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METHOD FOR DETERMINATION OF RHEOLOGICAL PROPERTIES OF CHEMICAL ADHESIVES USING A DYNAMIC SHEAR RHEOMETER

CAUTION: Prior to handling test materials, performing equipment setups, and/or conducting this method, testers are required to read "**SAFETY AND HEALTH**" in Part 2 of this method. It is the responsibility of the user of this method to consult and use departmental safety and health practices and determine the applicability of regulatory limitations before any testing is performed.

A. SCOPE

This test method covers the use of a dynamic shear rheometer for determining the complex modulus and phase angle at different frequencies and the stress relaxation modulus of chemical adhesives used for bonding anchors into hardened concrete. These values are then used either directly or for calculating parameters that have been correlated with creep compliance as determined by California Test 681.

B. APPARATUS

1. A Dynamic Shear Rheometer that is capable of being programmed to run frequency sweeps from 0.1 to 100 radians per second and stress relaxation tests with programmable time zones. The device needs to hold a solid sample geometry of roughly 3 x 12 x 45 mm in a temperature controlled environment of 25° to 95° ± 0.1°C. Rheometrics ARES, RAA and RDA-III and TA Instruments AR-2000 rheometers are examples of acceptable devices.

2. A precision saw with a water-cooled diamond blade. Saws made by Buehler or Wale have been used.

3. A typical lab oven capable of 45°C

C. SAMPLE PREPARATION

A block of solid adhesive, nominally 50 x 50 x 12 mm, needs to be cast. A metal mold with removable sides or a silicone mold, both 12 mm deep are acceptable. Fill a mold with the adhesive using the dispensing device and mixing nozzle that accompanies the typical adhesive cartridge. If the adhesive is contained in glass or foil, open the outer container and separate the resin from the catalyst/hardener. With the resin in a small paper or plastic cup, add the second component and mix expeditiously and transfer immediately into the mold. For adhesives that set rapidly, cool the components in a refrigerator before mixing. Strike off the top of the mold to obtain a relatively smooth surface. Let the adhesive cure for 24 hours at 25° ± 2°C. Remove the sample from the mold and prepare four slices, 3 mm thick using the precision saw. Place the slices on a sheet of aluminum in the oven at 45° C for 24 ± 0.25 hours. Label each slice and record the thickness and width.

PART 1. DETERMINATION OF SHEAR SUSCEPTIBILITY AND STRESS RELAXATIONPARAMETERS AT 45°C

A. PROCEDURE

The basic procedure is to run a frequency sweep from 0.1 to 100 radians per second and a stress relaxation test on each sample, at 45°C. During the frequency sweep, record the complex modulus (G*), the phase angle (delta) and the frequency. During the stress relaxation test, record time, torque and G(t).

Install a slice in the testing tools using shims as necessary for a snug fit. During the tightening process, minimize subjecting the sample to torsion or tension by adjusting motor alignment and/or tool height. Measure the gage length of the installed sample. Zero the vertical distance indicator. Enable the auto tension or "hold" capability. Close the environmental chamber. Raise the temperature to 45°C. After the sample temperature has been at 45 ± 0.1°C for ten minutes, run a frequency sweep from 0.1 to 100 radians per second (5 increments per decade) using 0.003 % strain. After the frequency sweep, run the stress relaxation. Use 0.01 % strain and record the values listed above for the first 60 seconds of the relaxation.

B. CALCULATION AND REPORT

There are two shear susceptibility parameters to be calculated from the measured values:

1. Shear Susceptibility of G* (SSG*), also known as: M

$$\text{M} = \left(\log\left(G^* @ 100 \text{ rps}\right) - \log\left(G^* @ 1 \text{ rps}\right)\right) \Big/ 2$$

2. Shear Susceptibility of Delta (SSD):

$$\text{SSD} = \left(\text{delta} @ 1 \text{ rps} - \text{delta} @ 100 \text{ rps}\right. \Big/ 2$$

   Report M × sin (SSD) for each sample.

3. Fit a power equation to the stress relaxation data:

   G(t) = a × (time)^b

   Report the factor "b" for each sample.

C. PRECISION AND BIAS

The precision and bias have not yet been investigated per ASTM E-691. Table 1 presents the results of one device and operator using four replicates of seven different materials. A coefficient of variation greater than 25 % may be an indication of inadequate mixing.

PART 2. SAFETY AND HEALTH

Prior to handling, testing or disposing of any waste materials, testers are required to read: Part A (Section 5.0), Part B (Sections: 5.0, 6.0 and 10.0) and Part C (Section 1.0) of the Caltrans Laboratory Safety Manual.

Users of this method do so at their own risk.

END OF TEXT
(California Test Method 438 contains 3 pages)

Table 1

Material	Mean Relaxation Slope (b)	Standard Deviation of (b)	Mean M	Standard Deviation of M	Mean SSD	Standard Deviation of SSD
A	0.0157	0.00129	0.0113	0.00189	0.269	0.0382
B	0.0333	0.00577	0.0190	0.00216	0.459	0.111
C	0.0347	0.00064	0.0185	0.00173	0.598	0.105
D	0.0417	0.00483	0.0398	0.00150	0.483	0.103
E	0.0595	0.00070	0.0413	0.00189	0.626	0.051
F	0.141	0.00458	0.0675	0.0116	2.17	0.147
G	0.251	0.00909	0.171	0.00356	3.21	0.137
Pooled s_r		0.00482		0.00483		0.106