GTR: DSR Evaluation and Geometry Recommendation

Asphalt Binder Expert Task Group
Salt Lake City, UT
April 27, 2016

Darin Hunter
Anton Paar USA
Ashland, VA

Summary of Situation

Current Status

- **GTR modification of asphalt binder** provides environmental and performance benefits
- Current Superpave test methods (PG) are not suitable for rubber modified materials with **particle sizes up to 2+ mm** (mesh 10)
- Acceptance of new materials by highway agencies depends on suitable test methods for **rheological characterisation** (DSR)

Scope of Work

- Evaluation of **various DSR measuring systems**
- Investigation of the **variabilit**y in measured properties

Main Goals

- **Identify** practical and rheological **challenges**
- **Apply** relevant **engineering principles** such as measuring system standards
- **Suggest suitable measuring geometry** for PG measurements of GTR modified Asphalt Binder which provides the truest rheological data

Status

Scope

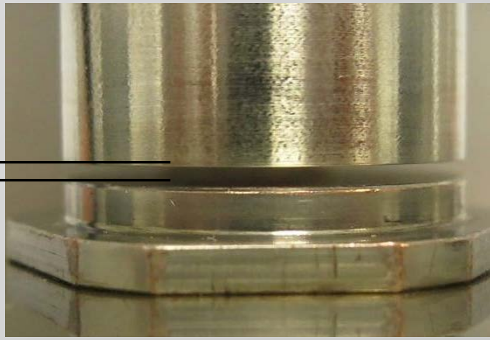
Goals

Summary of Problem and Solution Approaches

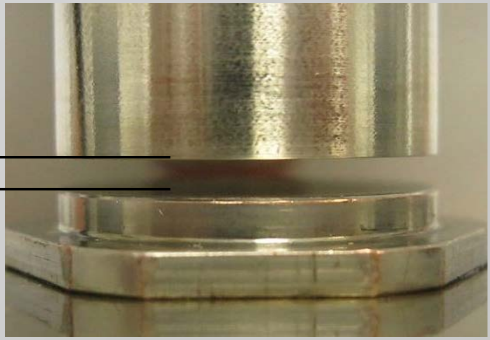
Problem

- Swollen 2 mm rubber particles do not fit in the 1 or 2 mm gap if AASHTO T315
- Particle interferences impact results
- Accepted Governing Rules for Good Rheology
  - Measuring gap should be at least 5X and preferably 10X the largest particle size
  - AASHTO T315 / ASTM D7175 (DSR): max. 400 µm particle size (2mm gap, 8mm ∅ plate)
- Questionable rheological data

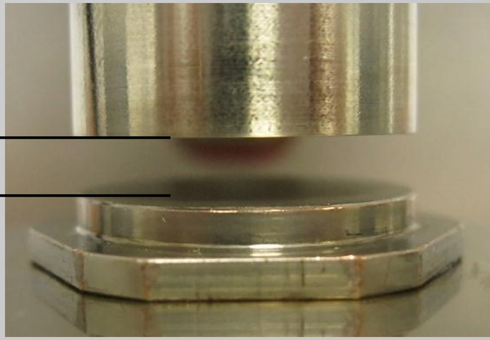
1 mm



2 mm



4 mm



Solution Approaches

- Increase the measuring gap
  - Plate-plate geometry with large gaps (up to 4mm)
  - Concentric cylinder geometry with large gaps
- Considerations:
  - Does gap matter?
  - Does the data agree with conventional measuring gaps?
  - Are new sources of error introduced?
  - Are new practical concerns introduced?

Plate-Plate Geometry: Pros and Cons

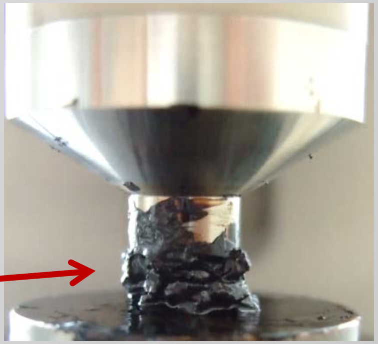
Anton Paar

Pros

- 👍 Less sample volume
- 👍 Faster heating/cooling rates and temperature equilibrium
- 👍 Accustomed and approved system

Cons

- 👎 Reliable trimming and gap filling
  - Larger influence with smaller plates
- 👎 Representative sample volume
- 👎 Ratio diameter / gap → Geometry factors, Calibration (ISO 6712-10, DIN 53019-1)
- 👎 Time effects due to sagging (flow) of binder out of the measuring gap



Concerns

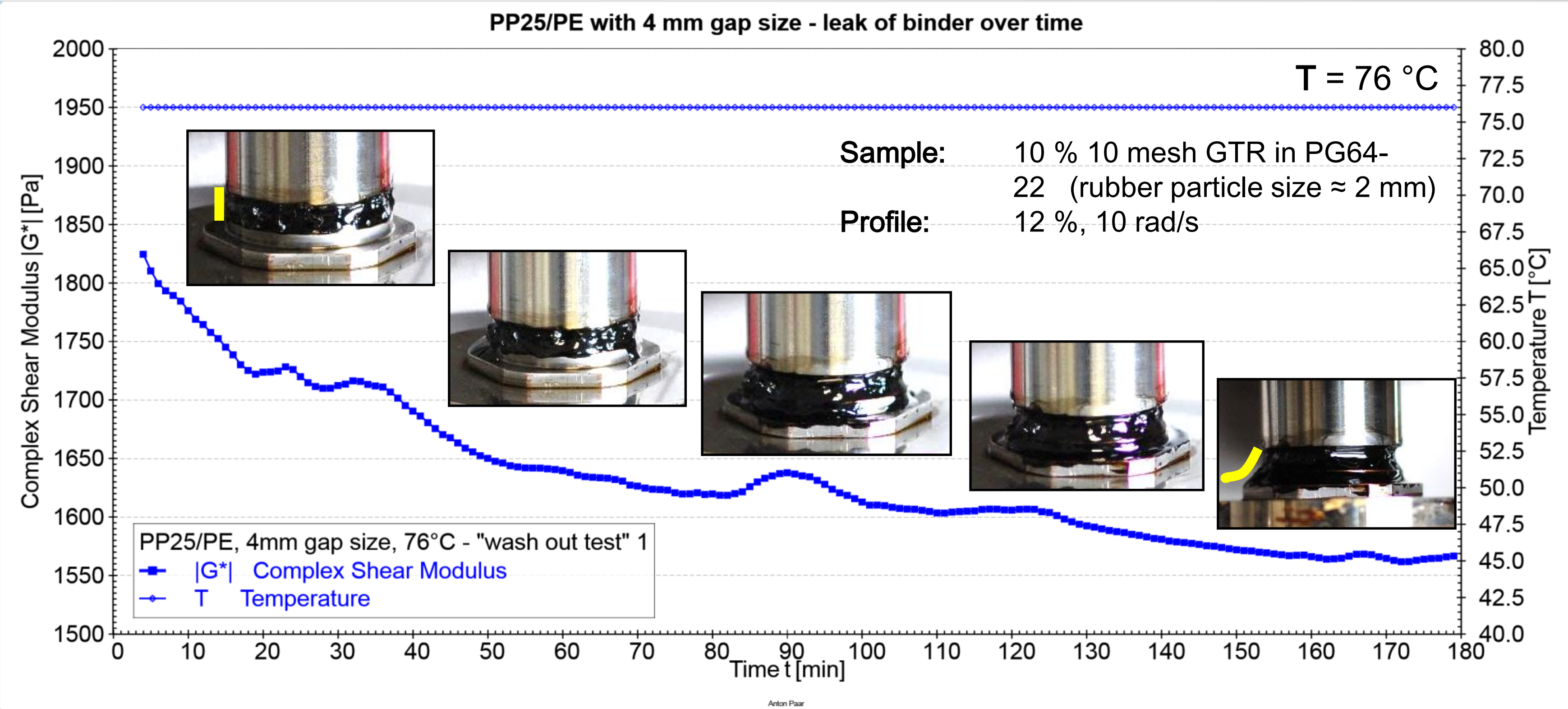
? Repeatability

? Reproducibility

? Reliability

Sample: 10 % 10 mesh GTR in PG64-22

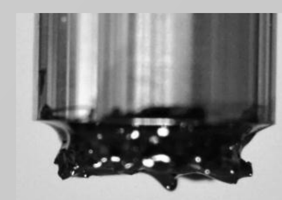
Plate-Plate Geometry: Sample Sag (Flow) Out of Gap



	IG*I [Pa] t = 0 min	IG*I [Pa] t = 30 min	IG*I [Pa] t = 60 min	IG*I [Pa] t = 180 min
	1824	1712	1640	1567
Deviation referred to t = 0 min [%]	---	- 6.1	- 10.1	- 14.1

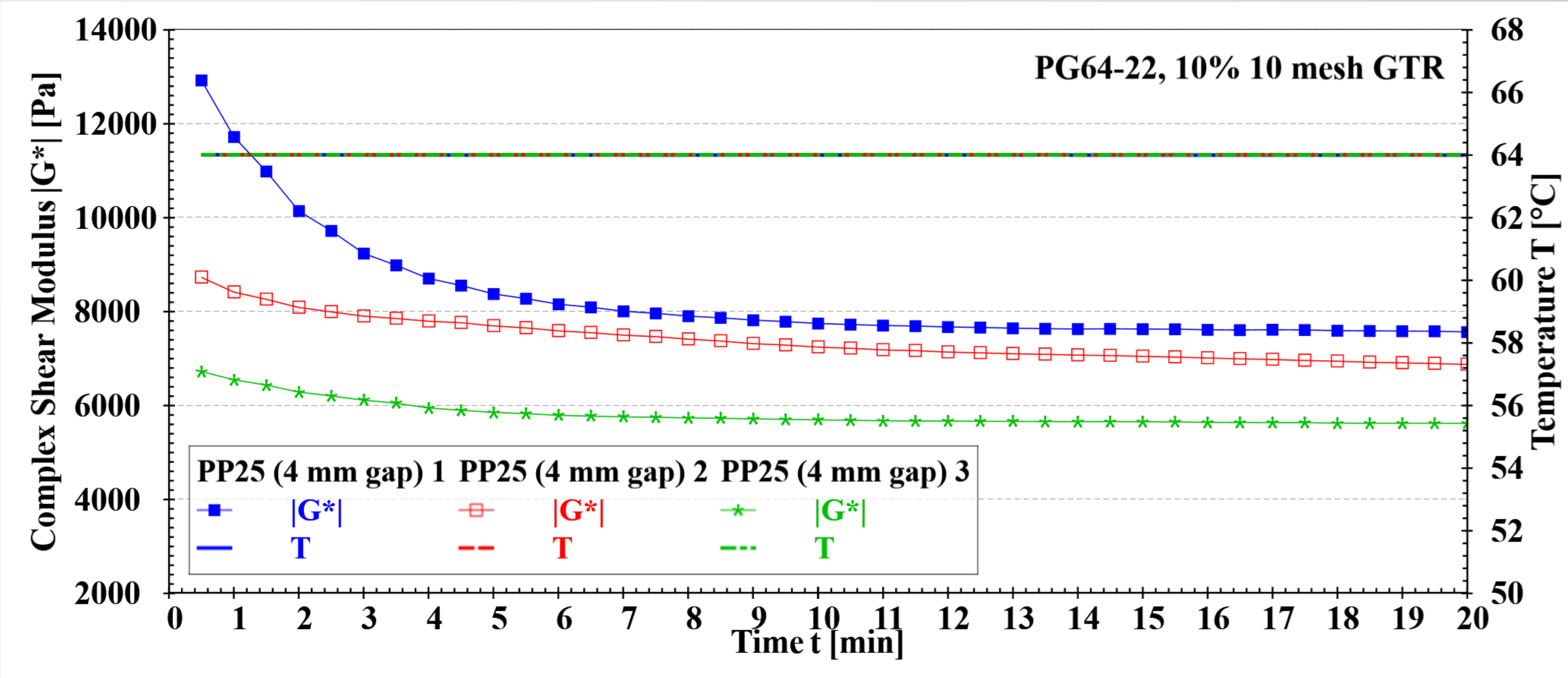
→ **Flow of sample out of gap produces increasingly lower measured results over time even within time scale of 10 min thermal equilibrium**

→ **Parallel plate system should be used with maximum gap size of 2 mm**



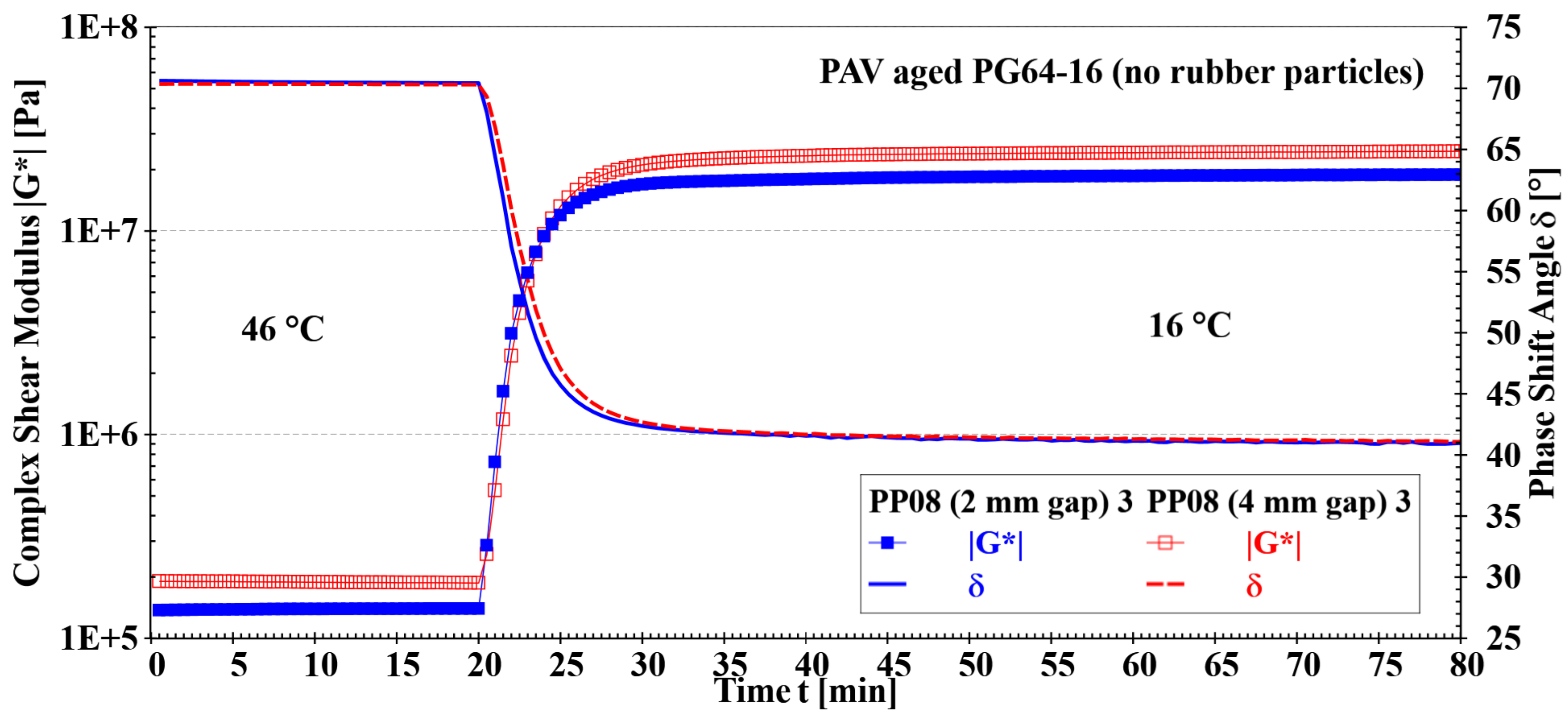
Remaining Rubber Matrix after 180 min Measurement (binder pooled on lower plate)

Plate-Plate Geometry: Repeatability



- Single, expert level user
- Issues contributing to lack of repeatability
  - Issues with; homogeneity, representative sample and trimming
  - Sample pulled out of gap and/or rubber particles at edge cannot be trimmed thus hang outside gap introducing additional drag
- New source of error introduced due to sampling and trimming issues

Plate-Plate Geometry: Reliable (Real) Measured Data



	Mean G* at 46 °C [Pa]	Mean G* at 16 °C [Pa]
PP08, 2 mm gap size	145537	19574667
PP08, 4 mm gap size	194573	24709667
Deviation with 4 mm gap size [%]	+ 33.69	+ 26.23

- Compared with data from the accepted 2mm gap
- No longer an absolute measuring geometry at large gap sizes
- Rheological data should be gap independent. When it ceases to be gap independent, the gap is too large.

Concentric Cylinder Geometry: Pros and Cons

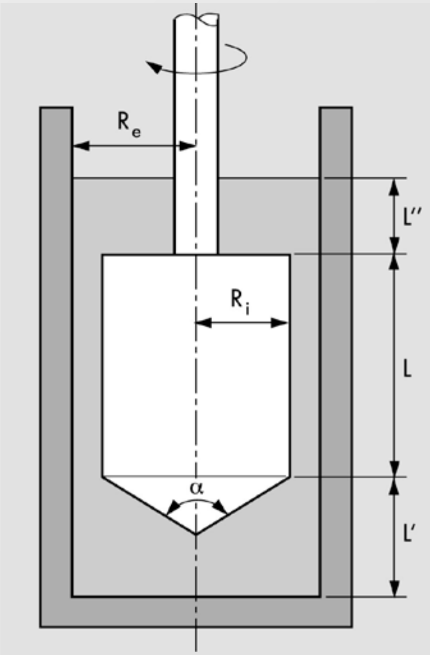
Pros

- Large gaps possible
- Sample cannot flow out of the shear gap
  - No binder sag errors
- No trimming required
- Representative sample volume
- No edge effects
- Disposable cups can be used

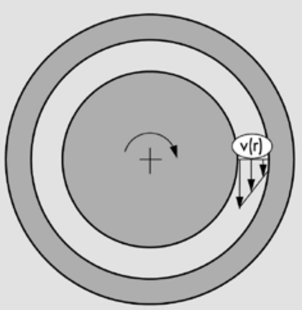
Cons

- Increased sample volume
  - Slower heating and cooling rates
  - Longer temperature equilibrium time (at least 30 min)
- Relative measuring geometry requires calibration with traceable standard (Cannon N2700000SP)

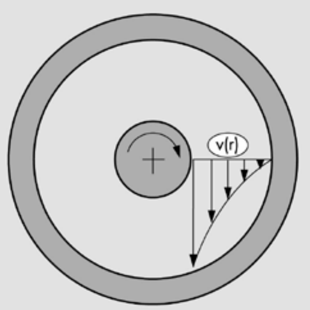
Concentric Cylinder Geometry: Overview



Small Bob in Large Cup



Absolute (req. small gap)



Relative (large gap)

CC17SP

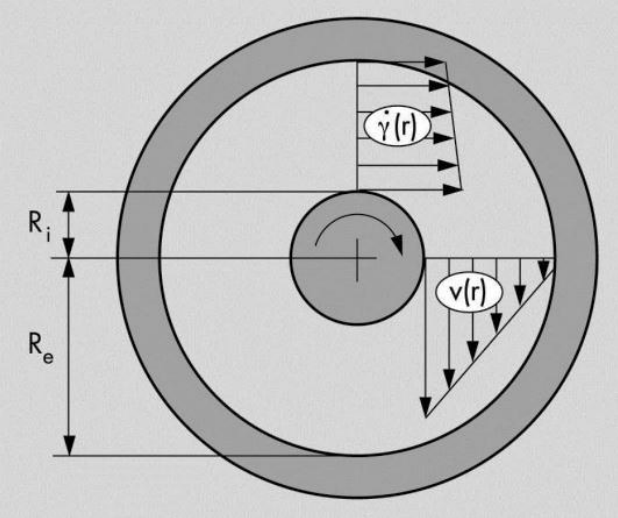
- Gap ≈ 6.2 mm
- Suitable for neat and RTFO asphalt binder (high PG temperatures)

CC10SP

- Gap ≈ 9.5 mm
- Suitable for PAV asphalt binder (intermediate PG temperatures)

Concentric Cylinder Geometries with Large Gap: Engineering Details

Concentric cylinder (CC) geometries with large gap (ratio δ_{cc} >> 1.0847)



B-CC17SP:

$$\delta_{cc} = \frac{R_e}{R_i} = \frac{14.46\ \mathrm{mm}}{8.33\ \mathrm{mm}} = 1.74$$

(Gap: ≈ 6.2 mm)

B-CC10SP:

$$\delta_{cc} = \frac{R_e}{R_i} = \frac{14.46\ mm}{5.00\ mm} = 2.89$$

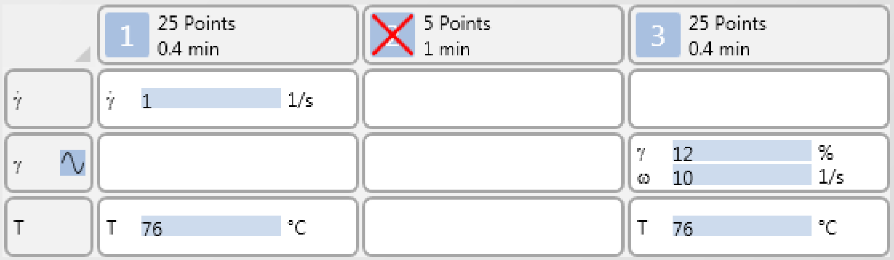
(Gap: ≈ 9.5 mm)

- The combination of smaller cylinders (bobs) in a CC27 cup results in relative concentric cylinder systems (non ISO 3219 compliant) due to ratio **δcc >> 1.0847**
- Compromise to be able to measure GTR modified asphalt binder
- Geometry constants must be calibrated
- Defined as relative measuring systems (same as PP with gap size > 2 mm is)
- Nevertheless, provides data comparable to absolute systems

Calibration of B-CC17SP

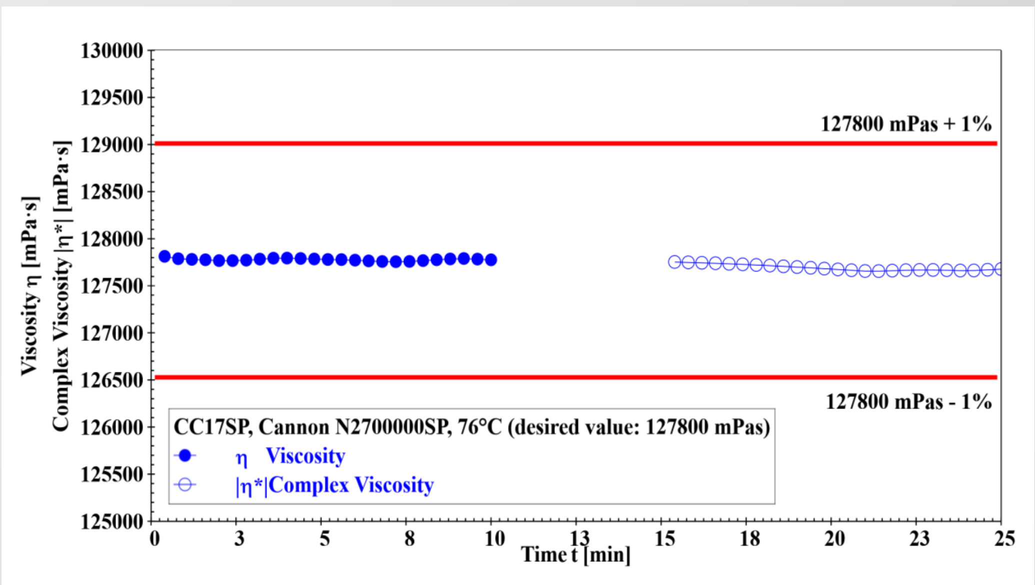
Anton Paar

Measuring profile used to calibrate and validate the geometry factors:



- 1 1/s in rotation was used to be within the newtonian range
- 12 % and 10 rad/s were used according to AAHSTO T315

Verification result after calibration:



- Shear viscosity and complex viscosity values are perfectly within ± 1 % of the viscosity value according to the calibration certificate of the used Cannon N2700000SP
- Limit according to AASHTO T315: ± 3 %

Viscosity Standard: **N2700000SP** *Lot Number:*

Certification/Issue Date: **06/05/2013** *Expiry Date:*

Temperature		Kinematic Viscosity	Dynamic Viscosity
°C	°F	mm²/s (cSt)	mPa·s (cP)
52.00	125.60		600400
58.00	136.40		395800
64.00	147.20		266700
70.00	158.00		183000
76.00	168.80		127800

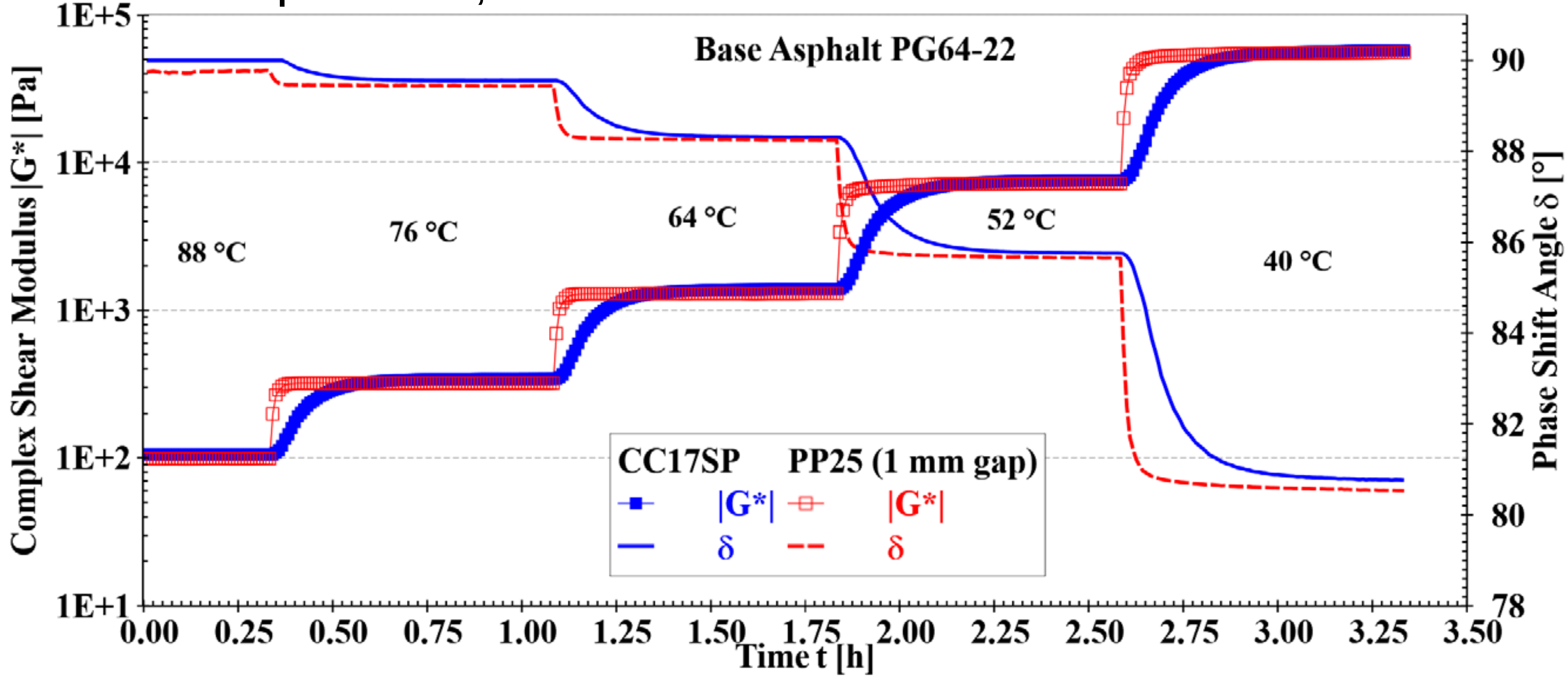
→ **No need to perform again at customer site!**

→ **Please Note: This calibration does not fit for low viscosity samples!**

Concentric Cylinder Geometry: Reliabe (Real) Data

Anton Paar

Neat Asphalt Binder, no rubber modification

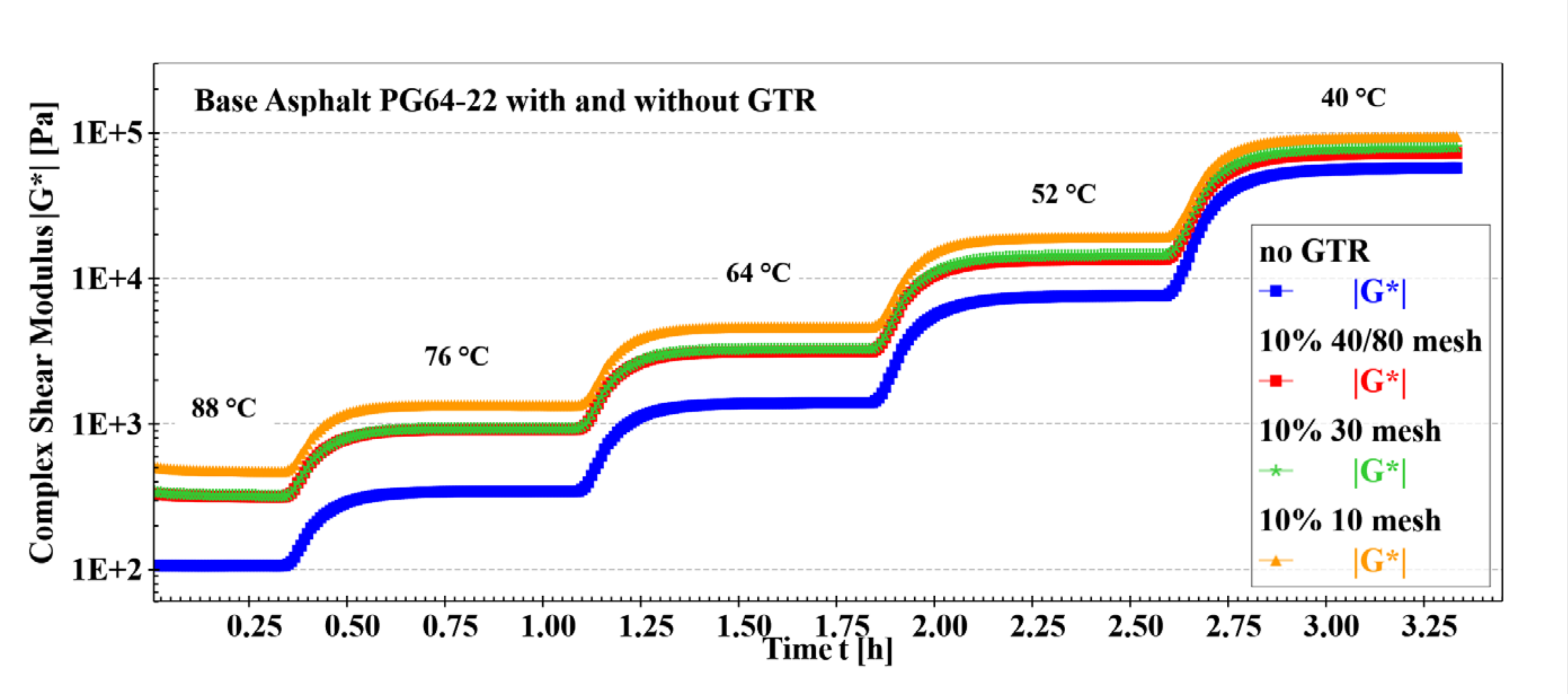


- CC compared with data from the accepted plate-plate, 1mm gap, 25mm dia
- Once thermal equilibrium is achieved, curve shapes are equal
- No significant difference in visco-elastic behavior (phase angle δ values)
- Relative CC system with large gap is comparable to absolute PP system (1 mm gap)

Concentric Cylinder Geometry: Proof of Concept Unaged Binder

Anton Paar

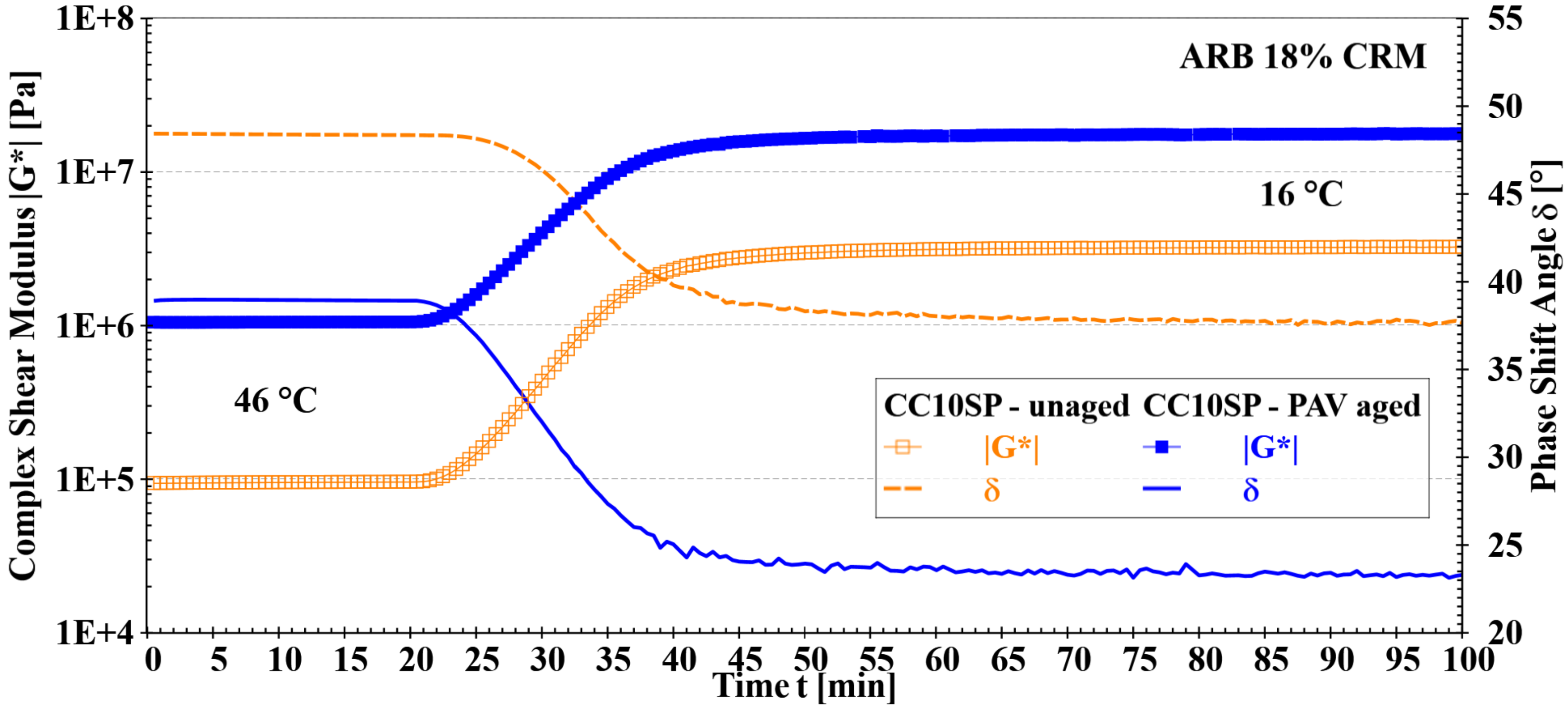
Influence of GTR mesh size on complex shear modulus G* (neat asphalt binder)



- Increasing G* values with increasing particle size
- Only slightly difference between 40/80 mesh and 30 mesh
- Overall stiffness (G*) is influenced by different rubber particles

Anton Paar

Influence of rubber modification on complex shear modulus G* and phase angle δ (PAV aged asphalt binder)



- Increasing stiffness (G*) with addition of GTR
- Shift to more solid-like behavior with addition of GTR
- Dramatic loss of damping ability (flow or liquid-like behavior) at intermediate temperature (delta going towards 0˚ or solid behavior)

Summary and Conclusions

Anton Paar

- Plate-plate geometry has intrinsic limits:
  - Questionable rheological data due to sample sag
  - Difficult to trim crosslinked rubber particles
  - Appropriate for smaller particle sizes (<< 2mm)
  - Okay for intermediate temperares where sample sag is not an issue
- Concentric cylinder geometries with large gap provide:
  - Eliminination of fundamental plate-plate limitations
  - Calibration procedure as for relative measuring systems
  - Easy, straight forward compensation for torsional compliance; thicker bob axis greatly reduces need for compliance compensation
  - **Performance Grading measurements at both high and intermediate temperatures are within reach**
- Compromises must be made, i.e. use of relative systems
- Final steps – collaboration partners currently working on repeatability with CC10 bob for PAV aged binder
- Practical considerations need to be addressed such as longer time to equilibrium, reproducible cup filling, etc...

Plate Plate

Concentric Cylinder

Final Steps

Some Impressions