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Rheological test methods — Fundamentals and interlaboratory comparisons — Part 2: Determination of the time-dependent structural change (thixotropy)

~~DTR stage~~

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Contents

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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.

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.~~

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Rheological test methods — Fundamentals and interlaboratory comparisons — Part 2: Determination of the time-dependent structural change (thixotropy)

1 Scope

This document gives ~~guidance~~information on an interlaboratory comparison for the determination of the time-dependent structural change (thixotropy) using rheological test methods. –Thixotropy is the reversible, time-dependent decrease of shear viscosity ~~η~~η at a constant shear rate ~~$\dot{\gamma}$~~ $\dot{\gamma}$ or shear stress ~~τ~~τ.

This document provides examples of ~~some~~ fields of application, in which important material properties can be characterized by the thixotropy. These fields of application include:

— effectiveness of rheological additives and thixotropic agents, respectively;

— stability of the structure at rest (e.g. behaviour when starting to pump);

— wet film thickness after processing;

— 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 B.~~

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, the latest 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

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

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4 Measuring technique for the determination of thixotropy

4.1 Conditions for the measuring technique

~~In~~ Clause ~~-~~ 4~~,~~, briefly describes methods that are currently in use ~~are briefly described~~. In principle, the thixotropy 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 and comparable evaluation; this applies especially for the level of shear load (shear rates, shear stresses, shear strain, oscillation frequencies), the duration of the individual measuring segments and the number of measuring points.

Thixotropy can be determined by rotational as well as by oscillatory tests. Measuring devices equipped with a mechanical bearing or air bearing are suitable for rotational tests. For oscillatory tests a rheometer with air bearing is used. It is essential to ensure that the measuring device is used in combination with the suitable measuring geometry, i.e. in accordance with the torque range, the torque resolution and the rotational speed range. Typically, rotational viscometers and rheometers that are subject to test equipment monitoring and are regularly calibrated and verified~~, ~~ (if necessary~~,~~), are used. Measuring results that are independent of geometry can only obtained by using absolute measurement geometries ~~in accordance with~~according to ISO 3219~~-~~-2.

If, independently of the measuring device and the measuring method used, no time-stable measuring results are obtained during the measurement of Newtonian standard samples, the measuring device, the measuring geometry or the measuring method is unsuitable. If the functional course of these time-stable measured values meets the reference values within the used measuring range, it is guaranteed that the measuring device, the measuring geometry and the measuring method are suitable for the investigation of the sample. Typically, this inspection is carried out under isothermal conditions in the expected viscosity range of the sample by using several Newtonian standard samples. If preliminary tests reveal that the viscosity of the measuring sample varies over three decades, then the verification of the measuring device and measuring geometry are performed with three Newtonian standard samples. This is carried out for all measuring temperatures. The influences due to application, e.g. sample filling, evaporation, shear heating, wrong choice of method~~,~~ and the sample material coming out of the gap ~~etc.~~, are mostly discernible and detected by this kind of verification.

~~At measurements~~Upon measurement, the possibility of evaporation is considered ~~in each case~~. A reduction of this influence can be reached e.g. by using a sample area coverage. All boundary conditions of the measurement are documented in the record, especially the usage of a coverage, the kind of sample trimming~~,~~ and the adjustment of the gap distance ~~and others~~. According to the specifications of the ~~described~~ measuring methods ~~below~~described in Clause 4, the methods are changed if influences on the measuring results occur. Another parameter ~~that~~ is ~~checked is that~~checking whether the duration of the load is shorter than the medium time scale of the structural changes of the sample. This is determined for each measuring method and its specifications by preliminary tests. ~~In addition, it is considered, however, that~~However, the duration of the load is selected ~~with respect to the practice to consider later~~in consideration of situations where conditions of application of the sample~~. Is~~ are later. If this duration is longer than the time scale of structural change of the sample, then a thixotropic behaviour will not be detected; nevertheless, the sample can be still thixotropic. In order to determine the thixotropy in a correct and reproducible manner, when filling the measuring geometry, it is considered that the time between filling and the start of the measurement has an influence on the measuring result. This time is distinctly longer than the time scale of the structural change. This can be determined by preliminary measurements in which the waiting time is varied. The waiting time is sufficiently high if the thixotropy of the sample is comparable for two measurements running each after a fresh filling within the limits of the requested precision in accordance with the measuring method chosen.

Measuring points can only be recorded if each single measuring point is controlled by the instrument according to the specification. At every change of the specified value, a transient process of the entire

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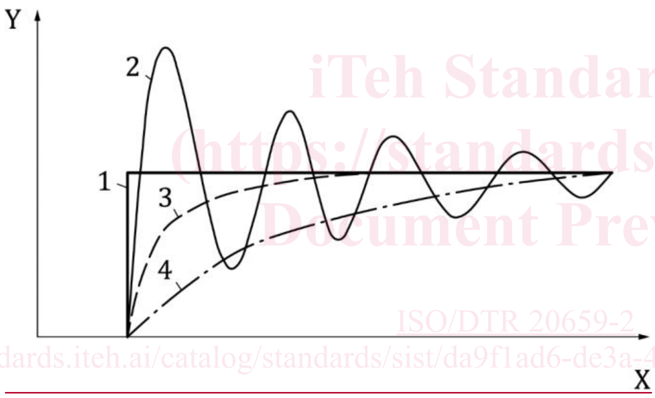
measuring equipment occurs towards the new specified value. This transient process can be different (see Figure 1).



Key

~~X time, *t*~~
~~Y shear stress, τ, or shear rate, $\dot{\gamma}$~~
~~1 specified function~~
~~2, 3, 4 different adjustment behaviours~~

X time, *t*
Y shear stress, τ, or shear rate, $\dot{\gamma}$
1 specified function
2, 3, 4 different adjustment behaviours

~~**Figure 1**~~ **Figure 1 — Different transient behaviour during the controlling of each individual measuring point**

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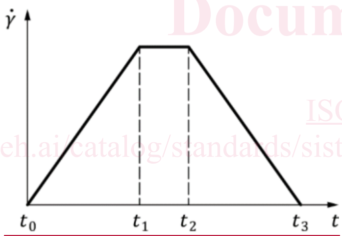
~~It is waited for the~~The measuring point ~~detection~~is not detected until the deviation between the specified value and the desired value is small enough. The integration time (time per measuring point minus adjustment time), which is considered for calculating the average of a data point, influences the measuring result. This condition is valid for constant shear load as well as for a time-dependent change of the load.

4.2 Flow curves, with evaluation of the hysteresis area (rotational test)

4.2.1 Specification of the measuring profile~~:~~

The specification is provided in the form of three measuring segments comprising a continuous or a step-like discontinuous upward ramp, a holding time and a downward ramp (~~Figure~~see Figures 2 and ~~Figure~~ 3). The shear rate ~~$\dot{\gamma}$~~ $\dot{\gamma}$ is specified as a function of time. Both ramp types can be used with linear and ~~with~~ logarithmic distribution. This is valid for the shear rate and for the time duration of the measuring points. At the beginning, this can also be preceded by a segment with defined pre-shear and/or a waiting time without shear (e.g. 5 min).



Key

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

$\dot{\gamma}$ shear rate

t time

~~**Figure 2**~~ **Figure 2 — Specified profile: shear rate/time function with the three measuring segments continuous upward ramp, holding time and continuous downward ramp**

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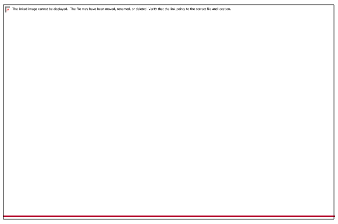

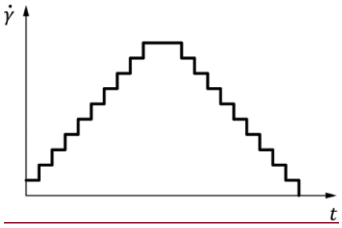

Key

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

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

~~**Figure 3**~~ **Figure 3 — Specified profile: shear rate/time function with the three measuring segments stepped upward ramp, holding time and stepped downward ramp**

Proposals for a typical measuring programme are:

Method A, with a linear ramp for the shear rate:

~~1)~~ 1) upward ramp with ~~$\dot{\gamma}$~~ $\dot{\gamma}$ = 0 s^{-1} to 1 000 s^{-1}, duration 3 min, with 45 measuring points;

~~2)~~ 2) holding time with ~~$\dot{\gamma}$~~ $\dot{\gamma}$ = 1 000 s^{-1} = constant, duration 1 min, with 15 measuring points;

~~3)~~ 3) downward ramp with ~~$\dot{\gamma}$~~ $\dot{\gamma}$ = 1 000 s^{-1} to 0 s^{-1}, duration 3 min, with 45 measuring points.

The period of time for the up- and down-ramps is the same. Moreover, it is defined whether the test is carried out with continuous or step-like ramp.

Method B, with a logarithmic ramp for the shear rate:

~~1)~~ 4) upward ramp with ~~$\dot{\gamma}$~~ $\dot{\gamma}$ = 0,1 s^{-1} to 1 000 s^{-1}, duration 3 min, with 61 measuring points. If a viscometer with a mechanical bearing is used, then 10 s^{-1} can be used as the minimum shear rate;

~~2)~~ 5) holding time with ~~$\dot{\gamma}$~~ $\dot{\gamma}$ = 1 000 s^{-1} = constant, duration 1 min, with 15 measuring points;

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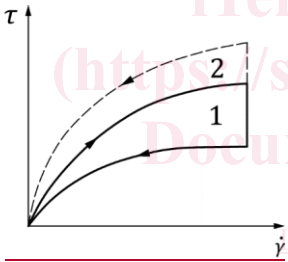
3) 6) downward ramp with $\dot{\gamma}$ $\dot{\gamma}$ = 1 000 s^{-1} to 0,1 s^{-1}, duration 3 min, with 61 measuring points.

The period of time for the up- and down-ramps is the same. Moreover, it is defined whether the test is carried out with continuous or step-like ramp.

4.2.2 Evaluation:

If the measuring sample displays behaviour that varies with shear load and time, a so-called hysteresis area is generated between the upward and downward flow curves. Here, hysteresis means curve loop. Flow curves are usually presented as shear stress τ τ (in Pa) against shear rate $\dot{\gamma}$ $\dot{\gamma}$ (in s^{-1}) (see Figure 4).



Key

τ shear stress
$\dot{\gamma}$ shear rate
1 hysteresis area with reduction of structural strength under shear load
2 hysteresis area with increase in structural strength under shear load

τ shear stress

$\dot{\gamma}$ shear rate

1 hysteresis area with reduction of structural strength under shear load
2 hysteresis area with increase in structural strength under shear load

Figure 4 **Figure 4 — Measuring result: flow curves with hysteresis area**

When the shear rate increases from zero to a maximum value and then decreases to zero following a defined time programme, the hysteresis curve is generated from the two resulting flow curves, which do not overlap.

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