PAV Degassing and Pan Warping – 4mm Testing

Dave Anderson

Asphalt Binder ETG

Bozeman, MT

September 19, 2017

1. PAV Degassing

- Hypothesis: Vacuum degassing does not affect the properties of the PAV residue as measured with the BBR or DSR
  - ✓ Ultimate property tests are not considered herein
  - ✓ Required for these tests
- Possible Action Item: Revise test methods so that vacuum degassing is an option at the discretion of the user

Vacuum Degassing - Historical

- Vacuum degassing was adopted to enhance repeatability of direct tension test data (19xx)
  - ✓ Not part of original DSR and BBR test protocols
  - ✓ Adopted after bubbles were shown to affect DTT results
  - ✓ Subsequently dropped when DTT was discontinued
- Vacuum degassing protocol was developed based on results of limited laboratory testing program
  - ✓ Preheating combined sample at 175°C for 10 ± 1 min
  - ✓ Vacuum at 15 ± 2.5 kPa (Absolute) for 30 ± 1 min
  - ✓ Included stirring and flashing steps

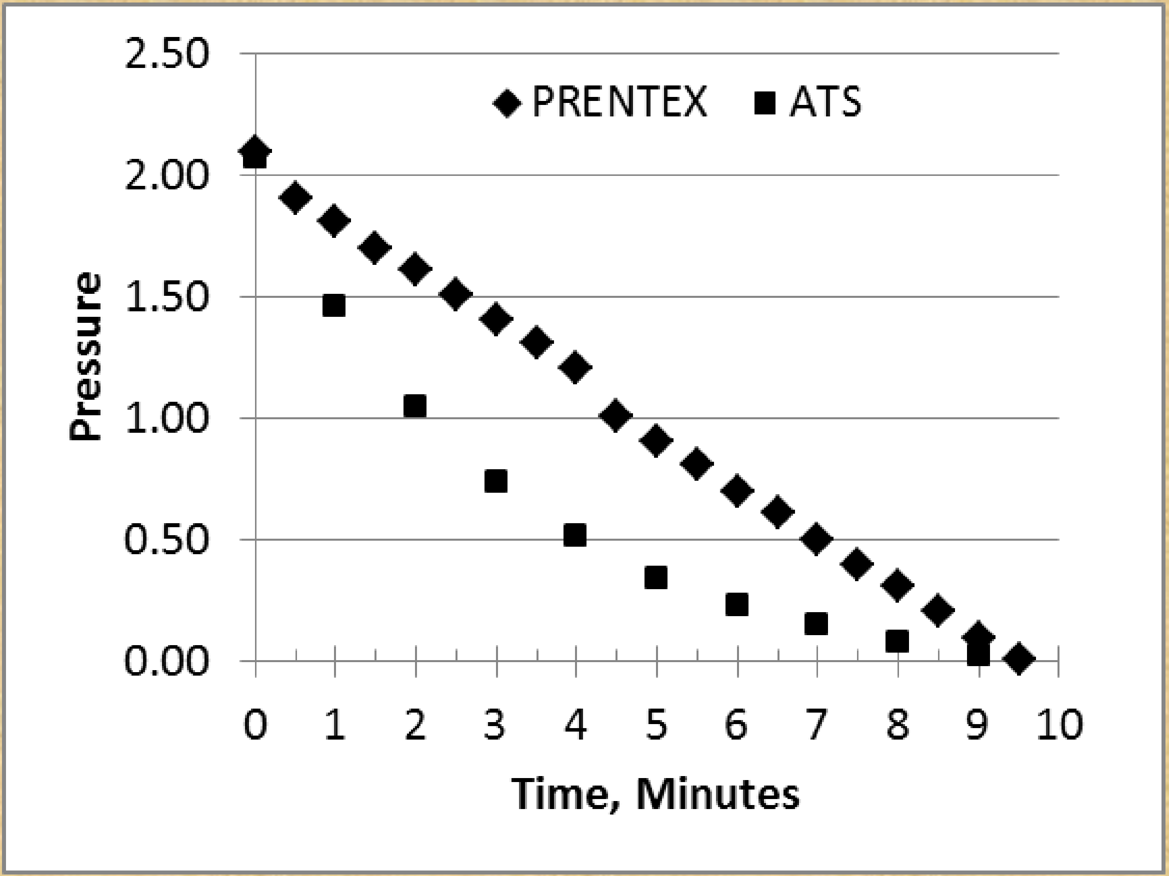
Experiment design

- Initially envisioned as simple study to validate previous decision that degassing should be optional
- Enlarged to include the following variables:
  - ✓ Rate of pressure release
  - ✓ Laboratory elevation
  - ✓ Binder source to include PMB’s
  - ✓ Manufacturer of PAV – degassing rate
- Experimental work and analysis as described above is now complete
  - ✓ Several recommendations

Linearity of Pressure Release Rate

- Reviewed as possible cause of excessive bubbles
- Pressure vs. release rate obtained from several labs
  - ✓ Prentex releases linearly in series of small bursts
    - Meets requirements of test method
  - ✓ ATS releases 50% in first 90 seconds
    - Does not meet requirements of test method
- Above verified by data from several laboratories
- Release rate from lab most vocal about degassing uses Prentex
- Analysis shows that pressure release rate is not a significant variable

Pressure Release Rate – Typical Results



Determination of Vacuum Level

- Laboratories routinely confuse any of barometric pressure with barometric pressure reported by the local weather station
  - ✓ Barometric pressure reported by local weather station is corrected to sea level
  - ✓ Useless for our purposes!
- During vacuum degassing the absolute pressure calculated in accordance with Eq. 4 shall be 5.0 ± 0.50 inches of mercury (17 ± 1.7 kPa). As a minimum the gage shall be read and reported to the nearest 0.5 in Hg (2 kPa).

Recommended Change

When using a vacuum gage to control the degassing pressure, the gauge readings given by Eq. 4 calculated using the laboratory elevation to the nearest 100 feet shall be used to control and report the vacuum during the degassing cycle. Equation 4 accounts for changes in atmospheric pressure with elevation. No additional corrections for laboratory barometric pressure, temperature, humidity, etc. shall be applied to the vacuum gage reading regardless of instructions supplied by any vendors, instrument software, or other source. The vacuum gage reading shall be reported and controlled to the nearest 0.5 in Hg (0.2 kPa).

Summary and Conclusions

- Barometric pressure is often reported erroneously
  - ✓ Tie vacuum level to elevation
  - ✓ Absolute vacuum gage should be specified
- Revise vacuum levels to more friendly and realistic values
  - ✓ Current tolerances cannot be read on gages
- Manufacturer’s different pressure release rate not significant
- Vacuum degassing should be allowed as option

Some Observations

- Bubbling continues through degassing cycle for some materials
- Recent work by Tom Bennert at Rutgers shows hydrocarbons released during degassing period
- Foaming during degassing is still a problem
- Suggest that task group continue to look at degassing protocol and make recommendations, if warranted, for further study and report back at next meeting

Equipment

- ThermoFisher Nicolet is50 FTIR (Fourier-Transform Infrared Spectroscopy.
  - TGA (Thermogravimetric analyzer) attachment
  - OMNIC Software
- Prentex VDO 9900 Degassing Oven
- Cox & Son's RTFO (Rolling Thin Film Oven)

76-22 PAV Binder Condition (NO RTFO) Spectra

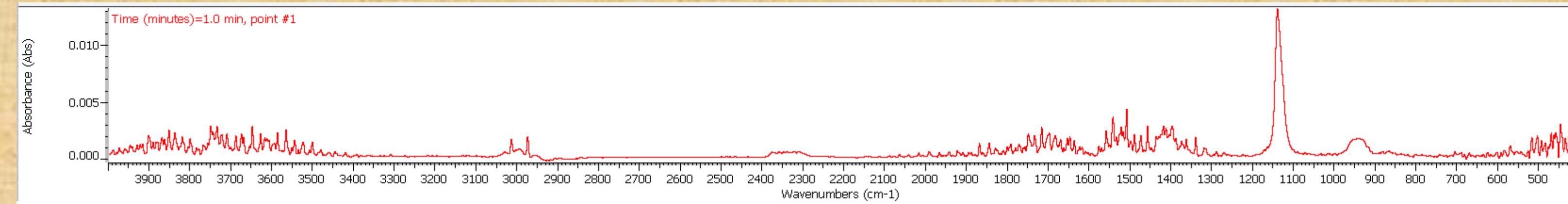


Figure 12: Spectrum collected at 1 minute from calibrated FTIR experiment; 40 minutes, 64 scans, 4 res, 0.6329 Optical Velocity. Asphalt Sample: 76-22 PAV only

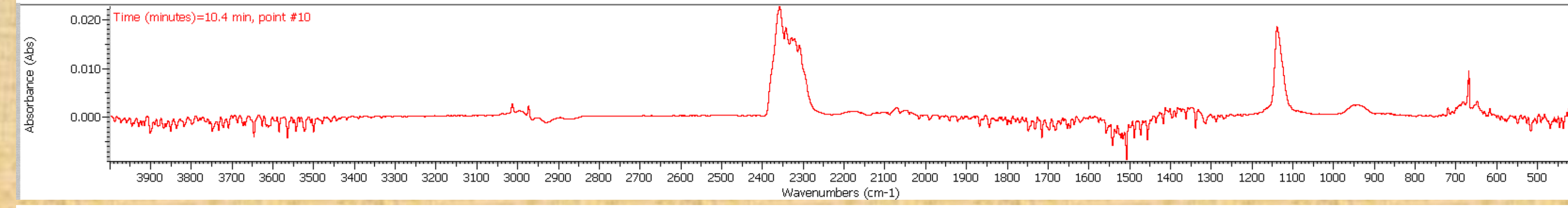


Figure 13: Spectrum collected at 10 minutes from calibrated FTIR experiment; 40 minutes, 64 scans, 4 res, 0.6329 Optical Velocity. Asphalt Sample: 76-22 PAV only

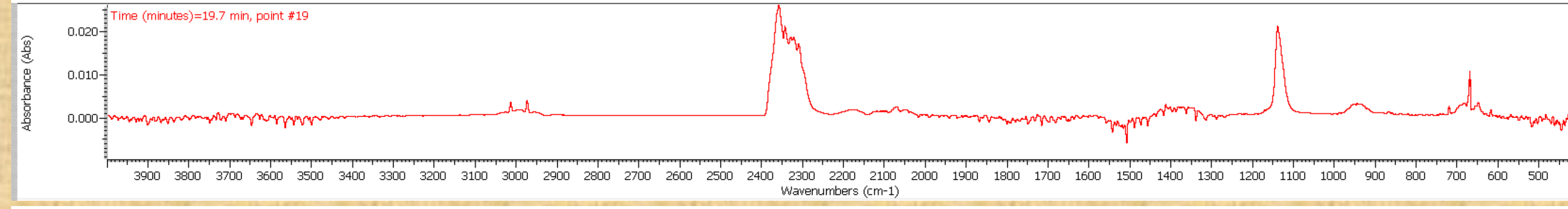


Figure 14: Spectrum collected at 20 minutes from calibrated FTIR experiment; 40 minutes, 64 scans, 4 res, 0.6329 Optical Velocity. Asphalt Sample: 76-22 PAV only

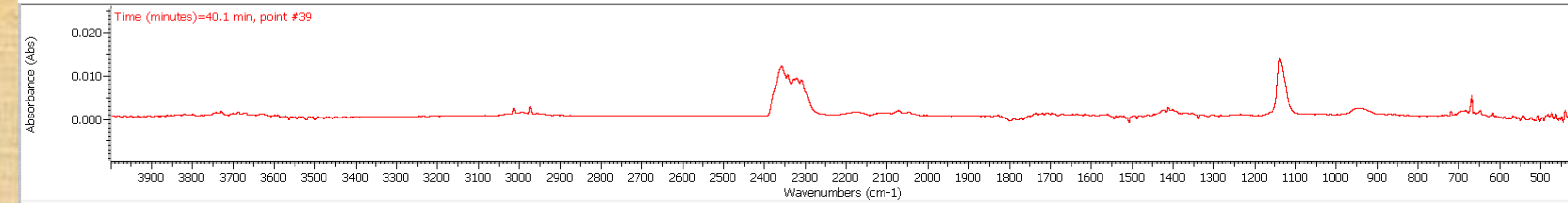


Figure 15: Spectrum collected at 40 minutes from calibrated FTIR experiment; 40 minutes, 64 scans, 4 res, 0.6329 Optical Velocity. Asphalt Sample: 76-22 PAV only

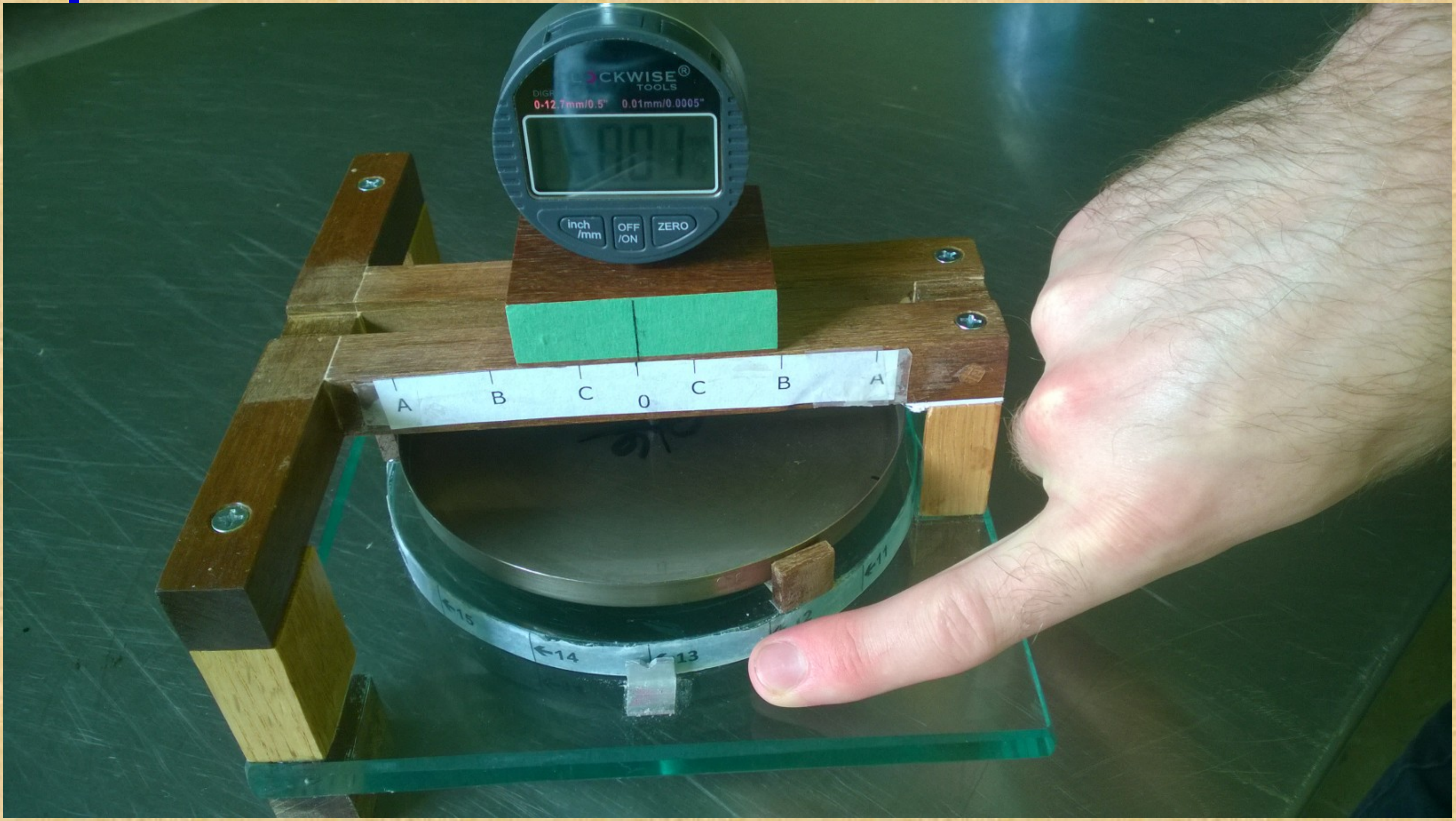
Bennert - Conclusions

- The degassing oven removes compounds such as phosphine oxide, alkyl chloride phosphine, and nitroso
- The compounds removed by the degassing oven from PAV binder indicates that aging is still taking place
- The vapor emitted from original binder being vacuumed in the degassing oven is nearly identical to the vapor emitted from original binder being aged in the RTFO
- Asphalt binder aged in the RTFO produces a vapor consisting of conjugated hydrocarbons, alcohols, amides and dienes
- The area of the carbonyl region increases throughout the duration asphalt binder is aged in the RTFO
- Original binder releases the most vapor in the degassing oven, followed by PAV only binder, followed by PAV + RTFO binder

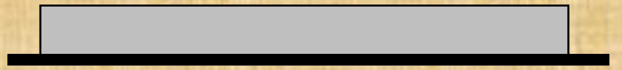
2. PAV Pan Warping and Levelness Issues

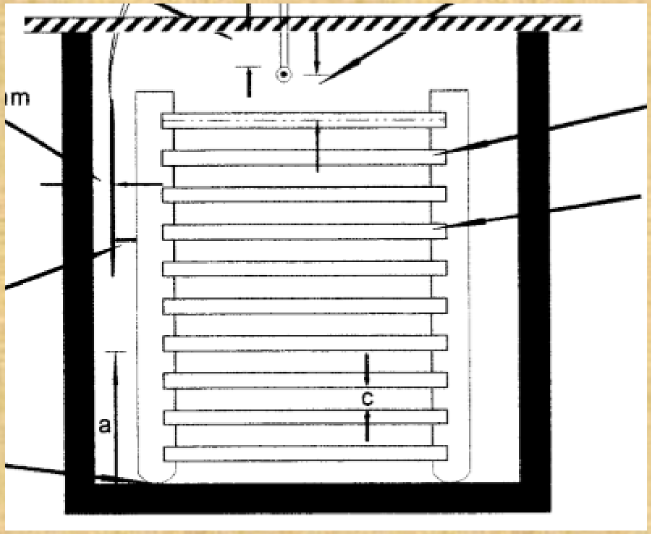
1. How can we measure pan flatness?
   a. Current techniques are not satisfactory
2. How do we obtain direct measure of flatness?
   a. Profile is needed
3. How do typical pan flatness errors affect test results?
   a. If flatness and property vs. thickness data are available it can be estimated
   b. Determined experimentally by using warped pans
4. How can we measure pan flatness in specification scenario?
5. What are realistic limits for specifying pan flatness?

Profile Gage



Three Support Techniques





Nature of Warping

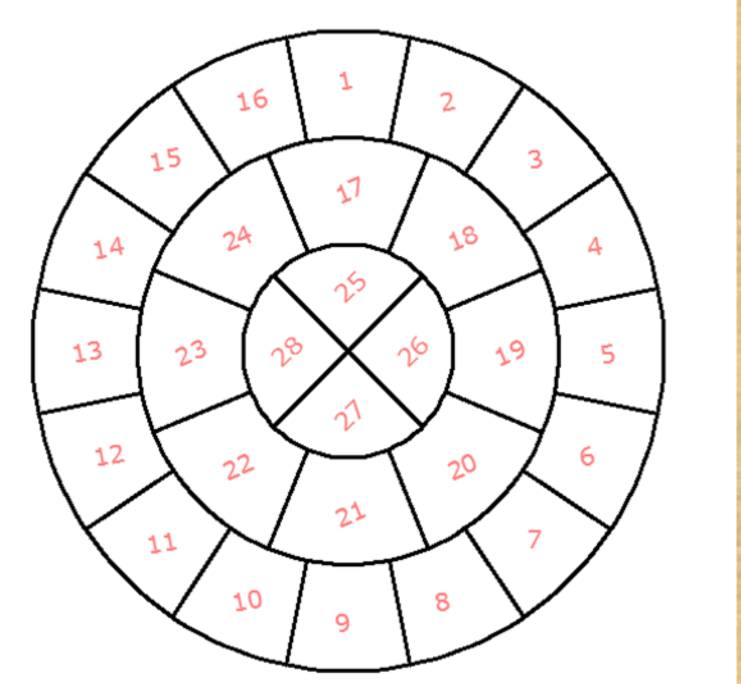
- Three Types Require Different Support
  - ✓ Shelf – support with quick-setting epoxy in stable position
    - One position
  - ✓ Three point - support on three studs
    - Rotate to multiple positions
  - ✓ Collar – support on collar
    - Need for multiple positions unknown
- Profiling jig has been modified to accommodate all three
- Warping may be a bow upward or downward
- More likely helical shape in perimeter
  - ✓ Like a compression washer

Pan Seeks Equilibrium Position on Shelf

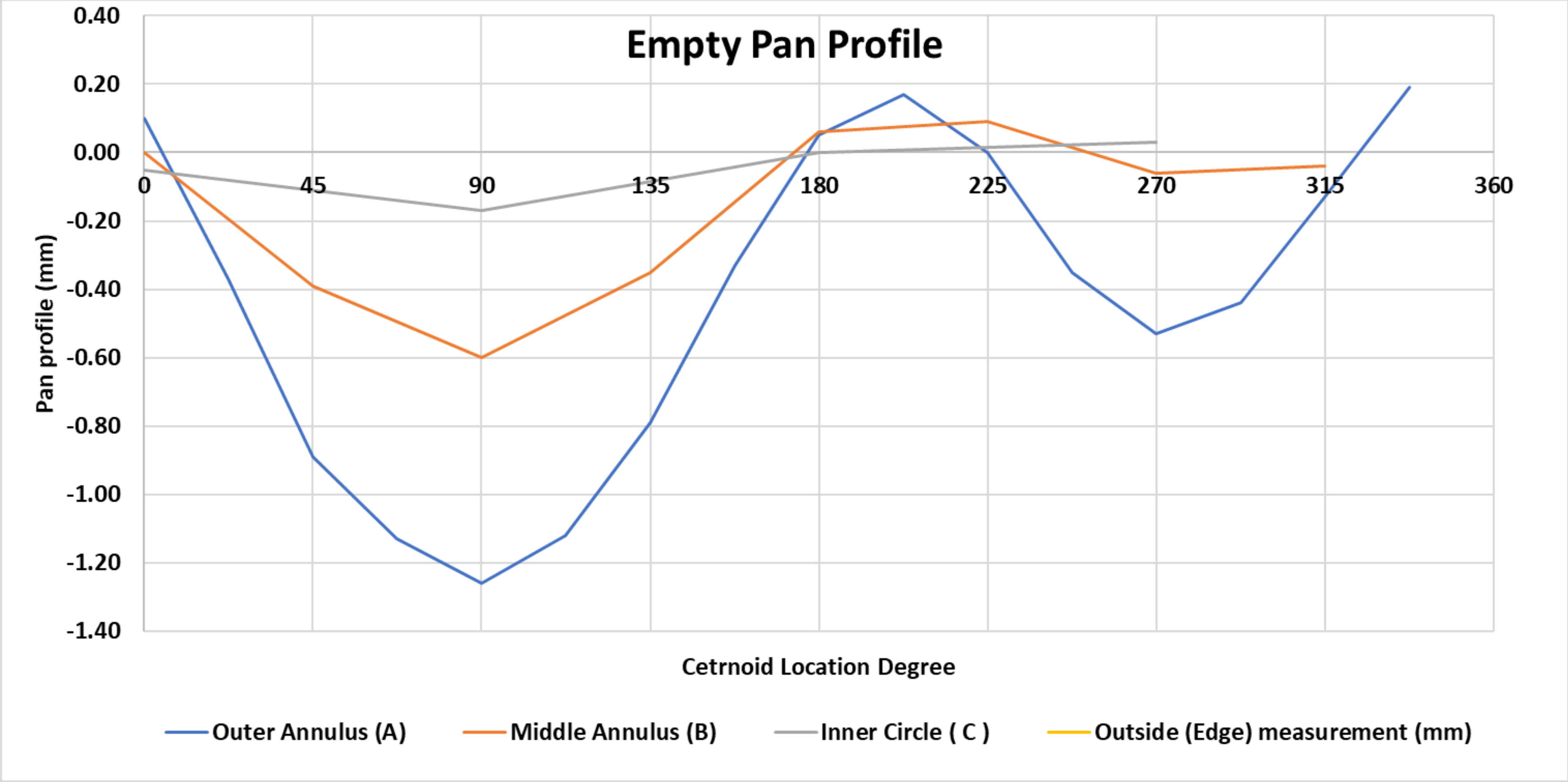
Pressure Point

Profiling and Calculated Effect

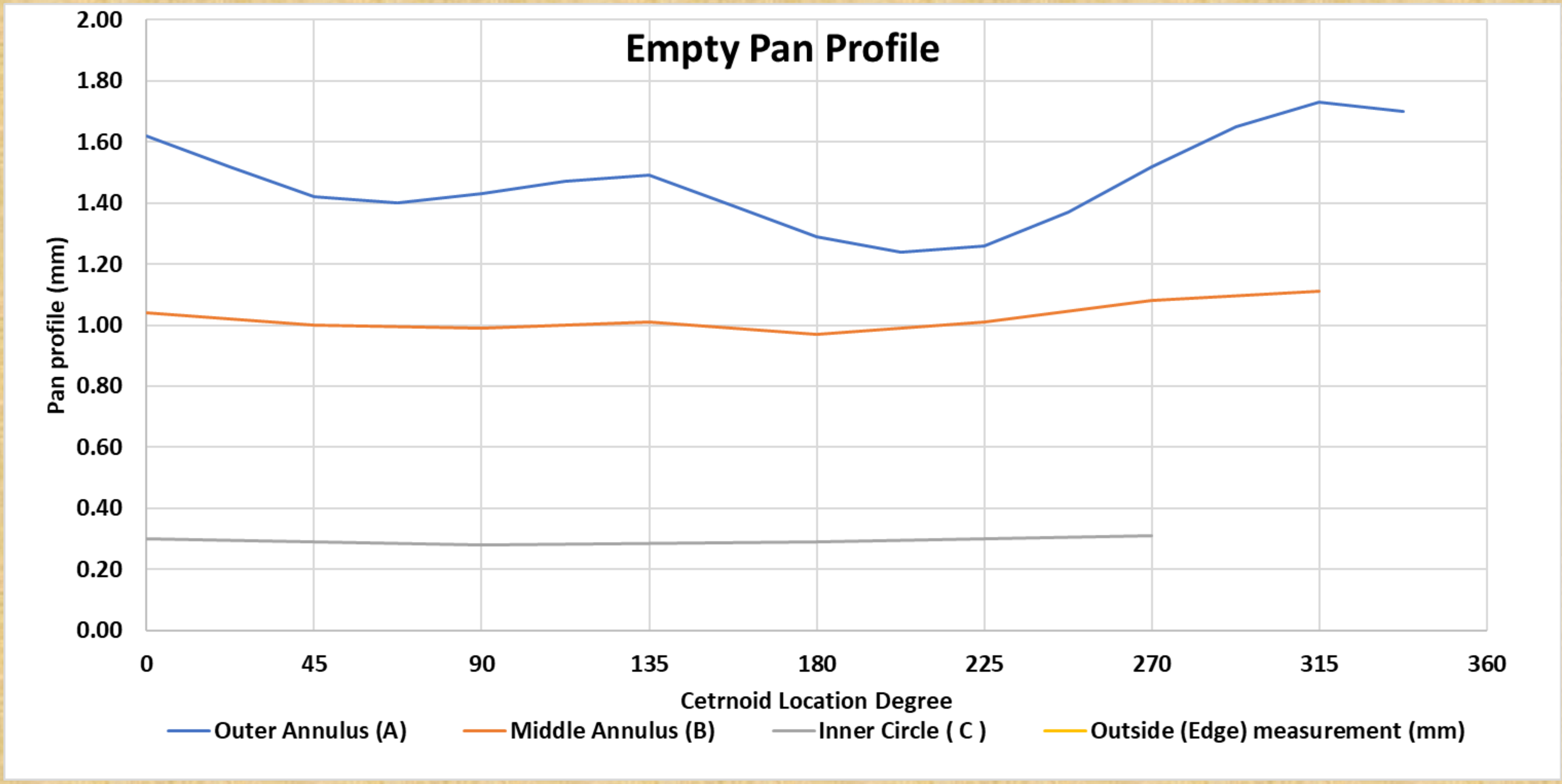
- Pan divided into 28 segments
- Profile measured at centroid of each profile
- Properties for each segment can be weighted as long as effect of thickness on properties is known
- Can also estimate effect of pan and vessel levelness



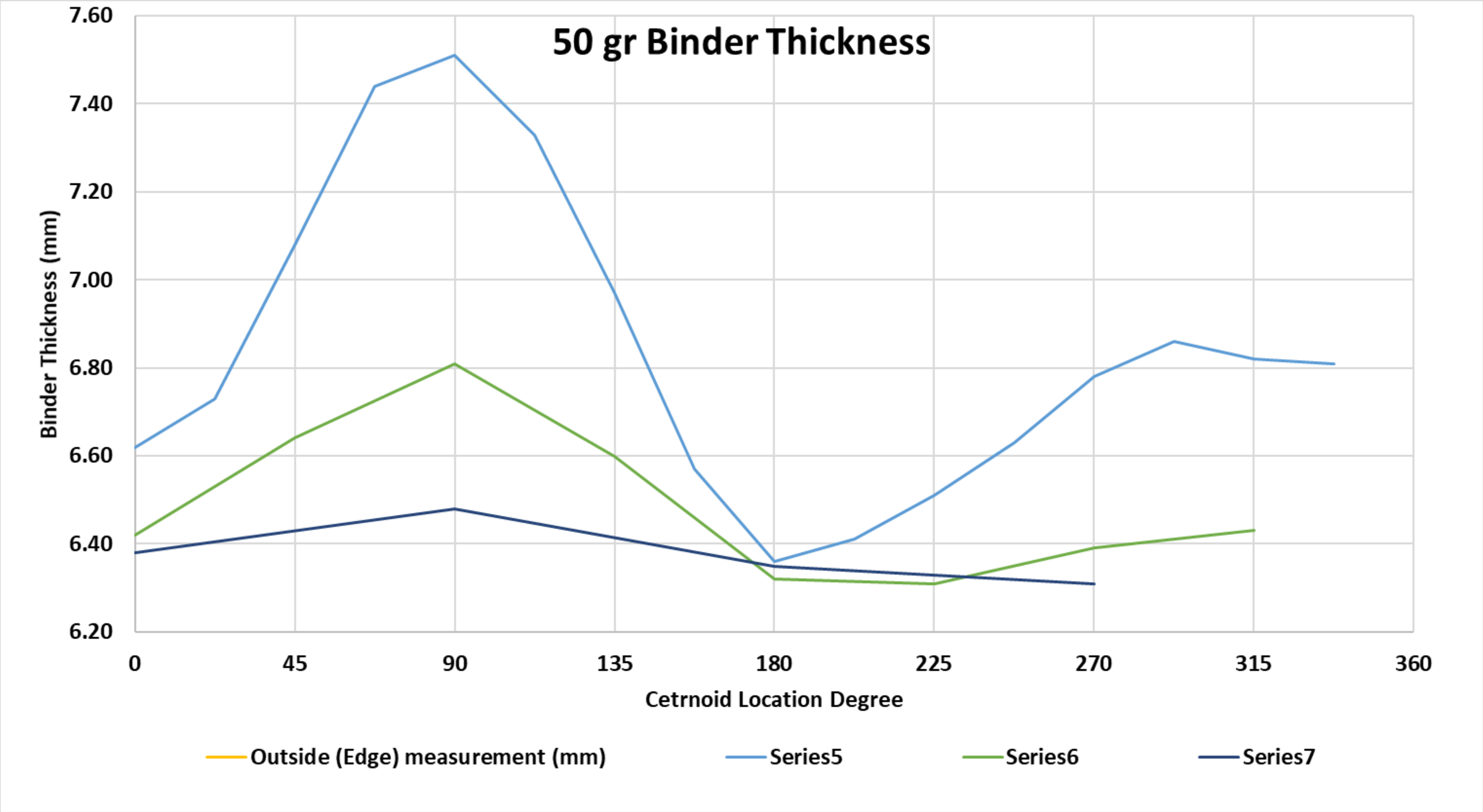
What Do the Profiles Look Like?



What Do the Profiles Look Like?

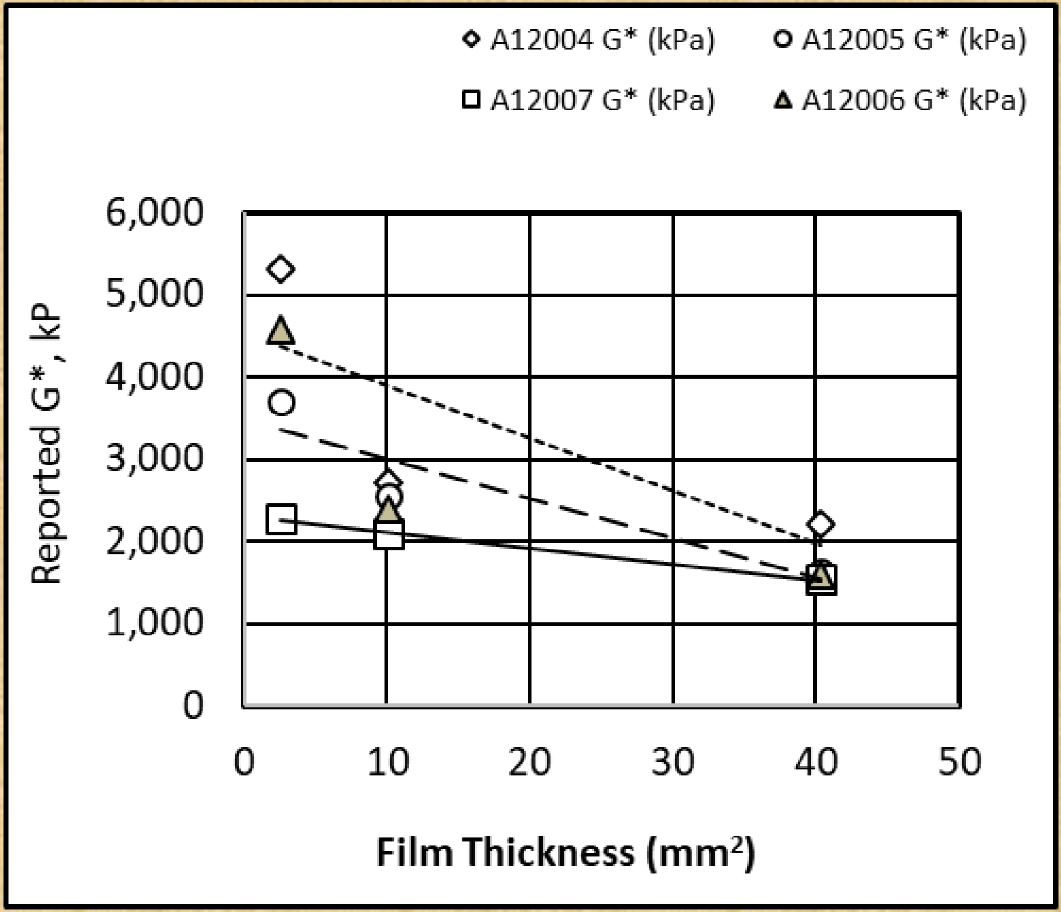


What Do the Profiles Look Like?



Available Data?

- ❑ Reliable data unavailable!
- ❑ Earlier conclusions based on viscosity data from TFO
- ❑ New data set is on its way courtesy of NCHRP 9-61



What Next?

- Complete the profiling with the different supports and updated profile jig
- Estimate the effect of thickness errors using the additional profiles and new data for SHRP AAC-1 and AAF-1
- Produce sufficient RTFOT residue for three participating laboratories
- Conduct experiments with three different pans, two binders and three laboratories
- Search out specification-suitable methods for estimating levelness
  - ✓ Three above laboratories to estimate levelness using specification-suitable methods

3. 4mm DSR Testing

- Task Force has been relatively inactive pending temperature control issues being investigated by Gerry Reinke
- Efforts to perform a ruggedness test and round-robin faltered due to lack of funding
  - ✓ Perhaps was premature until temperature control issues are solved.
  - ✓ Gerry will discuss on Wednesday
- General consensus is that Peltier plates are not sufficient for low temperatures without the addition of a convection oven

Work to date

- Procedures for measuring fixture compliance using both ice and epoxy have been written
- Two procedures for mounting test specimens have been written
  - ✓ This material can be circulated at this point
  - ✓ This can be done immediately
- Taskforces completed some limited round robin measurements
  - ✓ Designed to look at thermal equilibrium primarily
  - ✓ Questionable value given temperature control issues

Where Does the Task Group Go Next?

- ✓ Original charge also included a look at the 8 mm plate variability
  - This should be pursued given the ability to generate low temperature data on newer instruments with the 8 mm plate
- ✓ Further DSR Task Group activity should be pursued only with appropriate equipment upgrades
- ✓ DSR Task Group should pursue previous proposal for a ruggedness testing, training, and round robin
  - New source of funds for this effort appears promising