



Technical Report

ISO/TR 20659-1

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

**First edition
2024-03**

STANDARDSISO.COM : Click to view the full PDF of ISO/TR 20659-1:2024



COPYRIGHT PROTECTED DOCUMENT

© ISO 2024

All rights reserved. Unless otherwise specified, or required in the context of its implementation, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

Contents

Page

Foreword	iv
1 Scope	1
2 Normative references	1
3 Terms and definitions	1
4 Goal of the interlaboratory test	1
5 Metrological determination of the yield point	2
5.1 General	2
5.2 Shear rate-controlled rotational test	2
5.3 Yield point evaluation using flow curve regression models	2
5.4 Shear stress-controlled rotational test	4
5.5 Evaluation methods for yield points	4
5.5.1 General	4
5.5.2 Axis intercept for presentation of the flow curve using a linear scale	4
5.5.3 Plateau value for presentation of the flow curve using a logarithmic scale	5
5.5.4 Yield point evaluation at a reference value	5
5.5.5 Methods with regression lines for presentation in the $\lg \dot{\gamma} / \lg \tau$ diagram	6
5.5.6 Rotational test: viscosity maximum method	7
5.5.7 Tests with constant shear rate	8
5.5.8 Creep test	9
5.5.9 Oscillatory test: amplitude sweep	10
6 Results of the comparative testing programme	12
6.1 Performance of the tests	12
6.1.1 Preliminary tests	12
6.1.2 Comparative testing programme	12
6.2 Measuring samples	12
6.3 Method used for determination of the yield point	13
7 Result	14
8 Rheometer calibration and measurement uncertainty	15
Annex A (informative) Explanatory notes	16
Bibliography	17

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO document should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

ISO draws attention to the possibility that the implementation of this document may involve the use of (a) patent(s). ISO takes no position concerning the evidence, validity or applicability of any claimed patent rights in respect thereof. As of the date of publication of this document, ISO had not received notice of (a) patent(s) which may be required to implement this document. However, implementers are cautioned that this may not represent the latest information, which may be obtained from the patent database available at www.iso.org/patents. ISO shall not be held responsible for identifying any or all such patent rights.

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

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.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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](#).

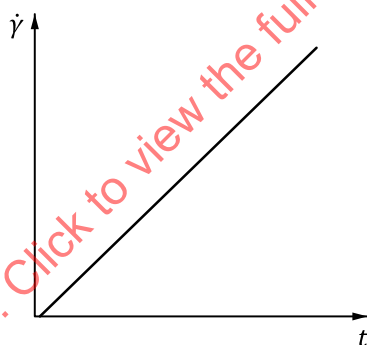
5 Metrological determination of the yield point

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](#).



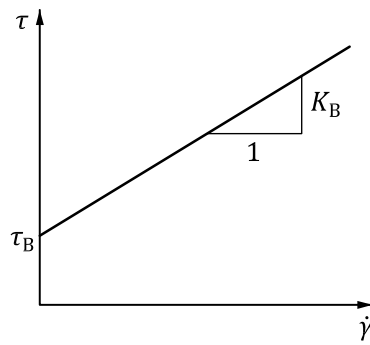
Key

$\dot{\gamma}$ 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 \(3\)](#):

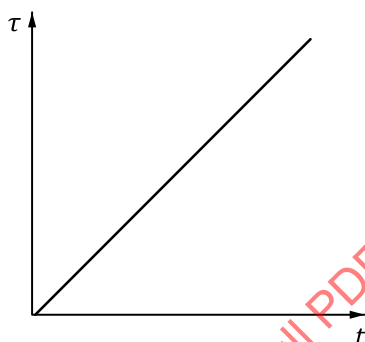
$$\tau = \tau_{HB} + K_{HB} \cdot \dot{\gamma}^p \quad (3)$$

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

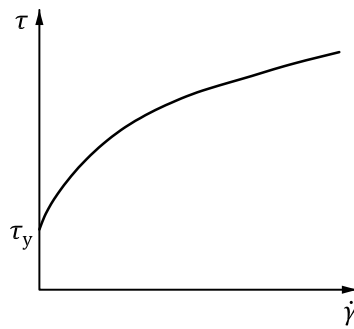
5.5 Evaluation methods for yield points

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](#)).

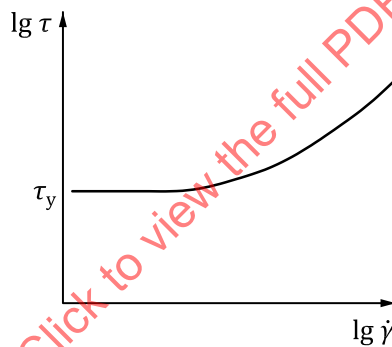
**Key**

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. $\dot{\gamma} = 0,01 \text{ s}^{-1}$.

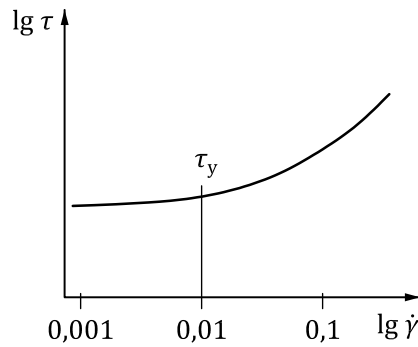
**Key** τ shear stress τ_y yield point $\dot{\gamma}$ shear rate

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

5.5.5 Methods with regression lines for presentation in the $\lg \gamma / \lg \tau$ 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](#)).

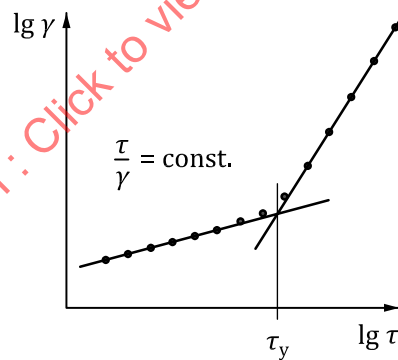
**Key** γ shear strain τ shear stress τ_y yield point

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.

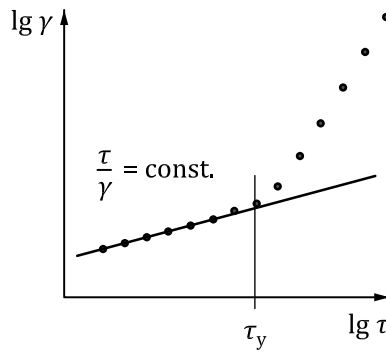
**Key** γ shear strain τ shear stress τ_y yield point

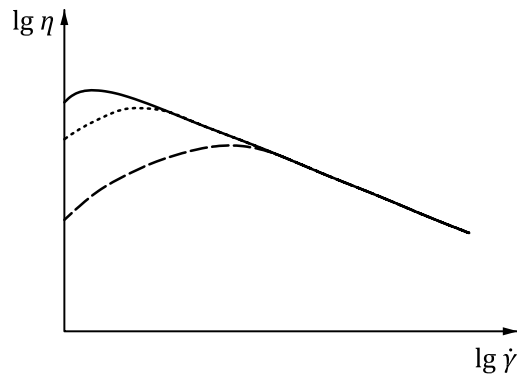
Figure 8 — Determination of the yield point τ_y using a regression line in the linear-elastic deformation range

5.5.6 Rotational test: viscosity maximum method

For rotational tests, the specification can take the form of a $\dot{\gamma}$ ramp or τ ramp. The yield point is determined at the maximum value $\eta_{\max}$ of the shear viscosity function $\eta(\tau)$ or $\eta(\dot{\gamma})$.

Due to time-dependent (transient) rheological effects, this yield point value depends strongly on the measuring point duration. The reason for this is inhomogeneous deformation behaviour and flow behaviour of the measured sample in the shear gap at very low shear rates during the transition from the state at rest to the flow state. This can be seen in [Figure 9](#), in which the results from three shear rate-controlled tests are presented. For the curve at the bottom, the shortest time for the ramp was specified. For the curve on top, the longest time for the ramp was specified. With increasing ramp duration, increasingly higher values for the relevant viscosity maximum $\eta_{\max}$ are obtained (until, at the end, it would even transition to an infinitely high initial value of the shear viscosity function at the shear rate of zero). The corresponding shear rate then shifts to increasingly lower values, as does the corresponding limiting shear stress (yield point). The yield point value, τ_y , is calculated by multiplying the viscosity maximum $\eta_{\max}$ with the shear rate $\dot{\gamma}$ according to [Formula \(4\)](#).

$$\tau_y = \eta_{\max} \cdot \dot{\gamma} \quad (4)$$

**Key** $\dot{\gamma}$ shear rate η shear viscosity

NOTE The longer the measuring point duration, the higher the resulting viscosity maxima, and the lower the corresponding shear rate value and therefore the lower the yield point value.

Figure 9 — Dependency of the yield point from the measuring point duration

5.5.7 Tests with constant shear rate

This test is mainly done with a vane rotor (see [Figure 10](#)) which is a relative measuring method for rapid routine tests, i.e. the results are relative viscosity values. The particular advantage of this test method is that when the measuring spindle (with its thin blade sheets) is immersed into the measuring sample, the structure of the measuring sample is disturbed very little during the in situ measurement.

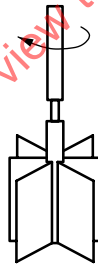
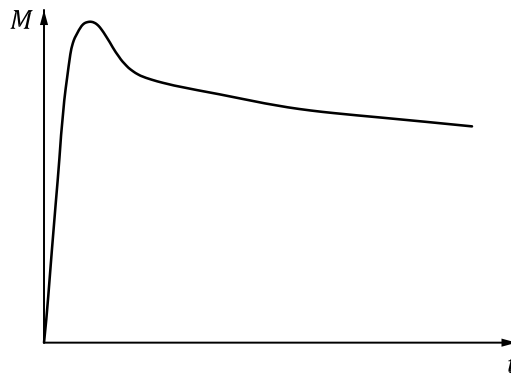


Figure 10 — Vane rotor

Two methods can be used:

- a) Specification: constant, low rotational speed n (or shear rate $\dot{\gamma}$)

Increasing strain is applied to the measuring sample. Here, the maximum torque $M_{\max}$ (or the maximum shear stress $\tau_{\max}$) of the time-dependent curve is evaluated as the relative yield point. After this, the shear stress maximum is exceeded and the structure-at-rest of the sample breaks (see [Figure 11](#)).

**Key** M torque t time

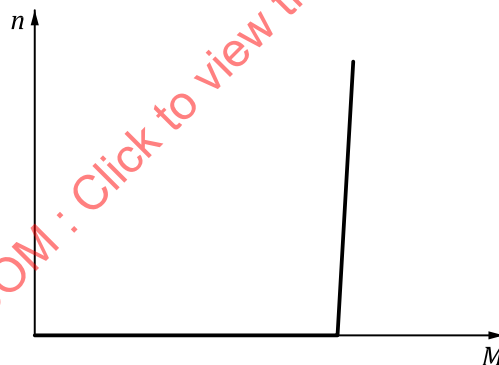
NOTE The maximum of the torque corresponds to the relative yield point.

Figure 11 — Test using a vane rotor with controlled rotational speed

By using an absolute measuring geometry, the curve is evaluated as the yield point.

b) Specification: linear increase over time of the torque M (or shear stress τ)

The structure at rest of the sample is deformed increasingly (it is “creeping”). Here, the M value (or τ value) at the start of the abrupt increase in the rotational speed curve (or shear rate curve) is evaluated as the relative yield point. The “breakaway point” is then exceeded and the sample now flows (see [Figure 12](#)).

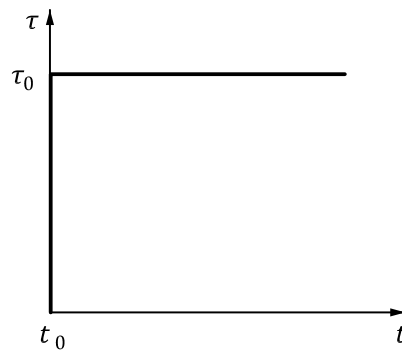
**Key** n rotational speed M torque

NOTE The relative yield point corresponds to the last torque value before the first rotational speed is detected (“breakaway point”).

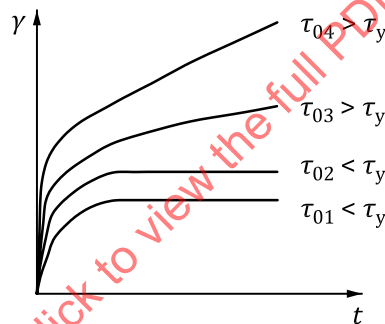
Figure 12 — Test using a vane rotor with controlled torque

5.5.8 Creep test

In the creep phase, a step to the shear stress τ_0 is specified; then the measuring sample remains exposed to this constant load (see [Figure 13](#)).

**Key** τ shear stress t time t_0 time at $\tau = 0$ **Figure 13 — Constant shear stress τ_0 as a specification for the creep test**

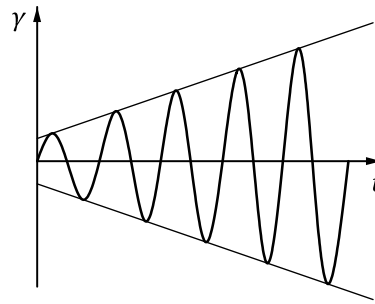
The determination of the yield point using the creep function is as follows: the yield point value is exceeded if the specified τ value leads to shear strain values that constantly increase over time, i.e. to stationary flow. Then the creep curve, i.e. the time-dependent shear strain curve $\gamma(t)$, will continue to rise steadily at the end (see [Figure 14](#)).

**Key** τ shear stress τ_y yield point t time γ shear strain

NOTE If the specified shear stress τ_0 exceeds the yield point τ_y then the $\gamma(t)$ curve will continue to rise steadily at the end.

Figure 14 — Creep curves for four different loads**5.5.9 Oscillatory test: amplitude sweep**

In the amplitude sweep, the magnitude of the oscillation amplitude varies while the oscillation frequency remains constant (see [Figure 15](#)).

**Key**

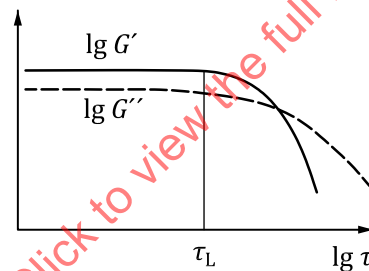
γ shear strain
 t time

Figure 15 — Specification for an amplitude sweep (with controlled shear strain)

For gel-like structures, determination of the linearity limit τ_L of the shear stress as the limit of the linear-viscoelastic range is the prerequisite for the existence of a frequency depending relative yield point.

Several methods for the evaluation of amplitude sweeps can be used:

- a) Linearity limit as τ_L value at the limit of the linear-viscoelastic range: the reversible elastic deformation behaviour ends when this limit is exceeded. At higher loads, the curves of G' and G'' will drop (see [Figure 16](#)). Alternatively, the value of the loss angle δ or of the loss factor $\tan \delta = G''/G'$ is considered. Above the limit of the linear viscoelastic range, the curves of these two parameters are rising.

**Key**

G' shear storage modulus
 G'' shear loss modulus
 τ shear stress
 τ_L linearity limit

Figure 16 — Result of the amplitude sweep: linearity limit τ_L as the shear stress limit at the boundary of the linear-viscoelastic range

- b) Regression lines can be fitted to all curves mentioned – one in the linear-viscoelastic range and one outside of it. Here, the yield point is the τ_y value at the intersection point between the lines.
- c) An alternative representation is the $\lg \gamma_A / \lg \tau_A$ diagram for yield point analysis with the aid of the regression lines. See [Figure 7](#) and [Figure 8](#) (however, the oscillatory test is shown with the shear strain amplitude γ_A and the shear stress amplitude τ_A).
- d) Shear stress value at the intersection point $G' = G''$, or with $\tan \delta = 1$. Then, in the viscoelastic behaviour of the sample, the liquid character will be predominant.

NOTE The linear-viscoelastic range with reversible elastic deformation behaviour has already been exceeded at this point.

6 Results of the comparative testing programme

6.1 Performance of the tests

6.1.1 Preliminary tests

The first comparative preliminary tests carried out by the working group showed the following results.

The differing measuring results of the preliminary tests can be due to the following reasons:

- Different preparation of samples or measurement with or without pre-application of shear.
- Samples stressed by shearing appeared not yet recovered after a period of rest. For this reason, the samples were taken and measured after periods of rest of 2 min to 10 min without pre-application of shear.
- The evaluation of the tests was carried out following the reference value method (see 5.5.4), via flow curve regression models and, additionally, via the intersection point between two regression lines (in the diagram shear strain $\lg \gamma$ over shear stress $\lg \tau$). When using automatic evaluation methods, the limits of commercially available software were sometimes reached when the operator could not influence given evaluation routines (yield point calculation by the intersection point method). Therefore, the members of the working group elaborated a further evaluation method. Also, the determination of the yield point from the value of the maximum viscosity was rejected and considered as unsuitable.

6.1.2 Comparative testing programme

Based on the experiences from the results of the preliminary tests, the following procedure for performance of the tests was agreed in January 2002.

These tests were only carried out with the test type shear stress ramp. The samples were prepared in such a way that one decade each was available for the evaluation below and above the assumed yield point, i.e. the shear stress ramp started at least one decade below the assumed yield point and ended at least one decade above the yield point value.

When using this method, a range from $\tau_{\min} = 0,1 \text{ Pa}$ to $\tau_{\max} = 100 \text{ Pa}$ can be covered. Specific details of the procedure are described as follows.

- The measurements were all carried out within a period of 2 weeks.
- Measurement geometry: cone-plate with a diameter of 50 mm to 60 mm and cone angles from 1° to 2° (see ISO 3219-2). The exact geometry was recorded in each case.
- Preparation of samples: fill the sample and then allow 5 min for temperature adjustment at 23°C without application of shear. Afterwards, the measurement was started straight away.
- Measurement: shear stress ramp from 0,1 Pa (or less) to 100 Pa within 5 min with a logarithmic increase. Here, 100 measuring points (equidistant in the $\lg$ scale) were recorded. The test was stopped when a shear rate of 50 s^{-1} was exceeded (criterion for stopping $\dot{\gamma} = 50 \text{ s}^{-1}$).

6.2 Measuring samples

Five samples were examined:

- Sample A and sample B: waterborne basecoats;
- Sample C and sample D: dispersions;
- Sample E: non-Newtonian reference sample.

Sample A was found to be unsuitable for the specified measuring method and for the requirements for performance of the tests. This sample showed very low viscosity with the yield point in a measuring range which could not be covered reasonably when using the agreed measuring method.

The measuring results returned for sample B and sample C were in relatively good agreement.

Sample D had a high solid matter content, which led to long standing times. As a result, this sample appears to be not well suited to the applied measuring methods and procedures.

The rheology of samples C and D is complicated. With sample D in particular, there can be different interpretations as to which measured values within the measuring result (shear stress ramp) are considered the definite yield point. In addition, this sample was at the upper end of the range covered by the specified procedure so that only insufficient quantification was possible.

6.3 Method used for determination of the yield point

The evaluation of the measuring curves was performed following the method with regression lines introduced in 5.5, with representation in the $\lg \gamma / \lg \tau$ diagram (see Figure 17).

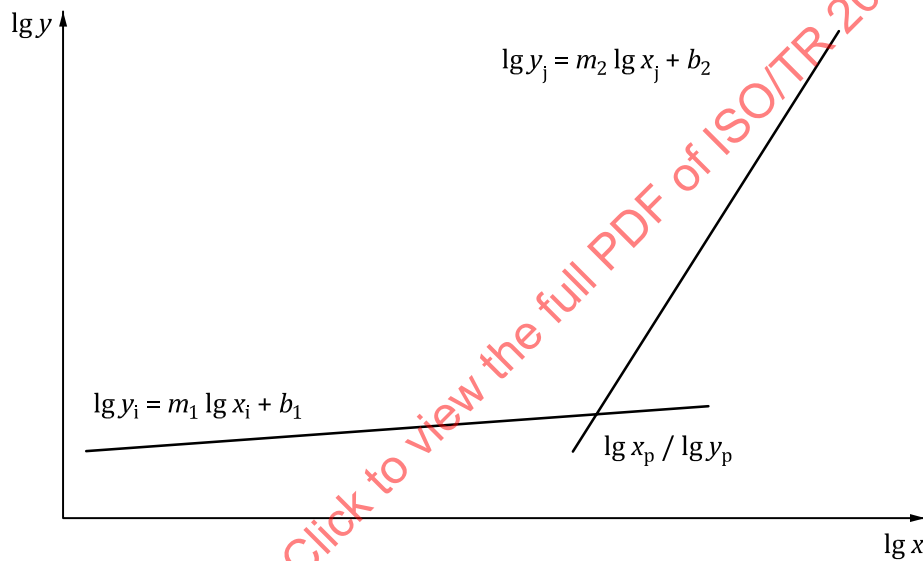


Figure 17 — Diagram for calculation of the intersection point

The intersection point can be calculated using the algorithm $\lg x_p / \lg y_p$.

Starting from the number of all measuring points h , Formulae (5) to (8) apply to the first regression line (with the measuring points 1 to k and the number of points k):

$$m_1 = \frac{\sum_{i=1}^k \lg x_i \sum_{i=1}^k \lg y_i - \frac{1}{k} \sum_{i=1}^k \lg x_i \sum_{i=1}^k \lg y_i}{\sum_{i=1}^k (\lg x_i)^2 - \frac{1}{k} \sum_{i=1}^k \lg x_i \sum_{i=1}^k \lg x_i} \quad (5)$$

$$b_1 = \frac{1}{k} \sum_{i=1}^k \lg y_i - \frac{m_1}{k} \sum_{i=1}^k \lg x_i \quad (6)$$

and for the second regression line (with the measuring points g to h and the number of points f):

$$m_2 = \frac{\sum_{g=1}^h \lg y_g \sum_{g=1}^h \lg y_g - \frac{1}{f} \sum_{g=1}^h \lg x_g \sum_{g=1}^h \lg y_g}{\sum_{g=1}^h (\lg x_g)^2 - \frac{1}{f} \sum_{g=1}^h \lg x_g \sum_{g=1}^h \lg x_g} \quad (7)$$