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Rheological test methods — Fundamentals and interlaboratory comparisons — Part 1: Determination of the yield point

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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~~/TR~~ 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 ~~guidance~~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.

~~Examples~~This document provides examples of ~~some~~ fields of ~~application~~applications, in which important material properties are characterized with the aid of the yield point~~ covered by this documents~~ . 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.

~~Some background information on the original round robin test is given in Annex A.~~

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~~https://www.iso.org/obp

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

4 Goal of the interlaboratory test

In the interlaboratory test, different possibilities for ~~the determination of~~determining the yield point using the ~~favourite~~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 ~~will be~~is determined.

Furthermore, a non-Newtonian reference sample from the the National Metrology Institute of Germany (PTB ~~Braunschweig~~) 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

~~In~~ Clause 5~~,~~ briefly describes all the methods ~~that are currently~~ in use ~~are briefly described~~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 ~~$\dot{\gamma}$~~ $\dot{\gamma}$ is specified in the form of a ramp, as shown in Figure 1.

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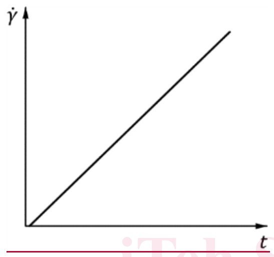

Key

~~$\dot{\gamma}$ shear rate~~
~~t time~~

$\dot{\gamma}$ shear rate
t time

~~Figure 1~~ 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 ~~$\dot{\gamma}$~~), $\dot{\gamma}$), the yield point is determined as the axis intercept on the τ axis (Figure 2).

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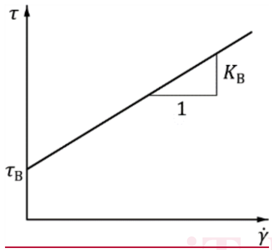

Key

~~$τ$ shear stress~~
~~$τ_B$ Bingham yield point~~
~~K_B consistency index according to Bingham~~
~~$\dot{γ}$ shear rate~~

$τ$ shear stress
$τ_B$ Bingham yield point
K_B consistency index according to Bingham
$\dot{γ}$ shear rate
1 chosen shear rate range

~~Figure 2~~ Figure 2 — **Flow curve regression according to Bingham**

~~As well as~~This yield point value depends not only on the specified ramp period, ~~this yield point value depends~~ but also on the chosen shear rate range and ~~on~~ 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):

~~$τ = τ_B + K_B \cdot \dot{γ}$~~ $$τ = τ_B + K_B \cdot \dot{γ} \quad (1)$$

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.

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):

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

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.

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):

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

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).

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