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Rheological test methods — Fundamentals and interlaboratory comparisons —

Part 1: Determination of the yield point

Méthodes d'essai rhéologiques — Principes fondamentaux et comparaisons interlaboratoires —

Partie 1: Détermination du seuil d'écoulement

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Foreword

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This document was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, Subcommittee SC 9, *General test methods for paints and varnishes*.

A list of all parts in the ISO 20659 series can be found on the ISO website.

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Rheological test methods — Fundamentals and interlaboratory comparisons —

Part 1:
Determination of the yield point

1 Scope

This document gives information on an interlaboratory comparison for the determination of the yield point, using rheological test methods. The yield point is the shear stress τ below which a material does not flow.

This document provides examples of fields of applications, in which important material properties are characterized with the aid of the yield point. These fields of application include:

— effectiveness of rheological additives;

— shelf life (e.g. with regard to sedimentation, separation and flocculation);

— stability of the structure at rest;

— behaviour when starting to pump;

— use in scraper systems;

— wet-film thickness;

— levelling and sagging behaviour (e.g. without brushmarks or sag formation);

— orientation of effect pigments.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3219-1, *Rheology — Part 1: Vocabulary and symbols for rotational and oscillatory rheometry*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 3219-1.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

4 Goal of the interlaboratory test

In the interlaboratory test, different possibilities for determining the yield point using the preferred methods were considered.

The samples used in the comparative testing programme consisted of different waterborne basecoats with lower yield points and dispersions with distinctly higher yield points. The samples also included the following limited cases:

— very low yield points (<1 Pa), at which the range of elastic deformation is so low that the material can also be approximately considered as a liquid at the state of rest;

— materials of which the internal structure is disintegrated only stepwise so that a transition range is occurring and a yield zone rather than a punctual yield point is determined.

Furthermore, a non-Newtonian reference sample from the the National Metrology Institute of Germany (PTB) was also included in the comparative testing programme.

Some background information on the original interlaboratory test is given in Annex A.

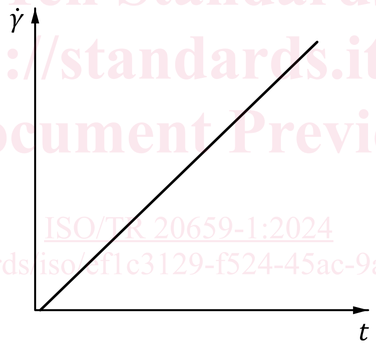
5 Metrological determination of the yield point

5.1 General

Clause 5 briefly describes all the methods in use at the time of publication. In principle, the yield point depends on the temperature, the pressure and the thermal and mechanical history of the material. A detailed specification of the measuring profile is therefore a precondition for reproducible measurements.

5.2 Shear rate-controlled rotational test

The shear rate $\dot{\gamma}$ is specified in the form of a ramp, as shown in Figure 1.



Key

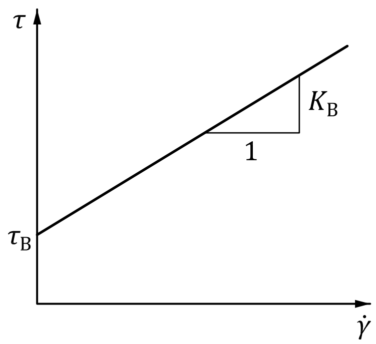
$\dot{\gamma}$ shear rate

t time

Figure 1 — Shear rate/time function as a ramp

5.3 Yield point evaluation using flow curve regression models

With a linear representation of the flow curve (usually the shear stress τ as a function of the shear rate $\dot{\gamma}$), the yield point is determined as the axis intercept on the τ axis (Figure 2).



Key

τ shear stress

τ_B Bingham yield point

K_B consistency index according to Bingham

$\dot{\gamma}$ shear rate

1 chosen shear rate range

Figure 2 — Flow curve regression according to Bingham

This yield point value depends not only on the specified ramp period, but also on the chosen shear rate range and the chosen regression model. In industrial laboratories, the models according to Bingham, Casson or Herschel/Bulkley are widely used.

The model function according to Bingham is given in Formula (1):

$$\tau = \tau_B + K_B \cdot \dot{\gamma} \quad (1)$$

where

τ is the shear stress;

τ_B is the calculated Bingham yield point;

K_B is the consistency index according to Bingham;

$\dot{\gamma}$ is the shear rate.

The model function according to Casson is given in Formula (2):

$$\sqrt{\tau} = \sqrt{\tau_C} + \sqrt{(K_C \cdot \dot{\gamma})} \quad (2)$$

where

τ is the shear stress;

τ_C is the calculated Casson yield point;

K_C is the consistency index according to Casson;

$\dot{\gamma}$ is the shear rate.

The model function according to Herschel/Bulkley is given in Formula (3):

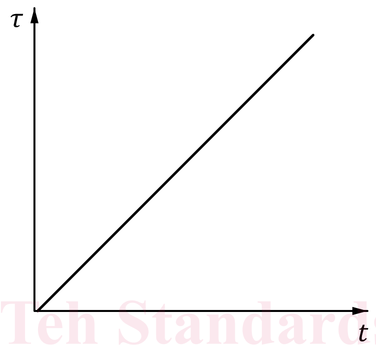
$$\tau = \tau_{HB} + K_{HB} \cdot \dot{\gamma}^p \quad (3)$$

where

τ	is the shear stress;
τ_{HB}	is the calculated yield point according to Herschel/Bulkley;
K_{HB}	is the consistency index according to Herschel/Bulkley;
$\dot{\gamma}$	is the shear rate;
p	is an exponent; if $p < 1$, the flow behaviour is shear thinning (structural viscosity, pseudoplastic), and if $p > 1$, the flow behaviour is shear thickening (dilatant).

5.4 Shear stress-controlled rotational test

The shear stress, τ, is specified in the form of a ramp, as shown in Figure 3.



Key

τ shear stress

t time

Figure 3 — Specified profile: shear stress/time function as a ramp

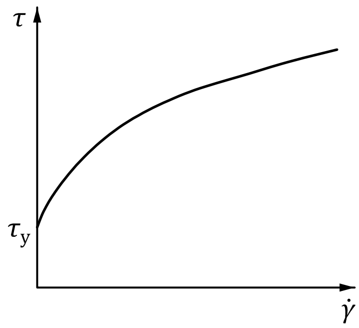
5.5 Evaluation methods for yield points

5.5.1 General

Besides the specified ramp period, the yield point value above all depends on the resolution of the rheometer for the lowest rotational speed. At shear rates of $\dot{\gamma} < 1\ \text{s}^{-1}$, time-dependent (transient) effects are expected if the measuring point duration is too short.

5.5.2 Axis intercept for presentation of the flow curve using a linear scale

This is the “classic method” of the yield point determination. In the case of the upward ramp, the yield point τ_y is determined as the last τ value at which the rheometer does not yet detect movement of the measuring system, i.e. at which $\dot{\gamma} = 0\,\text{s}^{-1}$ is still measured. By contrast, in the case of the downward ramp, the yield point is determined as the first τ value at which the rheometer no longer detects movement, i.e. at which $\dot{\gamma} = 0\,\text{s}^{-1}$ is measured (see Figure 4).



Key

τ shear stress

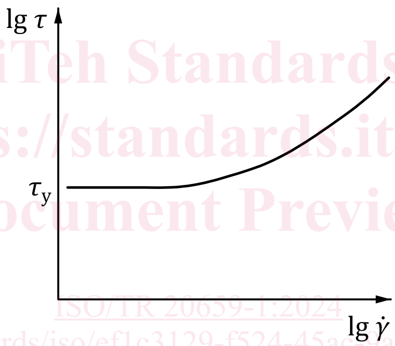
τ_y yield point

$\dot{\gamma}$ shear rate

Figure 4 — Flow curve in a linear scale with the yield point as an axis intercept on the τ axis

5.5.3 Plateau value for presentation of the flow curve using a logarithmic scale

If the flow curve approaches a plateau value in the range of low shear rates, this τ value is taken as the yield point τ_y, as shown in Figure 5.



Key

τ shear stress

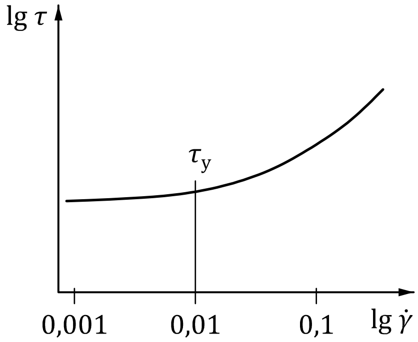
τ_y yield point

$\dot{\gamma}$ shear rate

Figure 5 — Flow curve in a double-logarithmic scale with the yield point as a plateau value of the shear stress in the range of low shear rates

5.5.4 Yield point evaluation at a reference value

The flow curve specification can take the form of a $\dot{\gamma}$ ramp or a τ ramp. The yield value is determined as shown in Figure 6 as the τ value at a shear rate previously defined by the user, e.g. $\dot{\gamma} = 0{,}01\ s^{-1}$.



Key

τ shear stress

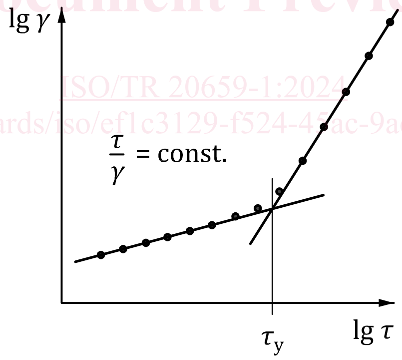
τ_y yield point

$\dot{\gamma}$ shear rate

Figure 6 — Flow curve, determination of the yield point τ_y as the τ value with $\dot{\gamma} = 0{,}01\ s^{-1}$

5.5.5 Methods with regression lines for presentation in the lg γ/lg τ diagram

If a yield point is present, then a straight line becomes visible in the range of low shear load because then the shear stress τ and the shear strain γ are proportional at low values. The measured sample then demonstrates reversible linear-elastic deformation behaviour in accordance with Hooke's law on elasticity. At higher loads the structure-at-rest disintegrates and the deformation then becomes disproportionately high, i.e. the material now demonstrates irreversible viscoelastic or viscous flow behaviour. The yield point is exceeded if the measuring points no longer lie on a straight line. If it is also possible to apply a second line through the measuring points in the flow range, i.e. when the deformation is high, the intersection point between the lines is evaluated as the yield point (see Figure 7).



Key

γ shear strain

τ shear stress

τ_y yield point

Figure 7 — Determination of the yield point using the method of the intersection point between two regression lines

If it is not easily possible to apply a second line, the regression line is only fitted in the lower range, i.e. in the linear-elastic range. The yield point is then the τ value at which the measuring curve deviates upwards from this line into the flow range (see Figure 8). If the internal structure of a material is disintegrated stepwise only so that no sharp edge but a transition range becomes visible, it is preferred to talk of a "yield transition zone" instead of a "yield point". In this case, the evaluation method shown in Figure 8 is preferred.