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Testing Asphalt Binders with Emphasis on Modified Asphalt Characterization

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"Understanding Modified Asphalt Binder Technology"

November 2014

How Asphalt Behaves

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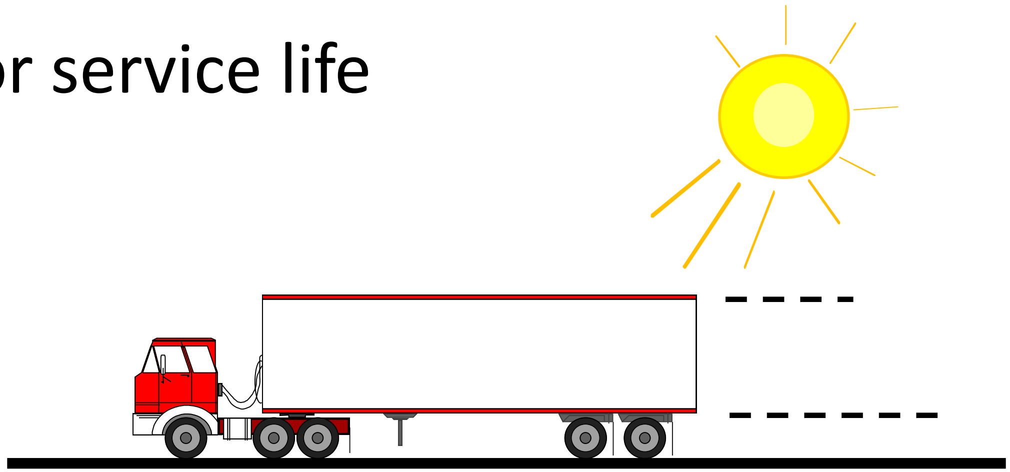
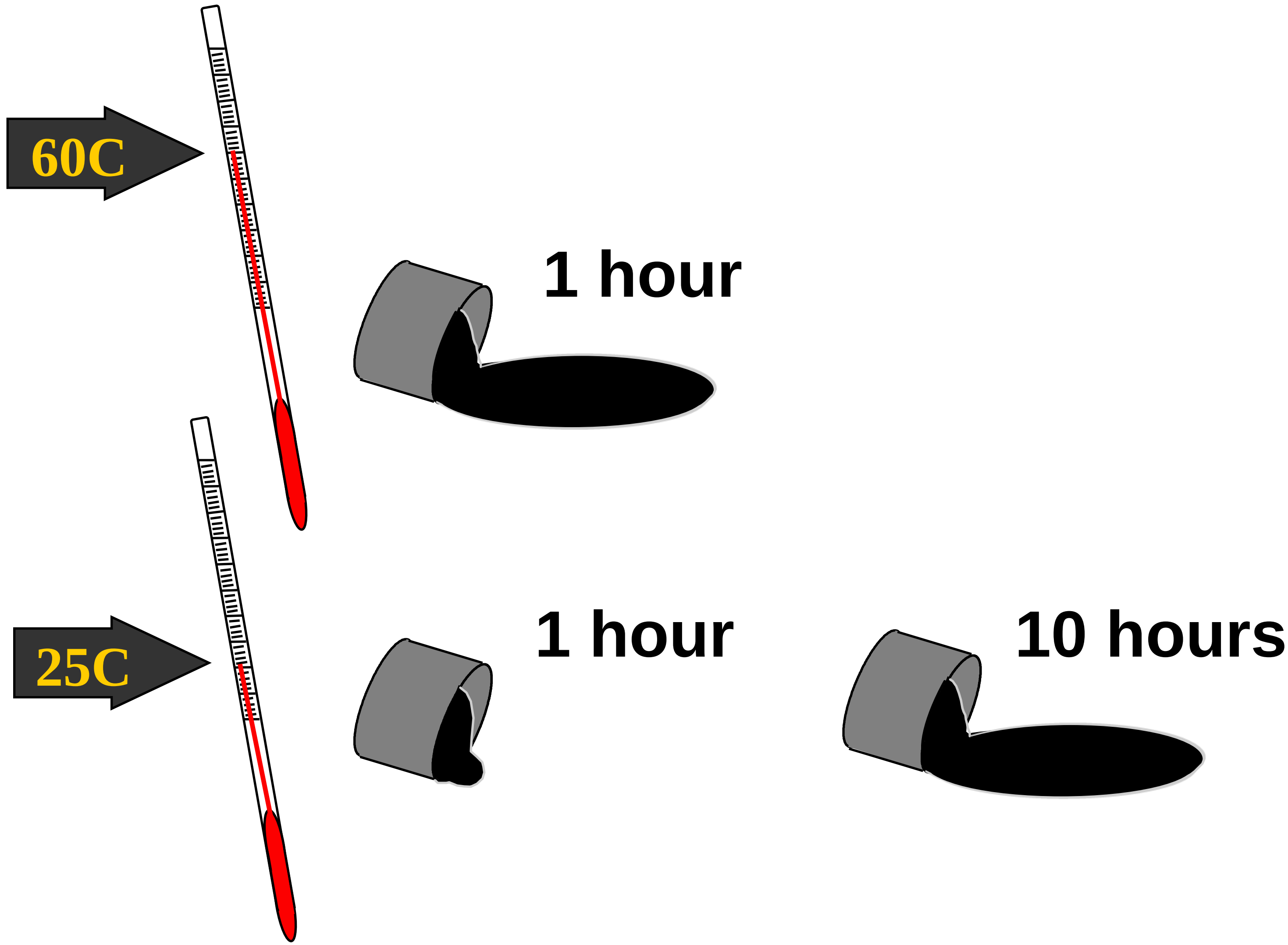
- Behavior is affected by :
  - Temperature
  - Time of Loading
  - Age of pavement or service life



Time-Temperature Superposition



Pavement Behavior – High Temperature

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- Permanent Deformation
- Mixture is Plastic
  - wheel path rutting
  - shoving at intersections
- Depends on…
  - asphalt cement (some)
  - mineral aggregate (some)
  - volumetric proportioning (some)


Rutting

Low Temperature Behavior

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- Low Temperature
  - cold climate
  - winter
- Rapid Loads
  - fast moving trucks

→ ***elastic solid***

Pavement Behavior - Low Temperature

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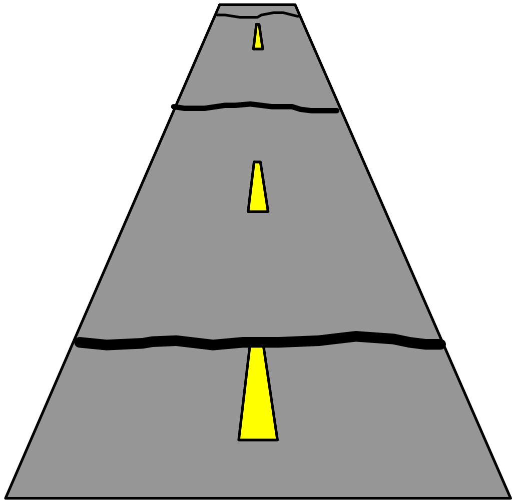
- Thermal Cracks
  - internal stresses induced by temperature change
  - stresses exceed strength
- Mixture is Brittle
  - transverse cracks
- Depends on…
  - asphalt cement (lots)
  - mineral aggregate (little)
  - volumetric proportioning (some)



Low Temperature Cracking

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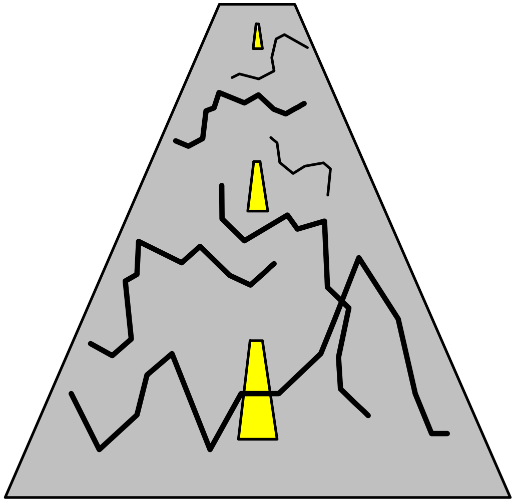
Binder Behavior - Aging

- Asphalt Reacts with Oxygen
  - “oxidative” or “age” hardening
  - During Construction - Short Term
    - hot mixing
    - placement and compaction
  - In Service - Long Term
    - hot climate worse than cool climate
  - Volatilization - Short Term
    - volatile components evaporate during construction

Pavement Behavior - Aging

- Durability Cracks
  - Mixture is brittle
  - Random, wandering cracks
  - Longitudinal
  - Depends on…
    - asphalt cement (some)
    - mineral aggregate (little)
    - volumetric proportioning (some)



Fatigue Cracking

Laboratory Handling and Storage

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- The properties of asphalt binders can be altered during handling and storage
  - These changes may be reversible or non-reversible depending upon cause
- The causes include…
  - Damaged or partially open cans
  - Heating during handling
  - Proper long-term storage has minimal effect
- Other factors

Causes of Change – Oxidation

- Oxygen reacts with asphalt cement
  - Molecular size increases
  - Polarity increases
  - The binder becomes stiffer
  - This reaction is not reversible
- Rate of reaction is highly dependent upon temperature
  - Rule of thumb – reaction rate doubles for every 10°C increase in temperature

Causes of Change – Volatilization

- Loss of lighter weight or more volatile molecules caused by heating
    - Causes the binder to become stiffer
    - This reaction is not reversible
- Minimized by following same precautions as listed above for oxidation
- Volatilization is probably of less concern than oxidation

Causes of Change – Steric Hardening

- Steric hardening is a reversible process that occurs at room temperature
  - Polar molecules become structured with time
  - This structuring increase binder stiffness
  - Steric hardening starts immediately upon cooling and continues at a reduced rate for an extended period of time (months? years?)
- The amount of steric hardening that occurs is binder-specific
  - Some binders show relatively small amounts

Controlling Steric Hardening

- Steric hardening is destroyed by heating
  - Referred to as annealing in test methods
- Always control the amount of time between sample pouring and testing
  - Limit the amount of time asphalt binder is held in silicone molds before testing
  - Holding a DSR test specimen in a silicone mold for several hours or more may be sufficient to pass a binder that would otherwise fail

Polymer Degradation

- Some polymers may degrade when heated at high handling temperatures
  - Temperature is polymer-specific
  - Use manufacturer recommendations for handling and do not overheat
- Polymer degradation cannot be reversed
  - Degradation causes a softening of the asphalt binder

Polymer Separation

- Separation during storage
  - Tendency is binder- and modifier-specific
  - Some systems are more stable than others
  - Separation may occur during storage
- Polymer tends to float to top giving “scum”
  - If this scum persists with stirring test results may not be representative
  - Removing “scum” removes polymer and test results are no longer representative

Heating Asphalt Binders – Precautions

- Always heat asphalt samples/cans in an oven
  - Avoid hot plates, heating mantles, etc.
- Always heat at the lowest temperature and for the shortest time possible
  - Follow manufacturer's instructions
- Avoid heating in thin layers
  - e.g., near-empty cans, shallow tins
- Cover the heated container when possible
  - The cover may not be effective in reducing oxidation but it will help prevent contamination from dust, etc.

Testing of Asphalt Cements

- Characteristics of Asphalt Cements
  - Consistency
    - term used to describe the viscosity or degree of fluidity of asphalt at any particular temperature
    - varies with temperature
      - necessary to define an equivalent temperature or an equivalent consistency when comparing temperature-consistency characteristics of asphalt cements

Testing of Asphalt Cements

- Characteristics of Asphalt Cements
  - Purity
    - Refined petroleum asphalts are usually more than 99.5% soluble in carbon disulfide
    - Organic material and impurities are inert
  - Safety
    - Free from moisture (foaming)
    - Increase in temperature leads to increase in fumes
      - Increased potential for flash in presence of spark or flame

Traditional Tests

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- Viscosity
  - Absolute
  - Kinematic

  Consistency
- Penetration *Consistency*
- Flash Point *Safety*
- Aging
  - Thin Film Oven (TFO)
  - Rolling Thin Film Oven (RTFO)

  Consistency
- Ductility *Consistency*
- Solubility *Purity*
- Specific Gravity *???*

Viscosity

- Absolute Viscosity
  - ASTM D2171 (AASHTO T202)
  - Conducted at 60°C (140°F)
  - Uses partial vacuum to induce flow through capillary tube
- Kinematic Viscosity
  - ASTM D2170 (AASHTO T201)
  - Conducted at 135°C (275°F)
  - Uses gravity to induce flow through capillary tube

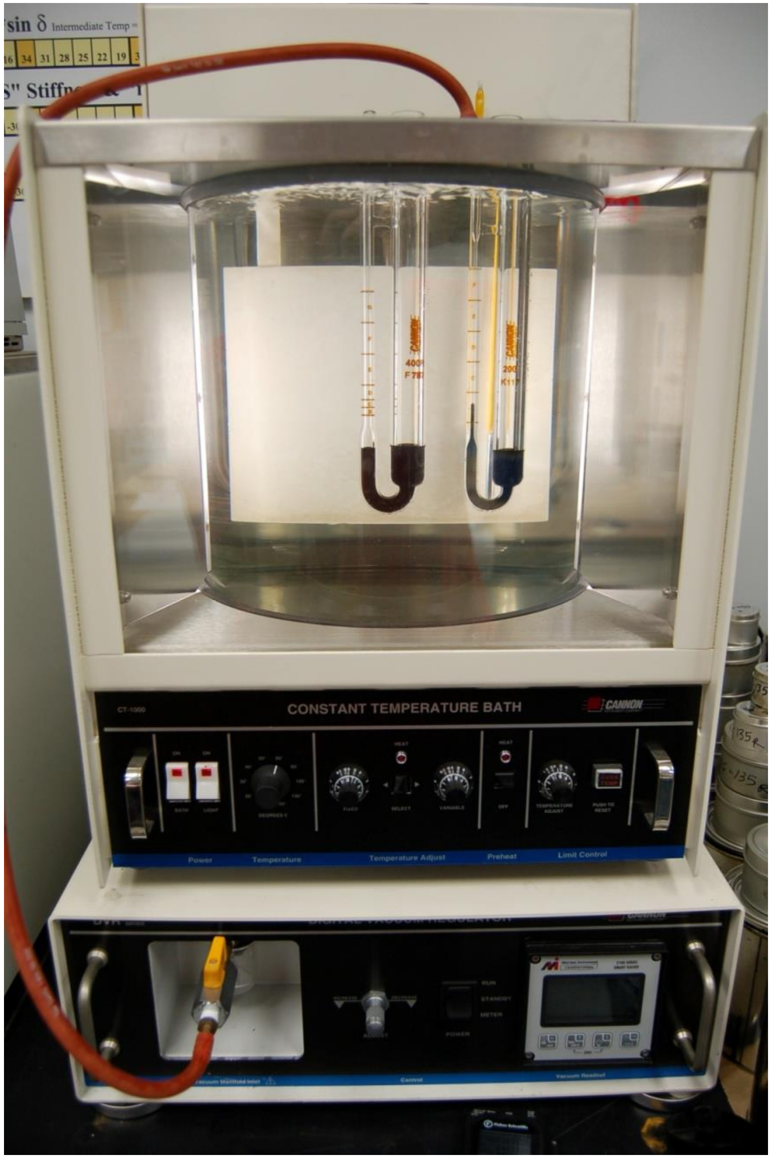
Viscosity

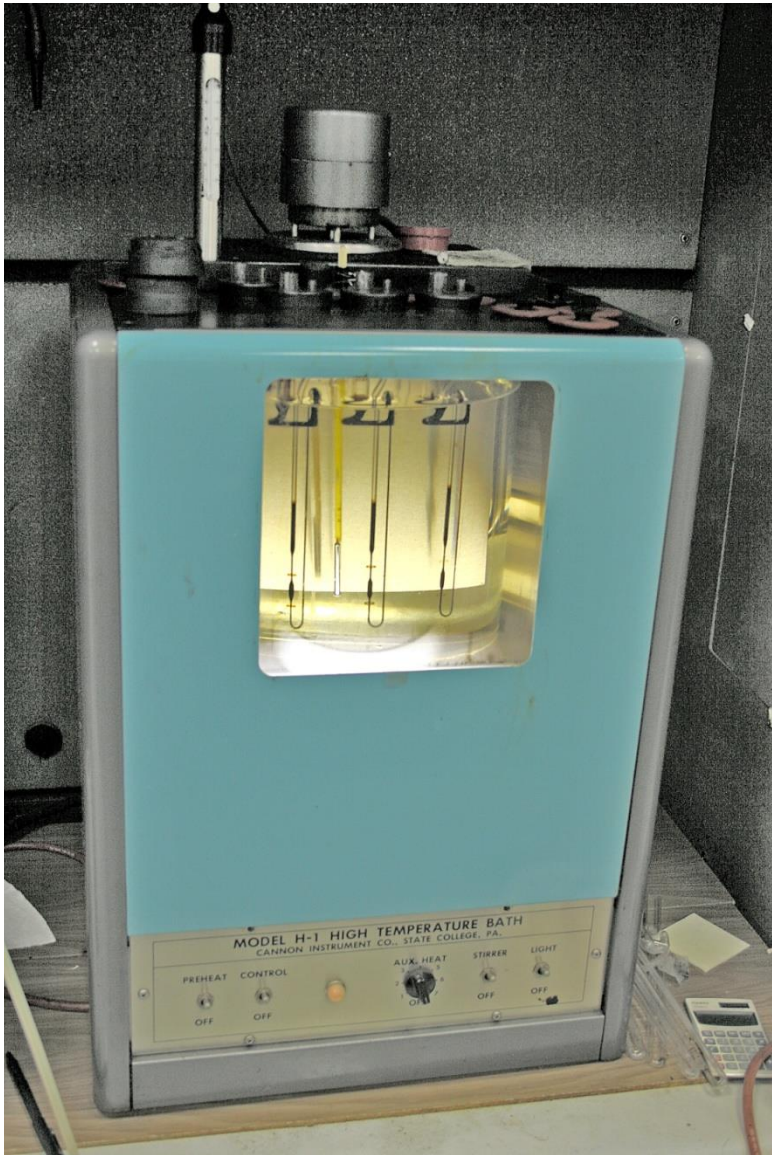
- Apparent Viscosity
  - ASTM D4957
  - Conducted at 60°C (140°F)
  - Uses partial vacuum to induce flow through capillary tube
  - Collect data through all bulbs
    - Not just bulb where the time equals or exceeds 60 seconds

Asphalt Cement

- Viscosity Graded Asphalt
  - 60°C (140°F) selected to simulate in-service temperature of asphalt pavements
  - 135°C (275°F) selected to simulate mixing and laydown temperature for HMA

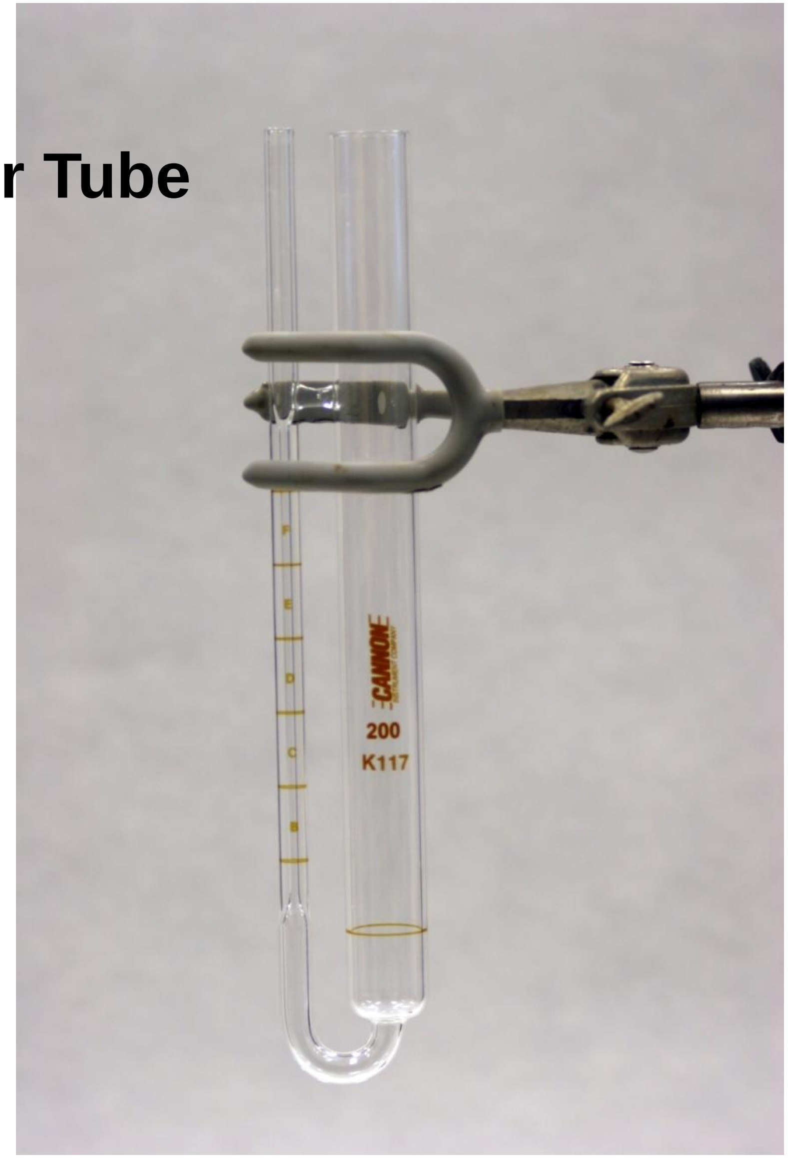
Viscosity


CONSTANT TEMPERATURE BATH
CANNON
Power
Temperature
Temperature Adjust
Preheat
Limit Control


MODEL H-1 HIGH TEMPERATURE BATH
CANNON INSTRUMENT CO., STATE COLLEGE, PA.
PREHEAT
CONTROL
AUX. HEAT
STIRRER
LIGHT
OFF

Viscosity

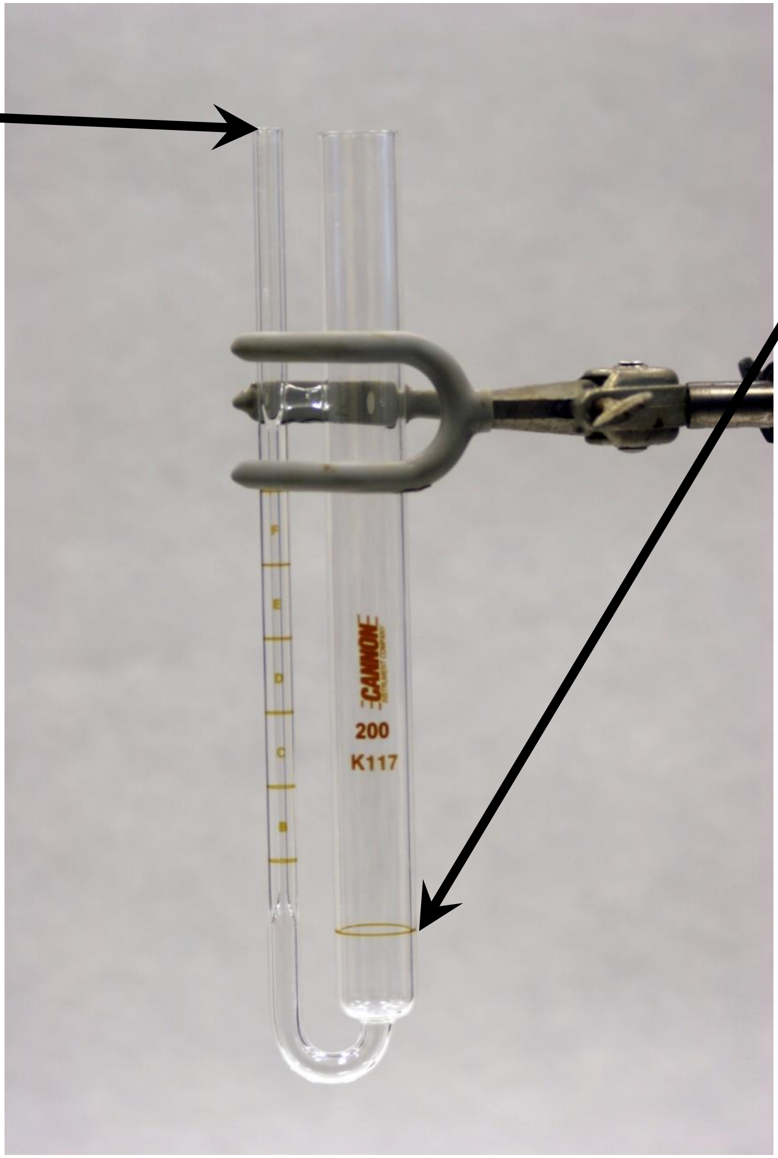
Vacuum Viscometer Tube



Zeitfuchs Cross-Arm Viscometer Tube

Absolute Viscosity

Vacuum applied to capillary tube to draw asphalt upwards



Asphalt poured in big end of tube to filling line

Absolute Viscosity

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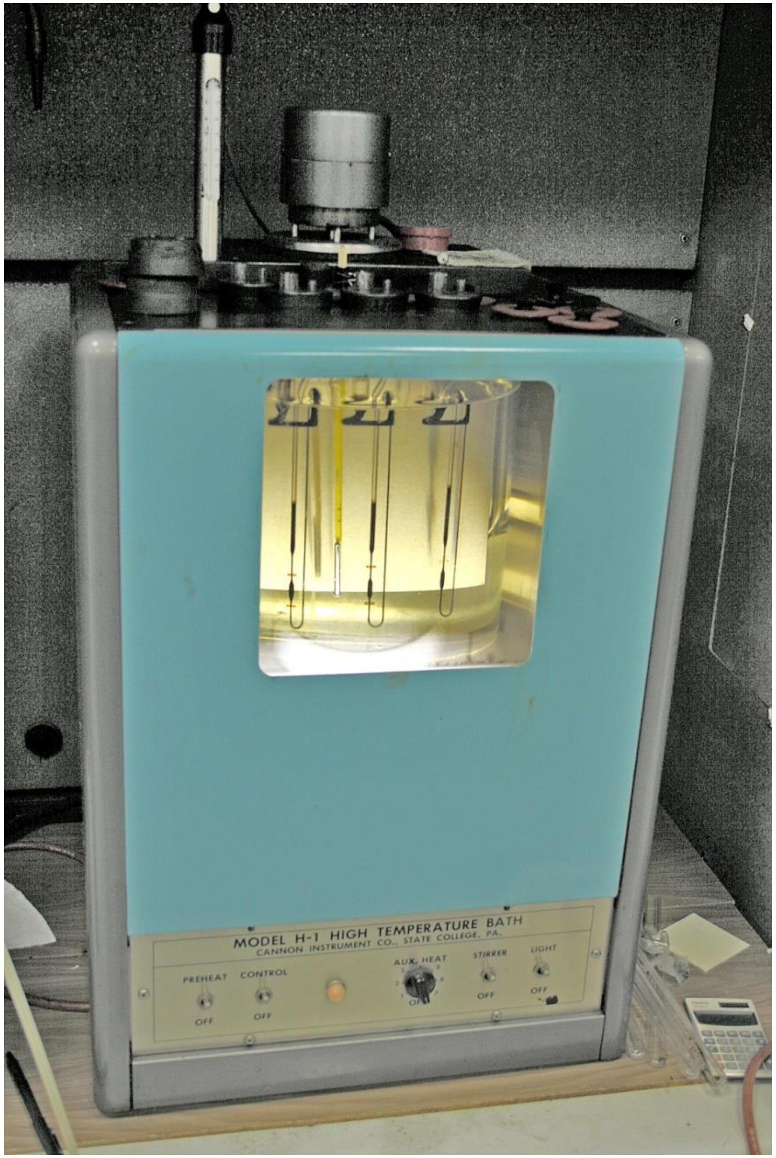
- Viscometer Tubes
  - Calibrated “bulbs”
    - Calibration constants
  - Time to flow through a “bulb” (between timing marks) measured
    - Minimum 60 seconds
    - Viscosity = Constant x Time
  - Different Tube Sizes
    - 50, 100, 200, 400R, 800R
    - Smaller tube = softer asphalt binder

Kinematic Viscosity

- Viscometer Tubes
  - Different shape and designation, but same purpose
  - Time to flow through a “bulb” (between timing marks) measured
    - Minimum 60 seconds
    - Viscosity = Constant x Time
  - No Vacuum
  - No Water Bath
    - Clear oil



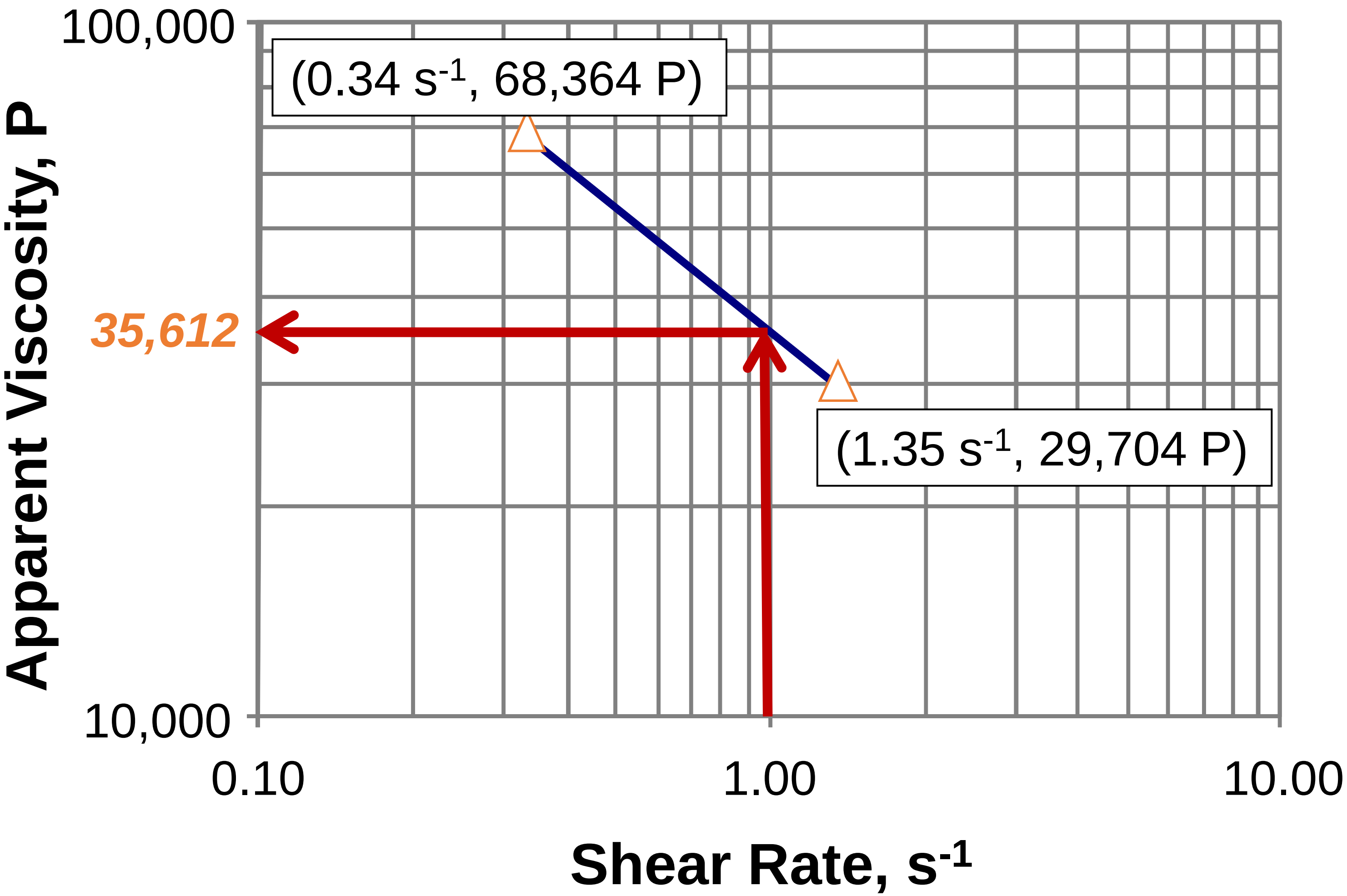
Apparent Viscosity

- Absolute Viscosity
  - Assumption that the material is Newtonian
    - viscosity remains constant regardless of the shear rate
  - Some modified asphalt binders can exhibit significant non-Newtonian behavior
    - measured viscosity is a function of the shear rate
    - different shear rate = different viscosity result

Apparent Viscosity

- Apparent Viscosity (ASTM D4957)
  - allows the user to better understand the behavior of non-Newtonian asphalt binders
- Procedure
  - Similar to absolute viscosity
  - Preheated asphalt sample poured into vacuum capillary viscometer tube until its level reaches the filling line.
  - Filled viscometer tube placed back in an oven for a short time.

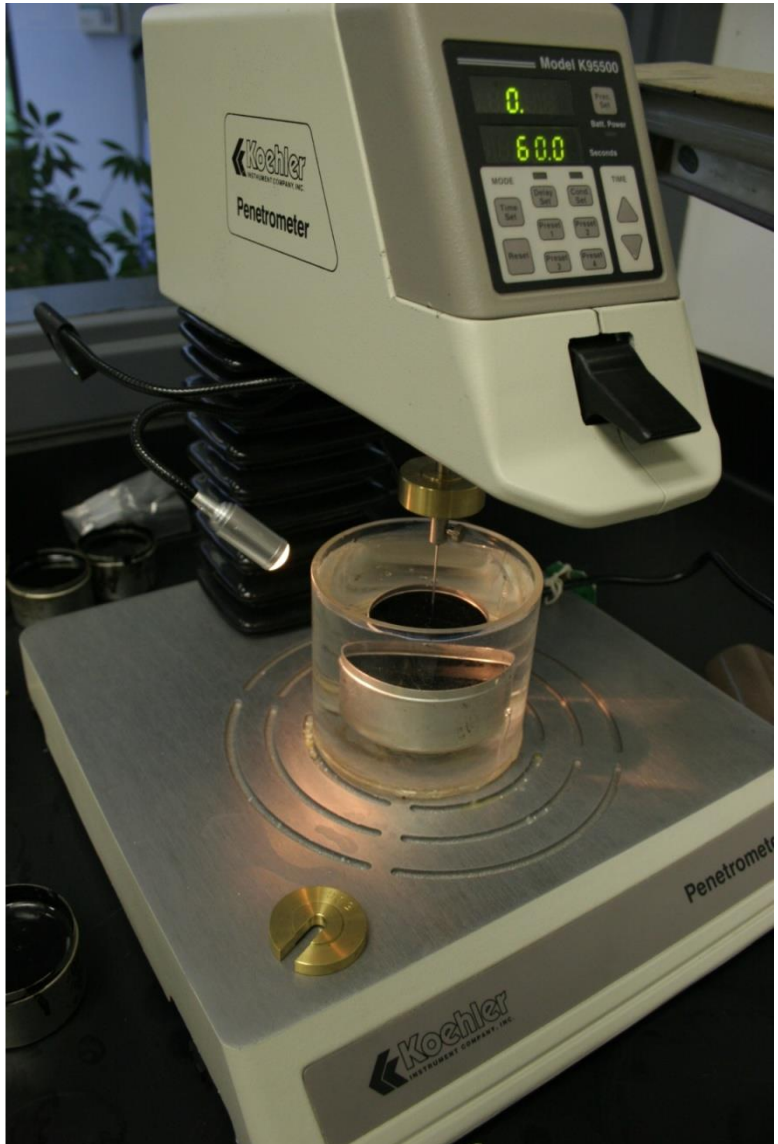
Apparent Viscosity



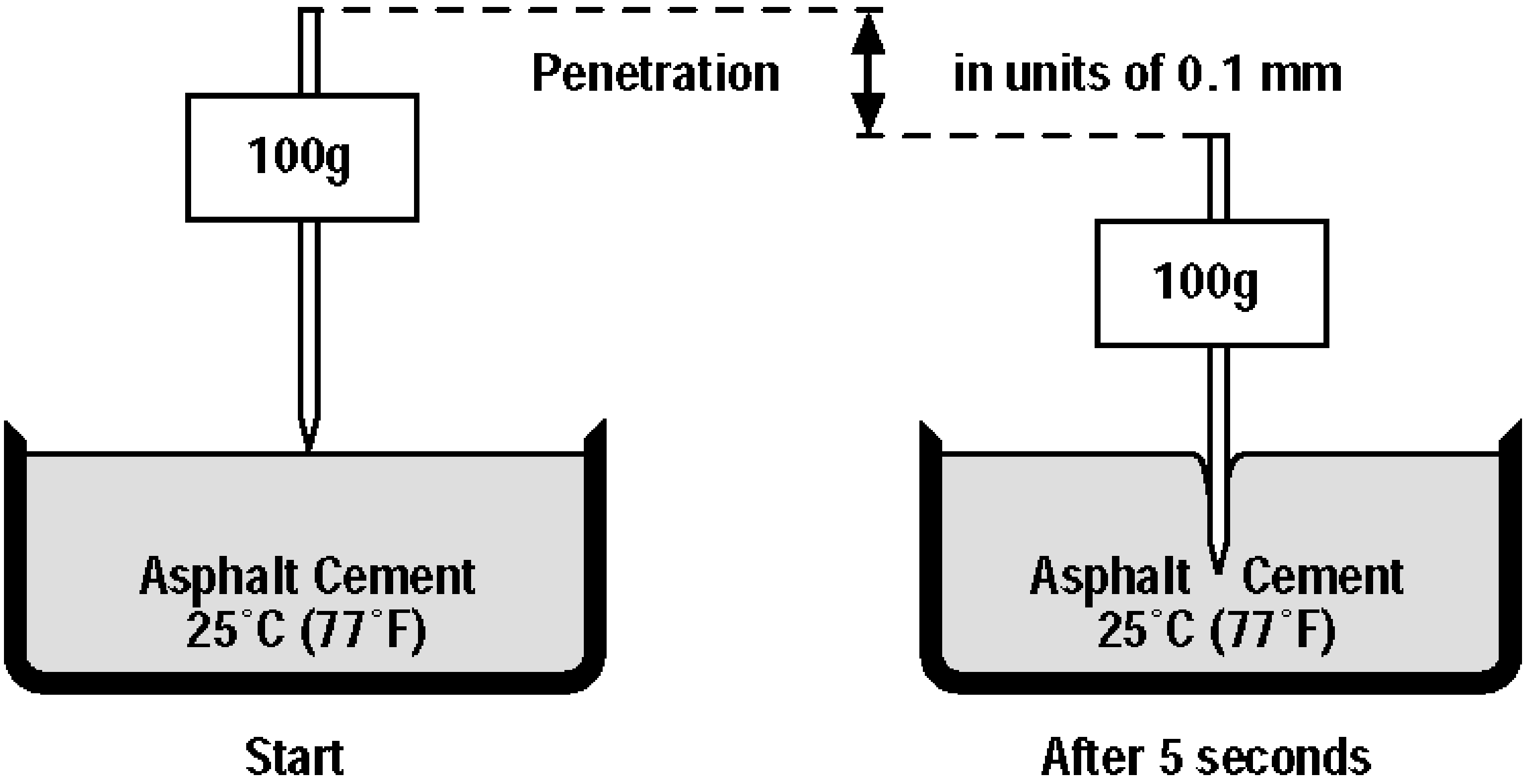
Penetration

- Penetration
  - ASTM D5 (AASHTO T49)
    - One of oldest asphalt tests
  - Standard needle allowed to penetrate into sample under specified loading conditions
    - 25°C – 100 grams, 5 seconds
    - 0°C – 200 grams, 60 seconds
    - 46°C – 50 grams, 5 seconds
  - Depth of penetration is recorded in 0.1-mm units (dmm)
  - Three penetration readings per test

Penetration



Penetration



Flash Point

- Cleveland Open Cup (COC) Flash Point
  - ASTM D92 (AASHTO T48)
  - Heat sample at prescribed rate
    - Rate changes as temperature approaches flash point
      - Initially 10°C/min to 20°C/min
      - When within ± 56°C of expected flash point reduce rate to 4°C/min to 7°C/min
      - Rate must be 4°C/min to 7°C/min for last 28°C before flash point

Flash Point

- Cleveland Open Cup (COC) Flash Point
  - Record temperature at which flash occurs
    - Occurs instantaneously
    - Must be closely observed
  - Use caution…asphalt products containing water or moisture may foam when heated

Flash Point:
Occurrence of Flash

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Aging

- Asphalt Binders Used for HMA
  - Subjected to heat and air during production
    - Thin films coating aggregate
    - Causes oxidation, volatilization
      - Loss of "light end" mass during process
- Aging Tests
  - Thin Film Oven (TFO)
  - Rolling Thin Film Oven (RTFO)

Aging

- Thin Film Oven (TFO)
  - ASTM D1754 (AASHTO T179)
  - Historically more widespread use
  - Used to determine mass loss
    - Volatilization that can be expected to occur during HMA production
    - Now termed "mass change"
      - Oxidation during test (mass gain) overcomes volatilization (mass loss)
  - Used to produce aged residue for other physical property tests

Thin Film Oven Aging

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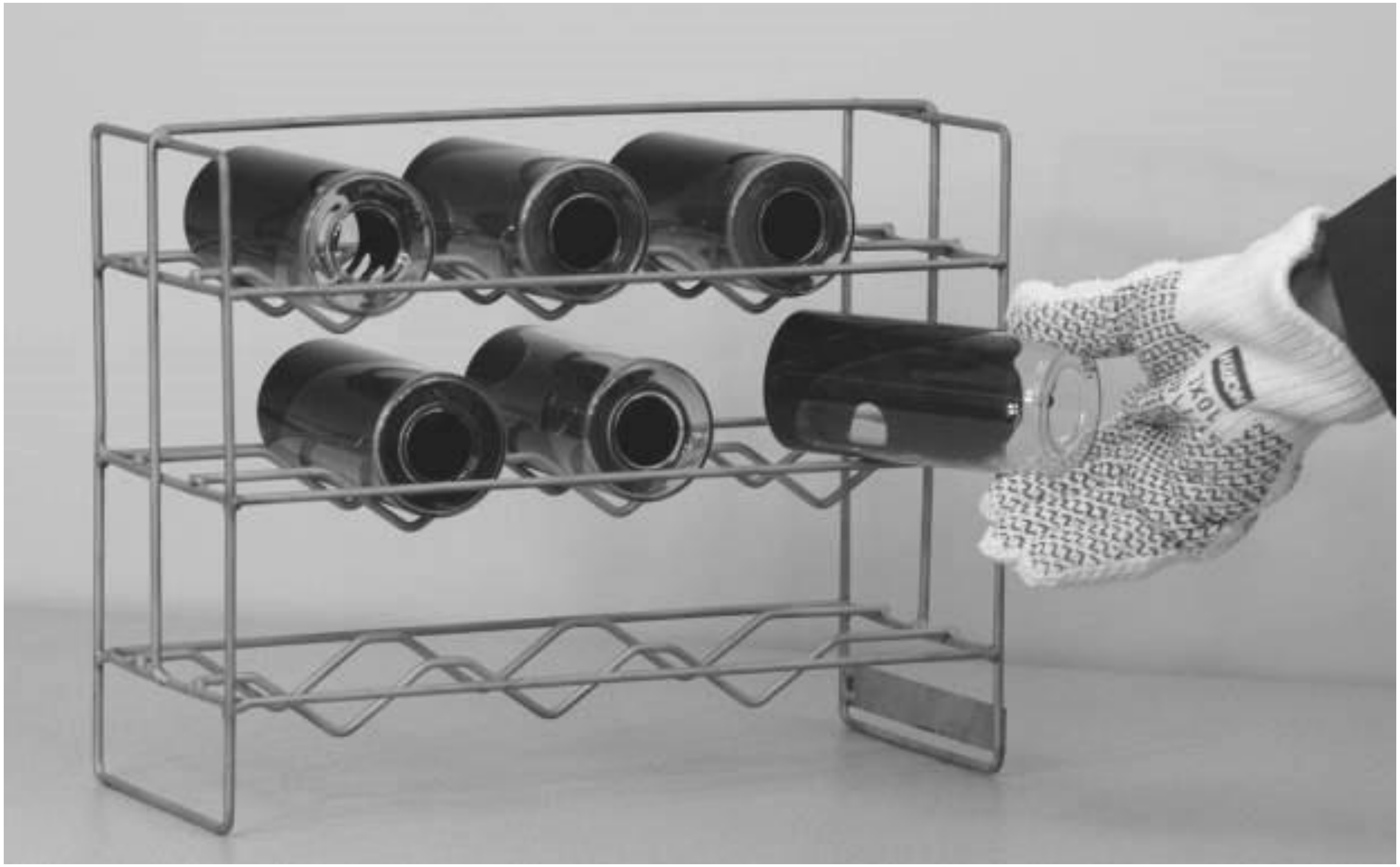
Aging

- Rolling Thin Film Oven (RTFO)
  - ASTM D2872 (AASHTO T240)
  - Historically used in Western United States
  - Used to determine mass loss
    - Volatilization that can be expected to occur during HMA production
    - Now termed "mass change"
      - Oxidation during test (mass gain) overcomes volatilization (mass loss)
  - Used to produce aged residue for other physical property tests

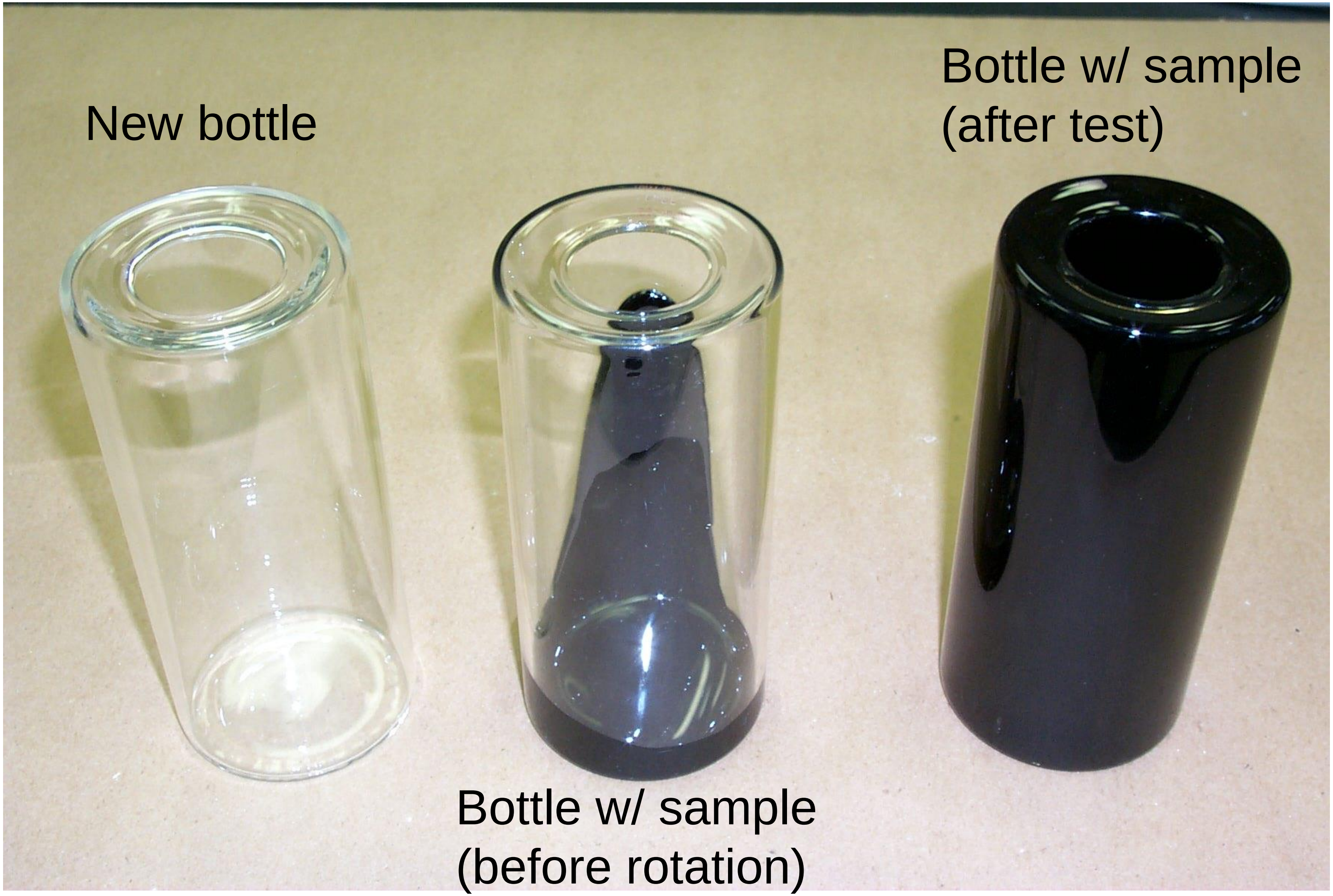
Rolling Thin Film Oven Aging

RTFO



Containers at multiple levels (AASHTO)

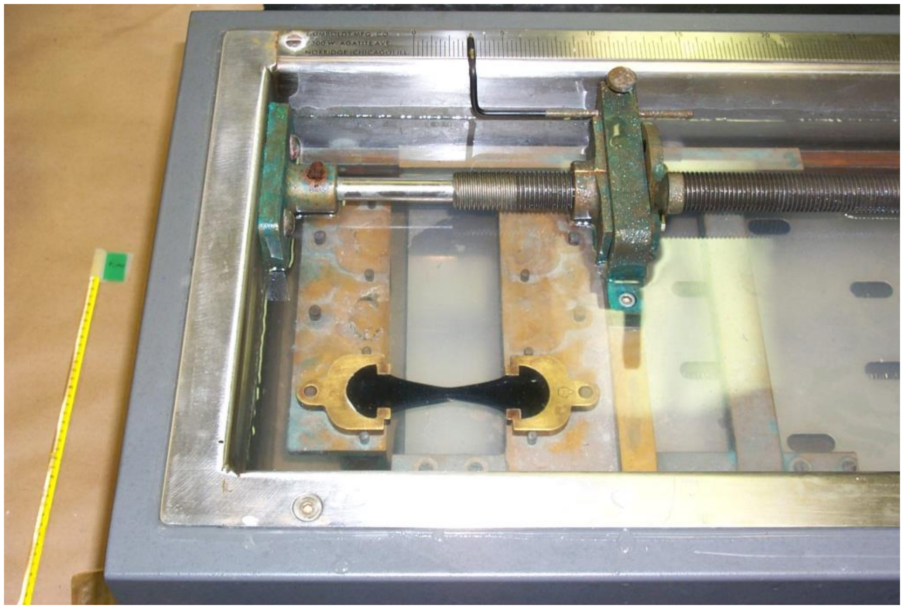
RTFO Aging

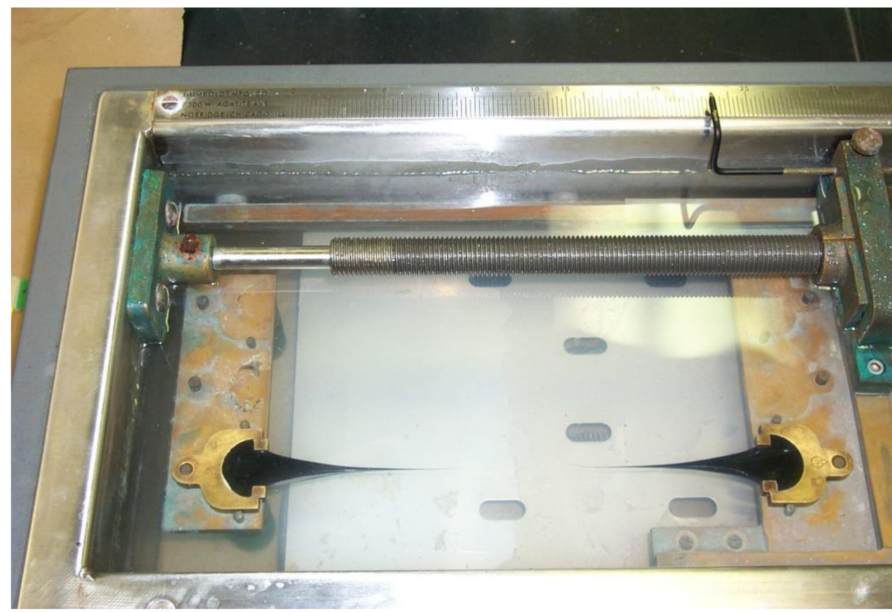

New bottle
Bottle w/ sample
(after test)
Bottle w/ sample
(before rotation)

Ductility

- Procedure
  - ASTM D113 (AASHTO T5)
- Purpose
  - Determine degree of ductility (i.e., ability to stretch without fracture) at intermediate temperature
    - Considered an important characteristic of asphalt binders by some engineers
    - Presence or absence of ductility often considered more significant than the actual degree of ductility
      - Some asphalt binders having a high degree of ductility have also been found to be more temperature-susceptible

Ductility



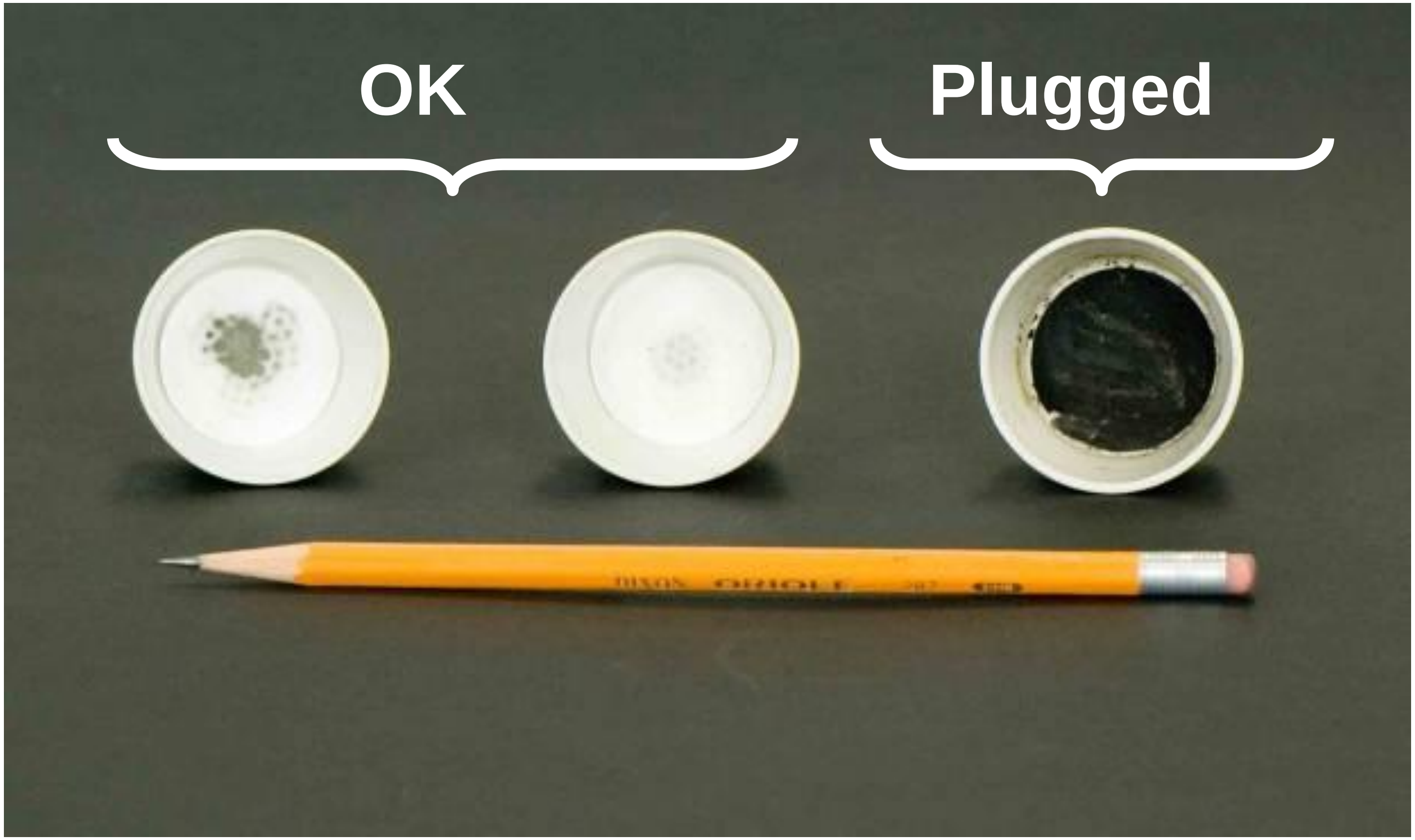


- “Dog-bone” shaped specimen
- Tested at 25°C
- Displacement Rate of 5 cm/min

Solubility

- Procedure
  - ASTM D2042 (AASHTO T44)
- Purpose
  - Measure of the purity of the asphalt binder
    - Portion of the asphalt binder that is soluble in carbon disulfide (trichloroethylene) represents the active cementing constituents
    - Inert components—such as salts, free carbon, or non-organic contaminants—are insoluble

Solubility: Examples of Filters

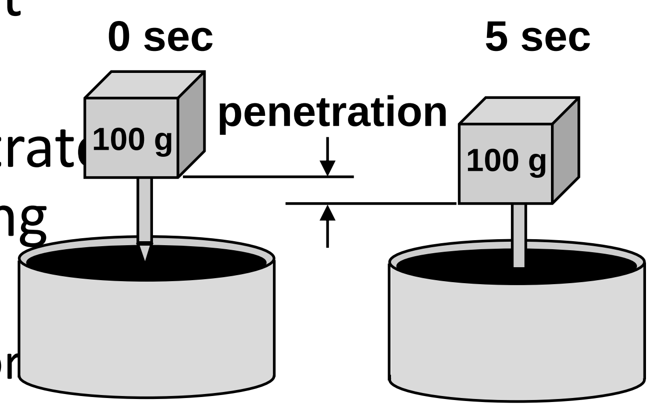


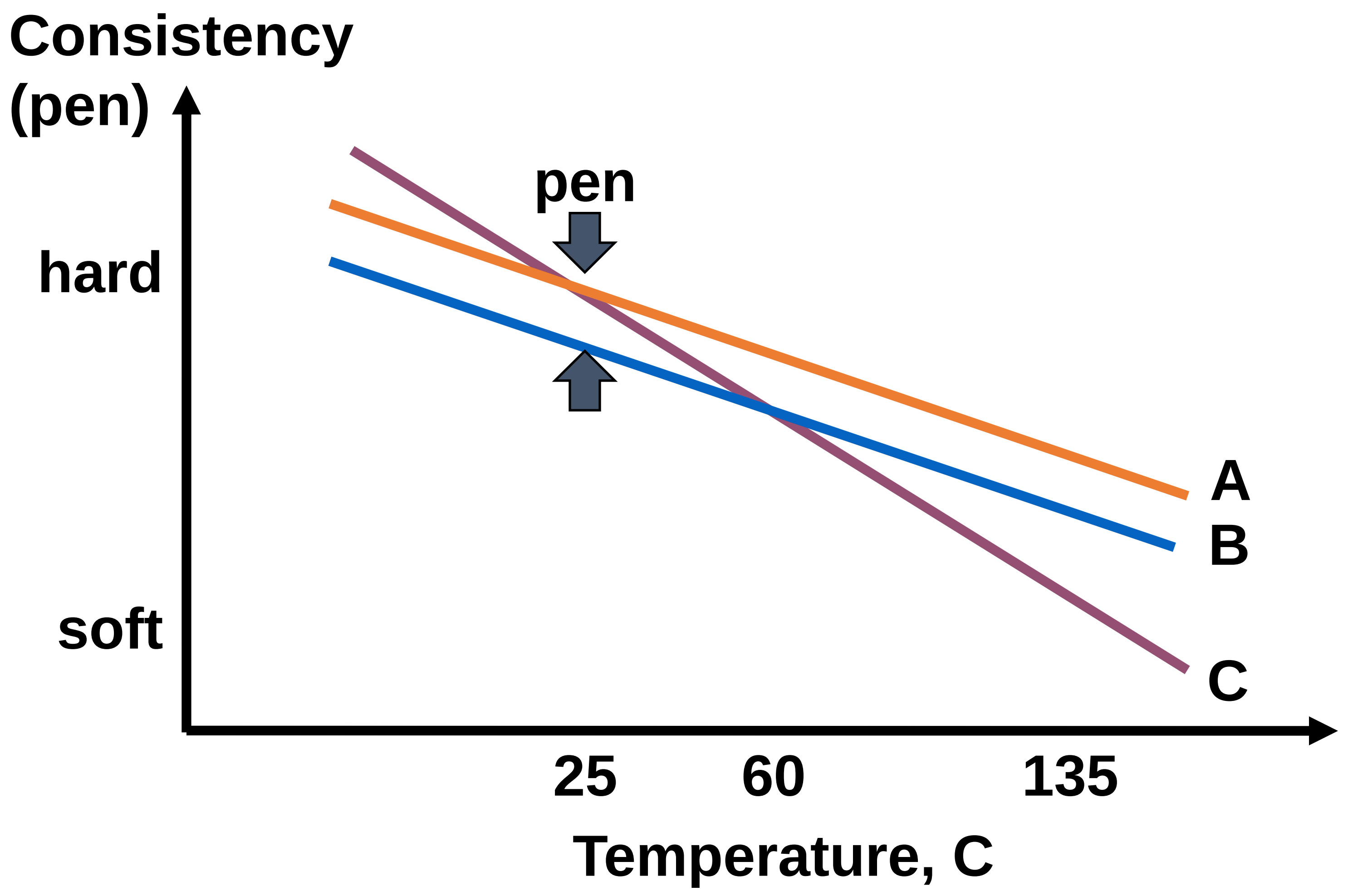
Specifications: Asphalt Cement

- Graded by several systems before the Strategic Highway Research Program
  - Viscosity (AC)
  - Viscosity (AR)
  - Penetration (PEN)
- Primarily used for hot mix asphalt (HMA) paving

Specifications: Asphalt Cement

- Penetration Graded Asphalt (PEN)
  - ASTM D946 (AASHTO M20)
  - Grading based on Penetration test at 25°C
    - Standard needle allowed to penetrat into sample under specified loading conditions
      - at 25°C, load of 100 grams is used for seconds
    - Original (unaged) asphalt is tested
    - Empirical test

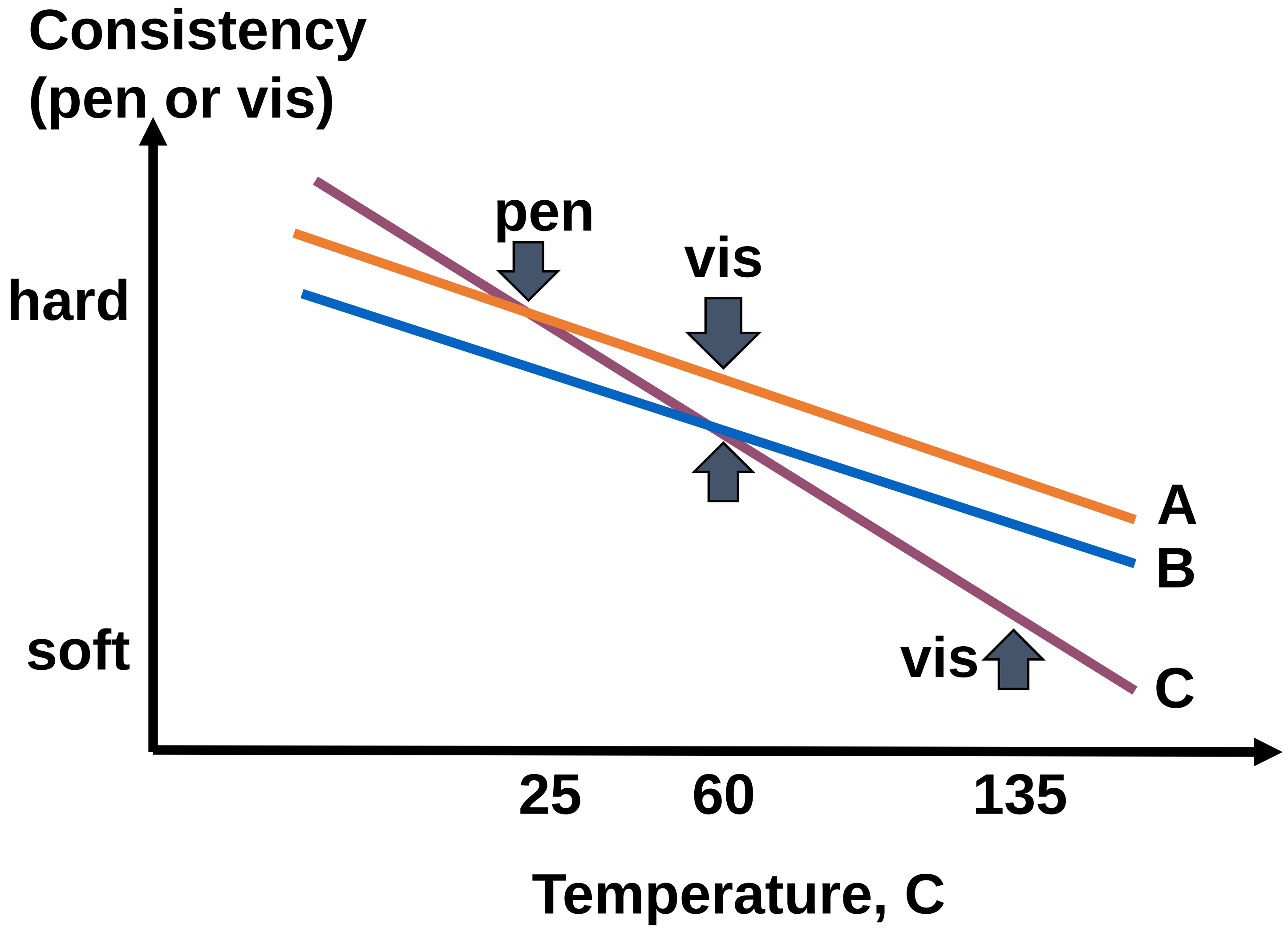




Consistency
(pen)
pen
hard
soft
A
B
C
25
60
135
Temperature, C

Specifications: Asphalt Cement

- Viscosity Graded Asphalt (AC)
  - ASTM D3381 (AASHTO M226)
    - Tables 1 and 2
  - Most commonly used (pre-SHRP) classification system in US
  - Based on Viscosity
    - Measure of the resistance of a material to flow
    - Absolute viscosity at 60°C (140°F)
    - Kinematic viscosity at 135°C (275°F)


Consistency
(pen or vis)
hard
soft
pen
vis
vis
A
B
C
25
60
135
Temperature, C

Problems with Previous Systems

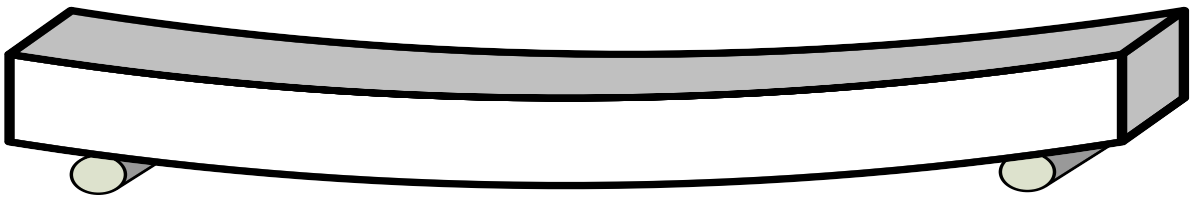
- Penetration
  - empirical measure of viscous and elastic effects
- Viscosity
  - viscous effects only
- No Low Temperature Properties Measured
- Problems Characterizing Modified Asphalt Binders
  - Specification proliferation
- Long Term Aging not Considered

PG System - Based on a New Concept

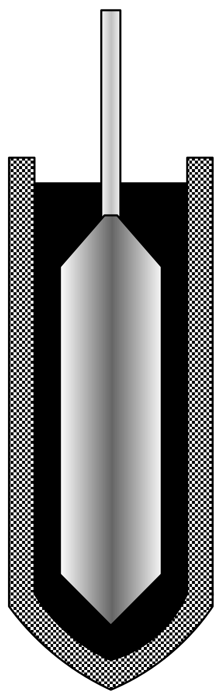
"the values of the specification criteria that warrant against distress are independent of temperature, but the values must be obtained at different temperatures according to climate."

This implies test measurements at temperatures and loading rates consistent with conditions existing in the pavement

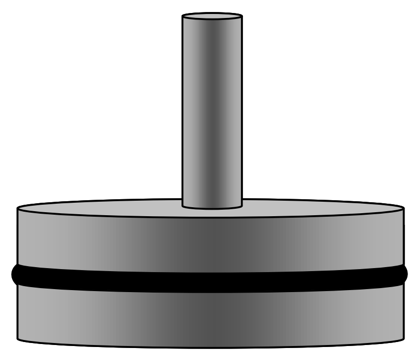
The SHRP PG Binder Tests



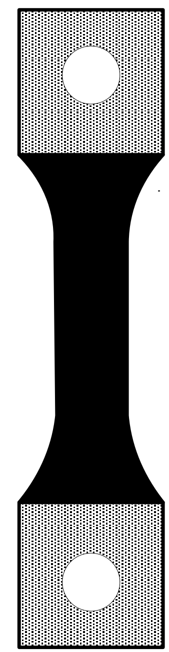
BBR



RV



DSR



DTT

The PG Binder Tests

- Rotational Viscometer (RV), ASTM D4402 (AASHTO T316)
  - for measuring viscosity at elevated temperatures
- Dynamic Shear Rheometer (DSR), ASTM D7175 (AASHTO T315)
  - for determining the modulus (stiffness) of asphalt binders at intermediate and upper pavement temperatures

The PG Binder Tests

- Bending Beam Rheometer (BBR), ASTM D6648 (AASHTO T313)
  - to determine the modulus (stiffness) of asphalt binders at lower pavement temperatures
- Direct Tension Test (DTT), ASTM D6723 (AASHTO T314)
  - to determine the tensile strength of asphalt binders at lower pavement temperatures

The PG Binder Tests

- Rolling Thin Film Oven Test (RTFOT), ASTM D2872 (AASHTO T240)
  - to simulate the aging that occurs during mixing and compaction
  - Adoption of existing test
  - Replaced the Thin Film Oven Tests (TFOT)
- Pressurized Aging Vessel (PAV), ASTM D6521 (AASHTO R28)
  - to simulate long-term in-service field aging
  - Adoption of existing research test

Pavement Distress in the PG System

- Rutting in the upper layers caused by inadequate shearing resistance
- Load-associated fatigue cracking that progresses from the underside of the pavement to the surface
- Low-temperature thermal cracking caused a single drop in the pavement temperature

Aging Considerations

- PG binder specification is designed to test materials that are representative of in-service conditions
  - Requires laboratory conditioning procedures to simulate binder conditions immediately after construction and after in-service aging

Short-Term Aging

- RTFO to represent short-term aging
  - Adapted from an existing California method
  - Simulates a batch plant operating at ±150°C
    - Represents a typical condition
    - May not represent drum plants operating at lower temperatures

Long-Term Aging

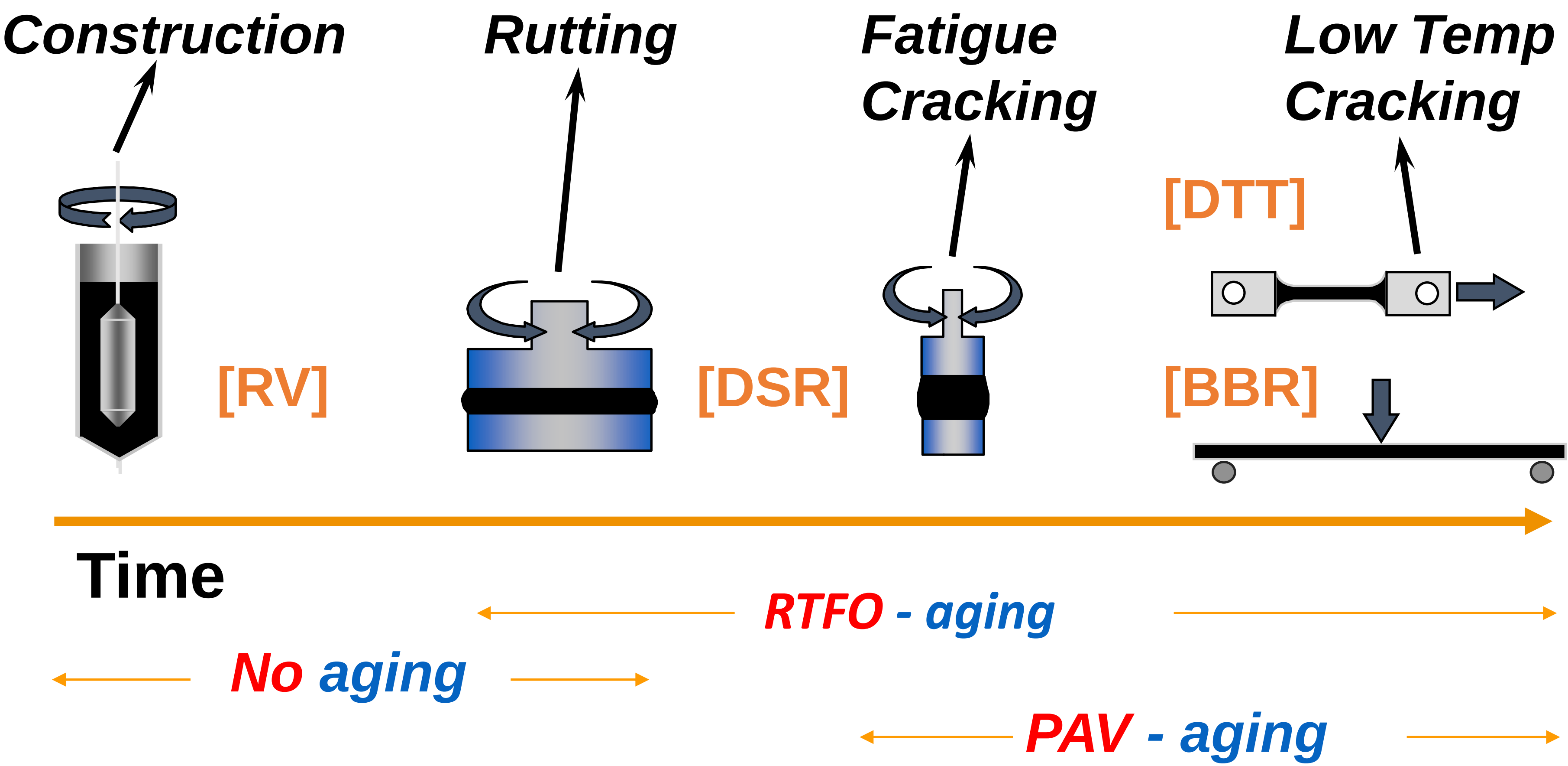
- PAV to simulate long-term aging
  - Increased temperature and pressure accelerates aging
  - Increased temperature increases the rate of aging
  - Increased pressure makes oxygen available to asphalt cement molecules thereby increasing rate of aging

Performance-Related Requirements

- Shearing resistance to resist traffic loads
  - Upper specification temperature
  - $G^*/\sin\delta \geq 1.00$ kPa (Tank)
  - $G^*/\sin\delta \geq 2.20$ kPa (RTFO residue)
- Resistance to increased stiffness after long-term aging
  - Intermediate specification temperature
  - $G^*\sin\delta \leq 5000$ kPa (PAV residue)

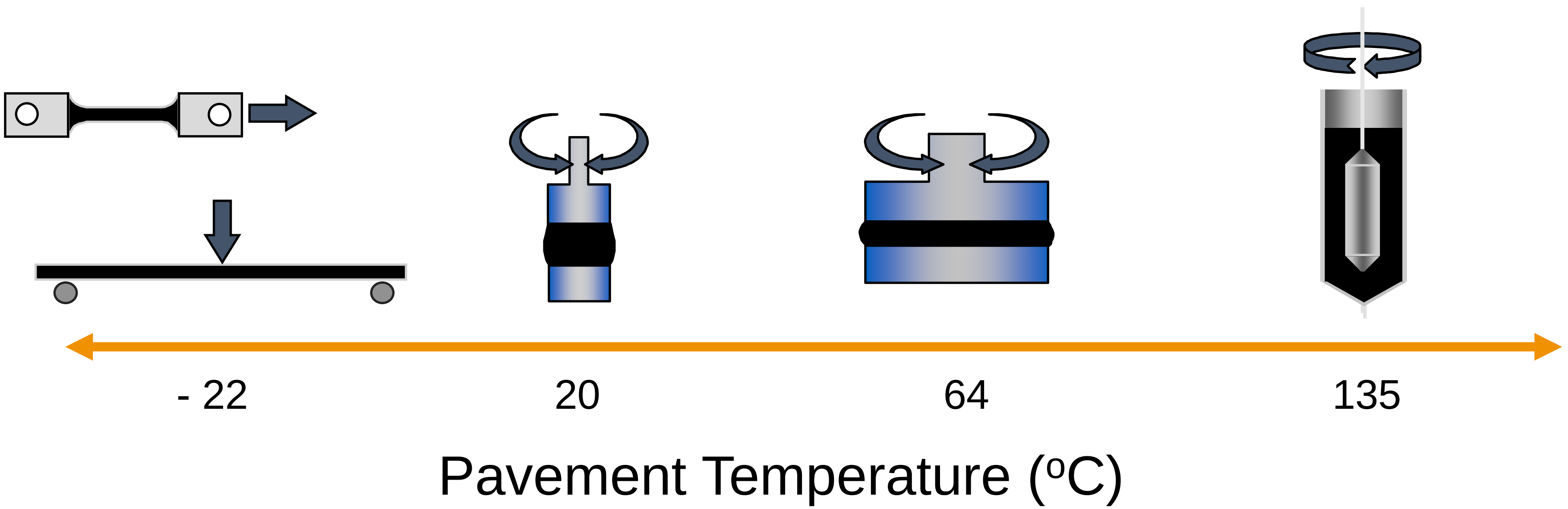
Performance-Related Requirements

- Low-temperature cracking resistance
  - Lower specification temperature
  - Stiffness after 2 hours loading ≤ 300 MPa
  - Changed to stiffness after 60 s loading and lower specification temperature +10°C
  - m-value after 60 s loading ≥ 0.300
- Table 2
  - Requires that pavement tensile strength be greater than thermal shrinkage stresses
  - Based on T_{CR} where strength and shrinkage stress are equal


Construction
Rutting
Fatigue
Cracking
Low Temp
Cracking
[RV]
[DSR]
[DTT]
[BBR]
Time
RTFO - aging
No aging
PAV - aging

Superpave Binder Testing

- Performance-Based Physical Properties Measured by
  - Rotational Viscometer (RV) (high temps)
  - Dynamic Shear Rheometer (DSR) (high, intermediate temps)
  - Bending Beam Rheometer (BBR) (low temps)
  - Direct Tension Tester (DTT) (low temps)

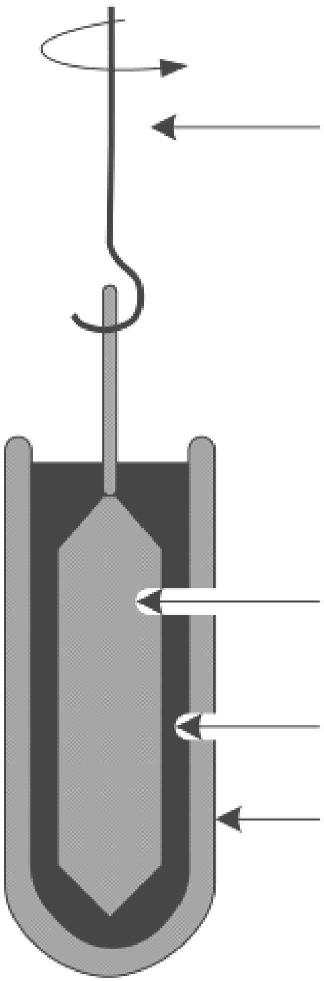


Rotational Viscosity

- Rotational Viscosity
  - Provides viscosity of asphalt binders in the range of temperatures from 60°C to over 200°C
  - The measured values are used to grade binders in accordance with AASHTO M 320 and AASHTO R 29

Rotational Viscometer

Rotational Viscosity:
Test Summary and Fundamentals



- Asphalt binder is placed between spindle and sample chamber
- Spindle rotates at constant speed
- Required torque is measured

Dynamic Shear Rheometer

- Test procedure results in complex modulus and phase angle
  - Specification test is conducted at 10 rad/s
  - Temperature range from 3°C to 88°C
- Parallel plate geometry
- Valid for linear viscoelastic materials
  - Materials with moduli that are independent of applied stress or strain
- Particles must be < 250 microns

Dynamic Shear Rheometer (DSR)

Dynamic Shear Rheometer

- Two plate sizes are used
  - 8 mm from 3°C to 40° C
  - 25-mm from 46°C to 86°C
- Complex shear modulus and phase angle provide a measurement of the deformation resistance of asphalt binders
  - Used to grade asphalt binders at the upper and intermediate specification temperatures

Bending Beam Rheometer

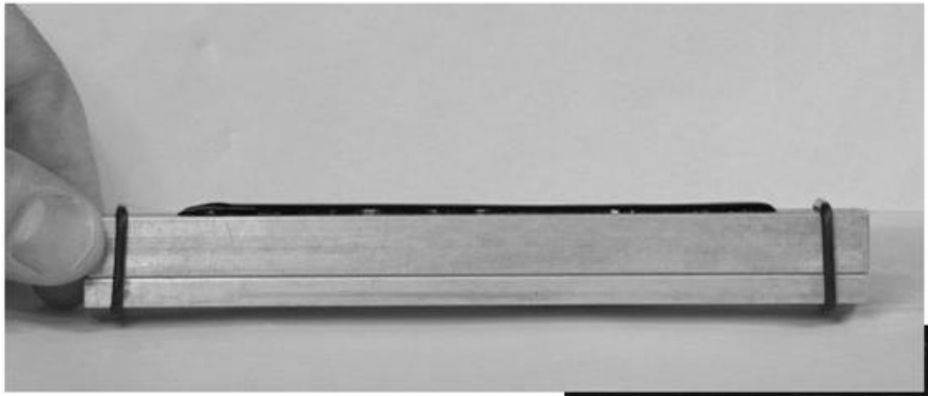
- BBR provides a means for measuring the flexural creep stiffness of asphalt binders
- Design of equipment limits its measurement range to 1 MPa – 20 GPa
- Test results for beams that deflect less than 0.08 mm or more than 4 mm are not considered valid

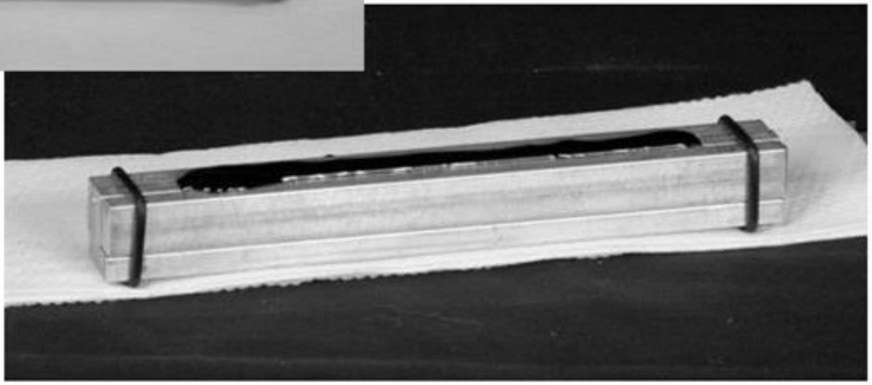
Bending Beam Rheometer (BBR)

BBR Test Summary and Fundamentals

- The load and deflection are used to calculate the maximum stress and strain in the beam
- Stiffness is calculated by dividing the maximum stress by the maximum strain
- m-value is calculated at 60 s as the slope of the log of stiffness versus the log of time

Proper Filling – Slightly Overfilled





Trimming

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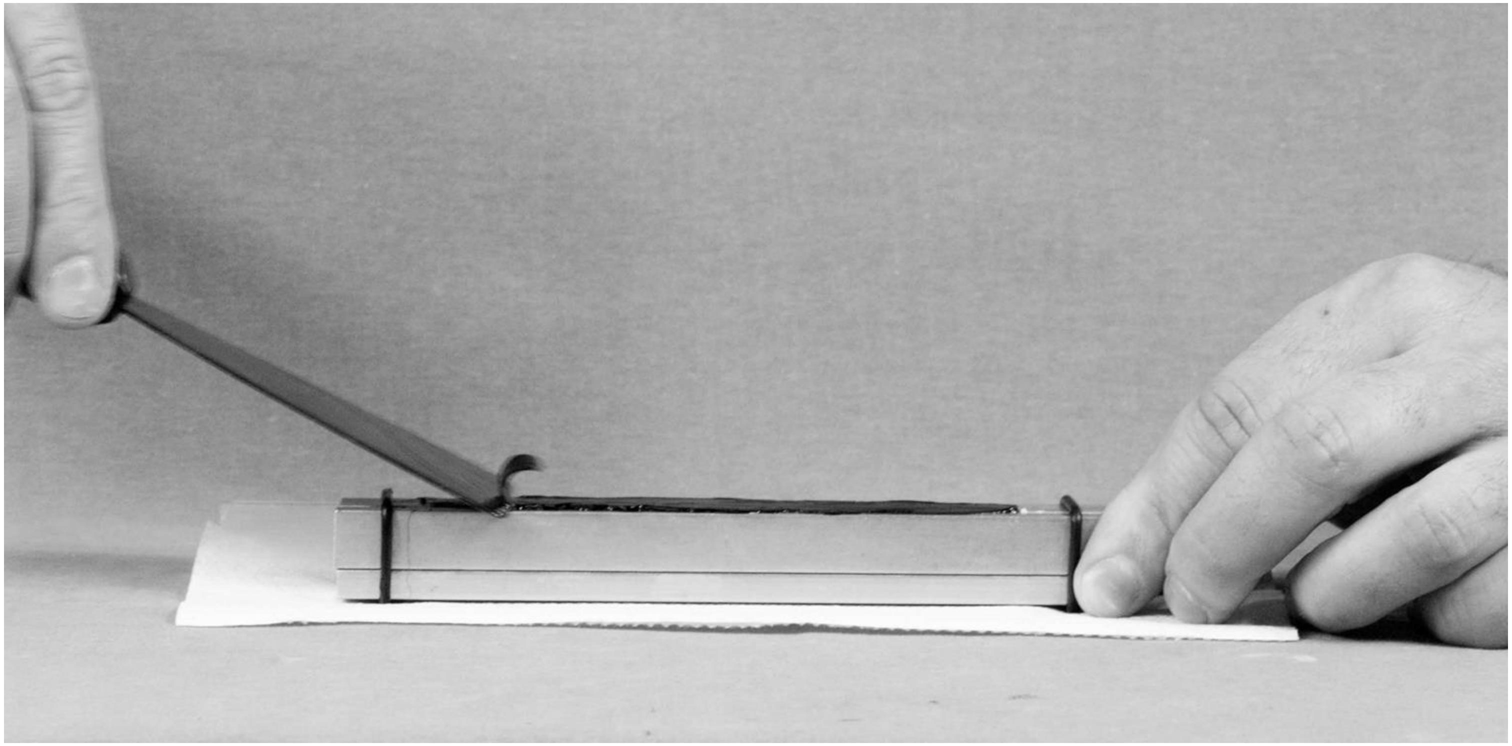
- Trim in one continuous motion



Pressure Aging Vessel

- Oxidation causes asphalt binders to harden (age) during field exposure
  - The PAV is used to simulate this field aging
- Aging in PAV is accelerated by increasing the conditioning pressure and temperature
  - Pressure forces oxygen into the binder
  - Temperature increases the rate of reaction
- PAV aging simulates the binder-specific aging that occurs in 5-10 years in the field

Pressure Aging Vessel (PAV)

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Field Aging is Binder Specific

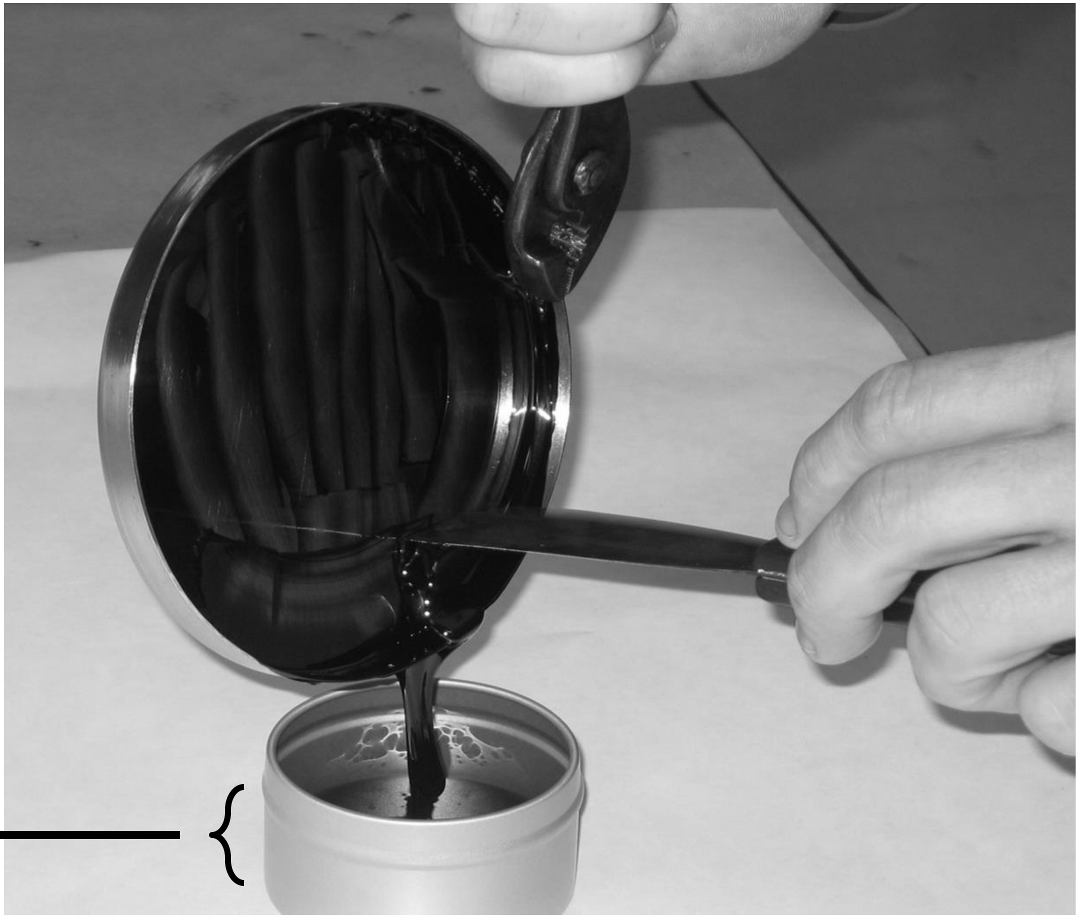
- The amount of aging that occurs is binder specific
  - Modulus increases approximately 8 times at the intermediate specification temperature
- As per test method

  "….it is not possible to select a single PAV aging time and temperature that will predict the properties of all asphalt binders after a specific set of in-service exposure conditions."

Transferring Binder to Container

Pour and then scrape binder from pan

15 – 40 mm thick when all of the binder is added



Modified Asphalt Tests

- Additional Tests
  - Separation
  - Solubility
  - Recovery and Stress-Strain Tests
    - Elastic Recovery
    - Force Ductility
    - Toughness and Tenacity
    - DSR Phase Angle

Modified Asphalt Tests

- Separation
  - Purpose
    - Used to assess polymer-asphalt compatibility
      - generally referred to as the “ointment” or “cigar” tube test.
  - Procedure
    - aluminum ointment tube is filled with 50 grams of modified asphalt binder
    - The filled tubes are then sealed and allowed to stand vertically in an oven operating at 163°C for 48 hours.

Modified Asphalt Tests

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- Separation
  - Procedure
    - The tubes are then transferred to a freezer for four hours
    - After freezing, the tubes are removed and cut into thirds
    - The top third and bottom third are separated into tins and tested
      - Using R&B softening point or DSR tests
      - determine if a significant difference exists in the properties of the top and bottom thirds.

Separation Test

Modified Asphalt Tests

- Solubility
  - Purpose
    - used to ensure that an asphalt binder does not contain insoluble organic and inorganic matter
      - doesn’t contribute to the “active cementing constituents” of the asphalt binder.

Modified Asphalt Tests

- Solubility
  - Different solubility test for polymer-modified asphalts
    - ASTM D-5546
      - similar to the standard solubility test except that it uses a centrifuge and toluene as the solvent.
      - some polymer modifiers did not dissolve adequately during the standard solubility test procedure
        - resulting in some polymer-modified asphalt binders failing the solubility criterion.

Modified Asphalt Tests

- Recovery and Stress-Strain Tests
  - Purpose
    - To evaluate whether an asphalt binder has been modified with an elastomer, specifically a block copolymer such as
    - designed to measure the ability of the asphalt binder to stretch when loaded and ultimately rebound to its original shape when unloaded

Modified Asphalt Tests

- Recovery and Stress-Strain Tests
  - Common Tests
    - Elastic Recovery
    - Force Ductility
    - Toughness and Tenacity
    - DSR Phase Angle
      - Indicator of relative proportion o f elastic and viscous components in asphalt binder

Modified Asphalt Tests

- Elastic Recovery
  - Based on ductility equipment
    - Same molds, but different side pieces
      - Rectangular (parallel), not V-shaped
    - Sample preparation like ductility test
  - Procedure
    - Load specimens at test temperature (usually 25°C)
    - Pull specimens to 10 or 20 cm
      - Defined by test procedure

Modified Asphalt Tests

- Elastic Recovery
  - Procedure
    - Cut specimens in center
      - May have a 5-minute wait period before cutting
    - Allow specimens to relax for 1 hour
    - Push specimen back together until cut ends touch
    - Record elastic recovery as the difference between the stretched and final position divided by the stretched position
      - Express as a %

Elastic Recovery

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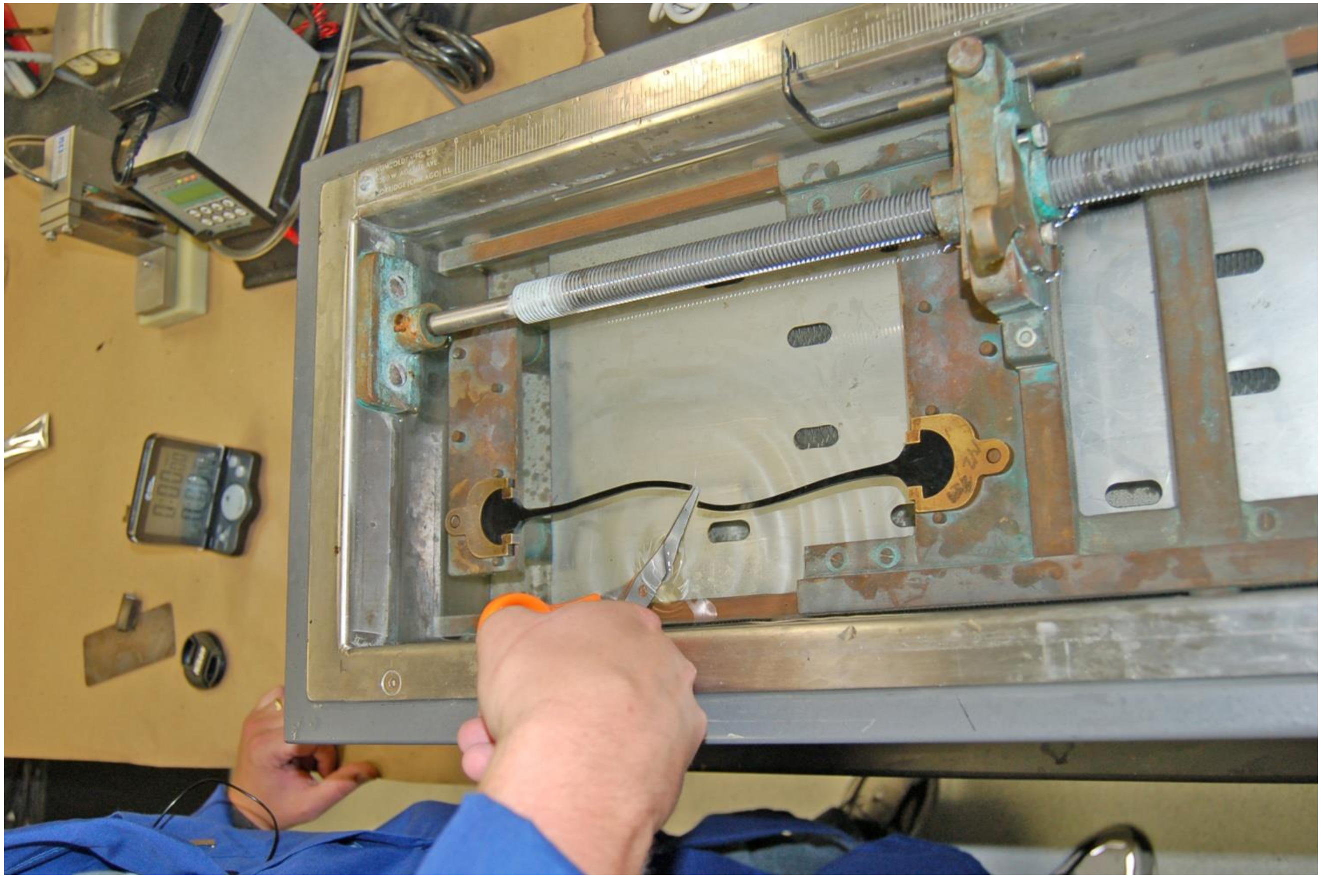
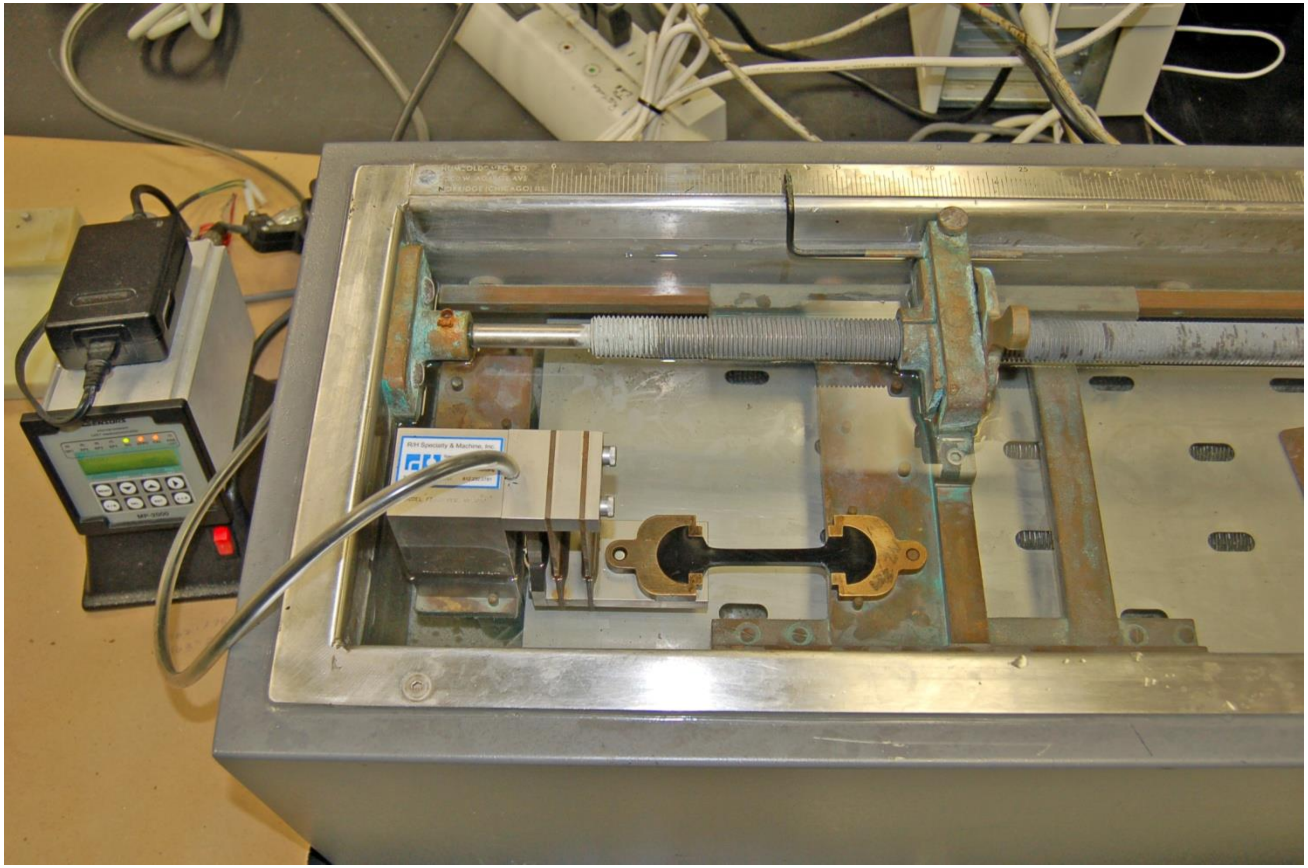


Modified Asphalt Tests

- Force Ductility
  - Based on ductility equipment
    - Similar to Elastic Recovery Test
      - Uses a load cell to measure stress
      - Conducted at 4°C
      - Usually unaged binder
  - Procedure
    - Load specimens at test temperature
    - Pull specimens to 30 cm
      - Measure stress

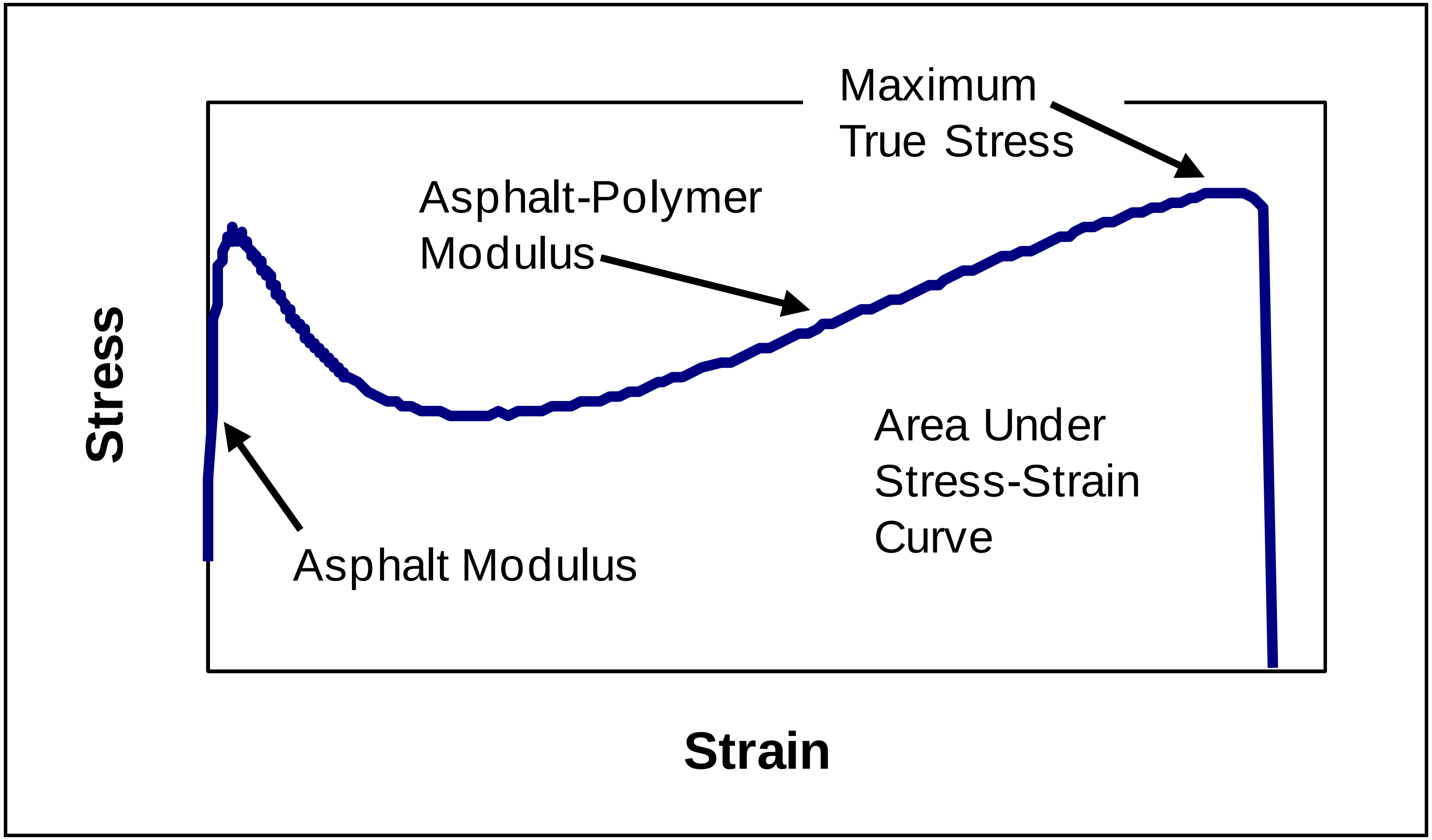
Modified Asphalt Tests

- Force Ductility



Modified Asphalt Tests

- Force Ductility



Modified Asphalt Tests

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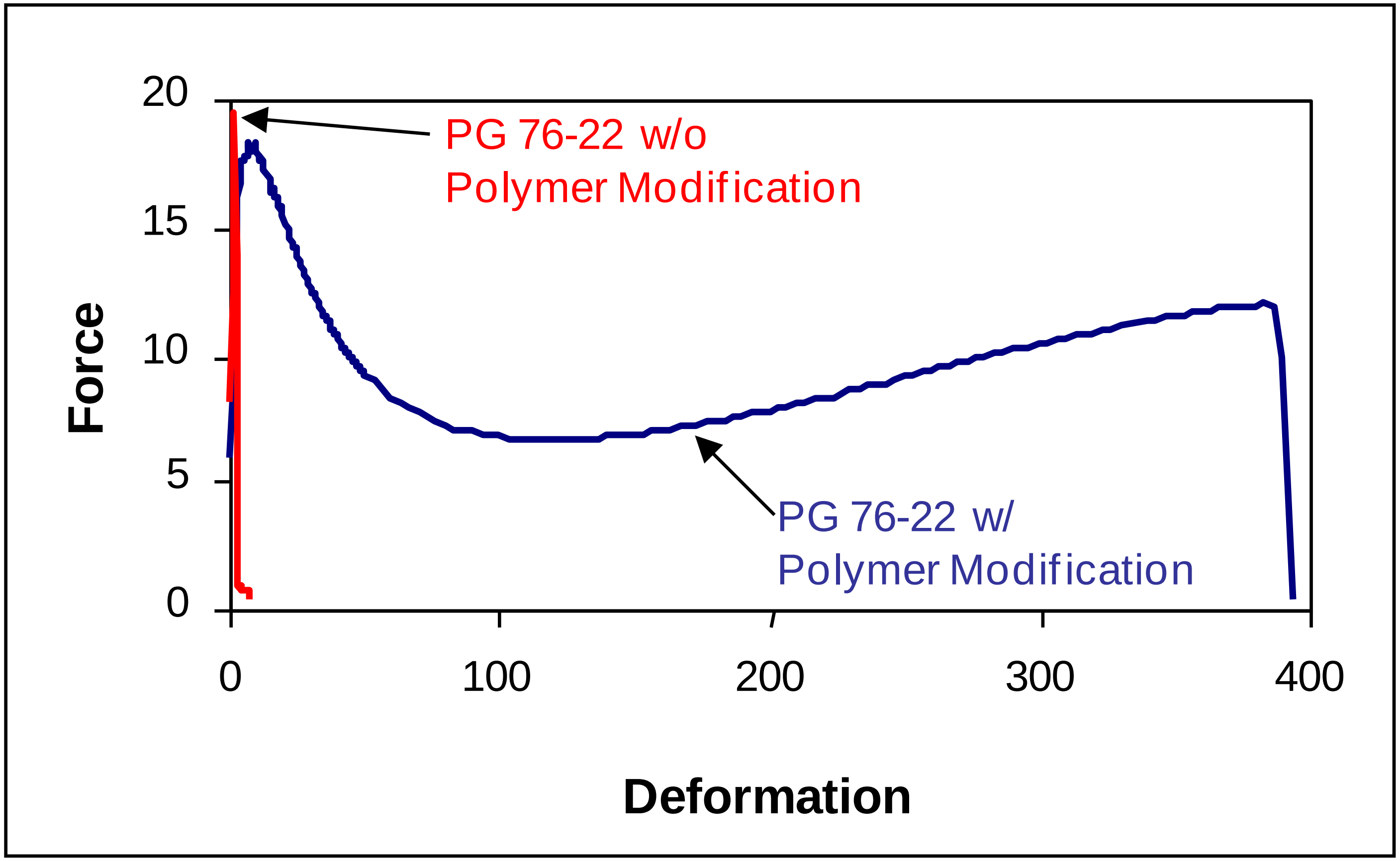
- Force Ductility

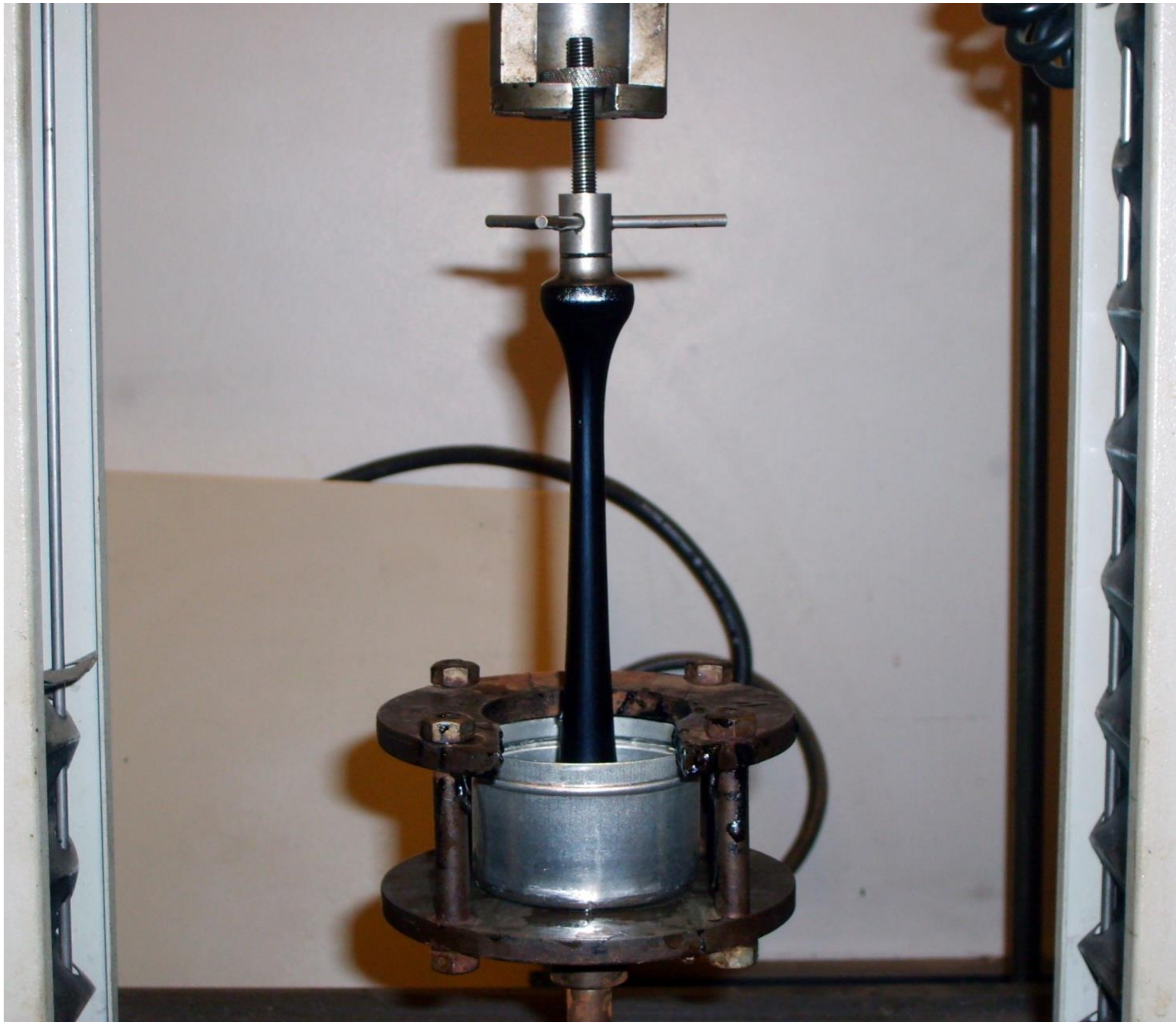


Modified Asphalt Tests

- Toughness and Tenacity
  - first introduced by Benson in the 1950s
  - Procedure
    - a metal hemispherical head is embedded in hot asphalt to a depth of approximately 11 mm
    - After cooling to 77°F (25°C), the head is attached to a tensile test machine and pulled from the asphalt binder at a rate of 51 cm/min.
    - The load is measured throughout the test
      - a load-deformation curve is plotted
      - Toughness and tenacity values determined based on the area under different portions of the load-deformation curve

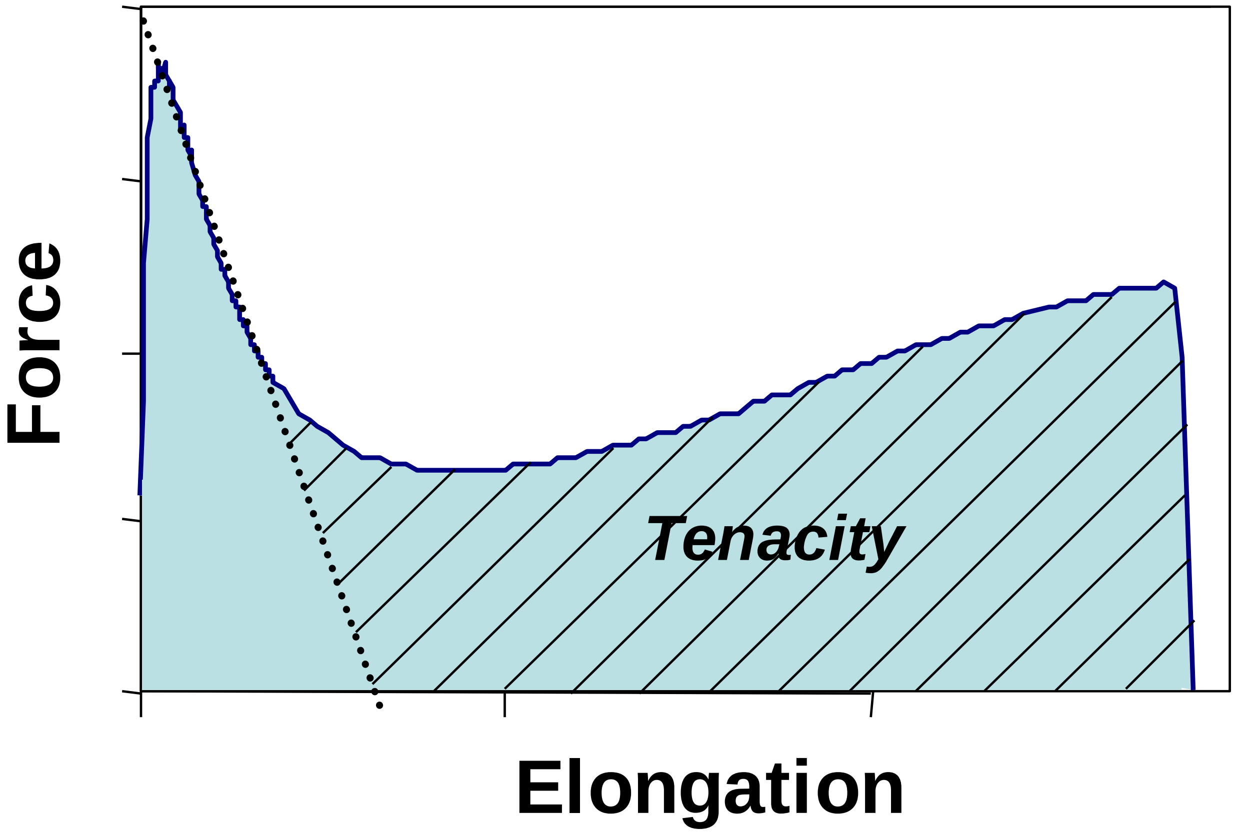
Modified Asphalt Tests

- Toughness and Tenacity



Modified Asphalt Tests

- Toughness and Tenacity



Modified Asphalt Tests

- DSR Phase Angle
  - Output from standard DSR testing
  - Maximum phase angle ensures elastic component in asphalt binder
    - Influenced by stiffness of asphalt binder

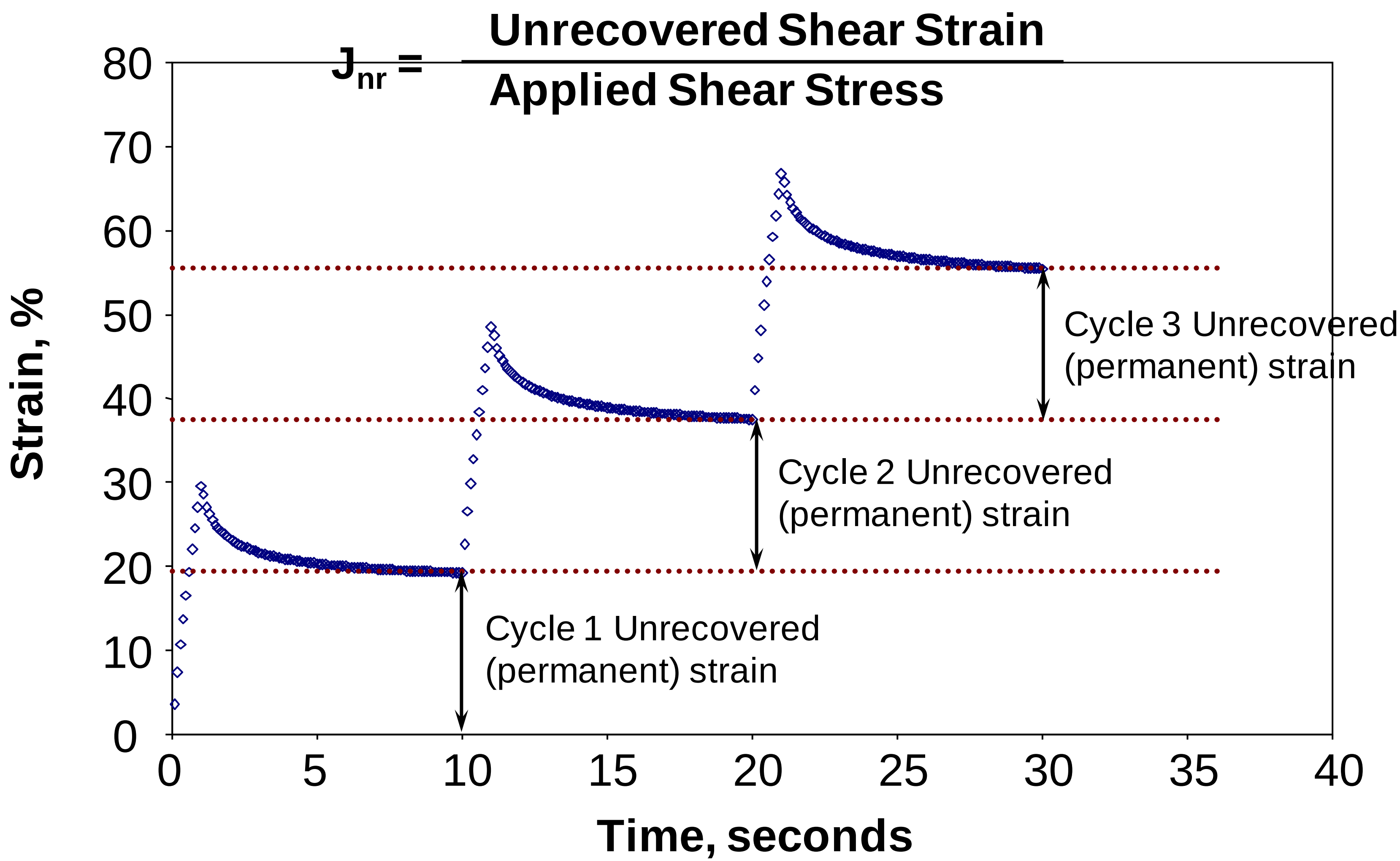
Multi-Stress Creep Recovery (MSCR) Test

- Performed on RTFO-aged Binder
- Test Temperature
  - Environmental Temperature
  - Not Grade-Bumped
- 10 cycles per stress level
  - 1-second loading at specified shear stress
    - 0.1 kPa
    - 3.2 kPa
  - 9-second rest period

MSCR

- Calculate Recovery for each Cycle, Stress
  - Difference between strain at end of recovery period and peak strain after creep loading
- Calculate Non-recoverable Creep Compliance (J_{nr})
  - Non-recoverable shear strain divided by applied shear stress
    - "J" = "compliance"
    - "nr" = "non-recoverable"

MSCR Strain Response



MSCR: Calculating J_{nr}

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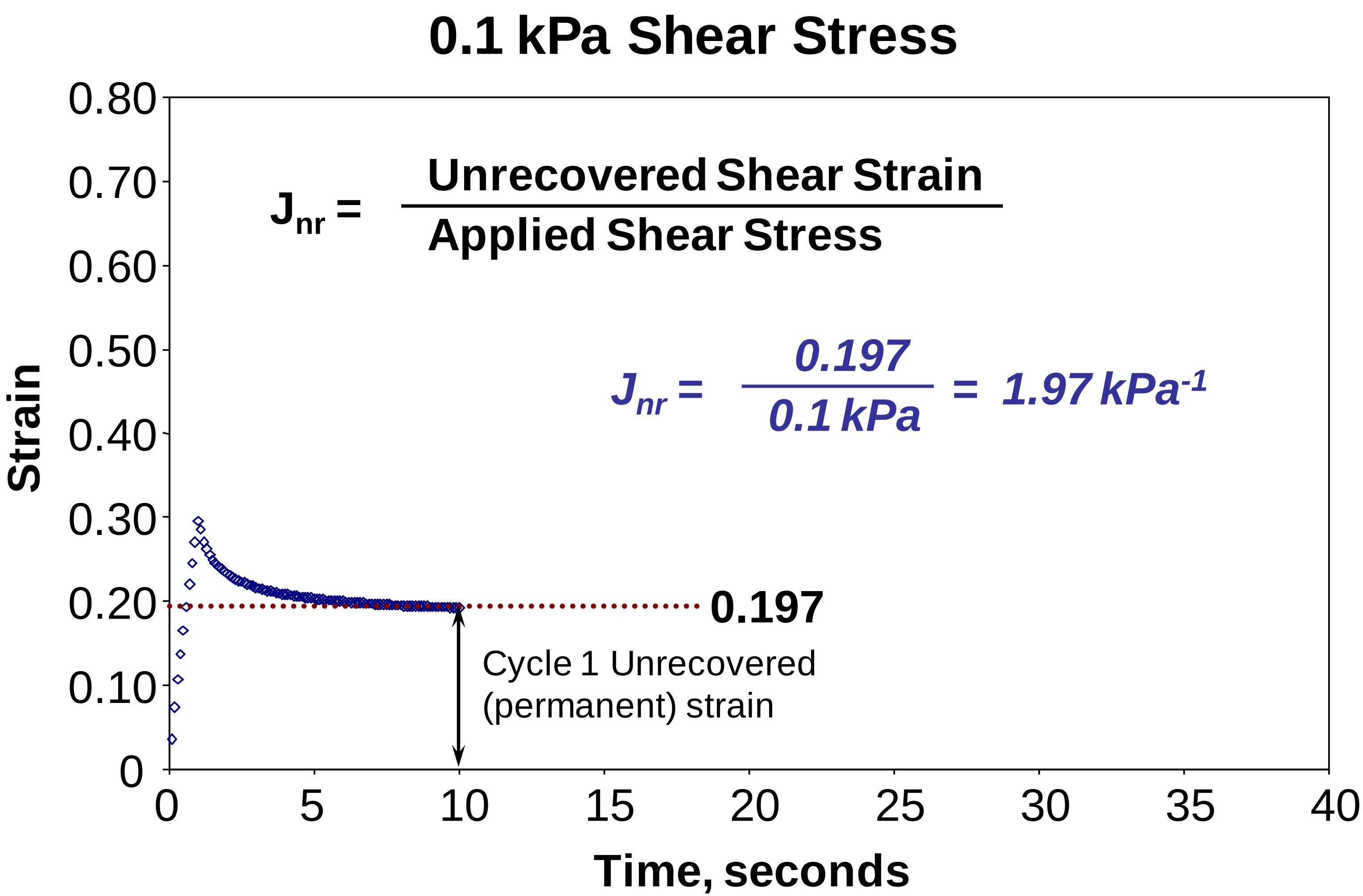
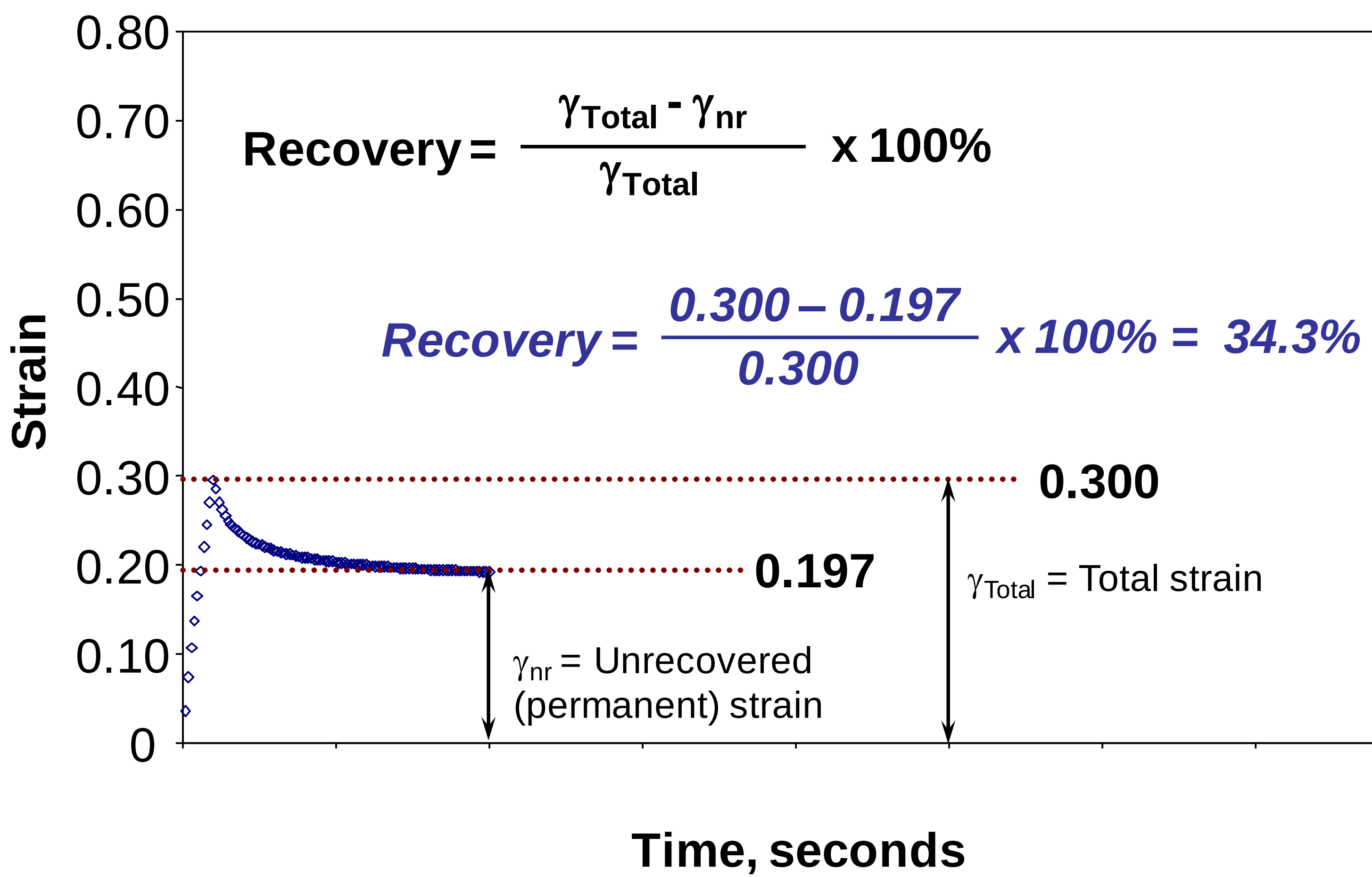
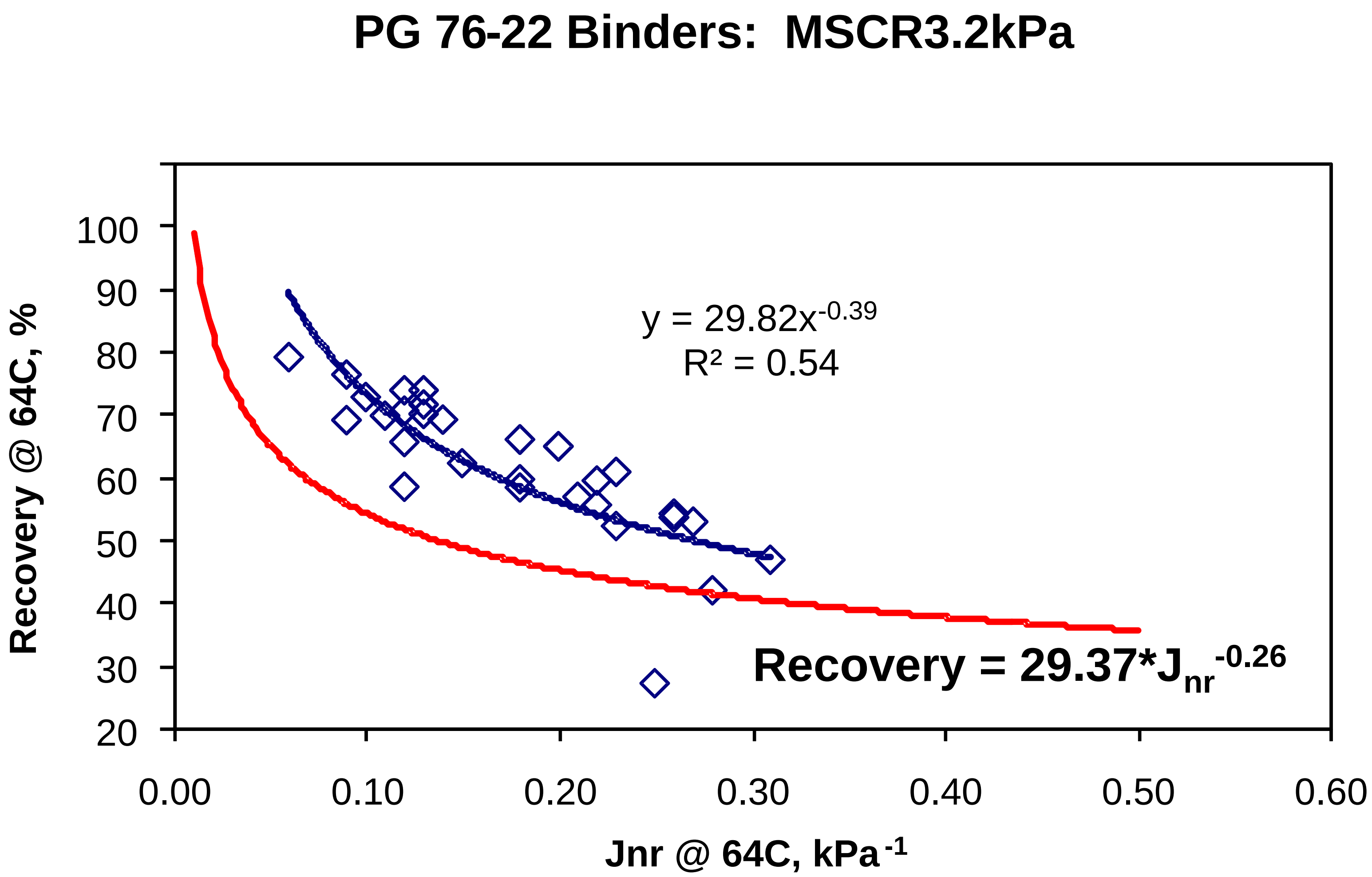


Modified Asphalt Tests

- MSCR Recovery



Validate Polymer Modification



Thanks !