Comments on Use of Reference Fluid to Verify DSR

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Reference fluid – how and why?

- Used to verify torque transducer *ONLY*
- How can we do this?
  - ✓ Compare viscosity measured with the DSR to viscosity published for reference fluid
- Why can we do this?
  - ✓ Measure viscosity of reference fluid with DSR at combinations of ***temperatures/frequencies/strains*** where response of fluid is Newtonian
  - ✓ Newtonian ⇨ viscosity is independent of shear rate
  - ✓ Elastic and viscoelastic response is therefore negligible and calculation for measured viscosity becomes straight-forward – more later

Sidebar - how is viscosity of reference fluid determined?

- Reference values determined with capillary viscometer in region of Newtonian flow
  - Viscosity independent of shear rate
  - Shear rate is NOT given on reference fluid container
- When reference fluid is calibrated supplier must exercise same cautions used when DSR measurements are made
  - Stay in Newtonian (linear) region
  - Avoid shear thinning (non-Newtonian region)
  - In capillary experiment limits defined by temperature and shear rate

Viscosity measurement with DSR – two options

- Steady state shear
  - ✓ Apply constant uni-directional torque or shear rate until reach steady state flow
  - ✓ Issues with geometric linearity
- Better approach – use sinusoidal oscillation and assume resistance to deformation is due entirely to viscous flow………….
  - ✓ Perform at small strains where shear thinning is absent
  - ✓ Limiting strain that will give Newtonian flow depends on temperature and frequency

Items that might affect accuracy of DSR measurements using fluid

- Measurement temperature
  - ✓ Always verify DSR thermometer before using fluid
- Fluid expiration date
  - ✓ Do not use beyond expiration date – check label
- Heating the fluid
  - ✓ Heating can cause it to deteriorate
  - ✓ Use proper storage temperatures
- Improper test specimen preparation
  - ✓ Incorporation of bubbles can reduce measured value
  - ✓ Gap and improper bulge – good laboratory technique
- Each of above easily controlled with proper laboratory procedures

Determining steady state capillary viscosity from oscillatory shear in DSR

- Invoke Cox-Merz rule (1958)
  - ✓ Well known rule used for may fluids
  - ✓ Well accepted in literature
  - ✓ Equates complex viscosity to capillary viscosity
- At low frequencies (oscillatory) and shear rates (capillary) complex viscosity = capillary viscosity
  - ✓ Under these conditions elastic effects become negligible

    $|\eta^*(\omega)|_{\omega \to 0} = \eta(t)|_{d\gamma/dt \to 0}$
  - ✓ $\eta^*(\omega)$ from DSR, $\eta(t)$ from reference fluid bottle
- Note test conditions!

Measurement of Complex Viscosity, η*

- Assuming only viscous response, $\eta^* = G^*/\omega$

  Substituting, $G^* = 2Th/4\pi r^4\Phi$

  $\eta^* = Th/2\pi r^4\Phi\omega$

where:

T = Torque applied to specimen, N-m

h = Specimen thickness, m

Φ = Angular rotation, rad

r = radius of test specimen, m

ω = frequency, rad/s

- If we assume other variables are accurately known uncertainty lies with measurement of torque, T

Assumptions that allow T to be only variable of uncertainty

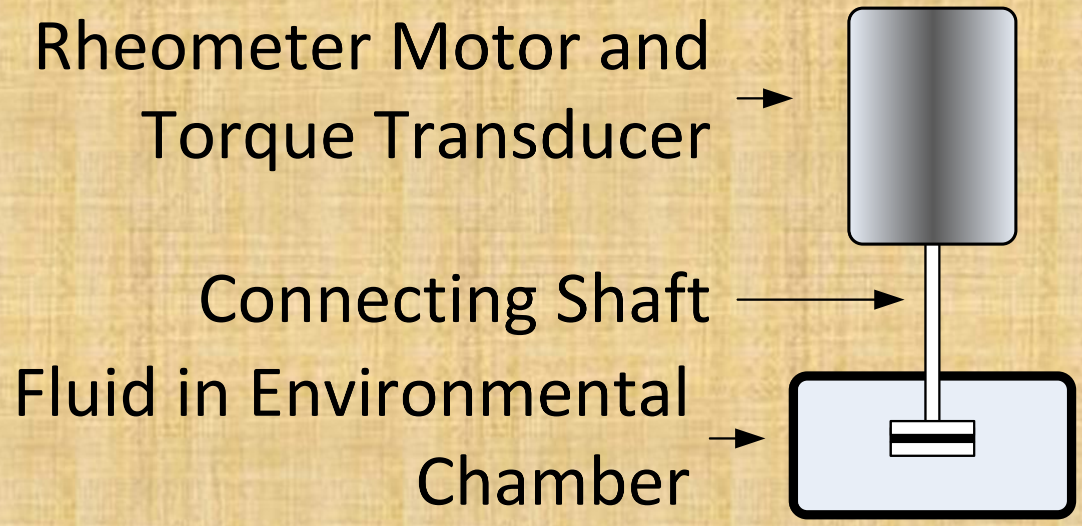
- Checklist

1. Incorrect specimen geometry – diameter, gap, bulge ✓
2. Improperly formed specimen – bulge, bubbles ✓
3. DSR thermometer not verified – verify thermometer ✓
   - Fluid temperature susceptibility ≈ ½ that of binder
4. Angular displacement transducer ✓
5. Internal instrument calibration ✓
6. Machine compliance ✓
   - Minimal for strains at 64 and 70 C

- Above assumptions reasonable at small strains developed with fluid using 25 mm plate at 10 rad/s
- Remaining item is accuracy of torque transducer

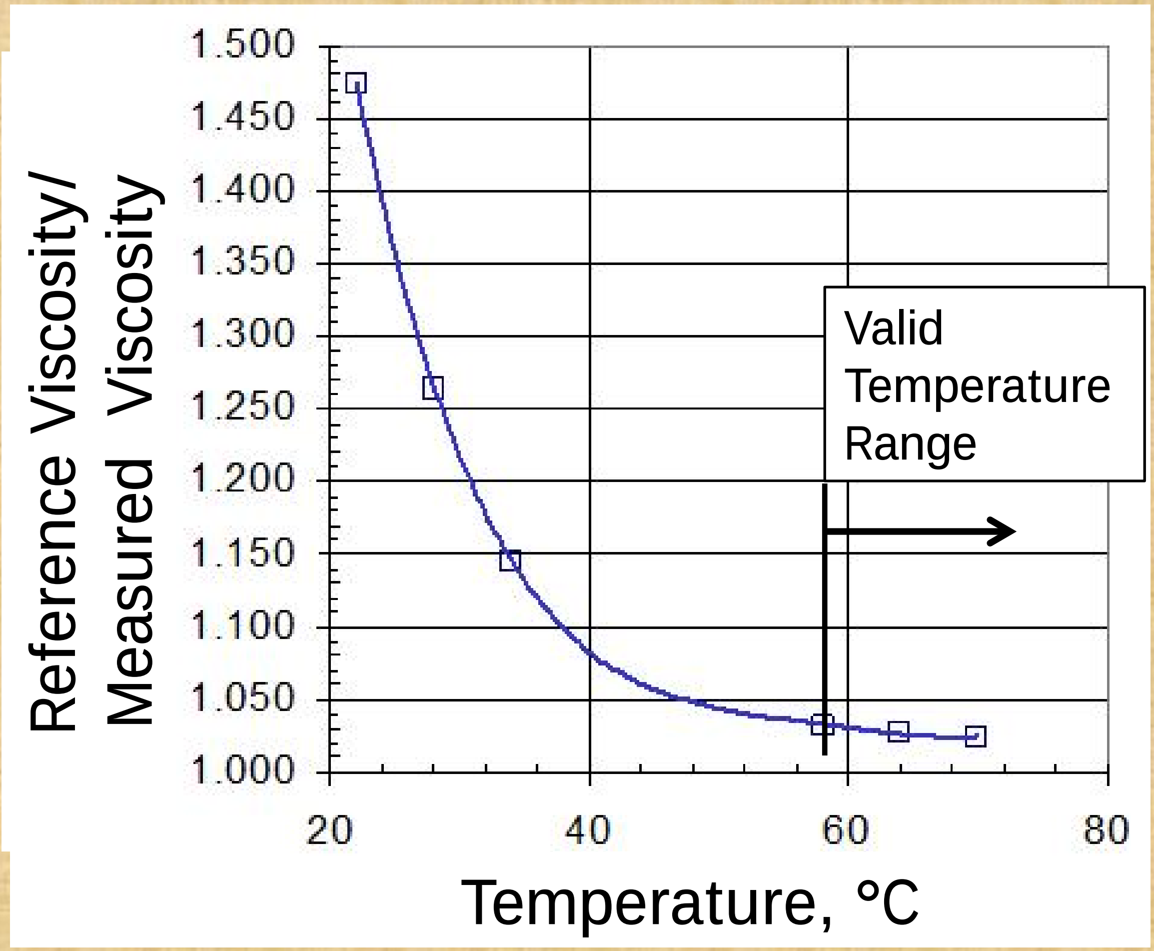
Why test at single temperature?

- Measurement of question is torque
  - ✓ Torque transducer is at ambient temperature
  - ✓ No reason for testing at temperatures
- Torque at 64°C, 10 rad/s, 10% strain =
  - ✓ Similar to torque used with 8 and 4 mm



Viscosity – Reference vs. Measured as f(T)

- Recommend testing at 64°C
- Errors become large at lower temperatures
- Binder flows from plates at higher temperatures



Conclusion

- If DSR viscosity equals reference viscosity DSR is likely working correctly and torque transducer standardization is verified
- If DSR viscosity differs from reference viscosity something is “wrong”
  - ✓ Could be torque transducer
  - ✓ Could be other internal DSR calibration items
  - ✓ Likely not machine compliance if strains are not small
  - ✓ Likely not temperature errors if temperature Was verified
  - ✓ Likely not specimen issues if technician is competent