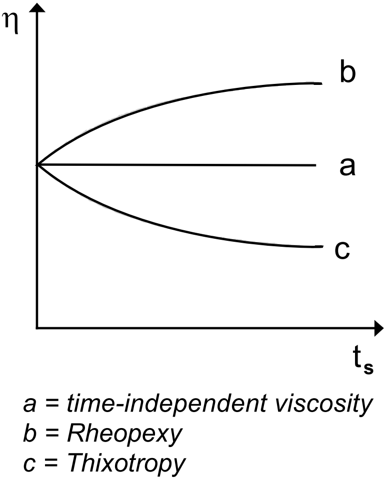


*Fig. 3 Viscosity curves of fluids*

It is divided into:

## Thixotropiy

Shear viscosity decreases at constant shear rate with increasing shear time. At rest, the internal structure of the sample builds up again, so that the original initial viscosity is achieved again after a certain rest time. Many paints or varnishes show thixotropic flow behavior in order to achieve optimal flow and leveling/sagging behavior.

## Rheopexy

Shear viscosity increases at constant shear rate with increasing shear time.

Rheopexy is observed, for example, in PVC plastisols. They are used for corrosion protection of metals. Rheopexic liquids are characterized by gradual structure formation under shear.

As with the thixotropy, rheopexy is also only present when the viscosity at rest is achieved after the shearing strain after a certain time.

## Viscoelasticity

The combination of viscous and elastic behavior leads to the designation of viscoelastic fluids. In particular, polymer melts and solutions show such properties, depending on the molecular structure. The description of viscoelastic behavior is a central component of the rheology of non-Newtonian liquids [1], but without significance for viscometry with glass capillary viscometers.

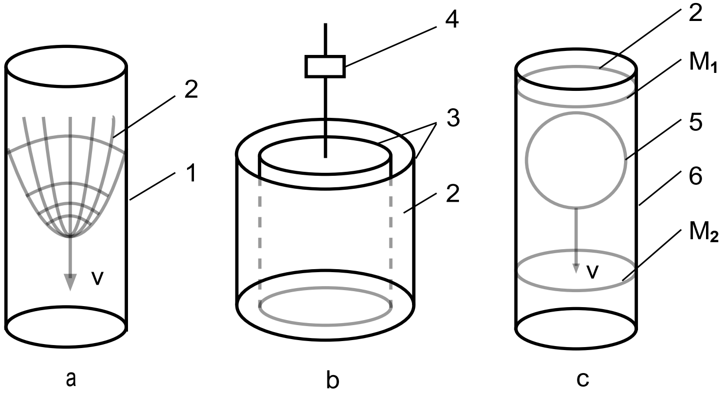
## 1.3 Principles of viscosity measurement

Rheological measurement procedures are mainly using mechanical methods, since they are based on the mechanical quantities stress and strain of the sample.

Viscosity measurement instruments usually generate a defined shear deformation (shear) and measure the required shea stress, or vice versa. The ratio of the two variables is, according to Eq. 1.3 the viscosity.

The most important possibilities for the realization of the deformation of the sample is shown in Fig. 4.

Perfection in manufacturing and sophisticated quality-assurance methods form the basis of standardized measurement systems which meet today's highest accuracy requirements as to reproduction uncertainties and absolute measurement uncertainty.



*a = Capillary viscometer*
*b = Rotational viscometer*
*c = Falling ball viscometer*

*1 = Capillary*
*2 = Sample*
*3 = Coaxial cylinder*
*4 = Torque transducer*

*5 = Measuring ball*
*6 = Glass cylinder*
*$M_1$, $M_2$ = Timing marks*

*Fig. 4  Measurement principles of viscometers*

# CHAPTER 2

## FUNDAMENTALS OF CAPILLARY VISCOMETRY

### 2.1 Measurement principle

Inside the capillary viscometers, the velocity gradient required for viscosity measurement is built up in the form of a laminar tube flow within a measurement capillary.

Under idealized conditions,

- laminar, isothermal, steady flow condition
- Newtonian flow behavior of the liquid
- pressure independence of the viscosity
- incompressibility of the liquid
- wall adherence of the liquid
- neglect of the flow influences at the entry and exit of capillary of sufficient length

the liquid moves in coaxial layers toward the pressure drop through the capillary, in which a parabolic velocity profile is formed (see Fig. 5).

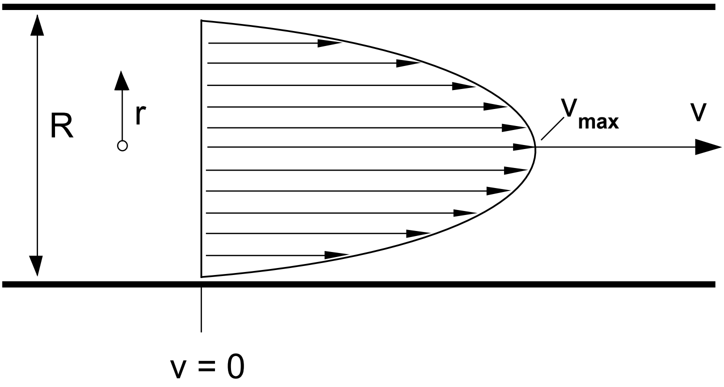


*Fig. 5 Velocity profile with laminar tube flow*

The Hagen-Poiseuille Law is the basis* for the description of the viscosity of all viscometers operating according to the capillary principle [3, 4]:

$$\frac{V}{t} = \frac{\pi R^4 \Delta \rho}{8 L \eta} \tag{2.1}$$

With regard to viscosity measurement, this results in two different fundamental measurement principles:

1) Measurement of the volume flow through the capillary at a given differential pressure
2) Measurement of the differential pressure at a constant volume flow of the liquid through the capillary.

The first measurement principle is used in capillary viscometry with glass capillary viscometers: The differential pressure is established as hydrostatic pressure in a simple manner and with very good reproducibility. Details regarding this measurement principle can be found in the next section.

Continuously operating viscometers can be built with the second measurement principle, whose measurement accuracy depends on the achievable accuracy in differential pressure measurement as well as by the stabilization of a defined volume flow.

Fields of application are, for example, relative measurements in polymer analysis, in which the pure solvent is used as a reference liquid in a first capillary. The sample is loaded, as in a chromatography system, in a sample loop between the first and a second capillary, which is connected in series with the first capillary - through this, the sample only flows through the second capillary and the volume flow is equal in both capillaries. The pressure drop resulting from the volume flow is measured across both capillaries and evaluated as a relative viscosity.

Another field of application of the first measurement principle is the measurement of the viscosity of polymer melts. The sample is pressed through the capillary under high pressure. Short capillaries are used here, frequently gaps with special geometry (high-pressure capillary viscometry).

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* For real tube flows in capillary viscometers, corrections may need to be performed, which are described in Appendix A

## 2.2 Designs of glass capillary viscometers

In low-pressure capillary viscometers, the viscosity is measured via the flow time of a defined volume of liquid through a measuring capillary.

The driving force is the hydrostatic pressure of the liquid column. It can also be operated with overpressure to achieve higher shear rates.

Irrespective of the specific design, the mostly U-shaped glass bodies have ball-shaped extensions, the volume of which determines the quantity of the sample. Measurement marks on the glass body, or accurately defined fixed sensors, allow the measurement of the passage time of the boundary layer between the sample and the air (meniscus), a process which enables the passage time of a sample volume defined in such a manner to be measured with measurement uncertainties < 1/10 s.

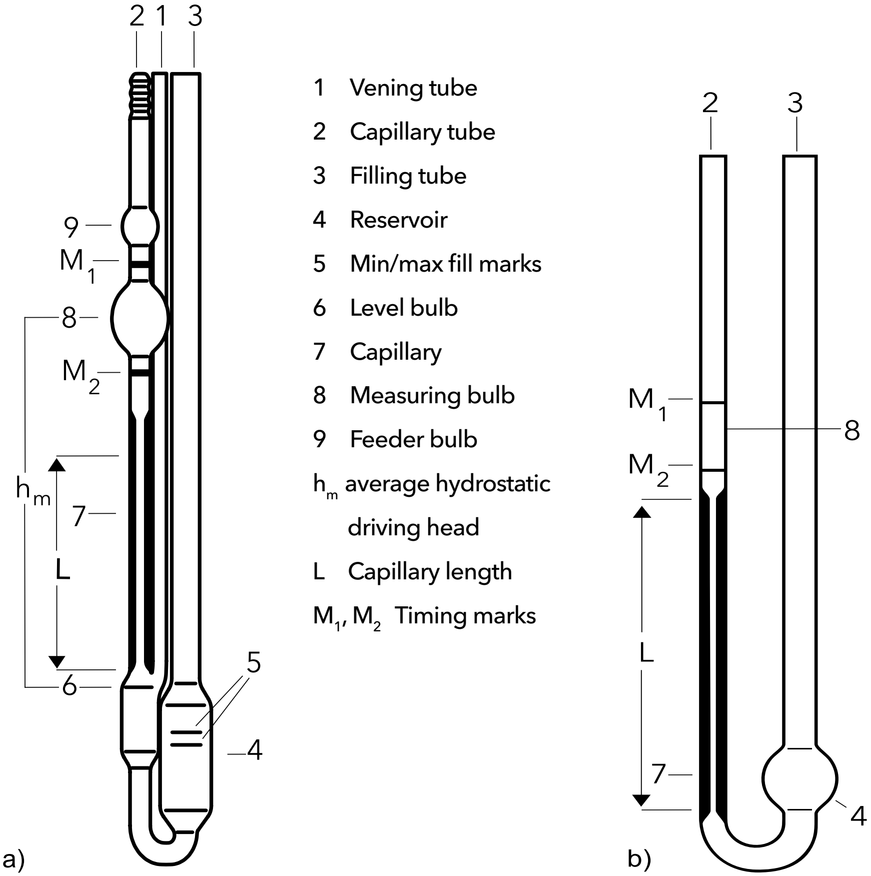
Fig. 6 shows the two fundamental types of viscometers according to OSTWALD and UBBELOHDE.

With both viscometers the liquid being examined is filled through the filling tube (3) into the reservoir (4). Since the mean pressure level in OSTWALD viscometers depends on the filling height, the prescribed measurement volumes must be strictly observed. A pipette is therefore used for filling. The sample is sucked into the tube (2) for measurement. It measures the time required for the meniscus to sink from timing mark $M_1$ to measurement mark $M_2$ (annular measurement marks).

With UBBELOHDE viscometers, the transition from the capillary (7) into the leveling bulb (6) is designed as a spherical cap. An additional venting tube (1) is connected to the leveling bulb (DIN 51562-1, ISO 3105 [2, 32, 33]). After the sample is filled via the filling tube (3) in the reservoir (4), the venting tube (1) is closed. Depending on the operational mode, i.e. pressing or sucking, the sample is filled by overpressure applied to tube (3) or by suction via the tube (2) into the capillaries (7), the measuring sphere (8), and at least up to half of the pre-run sphere (9).

After venting the tube (1), the liquid column in the leveling bulb (6) breaks off. At the exit of the capillary, the so-called suspended level develops (see also Fig. 22). For this reason, only a limited amount of sample may be filled in (between max .- / min. fill marks (5)).

During the measurement, the liquid flowing out from the capillary drains on the inner wall of the leveling bulb (6) as a film. In this way, the hydrostatic pressure of the liquid column is independent of the amount of substance filled in.

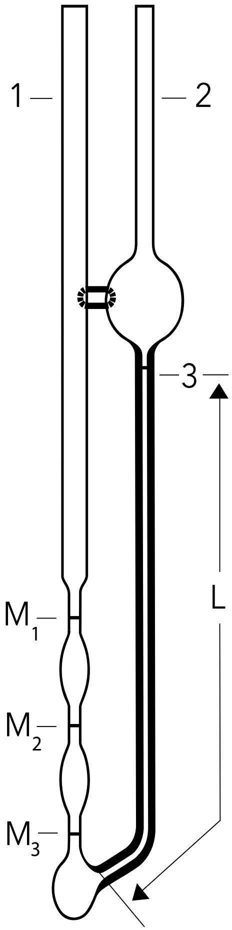


| | |
|---|---|
| 1 | Vening tube |
| 2 | Capillary tube |
| 3 | Filling tube |
| 4 | Reservoir |
| 5 | Min/max fill marks |
| 6 | Level bulb |
| 7 | Capillary |
| 8 | Measuring bulb |
| 9 | Feeder bulb |
| $h_m$ | average hydrostatic driving head |
| L | Capillary length |
| $M_1$, $M_2$ | Timing marks |

*Fig. 6 Glass capillary viscometers according to a) UBBELOHDE and b) OSTWALD*

In addition, owing to the geometrical shaping of the leveling bulb (6), the influence of surface tension on the measurement result is almost eliminated.

In the case of the UBBELOHDE viscometer, too, the measurement is aimed at the time required by the liquid meniscus to sink from the annular measurement mark $M_1$ down to the annular measurement mark $M_2$. In the case of very strongly tinted, opaque liquids, it can be possible that a visual detection of the meniscus passage through the measurement marks is impossible owing to the wetting of the tube. In this case, reverse-flow viscometers (see Fig. 7) are used for manual operation (DIN 51366, ISO 3105). The viscometer is filled standing over head, in which the capillary tube (2) is immersed in the sample and the sample is sucked to a fill mark (3) above the spherical bulb. The tube (1) is closed during the thermostatic control and opened for the start of measurement. To measure the viscosity, the flow time of the meniscus is captured by the measuring marks $M_1$, $M_2$ and $M_3$ on the riser tube (1). Additional nformation on handling can be found in Chapter 6.



| | |
|---|---|
| 1 | Riser tube |
| 2 | Capillary tube |
| 3 | Fill mark |
| L | Capillary length |
| $M_1$, $M_2$, $M_3$ | Ring measurement marks |

*Fig. 7 CANNON-FENSKE Reverse Flow Viscometer*

# CHAPTER 3

# MEASUREMENT OF THE FLOW TIME

## 3.1 Manual timing

In the simplest case, the flow time is captured by an observer using a stopwatch. Glass viscometers manufactured for this purpose have annular measurement marks burnt in above and below the measurement sphere (see Fig. 6, 7).

The disadvantages of this method are:

- Subjective observation errors or differences in the reaction time of the operator at the beginning and end of the timing lead to increasing repeatability uncertainties and, under certain circumstances, to systematic errors.

- In the case of opaque substances the meniscus cannot be seen. One must resort to Reverse-Flow viscometers with their more intricate handling and reduced accuracy.

## 3.2 Automatic timing

### Tasks and particularities

In the case of automatic capillary viscometers, an electric signal has to be generated during the passage of the air/sample or sample/air interfacial layer, respectively, through the measurement marks.

It is required as

- *a start and stop signal for the timing process,*

as well as

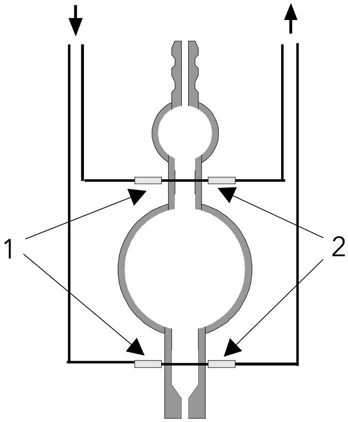
- *a status signal for automatic operation (pumping up the liquid into the measurement bulb, emptying of the viscometer).*

The detection and transformation of a time signal does not pose any metrological problems. In practical viscosity measurement, the measurement uncertainties are determined by the fluid-dynamic circumstances and the detection of the meniscus passage through the timing marks.

The manufacturer of the measurement device has to ensure by design and production provisions that the viscometer constant will not change even if the measurement conditions should deviate from the calibration conditions (e.g. measurement and calibration temperature).

## Detection of the meniscus passage

This task requires the use of sensors responding to the difference between the material properties of the air and the sample being analyzed during the passage of the meniscus through the measurement marks.



*1 = Optical fibre Input*
*2 = Optical fibre Output*

*Fig. 8 Arrangement of optical sensors*

## Optical sensors

During the meniscus passage, the optical conditions such as refraction and reflection within the detection plane are changing. This leads to a change in the radiation intensity of the light arriving from the transmitter at the receiver (see Figure 8).

For the measurement of time, for instance, the analogous signal provided by a photo diode is transformed into a pulse used for the start and stop of the time measurement. Specific threshold values of the analogous signal may be defined for the "filled" or "empty" status.

- Advantage:

Versatile application; simple set-up.

- Disadvantage:

Strongly colored or opaque liquids, particularly with strong wall adhesion, can not be measured.

Optical sensors are housed in a measurement tripod made of metal or plastic in viscometers from SI Analytics GmbH. The viscometer is fastened to a clamp connection in the tripod.

Fig. 8 shows the arrangement of the optical sensors in the measurement stand on the viscometer. The light is guided from the stand head via fiber optic cables in the stand to the upper and lower measurement level. The watertight sealing enables the measurement stands to be placed in liquid thermostats. Owing to high precision in the glass-technological and mechanical production as well as through measures of quality assurance, it is ensured that the glass bodies and tripods are freely interchangeable, with the certified viscometer constants remaining valid.

## Thermal conductivity sensors

Small thermistors melted in on the level of the measurement plane (NTC resistors) are heated up by an electric current. Due to the better thermal conductivity of the fluid, the thermistor cools off with the air/sample transition, thereby increasing its electrical resistance.

- Advantage:
  *Measurement signal formation is independent of tint, transparency and conductivity of the sample*

- Disadvantage:
  *Higher manufacturing costs (melting in of the sensors); risk of encrustation and soiling with thermally decomposable samples.*

Fig. 9 schematically shows a TC viscometer from SI Analytics. The glass-coated thermistors are clearly visible in the tube axis, whose diameter is <1 mm in melted-in head portion.

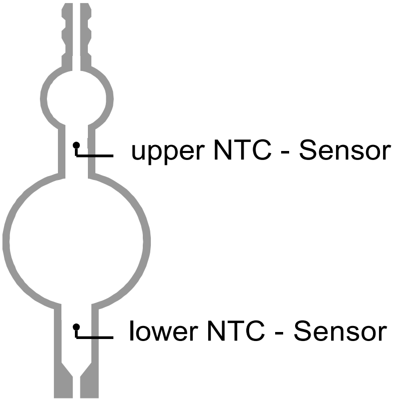


*Fig. 9*
*TC viscometer from SI-Analytics*

# CHAPTER 4

# METHOD FOR DETERMINING VISCOSITY

The absolute value of the kinematic viscosity is determined from the flow time using the calibration constant.

The model of a laminar tube flow forms the starting point, described by the Hagen-Poiseuille Law (see Equation (2.1)).

The hydrostatic pressure of the liquid column in the form of the mean pressure level $h_m$ serves as a driving head (Images 6, 7). Since the volume flow is detected by measuring the flow time $t$ the kinematic viscosity $\nu$ is obtained:

$$\nu = \frac{\eta}{\rho} = \frac{\pi \cdot R^4 g \cdot h_m}{8LV} t \qquad (4.1)$$

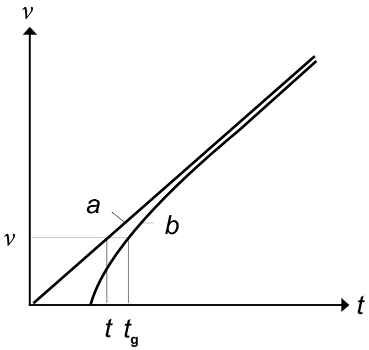
Other than the flow time, Equation (4.1) contains only constants and geometrical data.

They can be summarized to a characteristic variable for a given viscometer, the so-called viscosity constant $K$:

$$\nu = K \cdot t \qquad (4.2)$$

Due to the unavoidable tolerances when manufacturing the devices, only an approximate value of the constant $K$ can be calculated from the dimensions of the viscometer. The precise value of $K$ is determined by the calibration of each individual viscometer (see Chapter 5).

According to equation (4.2), there is a linear relationship between kinematic viscosity and flow time. In Fig. 10, this is represented in the form of the ideal characteristic curve (Curve a).



*a ideal and*
*b real viscometer characteristic curve*

*Fig. 10 Viscometer characteristic curve*

When using the flow model in the form of the Hagen-Poiseuille Law, additional pressure losses at the capillary ends are not considered. Due to the finite capillary length, the pressure losses in the inlet and outlet, however, have an impact on measurement accuracy. Due to these additional pressure losses, the measured flow time $t_g$ is greater than the time $t$, resulting from the Hagen-Poiseuille law.

The basic hydrodynamic processes were first examined by Hagenbach [5] and Couette [6]. The difference between the measured and theoretical flow time ($t_g$ - $t$) is therefore referred to as the kinetic energy correction time ($t_H$) .

$$t = t_g - t_H \tag{4.3}$$

This results in the following corrected equation for glass capillary viscometers:

$$\nu = K \cdot \left( t_g - t_H \right) \tag{4.4}$$

The smaller the flow time, the greater the kinetic energy correction time. Curve b in Fig.10 shows the real characteristic curve. In the practice of viscosity measurement, there are basically three options for taking the influence of kinetic energy correction into account and thus determining the kinematic viscosity of the measured substance.

## 4.1 Neglect of the kinetic energy correction

Long flow times are obtained by selecting viscometers with small capillary diameter (adapted to the sample viscosity). The kinetic energy correction then becomes so small that a correction can be omitted in the context of the required accuracy. The minimum flow time that must be achieved in order to be able to disregard the kinetic energy correction is different, depending on type of viscometer and capillary size. In addition, there are also differences depending on whether the absolute value of viscosity (e.g., for measurements of oils) or the relative viscosity (usually polymer analysis) is measured.

### 4.1.1 Absolute measurements

#### Ubbelohde viscometer according to DIN 51562-1

The following minimum flow times are applicable for DIN Ubbelohde viscometers:

| Size | Flow time |
|---|---|
| 0 | 1000 s |
| 0c | 800 s |
| 0a | 600 s |
| I | 400 s |
| Ic and larger | 200 s |

#### Micro Ubbelohde viscometer according to DIN 51562-2

Based on a smaller measurement volume and a larger capillary length, the kinetic energy correction for Micro Ubbelohde viscometers is smaller than for Ubbelohde viscometers of the standard size. The Hagenbach correction can be neglected in the following minimum flow times:

| Size | Flow time |
|---|---|
| MI | 70 s |
| MIc | 40 s |
| MII and larger | 30 s |

Short flow times below 100 s only permitted for measurement with automated instruments (i.e., not manually with stopwatch).

#### Ubbelohde viscometer according to ASTM D 446

The viscometer according to ASTM is similar to DIN-types, however, have a smaller measurement volume. It is 4 ml for capillary sizes 1 to 4B and 5 ml for size 5. The variables used for low viscosity samples 0, 0C and 0B have small measuring volumes of 1, 2 or 3 ml.

The small measurement volumes were chosen in order to keep the influence of the kinetic energy correction low. Therefore, the correction for all capillary sizes within the specified measurement uncertainty can be ignored if the flow time is a min. of 200 s.

### 4.1.2 Relative measurements

The ISO 1628 standard applies for relative measurements in polymer analysis. DIN or ASTM Ubbelohde viscometers are recommended for viscosity measurements here. Generally in ISO 1628-1, the kinetic energy correction is omitted, as long as it is a max. of 3 % of the shortest flow time - the blank value (solvent). The following minimum values of the flow times are specified:

| Size (DIN/ ASTM) | min. flow time |
|---|---|
| 0/0 | 150 |
| 0c/0C | 100 |
| 0a/0B | 100 |
| I/1 | 75 |
| Ic/1C and larger | 50 |

For automated devices, an undershooting of these values is also permitted.

## 4.2 Calculation of the kinetic energy correction

The manufacturer calculates kinetic energy correction times on the basis of the geometrical dimensions as a function of the flow time and states them in the device descriptions.

The basics of kinetic energy correction and its calculation are described in Appendix A.

The kinetic energy correction can only be calculated approximately, due to sample strew of the viscometer. For accurate absolute measurements, the calculated value may therefore be max. of 1 % of the flow time. If it is higher, viscometers with smaller capillary size have to be used - to extend the flow time - or the individual sample strew is determined for the viscometer.

## 4.3 Experimental determination of individual kinetic energy correction (according to DIN 51562-3)

The kinetic energy correction increases with decreasing flow times and strongly affects the measurement result. The viscometer characteristic curve is also influenced by after-flow effects of the liquid and by the beginning disturbance of the suspended level for UBBELOHDE viscometers.

If an undershooting of the measurement range recommended in the user instructions is unavoidable, an kinetic energy correction for the viscometer must be determined experimentally. To do this, two standard liquids of known viscosity are to be used, whereby the viscosity of the sample to be tested must lie between the viscosity values of the standard liquids. The smaller the difference of the viscosities, the more accurate the correction method.

### Implementation of correction procedure

1) Determination of individual values for the kinetic energy correction with the standard liquids:

$$t_{H_i} = t_{g_i} - \frac{\nu_i}{K} \qquad i = \{1;2\} \tag{4.5}$$

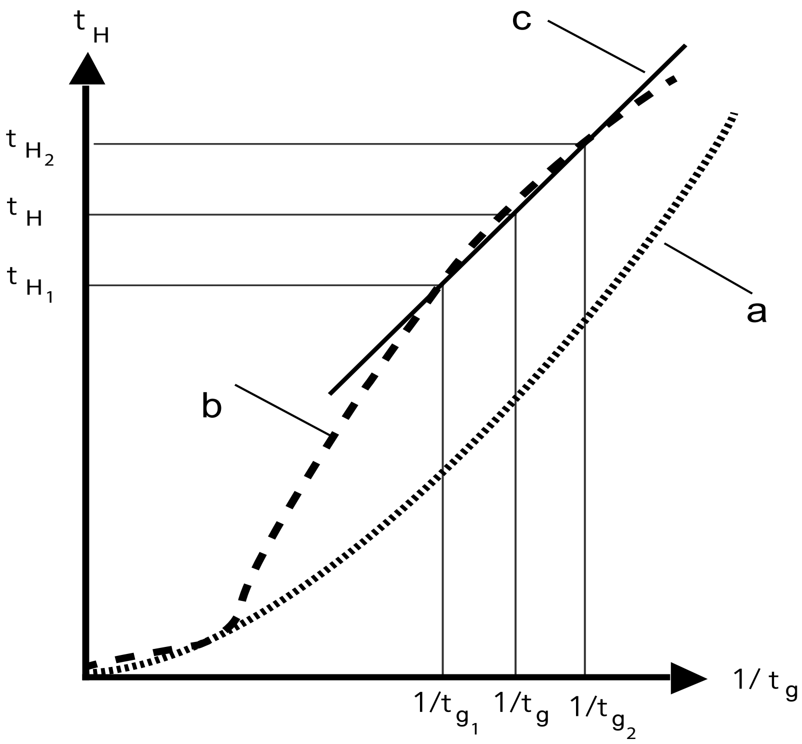
2) Determination of the kinetic energy correction $t_H$ for the flow time $t_g$ through linear interpolation between the values $t_{H1}$ and $t_{H2}$:

$$t_H = t_{H_1} - K_{12}\left(\frac{1}{t_g} - \frac{1}{t_{g_1}}\right) \tag{4.6}$$

$K_{12}$ thus describes the slope of the kinetic energy correction between the reciprocals of the measured slow times $tg_1$ and $tg_2$:

$$K_{12} = \frac{t_{H_1} - t_{H_2}}{\frac{1}{t_{g_2}} - \frac{1}{t_{g_1}}} \tag{4.7}$$

The correction method is graphically represented in Fig. 11.



- a Calculation of the kinetic energy correction as performed in Appendix A (see equation A 1.13)
- b real curve of the individual kinetic energy correction
- c Interpolation line

*Fig. 11 Individual kinetic energy correction* (DIN 51562-3)

## 4.4 Examples of viscosity determination

Viscosity measurement of n-decane at = 23 °C ($\nu \approx 1.21$ mm²/s) with UBBELOHDE viscometers

### 1st Case

Selection of a DIN Ubbelohde viscometer with the capillary 0

$K$ = 0.0009801 mm² / s²
$L$ = 90 mm
$V$ = 5.7 ml
$h_m$ = 130 mm
$D$ = 0.36 mm

Measurement range according user instructions:

0.2 ...1.2 mm² / s

mean measured flow time:

$t_g$ = 1234.57 s

The kinetic energy correction time is approx. 0.3 s. This corresponds to approximately 0.025% of the flow time. Therefore no significant change is recorded in the measurement result by neglecting the kinetic energy correction time.

Calculation of viscosity:

$$\begin{aligned}\nu &= K \cdot t_g\\ \nu &= 0.0009801\ \text{mm}^2/\text{s}^2 \cdot 1234.57\\ &= 1.210\ \text{mm}^2/\text{s}\end{aligned}$$

### 2nd Case

Selection of a viscometer with capillary I

$K$ = 0.01050 mm² / s²
$L$ = 90 mm
$V$ = 5.7 ml
$h_m$ = 130 mm
$D$ = 0.63 mm

Measurement range according user instructions:

1.2 ... 10 mm² / s

mean measured flow time:

$t_g$ = 116.05 s

Calculated kinetic energy correction time as shown in Appendix A (see equation (A 1.13))

$t_H$ = 0.69 s

Calculation of viscosity:

$$\begin{aligned}\nu &= K \cdot (t_g - t_H)\\ \nu &= 0.0105\ \text{mm}^2/\text{s}^2 \cdot (116.05 - 0.69)\ \text{s}\\ &= 1.211\ \text{mm}^2/\text{s}\end{aligned}$$

### 3rd Case

Selection of a viscometer with capillary Ic

$K = 0.03032\ \mathrm{mm^2/s^2}$
$L = 90\ \mathrm{mm}$
$V = 5.7\ \mathrm{ml}$
$h_m = 130\ \mathrm{mm}$
$D = 0.84\ \mathrm{mm}$

Measurement range according user instructions:

$3 \ldots 30\ mm^2/s$

mean measured flow time:

$t_g = 39.95\ s$

Calculated kinetic energy correction time (according to equation A 1.13)

$t_H = 1.03\ s$

The measurement range of the viscometer was undershot. In this case, a viscometer with a smaller capillary diameter should be used. If this is not possible, the individual kinetic energy correction time must thus be determined experimentally for precision measurements.

# CHAPTER 5

# CALIBRATION

The viscometer constant *K* is determined by individually calibrating each glass capillary viscometer. SI Analytics guarantees reproducible calibration of the highest precision through the use of reference measurement standards with very low measurement uncertainty.

## Measurement principle

SI Analytics determines constants by simultaneous flow measurement in the viscometers to be calibrated (test specimens) and in reference measurement standards, whose constants were determined by testing at the Physikalisch-Technischen Bundesanstalt (Physical Technical Federal Institution, PTB) in Brunswick.

This calibration procedure is referred to as calibration by direct comparison, in contrast to calibration with calibration liquids.

An essential advantage is that a deviation of the absolute temperature does not lead to an increase of measurement uncertainty, as long as the temperature in the thermostat bath is temporally and spatially constant. Nevertheless, at SI Analytics, the calibration temperature is set to 23.00 °C ± 0.02 °C checked by a certified thermometer.

## Realization

The flow time of a test liquid is measured by several glass capillary viscometers in a thermostat bath with a temperature uncertainty of max ± 0.02 °C.

Using these viscometers, there is a reference measurement standard, from whose flow time the kinematic viscosity of the test fluid is calculated.

Test liquids do not need to be reference standards when calibration is performed by direct comparison - the viscosity of the test fluids is actually determined in the reference viscometer.

However, Newtonian standard samples of the Deutscher Kalibrierdienst (German Calibration Service, DKD) are used at SI Analytics as test liquids. The measured viscosity of the test fluid can be checked for its plausibility in this manner.

The constants of the device being tested are determined from the kinematic viscosity of the fluid and the flow time (see Fig. 12).

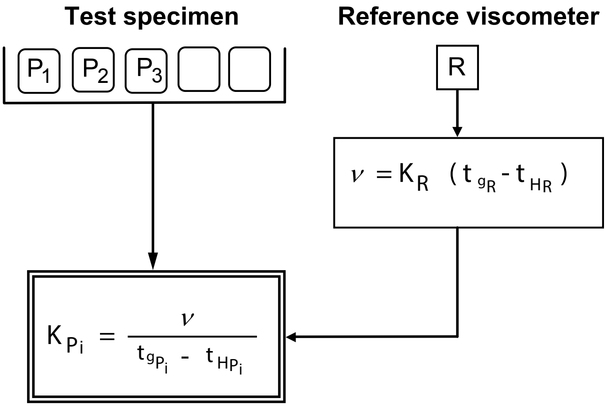


*Fig. 12 Performing the calibration of glass capillary viscometers*

Each calibration can guarantee the metrological accuracy of the viscometer constants for only a limited period. We therefore recommend that the constants are tested on a regular basis or to have it checked by the manufacturer.

The check can be performed either by comparative measurements with reference measurement standards (see above) or with reference standard liquids from a certifiedd institute (e.g. DKD in Germany). However, if regular oils are being used, the limitation of the accuracy of the test procedure caused by the uncertainty of the regular-oil viscosity indication should be noted.

See also DIN 51562-4: Viscometer calibration and determination of measurement uncertainty.

## Calibration of the viscometers at national metrological institutes

The calibration of viscometers is traced back to the viscosity of water. The following values are valid internationally (according to ISO / TR 3666:1998):

$\eta = 1.0016$ mPas (20 °C)

$\nu = 1.0034\ \text{mm}^2/\text{s}$.

At metrological institutes, master viscometers are used with a special capillary length of 400 mm. By comparing the flow time of water with those of higher viscosity calibration liquids in the identical viscometer as well as the flow times of the same liquids in viscometers of different capillary sizes (step-up procedure), the viscosity value of water is ultimately used as a reference standard for larger viscometer values in which water can not be measured.

Viscometers for industrial use usually have shorter capillaries (70-250 mm).

# CHAPTER 6

# HANDLING OF CAPILLARY VISCOMETERS

## 6.1 General guidelines for the selection of the measuring system

### Selection of the type of viscometer

The following viscometers from SI Analytics can be used for the measurement of transparent liquids:

- UBBELOHDE viscometer
- OSTWALD viscometer
- CANNON-FENSKE routine viscometer

These devices can therefor be used for manual or automatic measurements with optoelectronic detection of the meniscus passage. In addition, TC-UBBELOHDE viscometers may be used with thermistor sensors. UBBELOHDE viscometers are to be preferred in the majority of applications over other types because of the benefits mentioned in chapter 2.

Exception: Fewer bubbles occur in viscometers without a ventilation tube (OSTWALD and CANNON-FENSKE). These viscometers should be used in measurements of slightly foaming or bubbling liquids, since foam or bubbles impair the function of the photocells.

Due to the venting tube of the UBBELOHDE viscometer, this should not be used for samples that undergo chemical reactions with the ambient air. OSTWALD and CANNON-FENSKE routine viscometers are preferable here.

If only a little substance or solvent is available, the Micro UBBELOHDE or Micro OSTWALD viscometers can be used.

High and low temperature measurements should always be performed in UBBELOHDE viscometers for reasons of temperature-induced volume changes of the measurement substance.

CANNON-FENSKE reverse flow viscometers are available for manual measurement of opaque liquids. TC UBBELOHDE viscometers are suitable for automatic viscosity determination of opaque oils and emulsions. Since the thermistor sensors are glass-coated and sealed hermetically in the viscometer, conductive and highly aggressive liquids can also be measured.

## Selection of the capillary

The capillary diameter (0.25 ... 10 mm) determines the measurement range of the viscometer. Each capillary diameter is assigned a viscometry size number (Table 1).

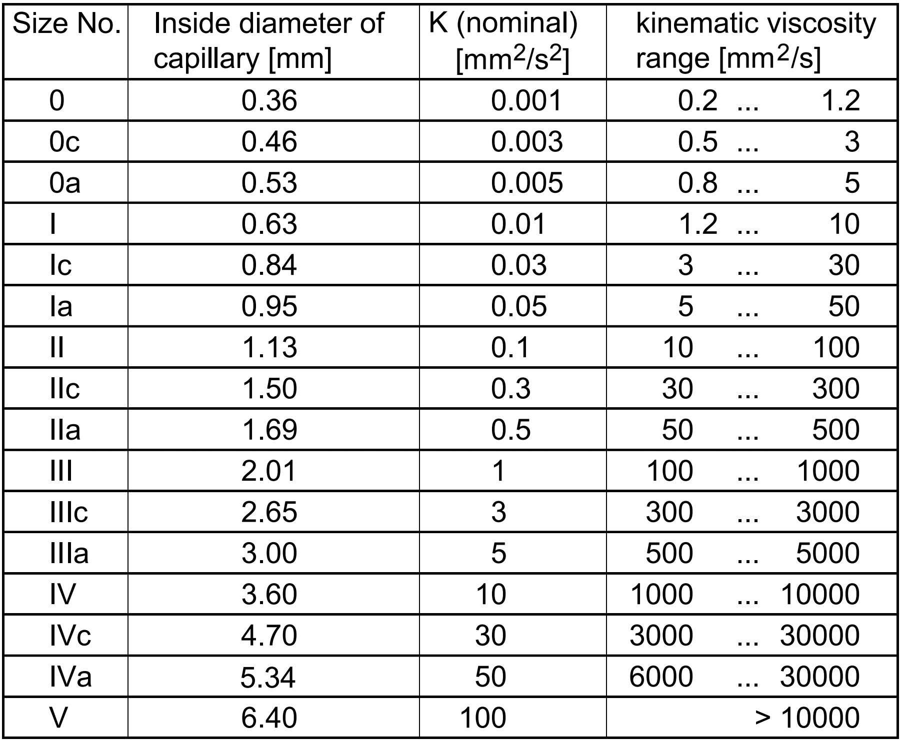
To select a viscometer, the viscosity of the substance to be tested must be estimated in which the flow time is roughly estimated (without kinetic energy correction) according to equation (4.2).

According to DIN 51562 / ISO 3105 and ASTM D446, the aspired minimum flow time for absolute measurements for most viscometers should be 200 s.

For relative measurements, flow times of at least 50 seconds are permissible according to ISO 1628-1 for the viscometer with larger capillary number. See the specifications in Section 4.1.

For Micro UBBELOHDE viscometers, the flow time can be shortened up to 35 s (viscometer no. MI) or 30 s (viscometer no. M Ic and larger). For use of the individual kinetic energy correction as well as automatic flow time measurement, flow times to approx. 10 s are possible using Micro UBBELOHDE viscometers according to some research results [34, 35]. Such short flow times should not be applicable in normal industrial laboratory practice.

Table 1 shows an example of the measuring ranges, depending on the capillary diameter for DIN-UBBELOHDE viscometers.



| Size No. | Inside diameter of capillary [mm] | K (nominal) [$mm^2/s^2$] | kinematic viscosity range [$mm^2/s$] |
|---|---|---|---|
| 0 | 0.36 | 0.001 | 0.2 ... 1.2 |
| 0c | 0.46 | 0.003 | 0.5 ... 3 |
| 0a | 0.53 | 0.005 | 0.8 ... 5 |
| I | 0.63 | 0.01 | 1.2 ... 10 |
| Ic | 0.84 | 0.03 | 3 ... 30 |
| Ia | 0.95 | 0.05 | 5 ... 50 |
| II | 1.13 | 0.1 | 10 ... 100 |
| IIc | 1.50 | 0.3 | 30 ... 300 |
| IIa | 1.69 | 0.5 | 50 ... 500 |
| III | 2.01 | 1 | 100 ... 1000 |
| IIIc | 2.65 | 3 | 300 ... 3000 |
| IIIa | 3.00 | 5 | 500 ... 5000 |
| IV | 3.60 | 10 | 1000 ... 10000 |
| IVc | 4.70 | 30 | 3000 ... 30000 |
| IVa | 5.34 | 50 | 6000 ... 30000 |
| V | 6.40 | 100 | > 10000 |

*Table 1 Measurement range of DIN-UBBELOHDE viscometers* [15]

## 6.2. Cleaning of capillary viscometers

Careful cleaning of the viscometer is a basic requirement for exact and reproducible results. Practice has shown that in the majority of cases, contaminants are the cause of increased scattering of the flow time. The smallest quantities of microscopic particles of dirt here can induce standard deviations up to several percent in the viscometer.

Particles which adhere firmly to the capillary wall and are frequently almost invisible are often the cause of systematic measurement errors. Such errors, which lead to an extension of the flow time, are difficult to see from the individual values of a measurement series. The larger the chosen capillary diameter, the lower the risk of contamination.

In addition to solid particles, oil or grease films adhering to the inner wall of the viscometer have an influence on the flow time.

Especially when measuring materials with high surface tension (e.g. aqueous media), there is the formation of liquid droplets during the start-up process for insufficiently cleaned viscometers, which adhere to the wall and falsify the measurement result. That is why it is advisable to measure only substances with similar properties in a viscometer. If this is not feasible, an especially thorough cleaning must be carried out.

It is recommended to filter all cleaning agents before use. Glass frits are suitable for this and syringe filters for nonhazardous liquids. Paper filters tend to detachment of fibers and are therefore not recommended.

### Initial or intensive cleaning

There may be contaminants from transport or storage. In addition, with a calibrated viscometer - despite cleaning at the manufacturer - residual calibration oil may be present. A thorough initial cleaning is therefore recommended.

Suitable cleaning fluids have been proven:

- ## Initial cleaning

For the removal of possibly oil existing residues of calibration, calibrated viscometers should be rinsed with a volatile petroleum spirit (e.g., boiling range 40 °C - 60 °C).

- ## Intensive cleaning

The classic cleaning agent for stubborn impurities is concentrated sulfuric acid with the addition of potassium dichromate (chromic acid).

ATTENTION!
In many laboratories, the use of chromic acid is forbidden! Extreme caution is always advised for: Chromium (VI) compounds are highly toxic and carcinogenic. Handling is therefore reserved to trained laboratory personnel in compliance with the applicable regulations. If possible, this chemical should be avoided.

In many cases, it is sufficient to use concentrated sulfuric acid without the addition of chromate. This is also dangerous and it is essential to observe the guidelines for hazardous materials.

A milder cleaning agent is a solution of 15% hydrochloric acid and 15% hydrogen peroxide. This mixture is oxidizing and can thereby remove certain organic and inorganic contaminants.

In many cases, a laboratory detergent such as Mucasol® is successful. Such laboratory cleaners are alkaline and therefore also attack glass at high concentration, at high temperatures and for long duration of action. So that the calibration constant of the viscometer is not changed, laboratory cleaners should only be used

- occasionally,
- as a dilute solution in accor dance with manufacturer's instructions,
- not at an elevated temperature
- and with an exposure time of max. 60 minutes.

## Method of cleaning

1. Complete filling the viscometer with the cleaning substances specified above.
2. Performance at room temperature. As exposure time of approx. one minute is sufficient in the case of petroleum spirit. For intensive cleaning with sulfuric acid, chromic acid or hydrochloric acid / hydrogen peroxide, it is recommended that the reagent fill the viscometer at least 12 hours.
3. Rinse the viscometer with distilled water. This does not apply if oils or similar hydrophobic sample residues are removed with petroleum spirit or similar.
4. Rinse with a filtered, water-miscible, highly volatile solvent (e.g., acetone). This is not nescessary if oil or similar hydrophobic sample residuals or similar are removed with volatile petroleum spirit.
5. For drying with a flow of air, it is preferably generated by connecting a vacuum pump, not by compressed air: This procedure is safer when the air is clean and oil-free.

The use of strong alkaline solvents leads to glass corrosion, by which the viscometer constant is changed.

## Daily cleaning

The viscometer should be cleaned with suitable solvents immediately after each measurement. The use of a vacuum pump has been proven useful here.

## Cleaning by using a vacuum pump

1. Connect the vacuum pump to the capillary tube via a liquid trap.
2. Pour in the cleaning liquid into the filling tube and the venting tube (for UBBELOHDE viscometers).
3. Periodically close the filling and venting tubes during aspiration of the liquid. A pulsating flow of liquid is created, which dissolves even stubborn contaminants.
4. Repeat the cleaning process if necessary (once or twice).
5. Rinse with highly volatile solvent.
6. Dry by sucking dry, dust-free air.

### Cleaning without using a vacuum pump

1. Pour cleaning fluid into the filling tube.
2. Suck the liquid several times into the measurement bulb.
3. Clean the remaining viscometer parts by shaking the viscometer.
4. Drain the viscometer.
5. Repeat the cleaning process two or three times.
6. Rinse with filtered, highly volatile solvents.
7. Dry by blowing dry, dust-free air or in a drying cabinet.

The cleaned viscometers should be kept free of dust. A cleaning operation should also be performed if the measured values (flow times) scatter by more than 0.2%.

To reduce the probability of occurrence of such errors from the outset, cleaning of the viscometer on a regular basis with intensive cleaning fluid at longer intervals is recommended.

## 6.3 Preparation of the measurement

### Sample preparation

Solid particles in the sample to be tested have similar effects on the measurement result as do impurities in the viscometer. For this reason, the following applies:

1. All parts coming into contact with the measured substance should be thoroughly cleaned and dried.
2. The samples have to be filtered.

### Filtration of low-viscosity samples

- Glass filter porosity 2 to 4 (10 - 100 µm).
- Syringe filters with porosity 5 µm: Such attachment filters may only be used for non-hazardous samples, since there is a risk that the attachment filter will come off during filtration and the sample thereby splashes around. Syringes with luer lock connections provide increased safety.
- For hazardous chemicals, particularly in polyme analysis: SI Analytics therefore offers a filter device ProClean II.

## Filtration of highly viscous samples

Sieve, mesh width 0.3 mm.
Paraffin or resinous products and substances in which the pour point is less than 30 °C below the testing temperature should be heated accordingly before the measurement. The measurement temperature must be at least 20 °C higher than the pour point.

## Filling of UBBELOHDE and OSTWALD viscometers

The substance to be tested is filled via the filling tube into the reservoir.

Considering that the driving head of the OSTWALD Viscometer depends on the filling quantity, the sample volumes for OSTWALD and Micro OSTWALD Viscometers indicated in table 2 are to be adhered to in any case. A pipette is therefore used for filling.

UBBELOHDE viscometers have two fill marks on the storage reservoir that designate the maximum and minimum filling quantity.

Only one mark is present for Micro UBBELOHDE viscometers, for which a tolerance range of about ± 1 mm should be observed. Accurate metering is therefore not necessary. It should only be ensured that the opening of the venting tube on the reference level vessel is above the liquid level.

Air bubbles can lead to increased measurement deviations during the measurement process. Therefore, make sure that the viscometer is filled without bubbles.

To do this, the viscometer is held somewhat obliquely and the liquid so poured in so that it flows down without bubbles on the filling tube into the reservoir.

| **Viscometer type** | **sample amount [ml]** |
|---|---|
| OSTWALD | 3 |
| Micro OSTWALD | 2 |
| UBBELOHDE | 15 - 20 |
| Micro UBBELOHDE | 3 - 4 |
| CANNON-FENSKE routine | ca. 7* |
| CANNON-FENSKE opaque | ca. 12* |

* Filling upside-down until filling mark

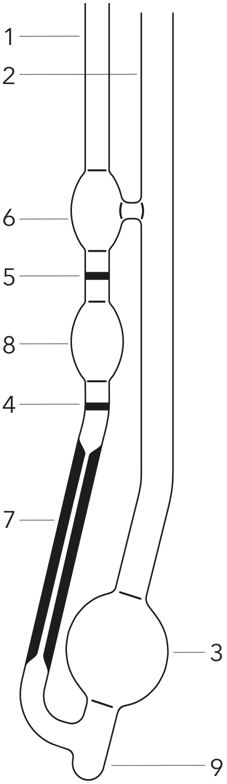
*Table 2 Filling quantities of various viscometers*

Especially when filling in substances of a higher viscosity into OSTWALD viscometers, the pipette should be immersed deeply into the filling tube in order to prevent errors due to flow-tailing.

## Filling CANNON-FENSKE routine viscometers

CANNON-FENSKE Routine Viscometers (see Fig. 13) are held upside down for filling. The capillary tube (1) immerses into the liquid to be measured, while suction is applied at the other tube (2) until the liquid has reached the timing mark $M_2$. After filling, the viscometer is placed in the normal measurement position.

Since the filling process of reverse flow viscometers is somewhat more complex, a reference should be made at this point to the standards DIN 51366, ISO 3105, ASTM D446 as well as to the user instructions.



| | |
|---|---|
| 1 | Capillary tube |
| 2 | Vent tube |
| 3 | Reservoir |
| 4 | Lower annular timing mark $M_2$ |
| 5 | Upper annular timing mark $M_1$ |
| 6 | Sphere |
| 7 | Capillary |
| 8 | Measuring bulb |
| 9 | Tube extension |

*Fig. 13 CANNON-FENSKE Routine viscometer*

### Suspending the viscometer in racks

SI Analytics offers brackets or holders for all types of viscometers, which ensure a stable vertical position of the viscometer in the thermostat bath. They also protect the viscometers from breakage. Before measuring, UBBELOHDE viscometers should be inserted in the brackets provided for this purpose (see Fig. 14), and fixed in position by pressing the spring downwards.

*Fig. 14 Using the viscometer in a bracket*

## 6.4 Performing the measurement

### Thermostatization

Viscosity depends strongly on the temperature. Therefore, the viscometers have to be treated in a thermostat during the measurement. The thermostats used are automatically controlled glass panelled viscothermostats. The viscometer has to be immersed until the bath liquid is at least 2 cm higher than the liquid meniscus in the viscometer in its highest position.

The test temperature should be kept temporally and spatially constant to ± 0.02 K in the range between + 15 °C to + 100 °C. Greater deviations cannot always be avoided outside the specified temperature range, but this should still not exceed ± 0.05 K. Calibrated precision thermometers with a resolution of 0.01 °C are recommended for temperature control. Such a high degree of accuracy is often achieved by certified mercury-glass thermometers with a scale division of 1/100 °C.

The alternatively available electronic platinum resistance thermometers with comparable precision are considerably more expensive. In addition, the validity of the calibration over a long period is difficult to realize. The liquid bath and especially the thermometer must be protected from direct light. Recommended bath fluids are:

| | |
|---|---|
| below 0 °C | alcohol |
| 0...80 °C | distilled water<br>+ tap water |
| 80...105 °C | water + glycol,<br>silicone oil |
| 105...200 °C | silicone oil, also of limited suitability:<br>polyglycols,<br>paraffin oil |

The transparent thermostats of the CT series developed by SI Analytics for capillary viscometry meet the requirements of viscometry in terms of temporal and spatial constancy of bath liquid temperature of ± 0.02 °C. They have openings or inserts for two (CT 72/2) or four capillary viscometers (CT 72/4). The capillary viscometers filled and inserted into the bracket or holder are suspended in the pre-tempered thermostat bath.

When using transparent thermostats of the CT series, special inserts for viscometer brackets are available for manual measurement.

Subsequently, the sample is exposed to thermostat treatment in the viscometer. For measurements with UBBELOHDE, OSTWALD or CANNON-FENSKE routine viscometers, it is recommended to pump the liquids at least three times into the measurement bulb in order to speed up the heat transfer. This is not possible with reverse flow viscometers. Their temperature adjustment should therefore be correspondingly longer. The following temperature-adjustment times are recommended:

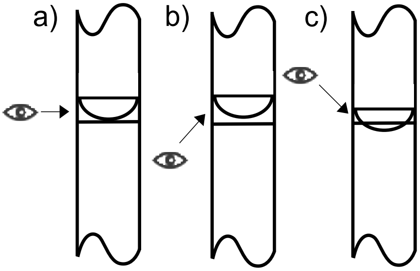
| | |
|---|---|
| 5 min | moving, low-viscosity substances |
| 10 min | moving, highly viscous substances, low-viscosity substances in the reverse flow viscometers |
| 15 min | highly viscous substances in the reverse flow viscometers |

The greater the measured temperature differs from the ambient temperature, the longer the temperature adjustment time has to be chosen.

## Manual measurement

To measure the flow times, the liquid is sucked into the measurement bulb by applying a vacuum to the capillary tube. When using viscometers with a feeder bulb, the latter should be filled at least up to its half.

Viscometers without a feeder bulb are filled until the liquid meniscus is approx. 20 mm above the upper timing mark. If UBBELOHDE viscometers are used, the venting tube must be closed prior to aspiration (e.g. with finger). After completion of the filling process, the suction hose is removed from the capillary tube and, for the UBBELOHDE viscometer, the venting tube released.



Perspective:

*(a)* – correct

*(b), (c)* – not correct, reading error due to parallax

*Fig. 15 Detection of the meniscus passage with manual measurement*

When measuring highly viscous samples, it is recommended that the capillary tube is kept closed after releasing the venting tube until the leveling bulb has run empty and the suspended level has built up.

When studying volatile substances, it is advantageous to realize the filling of the measurement sphere by overpressure at the filling tube in order to prevent evaporation. Closing and opening the venting tube in the case of UBBELOHDE viscometers should be done analogously.

The measurement involves the period of time over which the lower for vertex of the meniscus sinks from the upper edge of the upper annular mark down to the upper edge of the lower annular mark. The stop watch used for timing should have a resolution of at least 0.01 s.

When the meniscus passage is detected,it has to be made sure that the annual mark is at eye level (see Fig. 15).

In order to make the measurement values available for statistical evaluation, the measurement process should be repeated several times. Especially in the case of UBBELOHDE viscometers, in order to avoid any formation of bubbles, it should be noted that a renewed sucking or pressing up of the measurement substance must only begin when the drainage of the liquid from the capillary is completed.

When using reverse flow viscometers, sucking the liquid into the measurement sphere is not applicable. To perform the measurement, the tube that was closed after filling is opened on the side of the measurement sphere, and the time over which the liquid rises from the lower to the upper annular mark is subsequently measured.

The CANNON-FENSKE reverse flow viscometer is equipped with two measurement spheres one on top of the other , i.e., two measurement values are available after just one liquid passage. Reverse flow viscometers must be emptied, cleaned and refilled to repeat the measurement.

If the flow times of a series of measurements is scattered by more than 0.2-0.4%, one of the following causes of error is usually present:

- The sample is not homogeneous, or there are particles in the solution. Samples with particles can only be measured after filtration.
- The viscometer is contaminated.
- The sample is not sufficiently thermostatically controlled.

After cleaning the viscometer, the measurements must be repeated with a new portion of filtered sample. If only one "outlier" is present, it can be deleted, or better yet, be replaced by an additional measurement. An outlier test may need to be performed [17].

The calculation of the viscosity is based on the mean value of the flow times.

## Automatic measurement

For automatic viscosity measurement using UBBELOHDE, CANNON-FENSKE routine and MICRO OSTWALD viscometers, SI Analytics offers the automatic viscosity measurement devices of the AVS® series. Table 3 provides an overview of the device program.

The correct measurement stand AVS®/S, AVS®/SK or AVS®/S-CF for automatic viscosity measurement is chosen according to viscometer type and the bath liquid of the thermostats (metal stand for non-aqueous media, PVDF stand as a corrosion-free option).

| Instrument type | Meniscus detection | Way of pumping sample into measuring bulb | Remarks |
|---|---|---|---|
| **ViscoClock** | light barrier | vacuum or pressure, manual | No Cannon-Fenske viscometers |
| **AVS®370** | light barrier<br>NTC- sensor | vacuum or pressure, autonatic | PC controlled |
| **AVS®470** | light barrier<br>NTC- sensor | vacuum or pressure, automatic | Stand alone,<br>with printer,<br>without PC |
| **AVS®Pro III** | light barrier<br>NTC- sensor | vacuum, automatic | Full automated viscosity measuring system |

*Table 3  Automatic viscosity measuring instruments from SI Analytics*

No measurement tripod is required for measurement using the TC UBBELOHDE viscometer. The viscometer is clamped into a special bracket and suspended in the thermostat bath. The connection with the control unit is made using a cable which is plugged into a socket on the viscometer head. The viscometers are pneumatically connected to the AVS® device via silicone or PTFE hoses.

All automatically operating devices are microprocessor controlled. An RS-232-C interface allows for the connection of an external printer or computer. The parameterization and the start of the automatic measuring sequence occurs at the control unit. The displacement of the measuring liquid in the viscometer is performed by an internal pressure or suction acting micropump. It is so controlled that an optimal pumping pressure for the reproducible filling of the measurement system is set, depending on the viscosity of the sample.

The viscosity measuring devices are operated either via built-in software (AVS® 470) or via a PC with the appropriate PC software (AVS® 370, AVS® Pro III).

The AVS®Pro III Automatic Viscosity Sampler is a fully automatic viscosity measurement system for routine measurements. This device performs measurements of kinematic and relative viscosity up to calculation and documentation in an self-acting manner. Filling, discharing, and rinsing of the viscometers are integrated in the automatic course of the measurement. The viscosity limit of the sample is about 800 mm$^2$/s at 25 °C due to the automatic filling.

# CHAPTER 7

## SOURCES OF ERROR AND SPECIAL CORRECTIONS

The following information is not a substitute for an individual study of the fundamental standard DIN 53012 regarding this topic. We therefore strongly recommend acquiring more information.

Many of the influences and corrections named in the following section are negligible in general practice, since they change the measurement uncertainty only in the range of < 0.1%.

Certain factors may cause significant errors, however. Most important is the temperature control of the viscometer. In addition, the change in the gravitational acceleration at the site in relation to the calibration location as well as high surface tension of the samples may represent sources of error that should be corrected.

## 7.1 Correctable errors and corrections

### Surface tension correction

Surface tension causes the liquid which is wetting the tube wall to climb by a distance of Δh. The size of the relative error ε in terms of % can be calculated on the basis of the following formula:

$$\varepsilon = \frac{2}{g h_m}\left(\frac{1}{r_1} - \frac{1}{r_2}\right)\left(\frac{\sigma}{\rho} - \frac{\sigma_0}{\rho_0}\right) \cdot 100\% \qquad (7.1)$$

$h_m$ - mean pressure height
$g$ - acceleration due to gravity
$r_1$ - radius of the measurin bulb
$r_2$ - radius of the spherical cap of the bulb, flowing into the liquid from capillary
$\sigma$ - surface tension of the measurement substance
$\sigma_0$ - surface tension of the calibration substance
$\rho$ - density of the measurement substance
$\rho_0$ - density of the calibration substance

If the relation between surface tension and density of deviates considerably from sample to calibration substance, measuremnet uncertainty increases:

a) in the case of viscometers with a small pressure head, where the liquid flows from the upper container into another container the diameter of which is considerably different from the one of the upper container, e.g. CANNON-FENSKE and OSTWALD viscometer.

b) in all viscometers where the fluid flows out freely from the capillary.

In the case of UBBELOHDE viscometers, the correction will in general be no more than 0.1 to 0.2 % and can thus be neglected in most cases. This has been demonstrated experimentally [12].

## Thermal expansion of the capillaries and the measurement vessel

During high- and low-temperature measurements, the radius and the length of the capillaries, the volume of the measurement sphere, and the average pressure height of the viscometer will change owing to the large difference between the measurement and the calibration temperature. For this reason, the viscometer constant has to be corrected in the case of precision measurements.

The corrected device constant according to DIN 53012:

$$K' = K\left(1+\alpha\left(\vartheta-\vartheta_0\right)\right) \tag{7.2}$$

Viscometers from SI Analytics are calibrated at a temperature of $\vartheta_0$ = 23 °C. The coeffcient of longitudinal expansion $\alpha$ used for production by DURAN®-glass) is $3.3 \cdot 10^{-6}$ $K^{-1}$.

Even at a temperature difference of 150 °C, the change in the calibration constant according to Eq. (7.2) is less than 0.1 % and can therefore be neglected in general.

## Thermal expansion of the measurement substance

In the case of UBBELOHDE viscometers, no correction is required, since the measurement result is largely independent of the substance quantity being filled in. If, in the case of viscometers without suspended level, the substance temperature should deviate from the measurement temperature during the process of filling the viscometer, a volume change of the measurement substance leading to a change of the viscometer constants will occur during the temperature adaptation. The constants should then be corrected according to Eq. (7.3) for OSTWALD and CANNON-FENSKE routine Viscometer or according to Eq. (7.4) for reverse-flow viscometers (DIN 53012, ASTM D 446).

$$K' = K\left(1 + \frac{4V(\rho_2 - \rho_1)}{\pi D_m{}^2 h_m \rho_2}\right) \tag{7.3}$$

$$K' = K\left(1 - \frac{4V(\rho_2 - \rho_1)}{\pi D_m{}^2 h_m \rho_2}\right) \tag{7.4}$$

## Inclination error

Viscometers have to be used in the position in which they were calibrated. If the connection line between the center points of the reference bulbs deviates from normal position, the mean pressure head of the viscometer will change. If, instead of the initial angle $\Phi_0$ the connection line compared to perpendicular is at an angle of $\Phi$ the corrected viscometer constant is to be calculated according to:

$$K' = K\,\frac{\cos\phi}{\cos\phi_0} \tag{7.5}$$

The brackets or holders offered by SI Analytics ensure a perpendicular suspension of the viscometer with a deviation < 1°. This corresponds to a max. relative constant error of 0.02 %. This means that the inclination error can be neglected if these racks are being used.

$D_m$ - mean diameter of the liquid meniscus in the reservoir vessel
$\rho_1$ - density of the measurement substance at filling temperature
$\rho_2$ - density of the measurement substance at measurement temperature
$V$ - filling quantity
$h_m$ - mean pressure head

## Local dependence of gravity

The viscometers from SI Analytics are calibrated at a gravitational acceleration of 9.8105 m/s$^2$.

A correction is required if the acceleration of fall at the calibration place $g_0$ and the acceleration of fall at the measurement place $g$ are significantly different. Equation (7.6) is to be used to calculate the corrected device constant.

$$K' = K\,\frac{g}{g_0} \tag{7.6}$$

The acceleration due to gravity on Earth depends on the geographical latitude and the altitude above sea level: gravitational acceleration decreases with decreasing latitude and increasing altitude. According to WELMEC (Western European Cooperation in Legal Metrology) it can be calculated for different geographic locations using the following formula, which reflects the global gravity field [24, 37]:

$$g = 9.780318\,(1 + 0.0053024 \sin^2\phi - 0.0000058 \sin^2\phi - 0.000003085 \cdot h \ \mathrm{m/s^2})$$

$\phi$: geographic latitude
$h$: height [m] above the sea level

## Inaccurate adjustment and measurement of temperature

Errors caused by inaccurate temperature adjustment or insufficiencies in the temperature stability or temperature measurement are frequently very large, since the viscosity of most of the liquids varies largely as a function of temperature.

According to DIN 53012, it is permissible to correct the viscosity deviation to the target temperature at temperature deviations <1 °C:

$$U_\nu = -\frac{1}{\nu} \cdot \left(\frac{d\nu}{d\vartheta}\right) \tag{7.7}$$

The temperature coefficient $U_\nu$ is determined according to the corresponding DIN 53017. In practice, however, this is more complicated than working with the correct set temperature.

Fully immersed calibrated thermometers with a resolution of 0.01 K should only be used for temperature measurement. The requirements for the use of thermostats are described in section 6.4.

## 7.2 Uncorrectable errors

### Turbulence

Laminar flow is the basic requirement for viscosity measurement according to the capillary principle. The transition from laminar to turbulent tube flow occurs in capillaries (tubes) at a Reynolds number of about 2300. For capillary viscometers, turbulence occurs even sooner due to the influence of capillary ends (with sharp-edged capillaries, e.g., already at 1100).

For a given viscometer, the Reynolds number can be calculated according to the following numerical equation (DIN 53012):

$$Re = 63.7 \cdot \frac{V}{R \cdot K \cdot t_g^2} \qquad (7.8)$$

with the units

$[V]$ = cm$^3$

$[R]$ = cm

$[K]$ = mm$^2$/s$^2$

$[t_g]$ = s

Considering that kinetic energy correction will increase with an increase in the Reynolds number, one should work with a Reynolds number below 200 if this is possible in practice.

### Disturbance of the suspending level in the case of UBBELOHDE viscometers

If viscosity measurements are performed with short flow times, a deformation of the suspended level may occur. This will lead to systematic measurement errors, since the average pressure height of the viscometer will change. In addition, one has to reckon with an increased scattering of the measurement values within the limit ranges between the disturbed and the undisturbed suspended level, and the influence of surface tension on the measurement result will increase. Fig. 16 shows various stages of level disturbance.

Table 4 gives an overview of the limit values of the Reynolds numbers and the flow times up to which in general no disturbances of the suspended level will occur for UBBELOHDE viscometers (normal design).

| Size no. | 0c | 0a | I | Ic |
|---|---|---|---|---|
| $t_g$ [s] | 100 | 75 | 60 | 60 |
| Re | 500 | 500 | 300 | 100 |

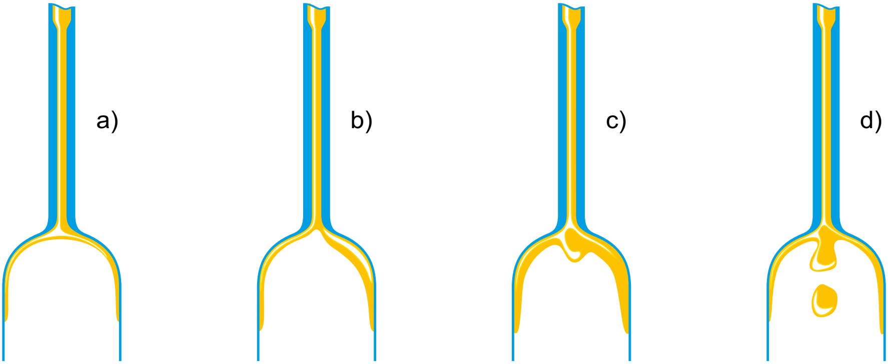
*Table 4 Limit values of $t_g$ and Re according to DIN 51562-4*

Considering further that the limits also depend on the surface tension of the liquid and the shape of the capillary outflow, disturbances of this kind may even occur in the case of somewhat longer flow times.

## Start-up length

One of the preconditions for capillary viscometry is a parabolic velocity profile. For this reason, the flow time has to be selected in such a manner that the start-length $l_e$ for the formation of the profile is considerably smaller than the capillary length. According to Schiller [10], the start-up length can be calculated as follows:

$$l_e = 0.015 \frac{V}{\pi \upsilon t_g} \tag{7.9}$$



a) no disturbance - measurement usable
b), c), d) disturbance - measurement not usable

*Fig. 16 Stages of disturbance of the suspended level with UBBELOHDE viscometers (according to DIN 51562-3)*

### Error due to delayed drainage

These errors are caused by the fact that a small liquid volume $\Delta V$ is adhering to the wall of the viscometer above the sinking liquid meniscus. $\Delta V$ will increase with the viscosity and the sinking velocity of the meniscus. The magnitude of the error is also influenced by the wettability of the wall, the surface tension of the liquid, and the geometry of the viscometer. Depending on the constructional shape of the device a shortening or extension of the flow times may occur.

### Radiation heat

To avoid an uncontrolled heating up of the liquid to be tested by heat radiation, the liquid bath is to be protected from direct exposure to the sun or light sources. Cold lights or light sources with a premounted infra-red filter should preferably be used for illumination.

### Uncertainty of timing

The relative error of measurement for the time measuring device must be less than 0.02 % and is to be included in the measurement uncertainty according to DIN 51562-4. The uncertainty of the timing device in practice is small compared to other uncertainties.

## 7.3 Trouble shooting table

Table 5 gives a summary of some of the major errors occurring during viscosity measurements using glass capillary viscometers, including their possible causes and ways of elimination. Errors which can be attributed to device defects as well as improper use of the automatic viscosity measurement devices are not considered.

| Measuring error | Cause of error | Elimination of error |
|---|---|---|
| Systematic error:<br>Flow time **too short** | Temperature of thermostatic bath liquid too high. | Check and - if necessary - adjust bath temperature |
| Systematic error:<br>Flow time **too long** | Dirt in the capillary | Discharge and clean viscometer (cf. chapter 6.2), repeat measurement |
| | Temperature of thermostatic bath liquid too low. | Check and - if necessary - adjust bath temperature |
| Systematic error:<br>Flow time **too long**<br>at short flow times | Kinetic energy correction not used or too small | For precise measurements choose viscometer with smaller capillary diameter or kinetic energy correction has to be determined experimentally with substances with similar viscosity and surface tension compared to the sample (cf. chapter 4) |
| Systematic error:<br>Flow time **too short**<br>at short flow times | Error due to flow-tailing,<br>Kinetic energy correction too high | Choose viscometer with smaller capillary diameter or kinetic energy correction has to be determined experimentally. |
| Systematic error:<br>Flow time **too short**<br>(OSTWALD-, CANNON-FENSKE-viscosimeter) | Too little sample filled into viscometer. | Refill viscometer after discharging and cleaning (cf. chapter 6.2 / 6.3). |
| Systematic error:<br>Flow time **too long**<br>(OSTWALD-, CANNON-FENSKE-viscosimeter) | Too little sample filled into viscometer. | Refill viscometer after discharging and cleaning (cf. chapter 6.2 / 6.3). |

*Table 5 Commonly occurring errors in the use of glass capillary viscometers*

| Measuring error | Cause of error | Elimination of error |
|---|---|---|
| Systematic error:<br>Flow time too short at short flow times (UBBELOHDE viscometer) | Disturbance of suspended level | Viskosimeter mit geringem Kapillardurchmesser wählen (siehe Kapitel 6.1 / 7.2) |
| Drift of flow times | Drift of bath temperature | Protect bath against radiation (cf. chapter 6.4); exchange thermostat, if necessary |
| | Thermostating of sample not finished | Prolong thermostating time until flow times are constant (cf. chapter 6.4) |
| | Evaporation of a highly volatile component | Liquid handling in viscometer by pressure mode instead of suction mode |
| Enhanced statistical scattering of flow times | Dirt in the viscometer | Discharge and clean viscometer, repeat measurement |
| | Particles in the sample | Discharge and clean viscometer, repeat measurement with filtrated sample – if required, use filter with smaller pore size. (cf. chapter 6.2/ 6.3) |
| | Air bubbles | For samples with negligible volatility and sufficient thermal resistance:<br>Casting out by short-time heating |
| | | Discharge and clean viscometer (cf. chapter 6.2)<br>At new filling, pay attention to avoid air bubbles (cf. chapter 6.3) |

*Continued Table 5  Commonly occurring errors in the use of glass capillary viscometers*

| Measuring error | Cause of error | Elimination of error |
|---|---|---|
| Very high statistical scattering of flow times – automatic measurements with light barriers,<br>up to complete malfunction. | Dirt at the optical sensors | Remouve measuring stand out of the thermostatic bath, clean sensor with fabric wetted with alcohol |
| | Malfunction of light barriers due to air bubbles, sludge or liquid lamellas | Viscometer with TC sensors instead of optical detection (cf. chapter 6.1) |
| Very high statistical scattering of flow times – automatic measurements with TC sensors,<br>up to complete malfunction. | Incrustation of TC sensors | Transparent samples: Optical detection<br>Opaque samples: Manual measurement with Reverse flow viscometers. |
| | Deterioration of sensors | Change viscometer |
| Enhanced statistical scattering at short flow times (UBBELOHDE viscometer) | Beginning disturbance of suspended level | Choose viscometer with smaller capillary diameter (cf. chapter 6.1 / 7.2) |
| Fluctuating flow times | Heating /cooling times of the thermostat too long | Improve control parameters of thermostat |
| | Thermostat defect | Improve control parameters of thermostat |
| Air bubbles are created when pumping liquid into measuring bulb of viscometer | Not enough sample in viscometer | UBBELOHDE viscometer:<br>Replenish sample<br>Alternatively: Discharge and clean viscometer, refill sample and repeat measurement |

*Continued Table 5  Commonly occurring errors in the use of glass capillary viscometers*

# CHAPTER 8
# SPECIAL APPLICATIONS

## 8.1 Testing of plastics

### Introduction

One of the major quality features of synthetic materials is the mean molecular weight of the polymer molecules. The molecular weight characterizes the chain length of the polymer molecules which has a decisive influence on the processing properties of a synthetic material as well as on the mechanical properities of the final products.

The stress exerted on the plastic by the processing process may lead to changes in the polymer (usually degradation of the chains). Under certain circumstances, the properties of the finished part might be changed to such an extent that it is no longer suitable for its intended purpose. The average molecular weight is determined by measuring the viscosity of a dilute polymer solution.

There are alternative methods for molecular weight determination (GPC, melt viscosity, osmosis, light scattering on solutions, field flow fractionation), but in the quality control of plastics, solution viscometry has established itself as the most important standard method:

It offers high accuracy (reproducibility), reliability and robustness, at relatively low cost of acquisition and operation.

There are different measuring tasks for different applications:

### Polymer chemistry (polymer manufacturing)

- *Determination of the mean chain length or mean degree of polymerization of the final product (raw granules)*

- Characterization of the final product
- Quality assurance
- Optimization of process parameters
- Preventing the production of faulty batches

## Polymer processing

- *Characterization of the properties of the starting material (raw granulate)*

- Incoming inspection
- Laying out systems for polymer processing
- Determining optimum process parameters

- *Determination of the chemical and physical properties of the finished parts*

- Final inspection
- Determination of damage to the polymer during processing: Optimization of process parameters

## Viscometry of polymer solutions

Determination of chain length, processing properties and quality of a plastic is carried out by viscosity measurements on solutions of the plastics in suitable solvents using capillary viscometers (solution viscometry). Learn about solvent, viscometer and used standards in Table 6.

The relative viscosity and the viscosity number (VN) based on it, the intrinsic viscosity (IV) or also the K value according to Fikentscher is calculated for polymer applications in general instead of the absolute viscosity of the solution.

The work flow for the viscometry of polymer solutions can be divided into three parts:

1. Sample preparation
2. Measuring the viscosity (flow time) of the sample and pure solvent (blank value)
3. Evaluation

For all three parts, the applicable standards must be considered for this polymer. This is particularly important during the sample preparation, but also in the evaluation. The measurement of the flow time is the least critical in many cases.

| Type | Abbr. | Solvent | Capillar | Operating temperature | Standard |
|---|---|---|---|---|---|
| Cellulose | C | EWN | | 20 °C | SNV 195598 |
| | I | Cuen (CED) | | 20 °C | DIN EN 60450 |
| | | Cuen (CED) | | 20 °C | ASTM D 4243 |
| | | Cuen (CED) | | 25 °C | ISO 5351 |
| | | Cuen (CED) | | 25 °C | AST; D 1795 |
| | | Cuen (CED) | | 25 °C | SCAN CM 15:99 |
| | | Cuen (CED) | | 25 °C | TAPPI T230-0M99 |
| Cellulose acetate | CA | Dimethylchloride/ methanol | 0c<br>I<br>I Micro | 25 °C | ASTM D817 |
| Polyamide | PA | Sulfuric acid (96 %) | II<br>IIc | 25 °C | ISO 307 |
| Polyamide | PA | Formic acid (90 %) | I<br>Ic | 25 °C | ISO 307 |
| Polyamide | PA | m-cresol | II<br>IIc | 25 °C | ISO 307 |
| Polybutylene terephthalate | PBT | Phenol/dichloro benzene (50 : 50) | Ic<br>II | 25 °C | ISO 1628-5 |
| Polycarbonate | PC | Dichloromethane | 0c<br>I | 25 °C | ISO 1628-4 |
| Polyethylene | PE | Decahydronaphthalene | I<br>Ic | 135 °C | ISO 1628-3<br>ASTM D 1601 |
| Polyethylene terephthalate | PET | m-cresol | II<br>IIc<br>IIc Micro | 25 °C | ISO 1628-5<br>ASTM D 4603 |
| Polyethylene terephthalate | PET | Phenol/dichloro benzene (50 : 50) | Ic<br>II | 25 °C | ISO 1628-5<br>ASTM D 4603 |
| Polyethylene terephthalat | PET | Dichloroacetic acid | II<br>IIc Micro | 25 °C | |
| Polymethyl methacrylate | PMMA | Chloroform | 0c<br>I | 25 °C | ISO 1628-6 |
| Polyproylene | PP | Decahydronaphthalene | I<br>Ic | 25 °C | ISO 1628-3 |
| Polystyrene | PS | Toluene | I<br>Ic | 25 °C | |
| Polysulfone | PSU | Chloroform | 0c | 25 °C | |
| Polyvinyl chloride | PVC | Cyclohexanone | Ic | 25 °C | ISO 1628-2<br>ASTM D 1243 |
| Styrene-acrylonitrile copolymer | SAN | Ethyl methyl ketone | 0c<br>I | 25 °C | |
| Styrene-butadiene copolymer | SB | Toluene | 0c<br>I | 25 °C | |

*Tabelle 6  Dilute solution viscometry in polymer analytics*

The solution should be always made according to the respective underlying standard. Very often inconsistencies in the end result are simply not based in the measurement or evaluation of the sample, but in the sample preparation.

The standards offer a variety of information, in particular regarding:

- the solvents
- the polymer concentration
- the dissolution temperature
- and the duration of dissolution

The exact composition should be observed for solvents. Thus, for example, according to ISO 307 96 % sulfuric acid or 90 % formic acid is used for polyamide. The water content is thus precisely defined in these two cases.

In some cases, multiple solvents are allowed (e.g., in ISO 307 for polyamide or in ISO 1628-5 for polyester). Companies or institutions that work together should therefore agree on the choice of solvent in order to guarantee the best possible comparability of results.

The polymer concentration is usually prescribed clearly in the standards. Often for the overall solution, the specification of 0.5 g polymer/100ml (= 5 g/liter ) should be found. An analytical balance with a resolution and accuracy of ± 0.1 mg is necessary for weighing these small sample amounts.

There are two procedures for the preparation of such a solution:

(a) The solution is prepared in volumetric flasks with defined volumes, for example, in 50 ml - flask. The calculated amount of polymer is weighed for this. In the example at a concentration of 0.5 g/100 ml, in 50 ml total volume these are 250.0 mg. Deviations from the calculated initial weight must be recorded, so that a corrected concentration can be calculated. The polymer is dissolved in about ¾ of the necessary amount of solvent. After dissolution, it is filled up to calibration mark of the volumetric flask with solvent (Fig.17).

It should be noted: The volume of the solution is temperature dependent. The measurement flask must therefore be filled at room temperature. This is particularly important when the sample was heated for solution beforehand - then the samples cool off to room temperature before filling.

If a magnetic stir bar was inserted for solution, it must be removed before filling. In fact, it must even be rinsed with the solvent and the rinsing solution collected in the measurement flask, to avoid losing adhering sample.

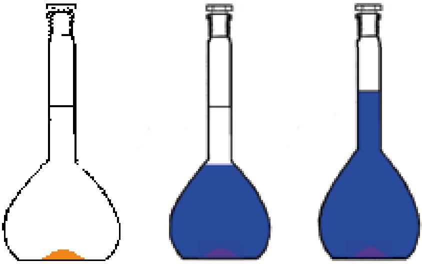


*Fig. 17  Sample preparation*

(b) The solution is manufactured in standard sample bottles using an piston burette, e.g. TITRONIC® 500 from SI Analytics (see Fig. 18).

In this procedure, the amount of sample can be weighed in a certain range, e.g., 200 to 300 mg - so an exact target value does not need to be reached. The sample weight is entered into the flask burette, which then calculates the amount of solvent and meters it to the polymer. This method is easier to use compared to method (a). In addition, it ensures a constant concentration.

Engineering plastics often contain glass fibers or other additives. These "impurities" must be taken into account for concentration calculation to obtain the pure polymer content. In order to obtain a polymer amount of 250 mg, for example, the quantity of sample weighed in a glass fiber reinforced plastic adds up to:

$$m_{\text{sample}} = \frac{250\ \text{mg}}{1 - \frac{w_{GF}(\%)}{100}}$$

*$w_{GF}$(%): count of glass fibres in %*

When preparing the solution according to method b using a flask burette, this formula is contained in the internal software to take account of the additives.

In practice, glass fibers most often occur as a foreign component. The glass fiber content is determined separately by incineration in accordance with standardized methods (usually ISO 3451).

When ashing, a plastic sample is burned in a crucible (usually of porcelain in the practice) and then annealed at temperatures of typ. 850 °C in a muffle furnace to constant weight.

*Abb. 18 Sample preparation with the piston burette TITRONIC® 500*

## 2. Measuring the flow time of the sample and pure solvent

In the viscometry of polymers, the flow time of the pure solvent (blank value) is also measured in addition to the flow time of the sample solution in almost all cases. Both results are converted into a relative viscosity $\eta_r$:

$$\eta_r = \frac{\eta}{\eta_0} = \frac{K \cdot \rho \cdot t}{K \cdot \rho_0 \cdot t_0} = \frac{t}{t_0}$$

| | |
|---|---|
| $\eta, \eta_0$ | viscosity of the sample and the solvent |
| $\rho, \rho_0$ | density of the sample and the solvent |
| $t, t_0$ | flow time of the sample and the solvent |

Since the flow time for sample and blank value is usually measured in the same viscometer, the calibration constant $K$ of the viscometer is omitted in the calculation of results.

In addition, the density of the sample and the solvent in the examined diluted solutions are approximately equal, so that the relative viscosity is simply the ratio of the flow times of sample and solvent.

Therefore uncalibrated viscometers can be used to measure the relative viscosity. In practice, a mean value is often calculated from three flow times, which is then converted into the relative viscosity. The automated (AVS®) devices from SI Analytics automatically perform repeat measurements and calculations. In most cases, the relative viscosity is converted even further into a final result (see section 3. Evaluation).

### Blank value

The blank value is determined for each viscometer, thus the transit time of the solvent. The blank value should always be the same in a given viscometer. If the blank value fluctuates with repeated measurements, this can be caused by:

- The viscometer is not clean, e.g., can still be carrying residues of a previous sample measurement in routine use. In this case, the blank value should be measured once again.

  If there are stubborn stains, the viscometer must undergo a suitable intensive cleaning (see chapter 6.2).

- The solvent has a slightly different composition, e.g., when a new bottle of solvent was begun. A change of the composition is possible, in particular with solvent mixtures such as phenol/ortho-dichlorobenzene (50/50, w/w), which is used in accordance with ISO 1628-5 for polyester (PET, PBT). With this solvent, there is also a risk that the phenol crystallizes when cooling down below room temperature and, in this way, changes the solvent composition.

The composition may change even with 96% sulfuric acid (ISO 307 for polyamide): The fluid is hygroscopic, that is, it takes on humidity while standing in air and dilutes itself in this way. The sulfuric acid must therefore always be kept tightly closed.

- When the temperature changes, the blank value also changes.

The blank value therefore gives experienced user information on whether everything is OK with the entire system: This mainly concerns the quality of the solvent, the cleanliness of the viscometer and measurement temperature. The blank value should be measured regularly, at least when opening a new bottle of solvent.

### Measurement of the sample

When measuring the sample solution, the run time is usually measured 3x as with the blank value.

According to ISO 1628-1 , the ratio of the transit time from the sample to the transit time of the blank value - thus the relative viscosity - should at least 1.2. The limit of 1.2 must be complied with in order to obtain sufficient accuracy for the difference between the measured flow times. The value 2 is recommended as the upper limit for the relative viscosity.

In practice, however, this value is exceeded in some cases, when polymers with high molecular weights are measured and the polymer concentration should not be changed because it is specified in the corresponding standards.

Samples containing particulates (e.g., glass fibers) must necessarily be filtered before filling into the viscometer. For filtering, see notes in Section 6.3.

If scattering of > 0.2% occur, it is probably because particles have entered the viscometer. In these cases, the measurement should be repeated with a filtered sample.

## 3. Evaluation

For the evaluation of the transit times, the relative viscosity is first calculated with Eq. (8.2) and other variable based upon it. They are listed in Summary Table 7.

Most important are:

- *the viscosity number* **VN**

synonymous terms:
- reduced viscosity $\eta_{red}$ or $I$
- according to DIN 1342-2:
Staudinger function $Jv$

- *Intrinsic velocity* **IV**

synonymous terms:
- abbr. $[\eta]$
- limiting viscosity number
- according to DIN 1342-2 :
Staudinger index $Jg$

In some cases, especially for PVC (according to DIN EN ISO 1628-2) the so-called

- *K value* according to Fikentscher is determined.

The evaluation and final result to be used are set out in the standards for historical reasons. Thus, as a rule, for the analysis of PVC, the K value according to Fikentscher is calculated, the intrinsic viscosity for PET and the viscosity number for polyamide.

Again, care should be taken when comparing the measurement results of different laboratories so that "apples are not compared with oranges". The type of evaluation is therefore to be documented.

| Quantity | Name |
|---|---|
| $\eta$ | Dynamic viscosity |
| $\nu = \eta / \rho$ | Kinematic viscosity |
| $\eta_{rel} = \eta / \eta_0$ | Relative viscosity |
| $(\eta - \eta_0) / \eta_0 = \eta_r - 1$ | Specific viscosity |
| $VN^* = 1/c \cdot (\eta - \eta_0) / \eta_0$ | Reduced viscosity, viscosity number |
| $\ln(\eta_{rel}) / c$ | Inherent viscosity |
| $IV^{**} = [\eta] = \lim\limits_{c \to 0} 1/c \cdot (\eta - \eta_0) / \eta_0$ | Intrinsic viscosity |
| $K = \frac{a - 1 + \sqrt{1 + a(2/c + 2 + a)}}{0.15 + 0.3 \cdot c}$<br>$a = 1.5 \cdot \log \eta_{rel}$ | $K$ value according to Fikentscher |

* *in some standards* $J_v$ *or I*
** *in some standards also* $J_g$ *or I*

*Table 7 Definition of terms in solution viscometry* [18]

## Intrinsic viscosity

The intrinsic viscosity is obtained by extrapolating the reduced viscosity (viscosity number) at concentration 0 ("infinite dilution"), s. Fig. 19.

For applications in quality control, the intrinsic viscosity measurements is determined from a single concentration (one-point measurement) and application of extrapolation formulas, e.g. the Billmeyer formula for PET (ASTM D4603) or the Martin formula for cellulose. The workload here for the determination of an IV is similar to that of a VN.

The intrinsic viscosity also has a special meaning in the scientific field: It is linked via the so-called Kuhn-Mark-Houwink relationship with the molar mass $M$:

$$[\eta] = K \cdot M^a \tag{8.3}$$

In this equation, the constants $K$ and $a$ are parameters that apply to a specific polymer, solvent, and a particular measurement temperature and enable the calculation of the molecular weight from the intrinsic viscosity.

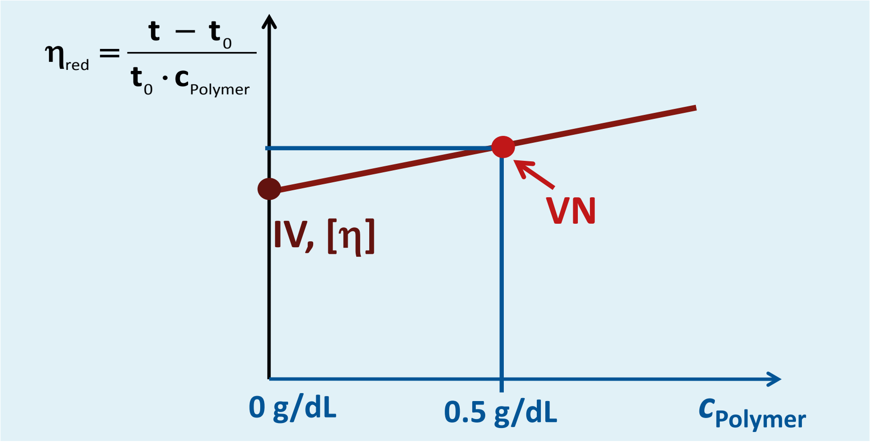


*Fig. 19 Comparison of viscosity number and intrinsic viscosity*

Since all technical polymers have a distribution of different molecular weights, a mean value is obtained - the viscosity average $M_\eta$ with the viscometric determination. The constants $K$ and $a$ can be found in the literature [16].

For accurate determination of the intrinsic viscosity, different concentrations of polymer sample solutions are manufactured (so-called dilution series [36]). The intrinsic viscosity is obtained from the extrapolation of the viscosity numbers at concentration = 0.

SI Analytics manufactures special viscometers for these series. Device software is also offered in parallel, which automatically dilutes and measures the solution in steps in these viscometers via connected burettes.

Due to the higher effort as compared to single-point measurements, the measurement of dilutions series mainly limited to research and development.

The description of the plastic analysis in this chapter is only a summary of the main points. For details regarding the individual methods - sample preparation, measurement and evaluation, please refer to the cited standards.

## 8.2 Viscosity determination of oils and additives

Mineral oils consist essentially of a mixture of hydrocarbons. They are used, among other things, as a lubricant, with additives, such as for the improvement of the viscosity index (VI), anti-wear, oxidation inhibitors, etc.

Viscosity is a decisive characteristic for the flowing and lubricating capabilities of an oil. Lubricating oils form a lubricating film between the rubbing parts in the engine which prevents direct contact of solid surfaces. The viscosity of the oil thus influences both the thickness of the lubricating film and thus the wear as well as the energy that is lost through friction.

The viscosity of a mineral oil varies greatly with the temperature:

- At low temperatures (e.g., in winter, when cold-starting the engine), it must be so low that the oil can be pumped to the lubrication points in the engine.

- At high temperatures (e.g., in summer, when fully opening the throttle; extreme loads such as driving in mountainous terrain), oil temperatures above 100 °C can occur. Sufficient lubrication film must still also be guaranteed here so that the lubricating film does not break at the friction points due to low viscosity.

The life of engine oil is limited, since in operation aging and external matter are building up on the one hand (e.g. caused by oxidation of the basic oil, metal abrasion, formation of soot), and the additives are becoming lean on the other (e.g. caused by the decay of the polymers owing to shearing action, oxidation, and thermal strain) [20, 21, 22].

The determination of viscosity plays a major role in the production and development of doped oils (basic oil / additive mixtures).

Regular viscosity measurement ensures adequate quality control in the course of production. As regards development on the other side, the focus is on the examination of the viscosity-temperature behavior of new oil/additive mixtures. In the case of used engine oils, the determination of viscosity can be used to determine whether the formation of the lubricating film will still be sufficient even at higher temperatures.

## Viscosity Index (VI)

One of the frequently used characteristics of viscosity-temperature behavior (VT behavior) of a lubricating oil is the viscosity index VI. The VI of an oil can be calculated on the basis of the viscosities at 40 °C and 100 °C by using tables (DIN ISO 2909).

The magnitude of the viscosity drop occurring with increasing temperature depends on the chemical composition of the respective oil. A minor temperature dependence of the viscosity leads to a higher viscosity index. Multi-grade, engine, and gear oils are characterized by a high VI [23].

The classification of an engine lubricating oil in so-called SAE viscosity classes is based on dynamic viscosity at -17.8 °C (0 °F) and kinematic viscosity at 98.9 °C (210 °F). Other lubricating oils should be measured at 40 °C according to ISO 3448.

Examples for viscometers and accessories from SI Analytics devices are summarized in Table 8.

| | automatic measurement | manual measurement |
|---|---|---|
| Viscometer | • UBBELOHDE<br>• CANNON Fenske routine<br>• TC-Viscometer<br>(for oils) | • UBBELOHDE<br>• CANNON Fenske routine<br>• CANNON Fenske opaque<br>(for oils) |
| Viscosity measuring system | • AVS® 370, AVS® 470<br>• AVS®Pro III<br>(up to $\nu \approx 800$ mm$^2$/s at room temperature) | • Stop watch |
| Accessories | • Thermostat und Cooler | • Thermostat und Cooler |

*Table 8 Measurement positions for viscosity measurements on oils and additives*

## 8.3 Food testing

The raw materials, semi-finished and finished products to be processed in the food industry have different rheological properties and are primarily non-Newtonian. They depend, e.g., on temperature, water content, particle size distribution (for suspensions/emulsions), mechanical processing, storage and transport conditions.

Such non-Newtonian samples are not measured with glass capillary viscometers, but with, e.g, rotational viscometers.

There are, however, liquids in food production which exhibit Newtonian flow behavior, and capillary viscometer can be used for their viscosity measurement.

### Examples of measuring tasks in the food industry

#### a) Determination of the viscosity of beer wort and beer [26]

Beers with a viscosity > 1.7 mPas are hard to filter, and this leads to a reduction of the production output. On the other hand, higher viscosity has a positive effect on richness and foam stability.

Objectives of viscosity measurement:

- optimization of the mashing properties
- selection of filtration strategy
- quality evaluation of malt, wort and beer

#### b) Determination of viscosity of fruit and vegetables juices

Raw-pressed juices with high viscosity are difficult to clarify. Viscosity is mainly affected by the pectin percentage which, in the case of concentrated fruit juices, may rise so high in the course of production that there is a danger of jellying of the contents of the tanks. Owing to the food-physiological importance, a complete decay of the pectin is not desired.

By way of an aimed pectinological decaying process in the course of the technological section of the fining and clarification process of the juices one tries to adjust an optimum pectin percentage [27].

Objectives of viscosity measurement:

- gathering of control parameters for the pectinological process of optimizing clarification and fining
- quality surveillance
- characterization of the jellying capabilities of pectins, inter alia by the determination of the limiting viscosity number [28]

### c) Viscosity determination in sugar industry

Information about viscosity is essential in the extraction and technical processing of sacchararose solutions, [29]. It increases in the form of an exponential curve with rising concentration and has thus a substantial influence on the crystallization readiness of sugar solutions. So it is that the crystallization of sacchararose solutions is favored with increasing concentration (state of over-saturation), but will decrease with the rise in the percentage of other than saccharide. Viscosity increases with the rise in the molecular mass of the solution components (mono- and disaccharides, glucose syrup [30]) and can be approximated according to a logarithmic mixing rule:

$$\eta = w_A \log\eta_A + w_B \log\eta_B \qquad (8.4)$$

w - weight fraction
A,B - components

Glucose syrups are characterized by different saccharide fractions, a fact which results in diverging viscous behavior patterns. Considering that they are used as crystallization inhibitors in the production of confectionery, viscosity is a major technological parameter.

Objectives of viscosity measurement:

- gathering of control parameters for processing sugar solutions
- quality surveillance
- development of recipes
- provision of information for the rating of appliances and apparatus for sugar industry

## d) Viscosity determination in milk industry

Owing to the differences in the provenience and composition of milk and dairy products, the rheologic behavior of dairy products differs greatly [31].

The viscosity of milk, cream, condensed milk etc. is influenced by the fat contents, the concentration of the dry matter, and, to a high degree, by the processing conditions, e.g. by homogenization. An addition of hydrocolloids (thickening, binding, and jellying agents) and stabilizers has a highly viscosity-raising effect. Viscosity measurement provides valuable information required to reveal their chemical structure and their effect in combination with components of milk.

Objectives of viscosity measurement:

- quality evaluation
- development of recipes

## e) Viscometry for special food applications

After examining the question of knowing whether the food liquid to be analyzed can be reasonably treated as a Newtonian fluid, all types of capillary viscometers can be used in principle.

There may be some difficulties in the detection of the liquid meniscus. Owing to their low degree of transparency and the after-flow effects, optical detection of dairy products is problematic.

The use of TC- Viscometers requires frequent, thorough cleaning, since the thermistors tend to soil as a result of incrustation. There are less problems in the viscosity measurement on beer, fruit juices and the like. Owing to the fact that these fluids have a tendency of foam formation, OSTWALD Viscometers and Micro OSTWALD Viscometers have proven their suitability for use [26]. Similarly good experiences have been made with viscosity measurements of fruit juices.

# APPENDIX A

## FUNDAMENTALS OF HC CORRECTION

The Hagen-Poiseuille equation (see. Chapter 2, Equation (2.1)) is not sufficient to describe the real flow in capillary viscometers.

Fig. A 1 shows the real pressure radient that occurs in the capillary [7]. The deviations from the ideal curve resulting from hydrodynamic processes in the entry and exit zones of the capillary. They are accounted for in the flow model (Fig. A 2) using additional terms.

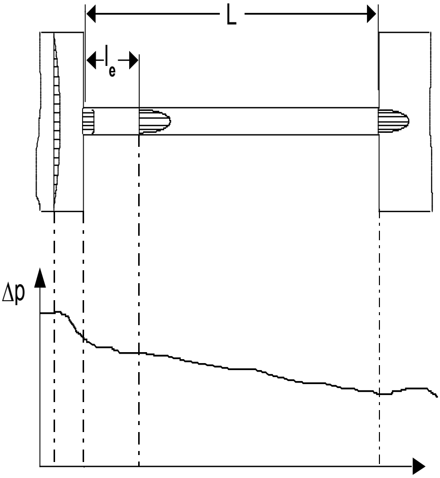
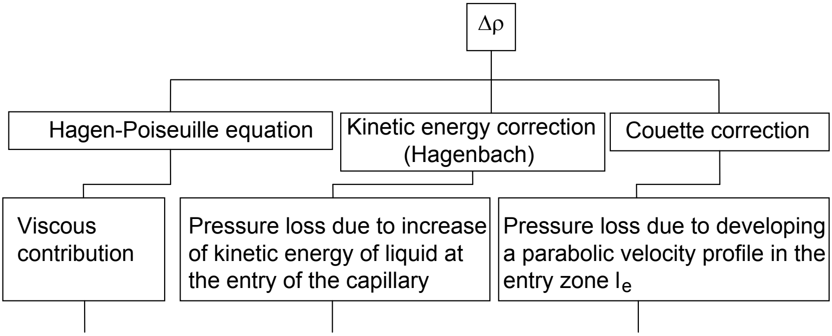


*Fig. A 1 axial pressure curve in the capillary*



$$p = \frac{8\,\eta\,\dot{V}\,L}{\pi\,R^4} + \frac{\rho\,\dot{V}^2}{\pi^2\,R^4} + \text{Pressure loss see text} \qquad \text{(A 1.1)}$$

*Fig. A 2 Flow model with correction terms*

Hagenbach [5, 14] for the first time pointed out that the kinetic energy of the formed capillary flow - for which part of the hydrostatic pressure must be applied - should be considered as a correction. This energy is not converted into heat the capillary by viscous friction, but only after the outlet.

This kinetic energy or the pressure loss can be calculated in principle (Fig. A2, Eq. A (1.1)). Since the hydrodynamics more complicated in practice, the kinetic energy correction term is expanded by a constant *m* [2, 8].

Couette described an additional pressure loss [6]: A capillary with trained parabolic flow profile has the lowest pressure loss - any other form of flow consumes more energy. Since the parabolic flow profile is formed only in the inlet zone, the pressure drop per capillary length is greater than in the subsequent capillary.

This effect was originally included by a fictitious extension of the capillary to a certain multiple *n* of the capillary radius *n·R*. The resultut is the following corrected Hagen Poiseuille equation:

$$\eta = \frac{\Delta p \pi R^4}{8\dot{V}(L+nR)} - \frac{m\rho\dot{V}}{8\pi(L+nR)} \qquad \text{(A 1.2)}$$

The values of *n* lie between 0.2 and 1.2 - funnel-shaped capillary ends have lower values than sharp-edged capillary ends. Since the Couette correction is hardly determined by measurement, it is not calculated inpractice with an extension n · R of the capillary. Instead the Hagenbach and Couette correction are summarized in the factor *m*.

With the equations

$$\Delta p = \rho g h_m \qquad \text{(A 1.3)}$$

and

$$\dot{V} = \frac{V}{t_g} \qquad \text{(A 1.4)}$$

For the kinematic viscosity can be written:

$$\nu = \frac{\pi R^4 g h_m}{8LV} t_g - \frac{mV}{8\pi L t_g} \qquad \text{(A 1.5)}$$

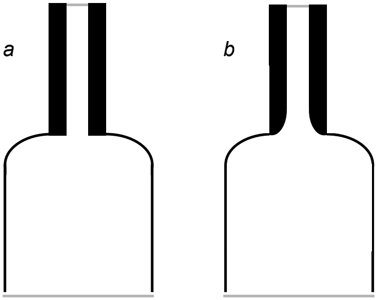
The parameter *m* is highly dependent on the shape of the capillary end and on the Reynolds number (Re).

The Reynolds number is an important dimensionless parameter for fluid mechanical descriptions of incompressible fluids:

$$\mathrm{Re} = \frac{\rho \cdot v_m \cdot 2R}{\eta} = \frac{\nu \cdot 2R}{\nu} \qquad \text{(A 1.6)}$$

$v_m$ = mean velocity

It characterizes the type of flow, laminar or turbulent, due to influences of inertia and friction (viscosity). Depending on the production technology, the capillary ends of viscometers can be sharp or funnel-shaped (Fig. A 3).



*a - sharp-edged* *b - funnel-shaped*

*Fig. A 3*
*Capillary ends of viscometers*

For sharp-edged capillary ends, a constant value of *m* = 1.12 was theoretically calculated [9, 10, 11]. This value is also specified in DIN 53012 as a maximum benchmark. Ideally, sharply cut capillary ends are not feasible for production reasons.

The calculated value has been experimentally confirmed for Re > 100. For Reynolds numbers below 100, m drops off sharply and has at Re = 25 only about 30 - 40% of its initial value [12]. At Re <10, m is negligibly small [13].

If the capillary ends are funnel-shaped, *m* is thus a function of the Reynolds number over the entire flow time range used mechanically. Cannon, Manning and Bell [14] found a proportionality between *m* and the root of Reynolds number:

$$m = 0.037\,\sqrt{\mathrm{Re}} \qquad \text{(A 1.7)}$$

Equation (4.15) represents the basis for calculating the kinetic energie correction by means of a universally applicable formula for viscometers with funnel-shaped capillary, see below. Thus, the following equations work for viscometers with sharp-edged or funnel-shaped capillary:

### sharp-edged capillary ends

$$\nu = K \cdot t - \frac{B}{t_g} \qquad \text{(A 1.8)}$$

$$B = \frac{1.12V}{8\pi L} \qquad \text{(A 1.9)}$$

$$t_H = \frac{B}{K t_g} \qquad \text{(A 1.10)}$$

### funnel-shaped capillary ends

Capillary viscometers from SI Analytics have funnel-shaped capillary ends for manufacturing reasons

For validity of the equation A (1.7) according to [14], the kinetic energie correction can be calculated as follows:

$$\nu = K \cdot t - \frac{E}{t_g^{\,2}} \qquad \text{(A 1.11)}$$

$$E = \frac{0.00166\, V^{\frac{3}{2}}}{L(2\,KR)^{\frac{1}{2}}} \qquad \text{(A 1.12)}$$

$$t_H = \frac{E}{K t_g^{\,2}} \qquad \text{(A 1.13)}$$

This calculation formula is used by SI Analytics by default both in the user instructions for the viscometer as well as in software.

The kinetic energie correction calculated according to this formula was tabulated in old versions of DIN 51562-1 for DIN Ubbelohde viscometers and is included in the still valid DIN 51562-2 of 1988 for Micro Ubbelohde viscometers.

In the current DIN 51562-1:1998 the individual determination of kinetic energie correction is recommended.

The ASTM D 446 still allows the use of the calculation formula, but also recommends an kinetic energie correction. The reason for this is that the kinetic energie correction of the individual viscometer is subject to significant model deviations, e.g., by the individual shape of the inlet and outlet funnel on the capillary ends. For this reason, the calculation formula of the kinetic energie correction should only be applied for small correction values, as indicated in the instructions of the viscometer.

## SYMBOLS AND UNITS USED

| | | |
|---|---|---|
| $A$ | Surface parallel to direction of flow | m$^2$ |
| $B$ | Constant of the kinetic energy correction with sharp-edged capillary ends | mm$^2$ s |
| $c$ | Concentration | g/cm$^3$ |
| $\dot{\gamma}$ | Shear rate | 1/s |
| $E$ | Constant of the kinetic energy correction with funnel-shaped capillary ends | mm$^2$ s |
| $F$ | Force in the direction of flow | N |
| $g$ | Acceleration of fall at the place of measurement | m/s$^2$ |
| $g_0$ | Acceleration of fall at place of determination of the viscometer constant | m/s$^2$ |
| $h_m$ | Mean hydrostatic pressure height | cm |
| $\Delta h$ | Capillary rising head of the liquid | cm |
| $IV$, $[\eta]$ | Intrinsic viscosity, limiting viscosity number | cm$^3$/g |
| $K$ | Viscometer device constant | mm$^2$/s$^2$ |
| $K_P$ | Viscometer device constant (device being tested) | mm$^2$/s$^2$ |
| $K_R$ | Viscometer device constant (reference viscometer) | mm$^2$/s$^2$ |
| $K'$ | Corrected viscometer device constant | mm$^2$/s$^2$ |
| $L$ | Length of the capillaries | cm |
| $l_e$ | Entry length of capillary | cm |

| | | |
|---|---|---|
| $m$ | Coefficient of the kinetic energy correction | - |
| $n$ | Coefficient of the Couette correction | - |
| $\Delta p$ | Acting pressure | mbar |
| $\Delta p_C$ | Pressure loss resulting from the Couette correction | mbar |
| $R$ | Radius of the capillaries | cm |
| $Re$ | Reynolds number | - |
| $r_1$ | Radius of the upper bulb on the liquid meniscus | cm |
| $r_2$ | Radius of the lower bulb on the liquid meniscus | cm |
| $s$ | Temperature error | K |
| $t$ | Flow time corrected according to Hagenbach-Couette | s |
| $t_g$ | Measured flow time | s |
| $t_{gP}$ | Measured flow time (device being tested) | s |
| $t_{gR}$ | Measured flow time (reference viscometer) | s |
| $t_H$ | Kinetic energy correction time | s |
| $t_{HP}$ | Kinetic energy correction time (device being tested) | s |
| $t_{HR}$ | Kinetic energy correction time (reference viscometer) | s |
| $t_s$ | Shear time | s |
| $T$ | Measurement temperature | K |
| $T_0$ | Calibration temperature | K |
| $U$ | Voltage | V |

| | | |
|---|---|---|
| $U_\nu$ | Temperature coefficient of kinematic viscosity | 1/K |
| $V$ | Flow through volumes | $cm^3$ |
| $\dot{V}$ | Volume flow rate | $cm^3/s$ |
| $\Delta V$ | on the inner walls of the viscometer adhering liquid volume | $cm^3$ |
| $v_m$ | Average flow velocity | m/s |
| $x$ | Coordinate flow direction | m |
| $y$ | Coordinate in the direction of the velocity gradient | m |
| $\alpha$ | Linear expansion coefficient | 1/K |
| $\varepsilon$ | Relative error of measurement value | % |
| $\eta$ | Dynamic viscosity | mPa s |
| $\eta_r$ | Relative viscosity | - |
| $\eta_0$ | Dynamic viscosity of the solvent | mPa s |
| $\nu$ | Kinematic viscosity | $mm^2/s$ |
| $\rho$ | Density of the liquid to be measured | $g/cm^3$ |
| $\rho_0$ | Density of the normal liquid or the solvent (in the polymer analysis) | $g/cm^3$ |
| $\sigma$ | Surface tension of the liquid to be measured | mN/m |
| $\sigma_0$ | Surface tension of the normal liquid | mN/m |
| $\vartheta$ | Temperature | °C |
| $\tau$ | Shearing strain | Pa |

| | | |
|---|---|---|
| $\Phi$ | Angle between the perpendicular and the line connecting the upper and lower the central point of the leveling bulbs during measurement | - |
| $\Phi_0$ | Angle between the perpendicular and the line connecting the upper and lower central point of the leveling bulbs during calibration | - |

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# STANDARDS

## Fundamentals

DIN 1342-1
Rheological concepts

DIN 1342-2
Newtonian liquids

DIN 53017
Determination of the temperature coefficient of viscosity of liquids

ASTM D 445
Standard Test Method for Kinematic Viscosity of Transparent and Opaque Liquids and Calculation of Dynamic Viscosity

## Metrology

DIN 51366
Measurement of kinematic viscosity by means of the Cannon-Fenske viscometer for opaque liquids

DIN 51562-1
Measurement of kinematic viscosity by means of the Ubbelohde viscometer

DIN 51562-2
Determination of kinematic viscosity using the Ubbelohde microviscometer

DIN 51562-3
Determination of kinematic viscosity using the Ubbelohde viscometer, viscosity relative increment at short flow times

DIN 51562-4
Measurement of kinematic viscosity by means of the Ubbelohde viscometer, Viscometer calibration and determination of the uncertainty of measurement

DIN 53012
Capillary viscosimetry of newtonian liquids
- Sources of errors and corrections

ISO 3105
Glass capillary kinematic viscometers
- Specifications and operating instructions

ASTM D 446
Standard Specifications and Operating Instructions for Glass Capillary Kinematic Viscometers

BS 188
Methods for determination of the viscosity of liquids

## Testing of mineral oils and used materials

DIN ISO 2909
Petroleum products - Calculation of viscosity index from kinematic viscosity

DIN ISO 3448
Industrial liquid lubricants - ISO viscosity classification

DIN 51563
Testing of Mineral Oils and Related Materials - Determination of Viscosity Temperature Relation - Slope m

DIN EN ISO 3104
Petroleum products - Transparent and opaque liquids - Determination of kinematic viscosity and calculation of dynamic viscosity

## Testing of plastics and cellulose

DIN EN ISO 1628-1
Determination of the viscosity of polymers in dilute solution using capillary viscometer - Part 1: General principles

DIN EN ISO 1628-2
Determination of the viscosity of polymers in dilute solution using capillary viscosimeters - Part 2: Poly(vinyl chloride) resins

DIN EN ISO 1628-3
Determination of the viscosity of polymers in dilute solution using capillary viscometers - Part 3: Polyethylenes and polypropylenes

DIN EN ISO 1628-4
Determination of the viscosity of polymers in dilute solution using capillary viscometers - Part 4:  Polycarbonat

DIN EN ISO 1628-5
Determination of the viscosity of polymers in dilute solution using capillary viscometers - Part 5: Thermoplastic polyester (TP) homopolymers and copolymers

ISO 1628-6
Bestimmung der Viskositätszahl und der Grenzviskositätszahl;
Teil 6: Methylmethacrylatpolymere

DIN EN ISO 307
Polyamides - Determination of viscosity number

EN ISO 1157
Cellulose acetate in dilute solution - Determination of viscosity number and viscosity ratio

DIN 54270-1
Testing of textiles; determination of the limit-viscosity of celluloses, principles

DIN 54270-2
Testing of textiles; determination of the limit-viscosity of celluloses, Cuen-procedure

DIN 54270-3
Testing of textiles; determination of the limit-viscosity of celluloses, $\text{EWNN}_{\text{mod(NaCl)}}$- procedure

ISO 5351
Pulps - Determination of limiting viscosity number in cupri-ethylenediamine (CED) solution

DIN EN 60450
Measurement of the average viscometric degree of polymerization of new and aged cellulosic electrically insulating materials (IEC 60450)

SNV 195598
Textilien; Prüfung auf Faserveränderung und Faserschädigung; Bestimmung der Viskositätszahl von Cellulose in EWN-Lösungsmittel

TAPPI T230 om-99
Viscosity of pulp (capillary viscometer method)

ASTM D 2857
Standard Practice for Dilute Solution Viscosity of Polymers

ASTM D 789
Standard Test Methods for Determination of Solution Viscosities of Polyamide (PA)

ASTM D 1243
Standard Test Method for Dilute Solution Viscosity of Vinyl Chloride Polymers

ASTM D 1601
Standard Test Method for Dilute Solution Viscosity of Ethylene Polymers

ASTM D 4603
Standard Test Method for Determining Inherent Viscosity of Poly (PET)

ASTM D 4243
Standard Test Method for Measurement of Average Viscometric Degree of Polymerization of New and Aged Electrical Papers and Boards

ASTM D 4878
Determination of Viscosity of Polyols

## Standards for liquid thermostats

DIN 12876-1
Electrical laboratory devices - Laboratory circulators and baths
- Part 1: Terms and classification

DIN 12876-2
Electrical laboratory devices - Laboratory circulators and baths
- Part 2: Determination of ratings of heating and refrigerated circulators

DIN 12876-3
Electrical laboratory devices - Laboratory circulators and baths
- Part 3: Determination of ratings of laboratory baths

# SI Analytics

a xylem brand

We express our core competence with our company name SI Analytics - the manufacture of analytical instruments. In addition, SI represents the main products of our company: sensors and instruments.

Arising from the history of SCHOTT® AG, SI Analytics has nearly 80 years of experience in glass technology as well as in the development of analytical instruments. As always, our products are manufactured in Mainz with high standards of innovation and quality.

As an independent company in Mainz for over 40 years, we are, as a former subsidiary of SCHOTT® AG, still connected to tradition and, like before, continue manufacturing as a Mainz glass maker.

In the future, our electrodes, titrators and capillary viscometers will also be at home everywhere know-how is required in analytical measurement technology.

Since 2011, SI Analytics has belonged to the publicly traded company Xylem Inc., headquartered in Rye Brook / NY, USA. Xylem is a leading global provider of solutions for problems relating to water.

# We are Xylem Analytics

Xylem consists of different business sectors - such as Applied Water Systems and Analytics. The following companies make up Xylem Analytics and act like SI Analytics in the chemical, pharmaceutical, biotech, food and plastics industries.

## Bellingham + Stanley

- Refractometers
- Polarimeters
- Certified Reference Materials

**www.bellinghamandstanley.com**

## ebro

- Oil Quality Measurement Devices
- Precision Thermometers
- Temperature, Pressure and Moisture Data Loggers

**www.ebro.com**

## OI Analytical

- Total Organic Carbon and Cyanide
- Phosphoric Organic & Organochlorine Pesticides
- Volatile Organic Compounds (VOC)

**www.oico.com**

More Xylem Analytics brands:

## Xylem |ˈzīləm|

1) The tissue in plants that brings water upward from the roots;
2) a leading global water technology company.

We're a global team unified in a common purpose: creating innovative solutions to meet our world's water needs. Developing new technologies that will improve the way water is used, conserved, and re-used in the future is central to our work. We move, treat, analyze, and return water to the environment, and we help people use water efficiently, in their homes, buildings, factories and farms. In more than 150 countries, we have strong, long-standing relationships with customers who know us for our powerful combination of leading product brands and applications expertise, backed by a legacy of innovation.

**For more information on how Xylem can help you, go to www.xyleminc.com**

SI Analytics
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