Testing a Viscoelastic PDMS Standard in Oscillation

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Introduction

For the user of a rheometer, running a test on a liquid with a certified viscosity is the easiest and most cost effective method to verify, whether the viscosity values measured with the instrument are reliable. Usually these viscosity standards are Newtonian or mostly Newtonian liquids, which show no or almost no elasticity. With the increasing use of rheological oscillation methods, the demand for viscoelastic standard materials has risen over the last years.
Thermo Fisher Scientific supplies a viscoelastic PDMS standard, which has been thoroughly characterized and is long time stable in its rheological properties. The accompanying certificate lists the values for the cross over frequency and the cross over modulus determined at 20 °C. As long as these values can be reproduced within the range of uncertainty given on the certificate, the instrument and the handling procedure can be trusted to give correct results.

Preparations

The results of this test are intended to be compared with the parameters printed on the certificate or in other words, with an external reference. Therefore the rheometer has to be prepared not only to give good data, which is consistent in itself but to give the absolute correct values of the test results. This preparation includes the use of the correct A- and M-factor for the test geometry used and the verification of the real sample temperature [1].
Especially when testing viscoelastic samples of higher viscosity, the handling of the sample prior to sample loading and the sample loading procedure itself have to be done with care for example to avoid the inclusion of air bubbles and to achieve a correct filling of the geometry[2].
Finally, the time needed for the sample to relax any mechanical stress from the sample loading and to achieve temperature equilibrium should be included into the test procedure to ensure that it is not shortened or forgotten.

The Test Method

For the tests done for this report, a plate with 35 mm diameter has been used. The gap was set to 1 mm. To reduce the mechanical stress on the sample, the lift was set to a lower speed when closing the gap. The Trimming Position was used to optimize the sample filling. Then within the test method the axial force on the sample was checked and the test only continued after the axial force fell below a certain value (Fig. 1). The combination of these three functions ensures the sample to always be in the same condition afterwards.

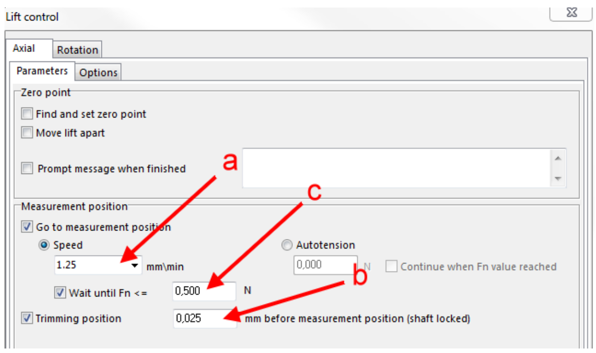

Fig. 1: Several features of the Thermo Fisher Scientific HAAKE Rheowin software have been used to ensure that after sample loading every sample was in the same condition: reduced lift speed (a), trimming position (b) and waiting for axial relaxation (c).

To characterize the viscoelastic standard, first an amplitude sweep has been run in controlled deformation (CD) mode. The range of deformation has been chosen to cover the linear viscoelastic range (LVR) and the onset of the non-linear behaviour (Fig. 2 left). The CD-mode has been chosen to be able to control the range of deformation under different circumstances like running the test with different temperatures. For the data acquisition an increased number of waiting periods and measuring repetitions have been chosen to improve the data quality. With 3 waiting periods and 7 repetitions (Fig. 2 right) one data point will take about 10 s.
The result of such an amplitude sweep looks like the graph shown in Figure 3. At 20 °C the viscoelastic standard shows a linear behaviour up to a deformation (γ) of approximately 0.1. The Phase angle (δ) in the LVR is around 60 °.

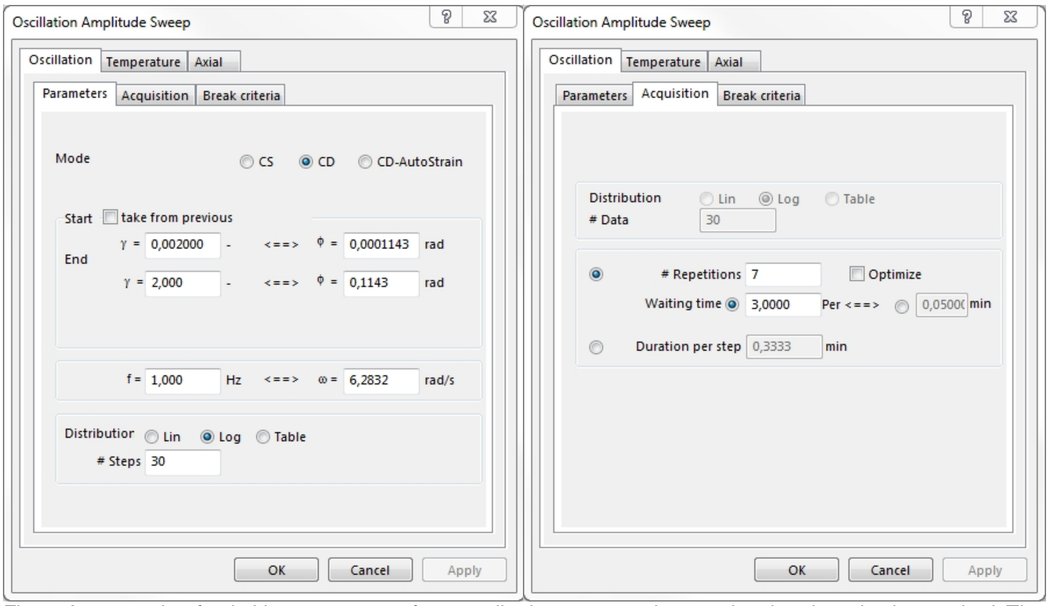

Fig. 2: An example of suitable parameters of an amplitude sweep to characterize the viscoelastic standard. The test runs in CD-mode over 3 orders of magnitude in deformation (left) and uses some more repetitions per data point to improve data quality (right).

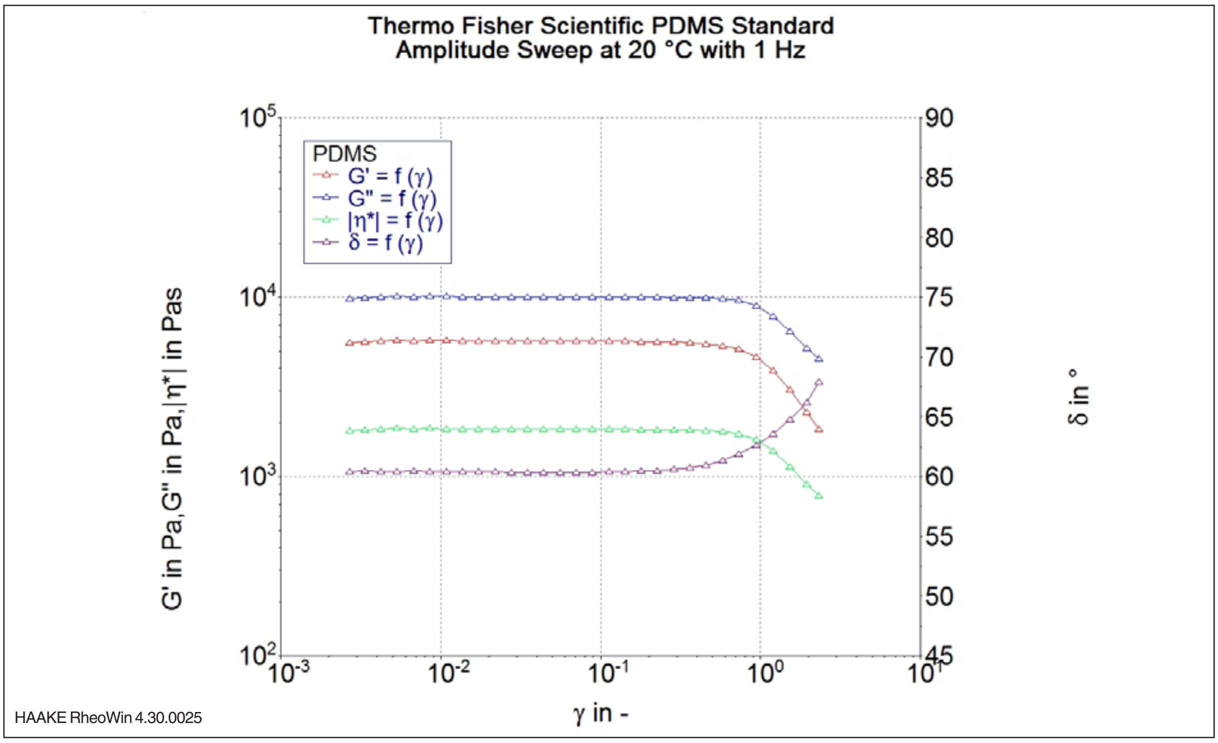

Fig. 3: Strain dependency of the rheological properties of the viscoelastic standard material. The linear viscoelastic range is clearly distinguished from the non-linear behaviour above a deformation of 0.01.

Based on the results of the amplitude sweep a deformation of γ = 0.01 has been selected for the frequency sweep (Fig. 4 left). For this report a wide range of frequencies has been used starting at 80 Hz (500 rad/s) and going down to 0.001 Hz (0.006 rad/s). To improve the data quality while not increasing the time necessary to run this test too much, the test method has been split into 3 parts with different parameters for waiting periods and repetitions. For frequencies down to 1 Hz 10 waiting periods and 25 repetitions (10|25) have been used like shown in the right part of Figure 4. Down to 0.1 Hz is was still 6|9 and below 0.1 Hz 1|3 have been used.

With these parameters the frequency dependency of the material's properties has been recorded over 5 orders of magnitude as shown in Fig. 5. Since such a test runs almost 5:35 h including sample loading and equilibration, this method is not suitable for a quick verification of the instrument's condition. If the test is reduced to the frequency range from 16 Hz (100 rad/s) down to 1.6 Hz (10 rad/s) the crossover can still be determined accurately and the whole test will take less than 15 min, again including sample preparation and equilibration.

At the end of the test the RheoWin software can determine the crossover automatically, compare the crossover frequency and the crossover modulus with the certificate values and document whether the test was successful or not. The crossover frequency should be around 5.1 Hz (32 rad/s) and the crossover modulus should be around 22000 Pa. The exact values always have to be taken from the certificate of the standard material currently in use.

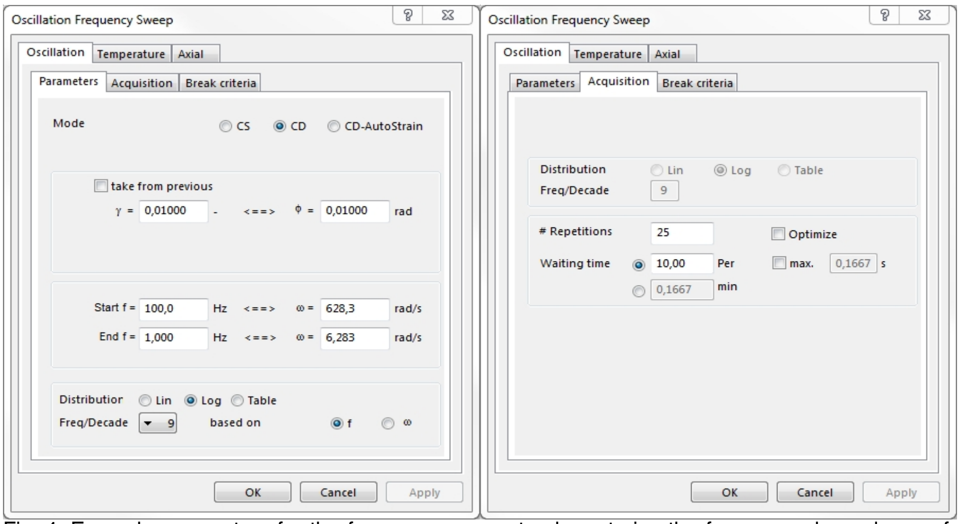

Fig. 4: Example parameters for the frequency sweep to characterize the frequency dependency of the visco-elastic standard's properties. Here, the parameters for the highest frequency range are shown. For lower frequencies the acquisition parameters have been adapted.

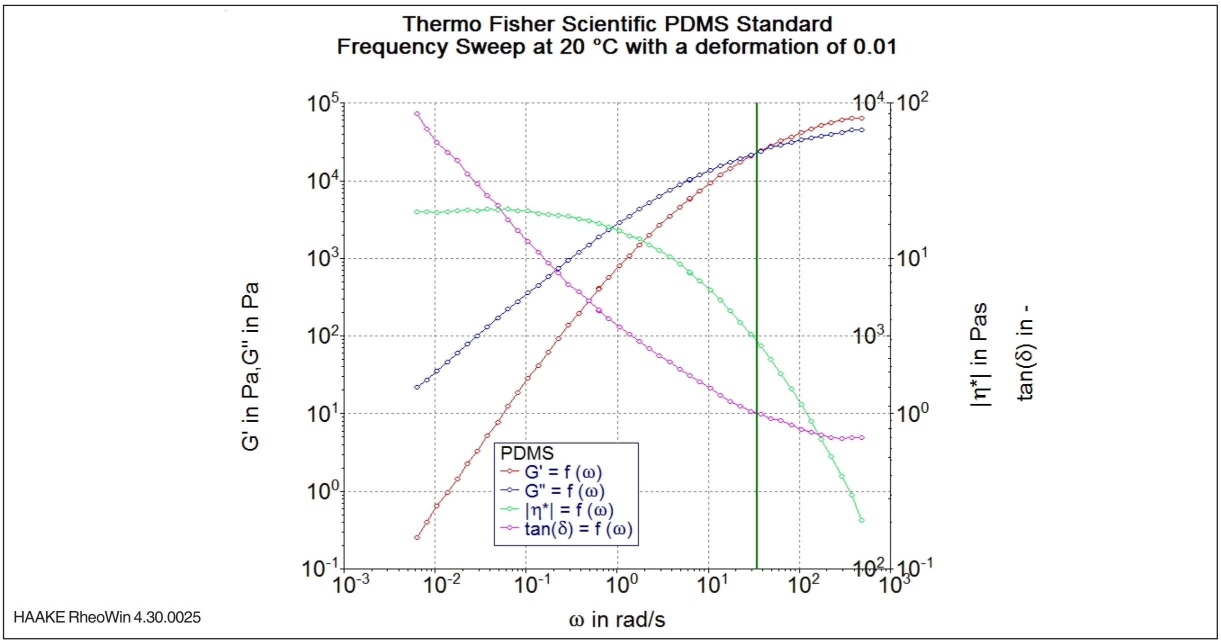

Fig. 5: Frequency dependency of the viscoelastic standard material's rheological properties. The crossover parameters have been calculated automatically by Rheowin.

Summary

The Thermo Fisher Scientific viscoelastic standard material and the relevant test methods have been presented. Due to the nature of the standard material, the sample preparation requires more care compared to handling purely viscous calibration fluids. With the viscoelastic standard material it is possible to verify the performance of a rheometer in oscillation mode and the accuracy of the sample handling and loading procedure in a very time and cost efficient way.

References

[1] Klaus Oldörp, Thermo Fisher Scientific application note V218 "Test liquids"

[2] Cornelia Küchenmeister, Klaus Oldörp, Thermo Fisher Scientific application note V248 "Well Prepared - Good Results"

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