**DEPARTMENT OF TRANSPORTATION**
**DIVISION OF ENGINEERING SERVICES**
**Transportation Laboratory**
**5900 Folsom Blvd.**
**Sacramento, California 95819-4612**

# 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 Section G of this method. It is the responsibility of whoever uses 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. 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. Precision saw with a water-cooled diamond blade. Saws made by Buehler or Wale have been used.

3. A forced draft oven capable of maintaining 45 ± 2°C.

4. Mold that can cast a specimen 50 x50 x 12 mm. Either a metal mold with removable sides or a silicone mold.

## C. SPECIMEN PREPARATION

1. A block of solid adhesive, nominally 50 x 50 x 12 mm, needs to be cast. Fill the mold with the adhesive using the manufacturers 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 2nd 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.

2. Let the adhesive cure for 24 hours at 25° ± 2°C.

3. Remove the sample from the mold and prepare four slices, 3 mm thick by 50 x 12 mm 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.

## D. PROCEDURE

1. This procedure will determine the shear susceptibility and stress relaxation parameters at 45˚ C. Run a frequency sweep from 0.1 to 100 radians per second and a stress relaxation test on each sample, at 45°C

2. Install a sample 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.

3. Zero the vertical distance indicator. Enable the autotension 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. Record the complex modulus (G*), the phase angle (delta) and the frequency.

4. Run the stress relaxation using 0.01% strain. Record time, torque and G (t) for the first 60 seconds of the relaxation.

## E. CALCULATIONS

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

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

   M = (log (G* @ 100 rps) - log (G* @ 1 rps))/2

2. Shear Susceptibility of Delta (SSD)

   SSD = (delta @ 1 rps - delta @ 100 rps)/2

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

   G (t) = a x (time)^b

   Report the factor "b" for each sample.

## F. PRECISION and BIAS

The precision and bias have not yet been formally investigated. 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.

**Table 1**

| Material | Mean Relaxation Slope (b) | Std. Deviation of (b) | Mean M | Std Deviation of M | Mean SSD | Std. 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 |

## G. SAFETY AND HEALTH

Prior to handling, testing or disposing of any waste materials, testers are required to read the Caltrans' Laboratory Safety Manual. Users of this method do so at their own risk.

**END OF TEXT**

California Test 681

(California Test Method 438 contains 3 pages)