Modern rheological test methods: Fundamentals and comparative testing programme for the determination of the yield point

**Report of the NPF/NAB-AK 21.1 "Rheology"
DIN Standards Committee "Pigments and extenders"**

SUMMARY

For characterising yield points of different products definitely, detailed test specifications have to be elaborated for each class of substances. It is not possible in each case to cover a wide rheological measuring range by one generalised measuring specification.

In the DIN Technical Report 143 the authors describe a method for the determination of the yield point of paints and dispersions. As one result it was found that the reference substance of the Physikalisch - Technische Bundesanstalt, Braunschweig, Germany (PTB) which is available in the meantime could be measured by all laboratories with very good reproducibility. The results of the determination of the yield point published in the DIN Technical Report 143 are based on comparative tests carried out by members of the Working Panel "Rheology" (NPF/NAB-AK 21.1) within the Standards Committee "Pigments and extenders".

In first preliminary tests, already in 2002, different waterborne Basecoats have been examined. It it was found that some methods showed unexpectedly good qualitative relationship. On the other hand, problems with the preparation of the samples were reported. Furthermore, automatic methods for the expression of results have shown their limits. In addition, certain methods of measurement have been found to be unsuitable for the samples to be examined. Also evaluations on the basis of traditional regression (fitting) methods have not been considered for further tests.

In the comparative testing programme the method "stress ramp" has exclusively been used. Below and above the assumed yield point one decade for the evaluation should be available, i.e. the shear stress ramp should begin at least one decade below the assumed yield point and should reach at least one decade beyond the yield point value. Exact test conditions have been definitely specified for all participants of the comparative testing programme (Round Robin Test). Two waterborne Basecoats, two dispersions and one sample of the PTB have been examined. With the waterborne Basecoats one sample could not be characterised unambiguously. In contrast to this, the results for the second water-based paint sample agreed well. The rheological behaviour of the two dispersions was very complex. Nevertheless, good results have been obtained.

As to the checking of the rheometer and the agreement of specifications for the measurement between different laboratories the reference liquid developed by the PTB was helpful. In the DIN Technical Report 143 all general known fundamentals for the determination of the yield point will be presented and discussed. The detailed conditions are given in DIN Technical Report 143.

RESULTS OF THE COMPARATIVE TESTING PROGRAMME

When using automatic evaluation methods, the limits of commercially available software have been reached. Therefore a further method has been elaborated by the Working Panel members. The measuring curves have been evaluated using the following tangent method to be presented in a double - logarithmic diagram. The calculation of the tangent crossover point is used to determine the yield point. For further details and derivation see the DIN Technical Report 143.

RESULT

Generally it can be said that the determination of the yield point was possible for all samples. Under the specified conditions a yield point could be indicated for each sample using the tangent crossover method proved in the comparative testing programme.

CALIBRATION OF A RHEOMETER AND MEASURING UNCERTAINTY

The calibration of a rheometer comprises the parameters being characteristic for the instrument, but provides no indication regarding the measuring deviations caused by the use of the instrument. For this, comparative testing programmes, as that described here, are indispensable, in order to obtain quantitative and practice-

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relevant information on these additional measuring deviations, caused by the use of the instrument.

The standard sample of the PTB used in this comparative testing programme is a non-Newtonian reference liquid with a well-defined yield point. These reference values and the affiliated values of the measuring uncertainty can be used to determine systematic deviations of a certain measurement procedure or of an evaluation method.

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