AF2903 Road Construction and Maintenance
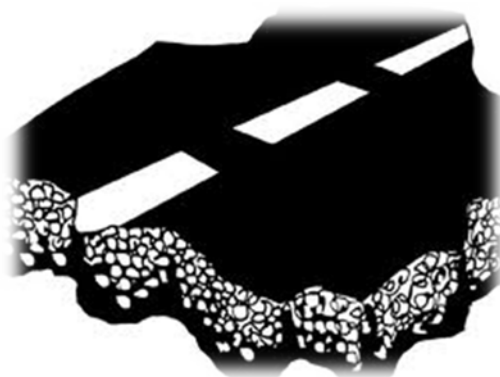
Asphalt Binder Characterization

ALI AZHAR BUTT

PhD Candidate

Highway and Railway Engineering

Asphalt Binder Characterization

➢ Introduction to the Binders

➢ Modified Binders

➢ Traditional Grading System

➢ Superpave (SHRP)

Standard Bitumen

- General

- Origin

- Production

- Test methods

- Properties

- Specifications

- Functional requirements

General

Bitumen

- The virtually in-volatile, adhesive and waterproofing material derived from crude petroleum, or present in natural asphalt, which is completely or nearly completely soluble in toluene, and very viscous or nearly solid at ambient temperatures. (EN 12597)

- Bitumen / Asphalt

Bitumen is a visco-elastic material

- At low temperatures and/or high frequencies: **elastic solid material**

- With increasing temperature and/or decreasing loading rate: **more viscous**

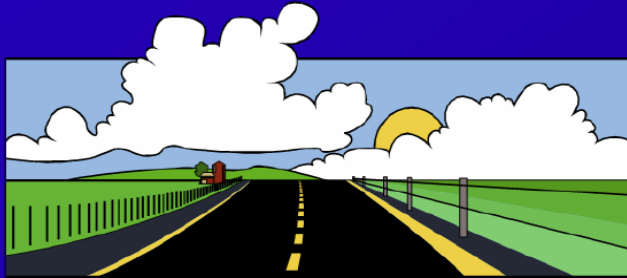- At sufficiently high temperature and/or long loading time: **like a fluid**

Mixture of millions of chemical components…

- 90-95% C and H

- 5-10% heteroatoms (N, O, S)

- Trace metals

- Salt, mineral particles

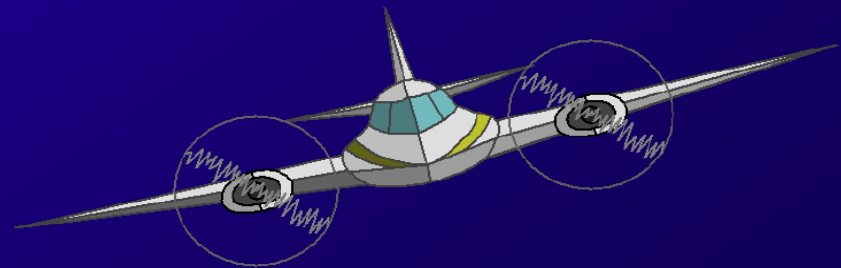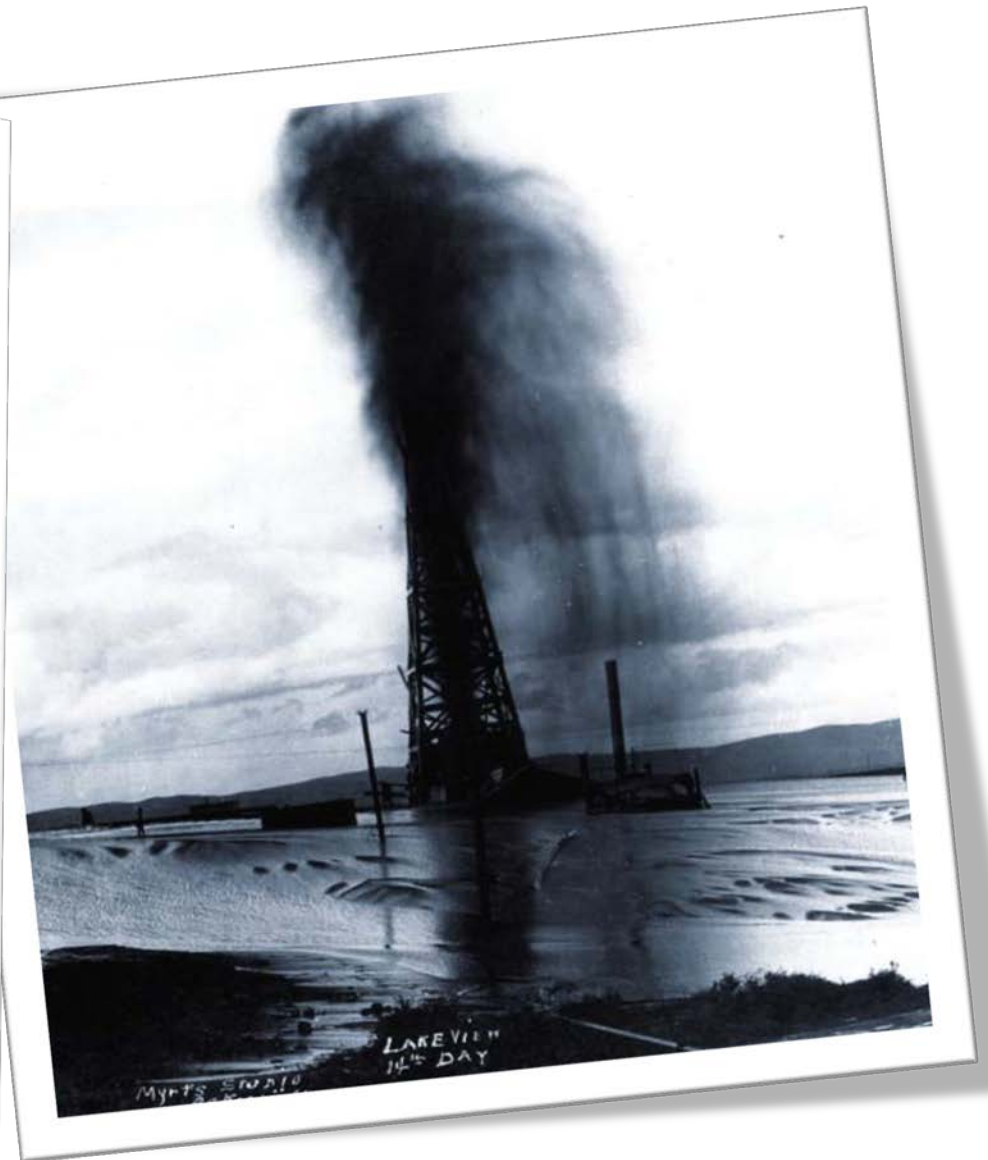# Bitumen chemically reacts with ......

- Oxygen in the air?

- Water?

- Chemicals?

- Solubility?

Origin

LAKEVIEW 14th DAY

Myrts Swald

Cyclic Steam Stimulation (CSS)

Diagrammatic Representation of the Cold Lake Plant for Bitumen Extraction Using Cyclic Steam Stimulation (CSS)

Stage 1
Steam Injection

Stage 2:
Soak
Time

Stage 3
Melted Bitumen Production
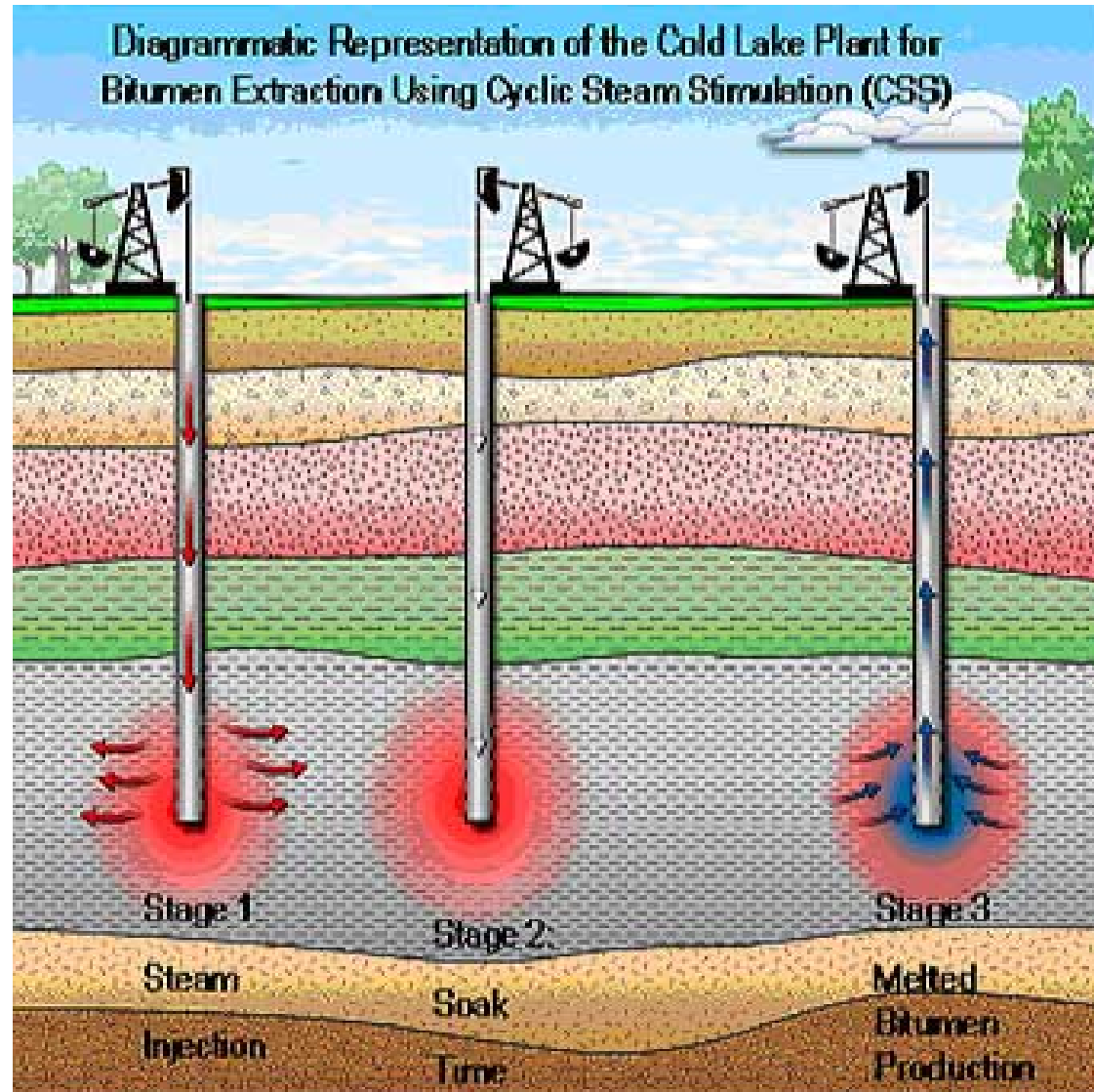
Steam Assisted Gravity Drainage

© McDaniel & Associates Consultants Ltd. 2006

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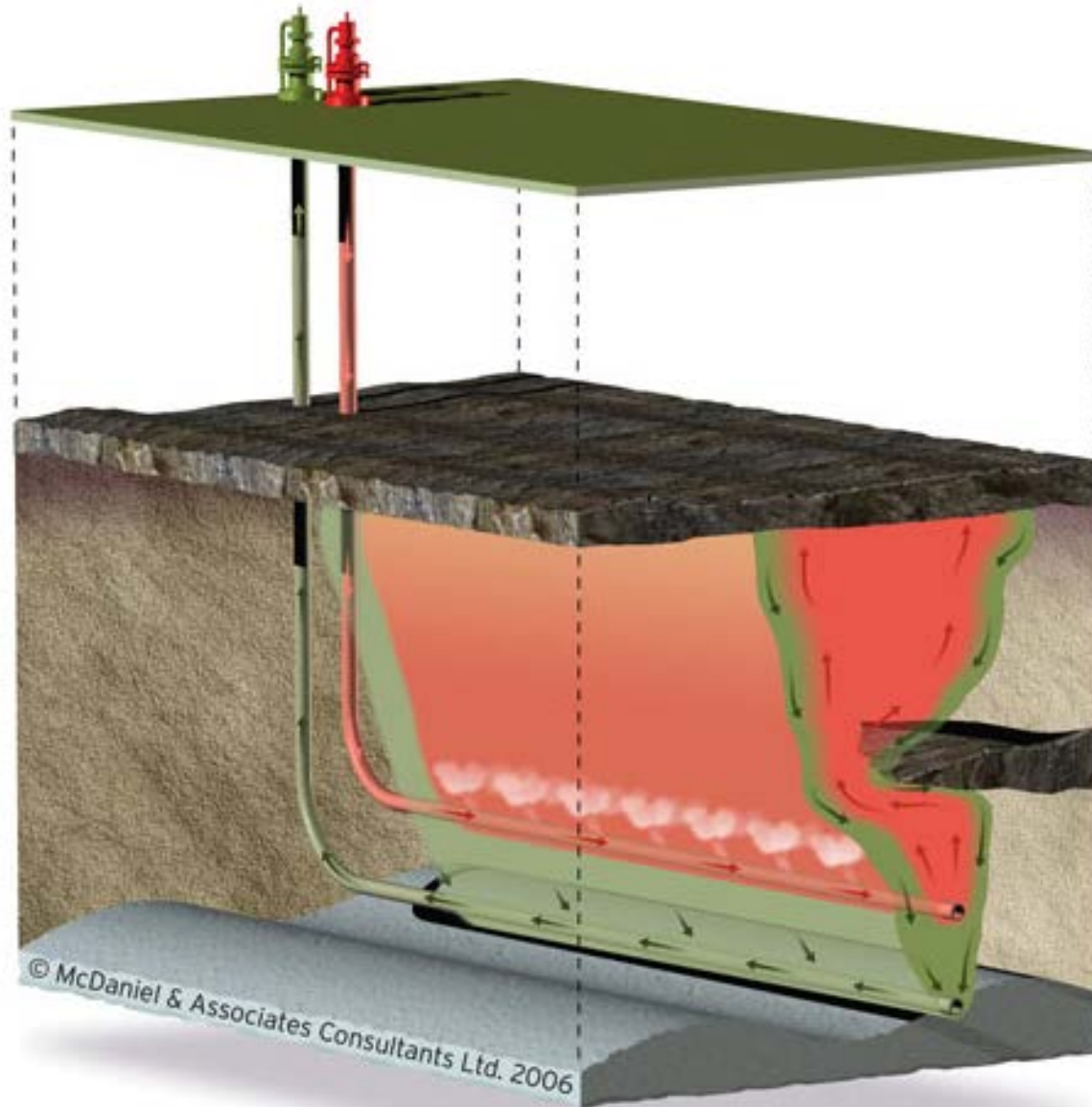
Crude oil products

+3 000 million tons of crude oil per year are produced in the world today

- Aviation fuel (Kerosine)

- Gasoline

- Fuel and lubricating oils (Diesel)

- Paraffin wax

- Approximately 3 % will be bitumen

Bitumen Markets

- World   80-90MT

- EU   20MT

- US   33MT
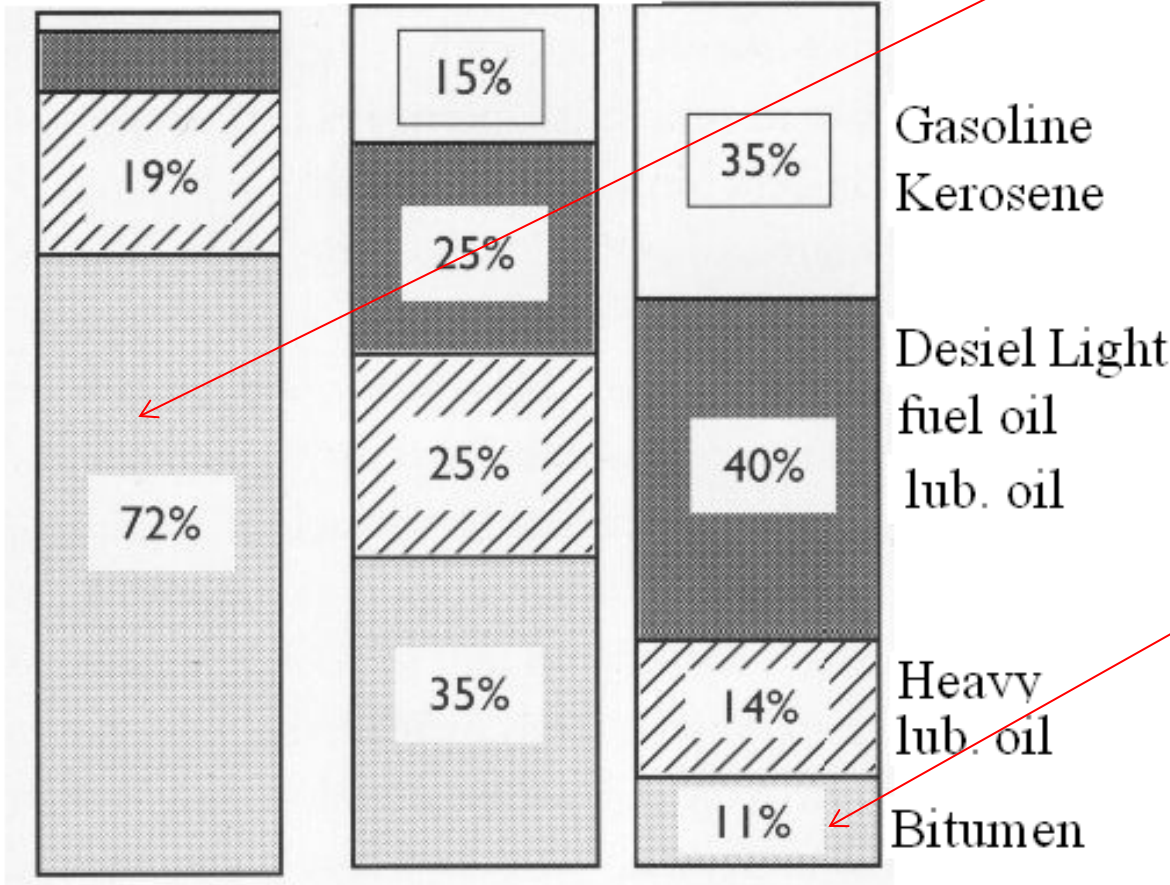
- CHINA   11MT

- UK   2MT

- Sweden   0,5MT

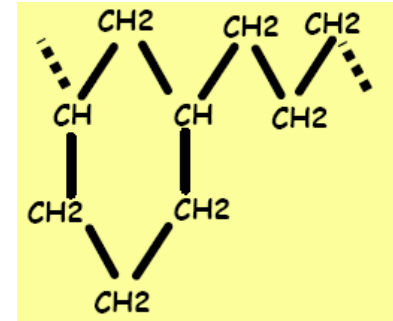90% ROAD
10% Roofing, selant, etc

Different types of crude oil
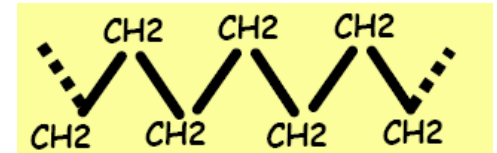
Production: Fractional distillation

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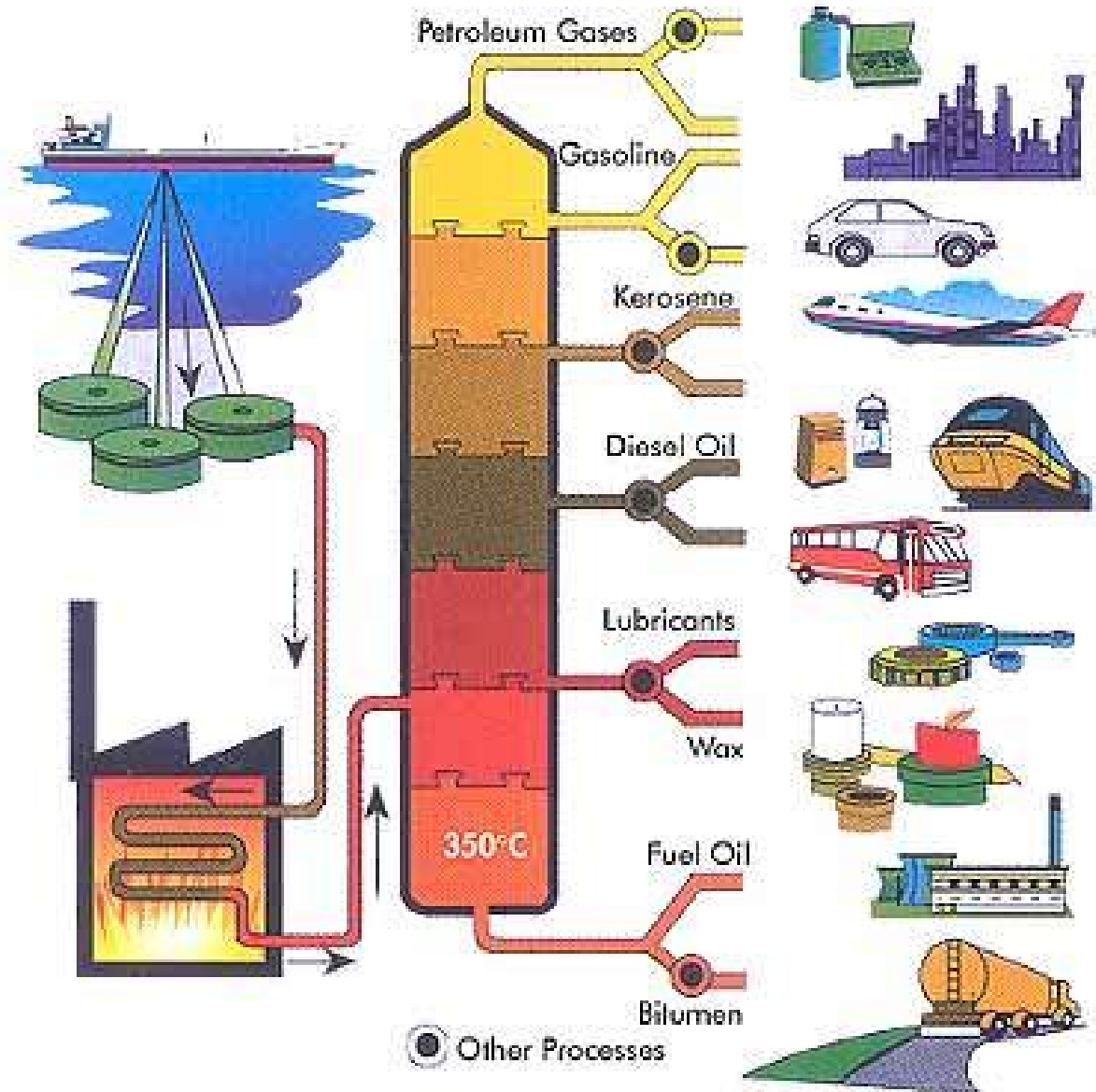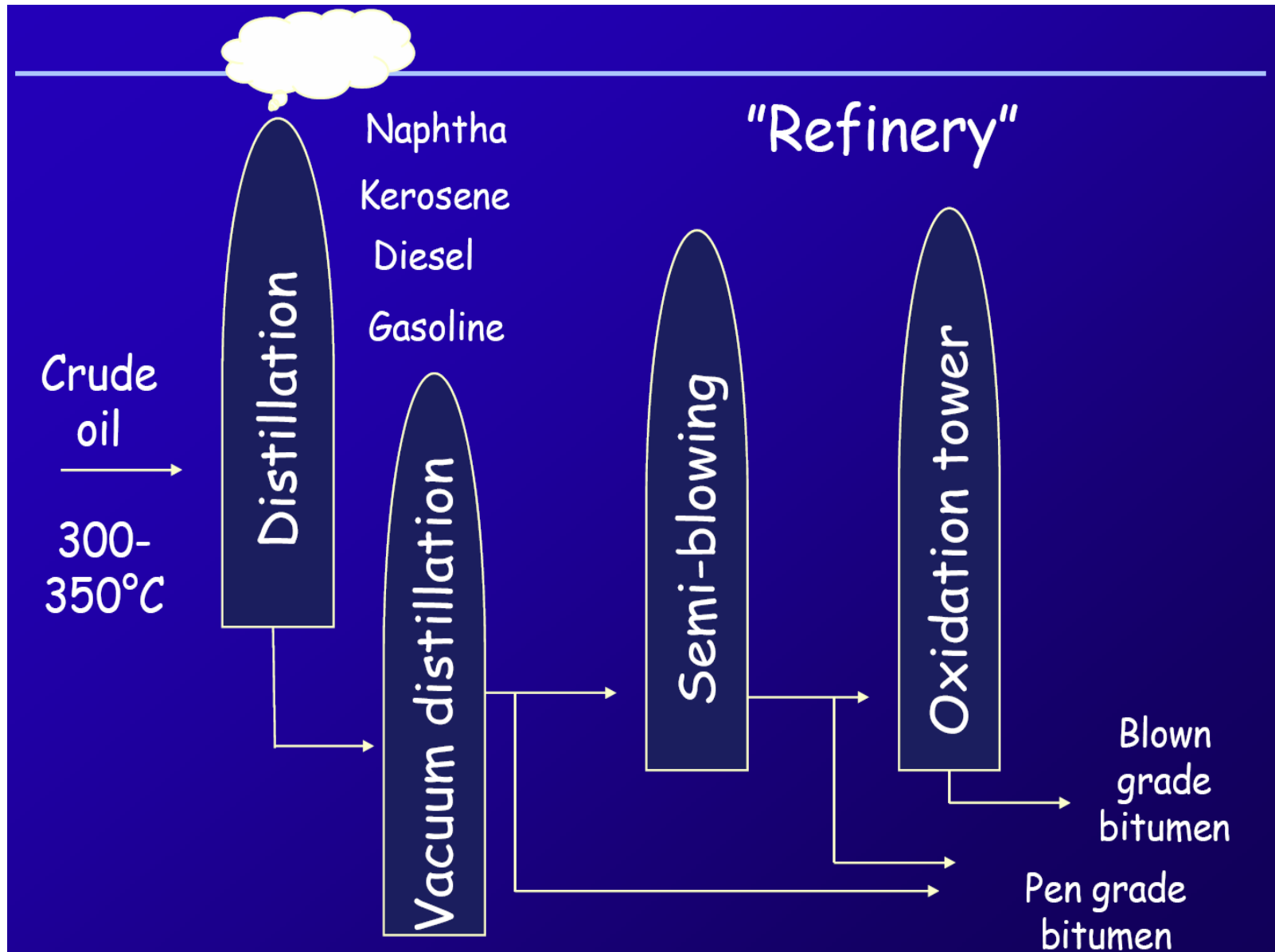
Refinery in Tarragona

Test methods

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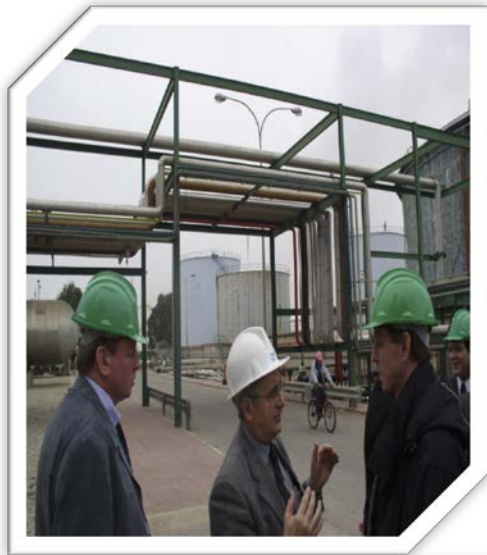
The state of bitumen is different at different temperatures.

- High Temperatures : **Viscous**

- Medium Temperatures : **Semi Solid**

- Low Temperatures : **Elastic Solid**

- Very Low Temperatures : **Glassy/Brittle**

Standard specification tests

- Softening Point (EN 1427)

- Penetration (EN 1426)

- Elastic Recovery (EN 13398)

- Breaking Point, Fraass (EN 12593)

- Viscosity Brookfield (EN 13302)

- Storage Stability (EN 13399)

Penetration (EN 1426)

- Index test

- Material Property

- Classification

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Softening point (EN 1427)

- Traditional way

- Change of state temperature measurment

Breaking Point, Fraass (EN 12593)

40,0 mm

36,5 mm
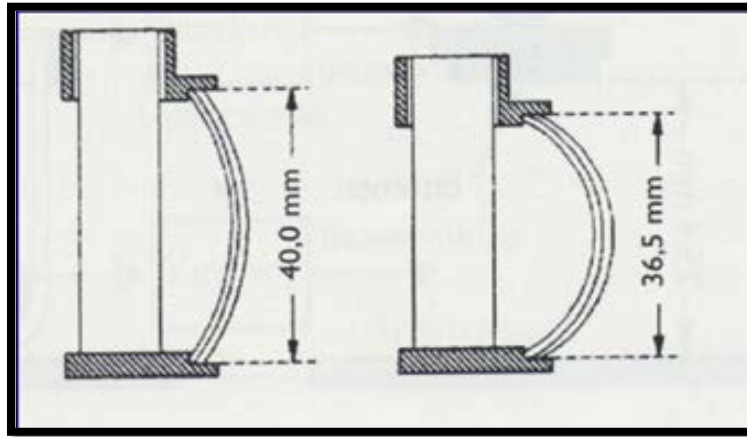
To measure the temperature at which the bitumen cracks/fails at low temperatures in the glassy phase

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Viscosity Brookfield (EN13302)

Measure of viscosity of the bitumen at high temperatures

Mixing and compaction temperatures can be determined for a particular bitumen.

Viscosity-Temperature Chart

PENETRATION, 0.1 mm

9,5 mm

Bitumen

25,4 mm

Penetration
i 0,1 mm enheter

100 g   100 g

Bitumen   Bitumen
25°C   25°C

Start   Efter 5s

40,0 mm   36,5 mm

PENETRATION, 0.1 mm

PI
−3 −2 −1 0 +1 +2 +3 +4 +5 +6 +7

VISCOSITY, POISES
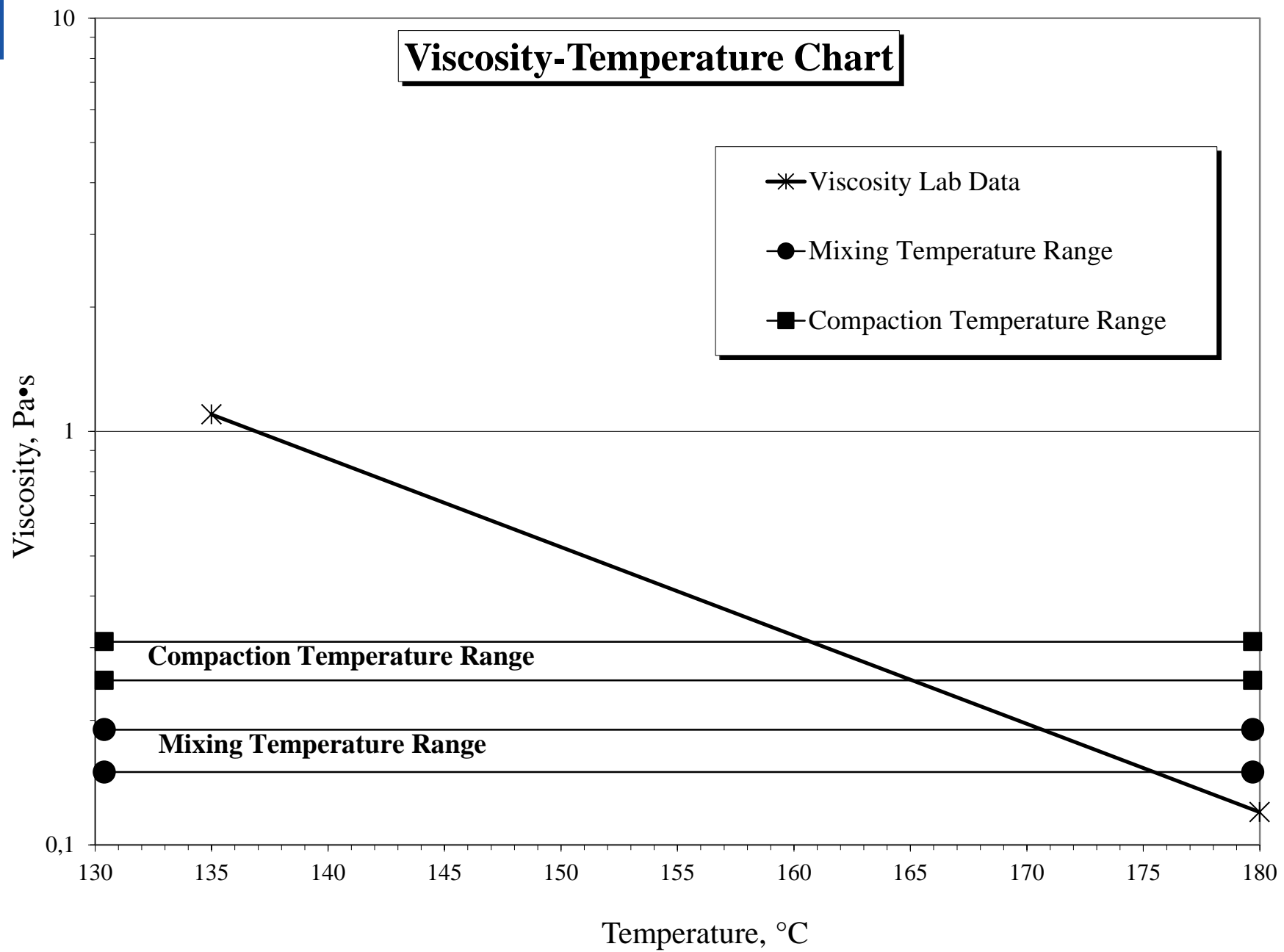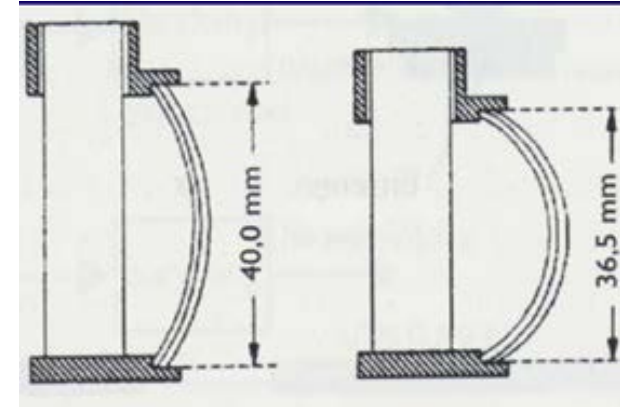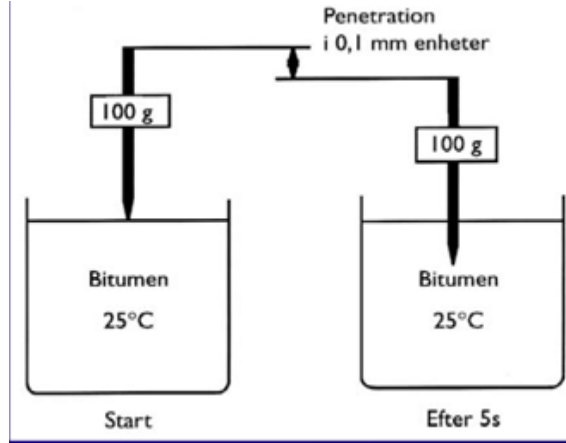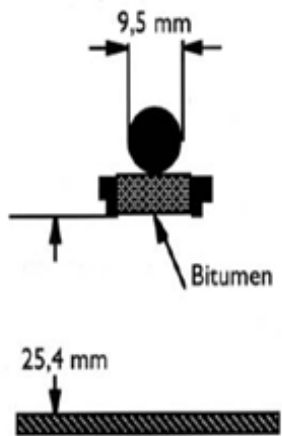
BITUMEN TEST DATA CHART
(Ref. W. Heukelom, J. Inst. Petr. 55(1969)404−417)

$T_{800\ pen}$

R&B

−40   −20   0   20   40   60

−50 −40 −30 −20 −10 0 10 20 30 40 50 60 70 80 90 100 110 120 130 140 150 160 170 180 190 200 210 220 230 240 250

TEMPERATURE
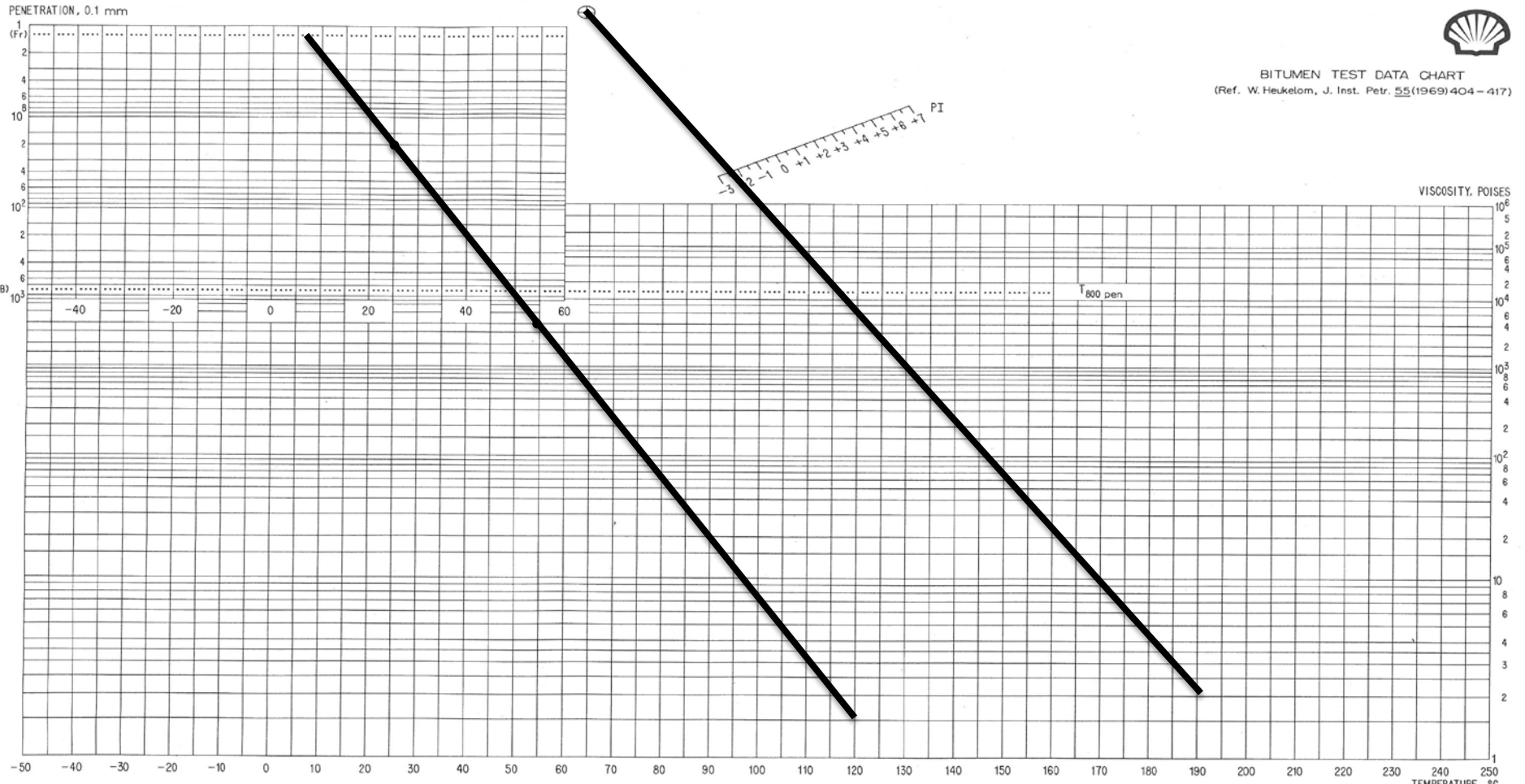
Heukelom's diagram
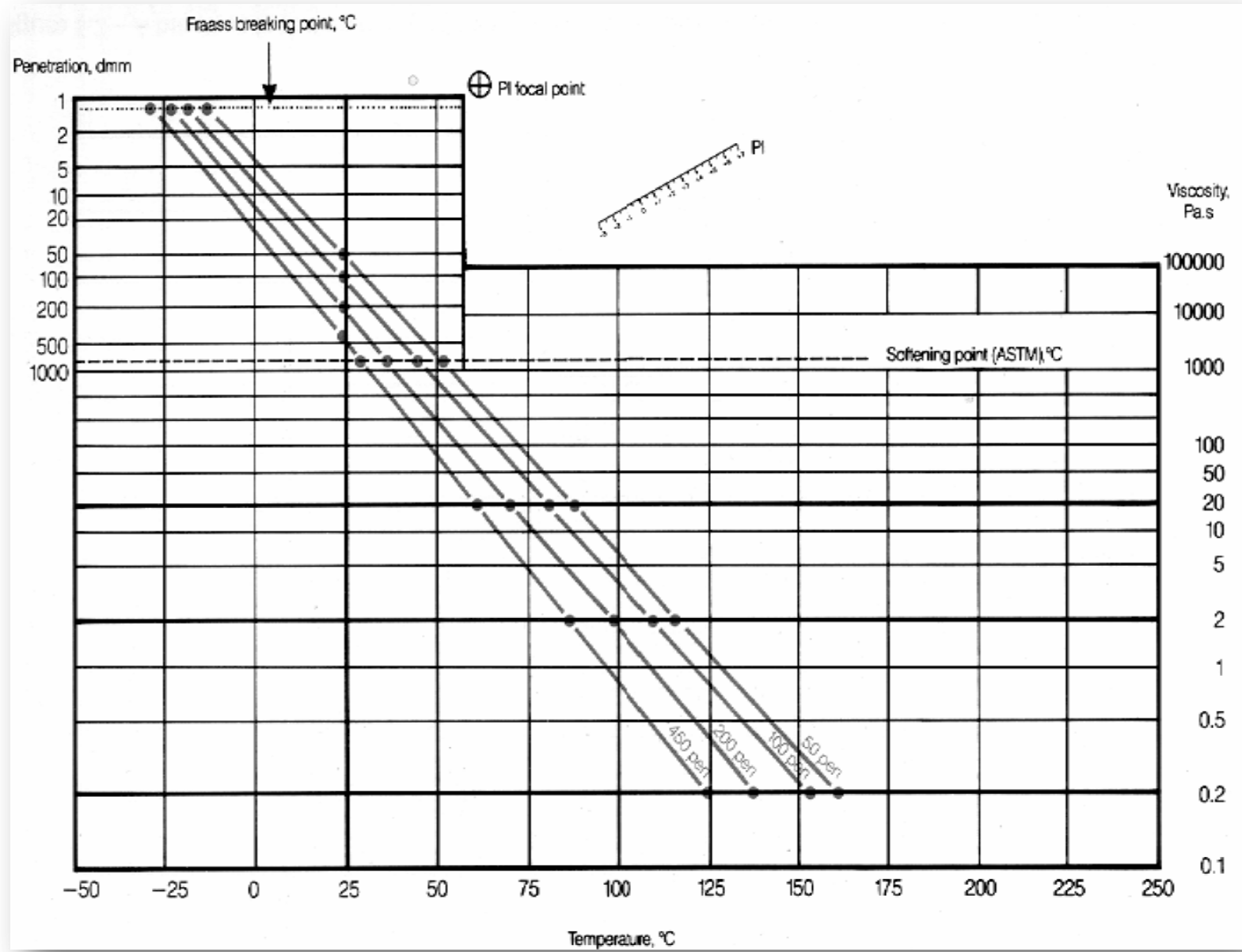
Same Bitumen Grade; different Source
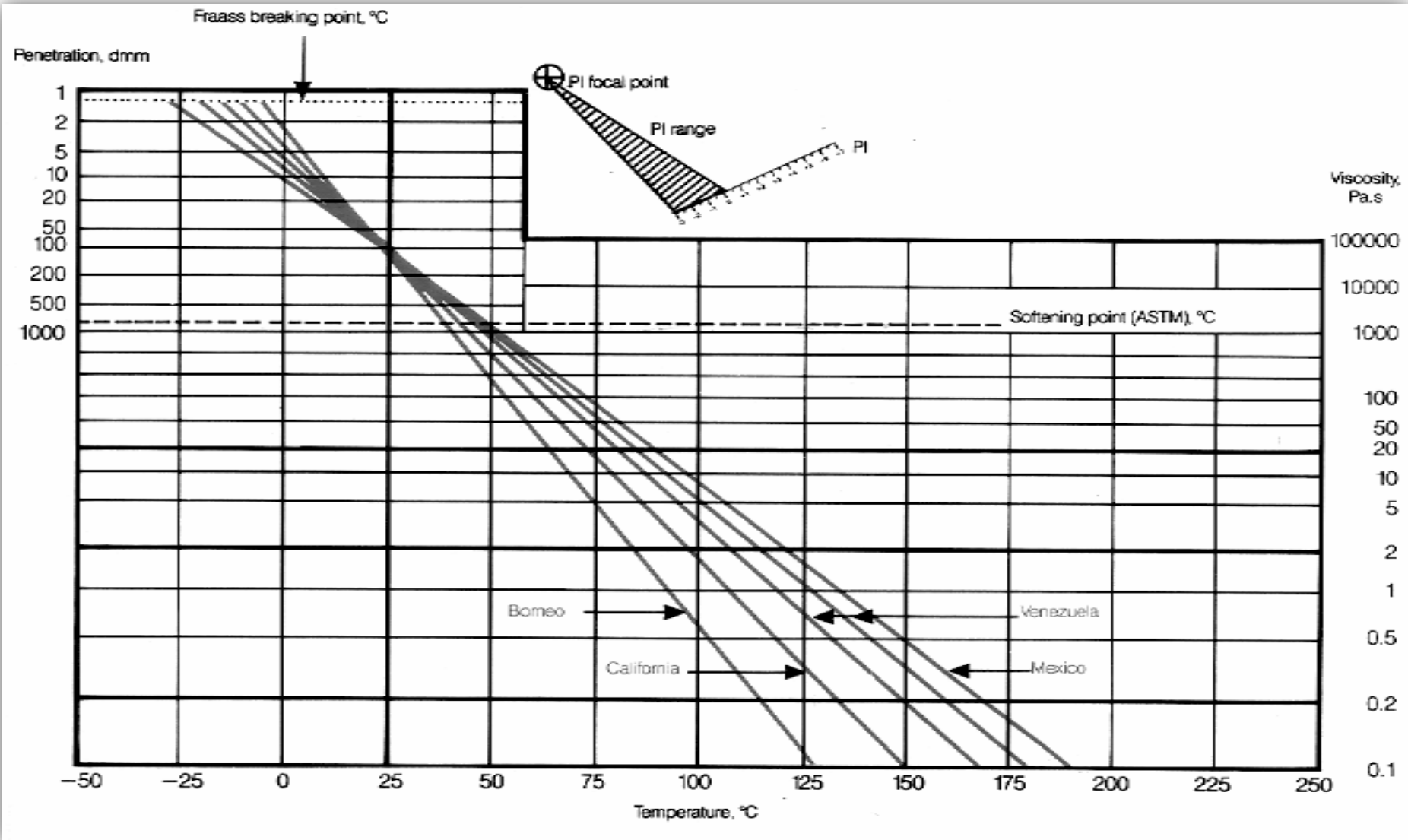
Rolling Thin Film Oven Test RTFOT

Short term aging

*EN 12607-1

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Pressure Aging Vessel PAV

Long term aging

*EN 14769

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FTIR Spectroscopy

Identify effect on different groups in a bitumen due to aging

Specifications

Penetration grade

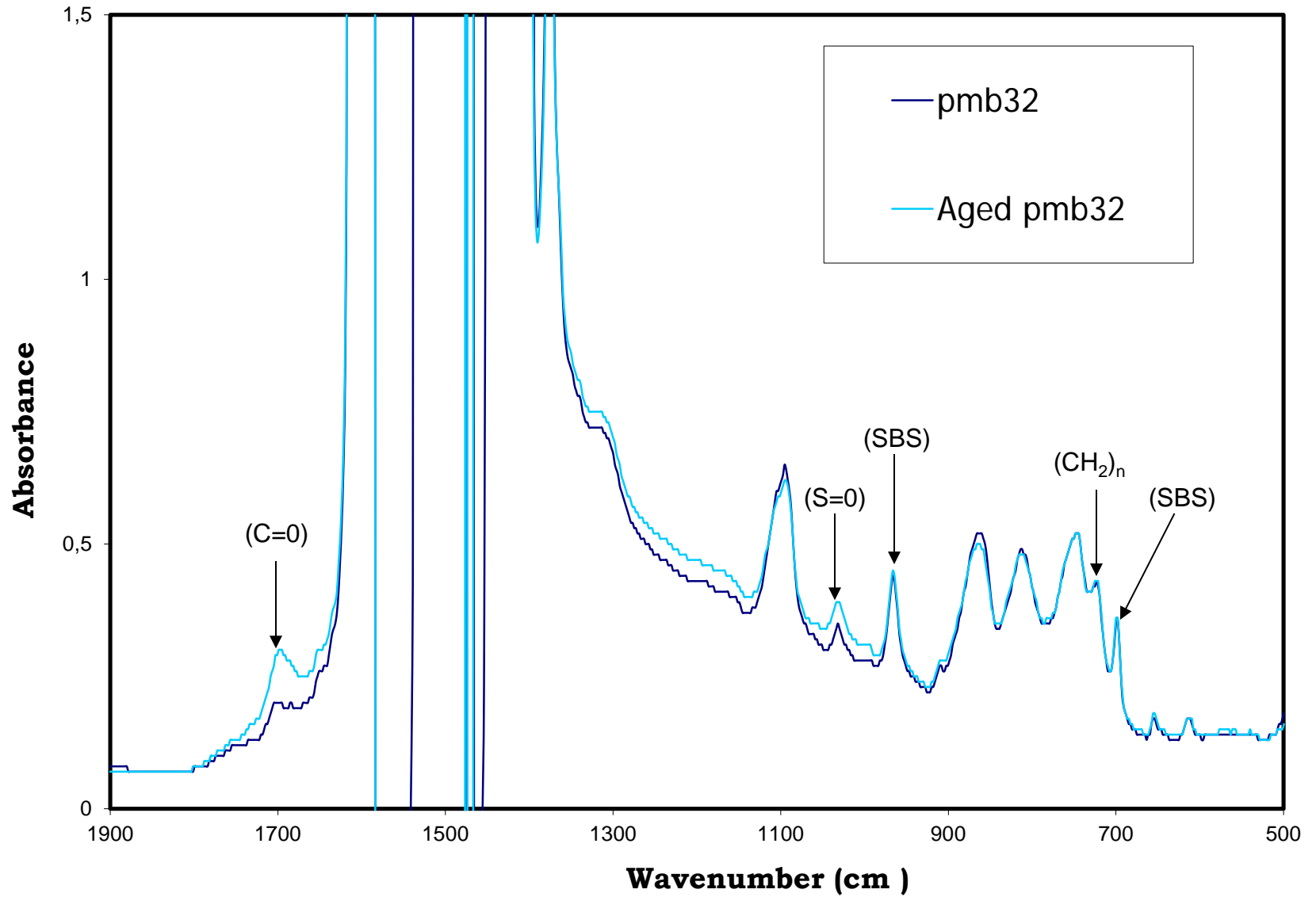| Egenskaper	Enhet	Testmetod SS-EN	Kvalitet				
			50/70	70/100	100/150	160/220	330/430
Penetration vid 25 C	x 0,1mm	1426	50-70	70-100	100-150	160-220	330/430
Penetration vid 15 C	x 0,1mm	-	-	-	-	-	90-170
Kinematisk viskositet vid 135 C, minimum	mm2/s	12595	295	230	175	135	85
Dynamisk viskositet vid 60 C, minimum	Pa s	12596	200	120	80	43	15
Mjukpunkt	C	1427	46-54	43-51	39-47	35-43	-
Brytpunkt Fraass, maximum	C	12593	-8	-10	-12	-15	-18
Löslighet, minimum	% (m/m)	12592	99,0	99,0	99,0	99,0	99,0
Flampunkt, minimum	C	22592 b)	230	230	230	220	-
Flampunkt, minimum	C	22719	-	-	-	-	180
Densitet	kg/m3	SS EN ISO 3838 c)	-	-	-	-	-
Viktförändring efter upphettning 163 C, maximum	%	12607-1 -3 a)	0,5	0,8	0,8	1,0	1,0
Mjukpunkt efter upphettning 163 C, minimum	C	1427 d)	48	45	41	37	-
Bibehållen penetration efter upphettning 163 C, minimum	%	1426 d)	50	46	43	37	-
Mjukpunktsökning efter upphettning 163 C, maximum	C	1427 d)	9	9	10	11	-
Viskositetskvot för viskositet vid 60 C, maximum		12596 d)	-	-	-	-	4,0

*ATB VÄG 2005 / VVTBT

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DSR (Dynamic Shear Rheometer)

Shearing between oscillating spindle and fix plate

Measures:

Complex modulus G*, total shear resistance

Phase angle δ, relationship between elastic and viscous part
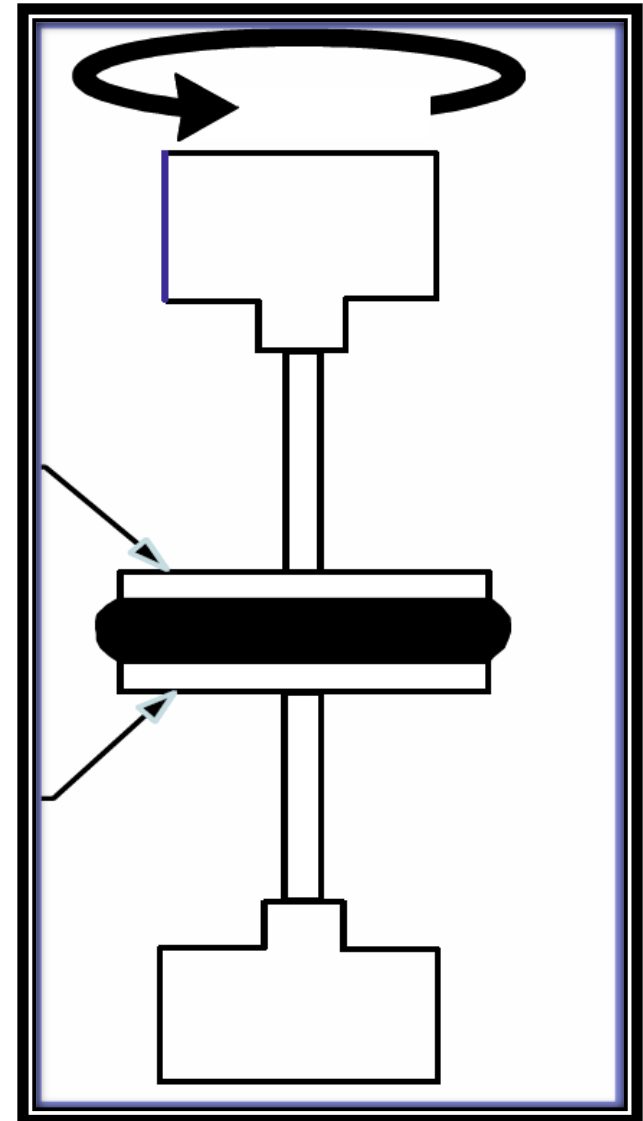
Complex modulus

$$\tau_{\max} = \frac{2T}{\pi r^3}$$

$$\gamma_{\max} = \frac{\theta r}{h}$$

$$G^* = \frac{\tau_{\max}}{\gamma_{\max}}$$
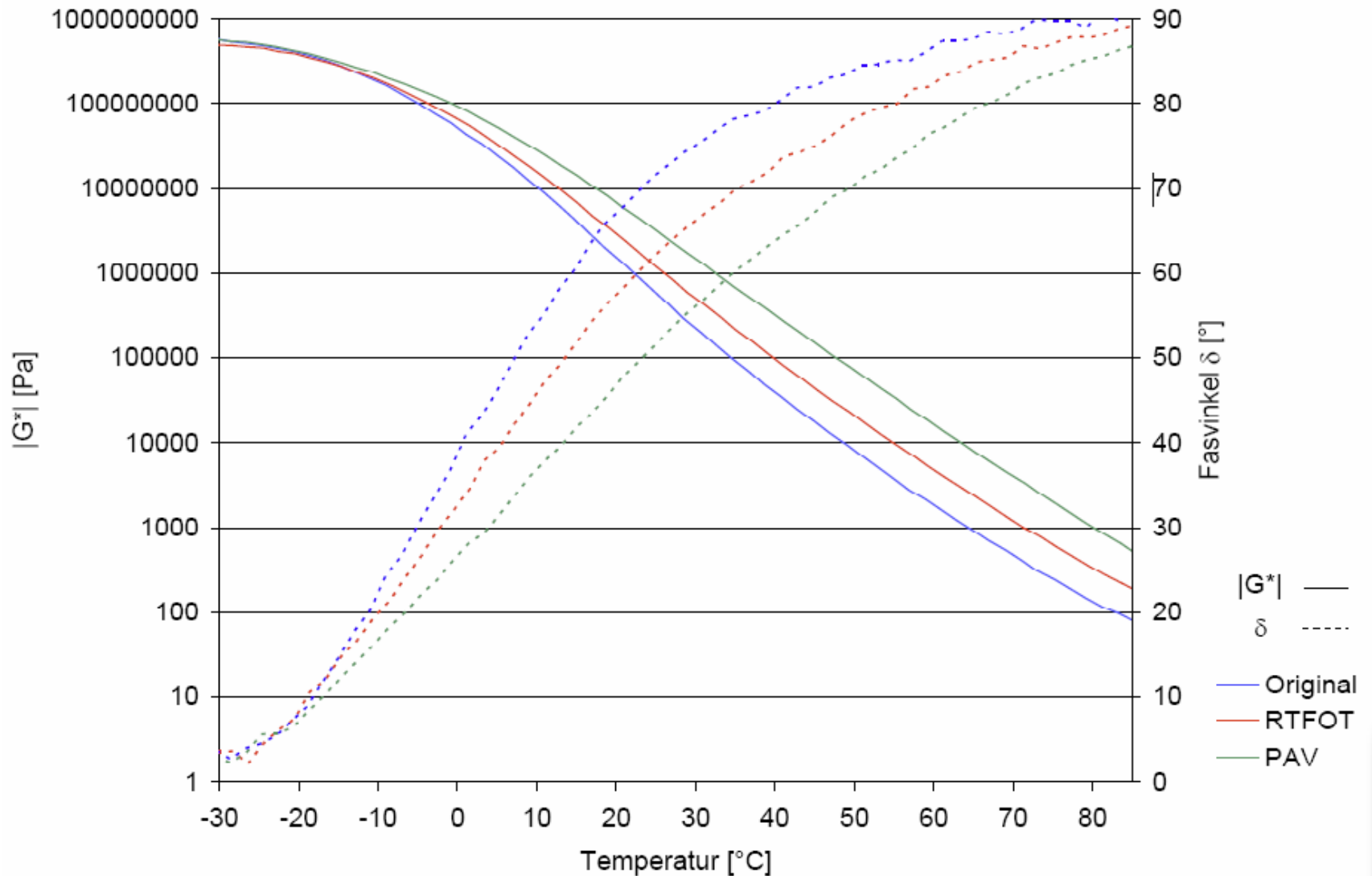
Binder Modification

Uses of binder Modification

Mitigate both traffic- and environmentally induced HMA pavement distresses

Rutting resistance

- Stiffer asphalt binders at warm temps

Thermal cracking

- Inhibit crack propagation

- Internal elastic network (polymers)

Most common modifiers

- Polymers
  - Rubber
  - Styrene-Butediene-Styrene (SBS)

- Waxes
  - Montan
  - Paraffin
  - Sasobit

- Fillers
  - Limestone

Modifier's effects

Bitumen	Penetration (dmm)	Softening point (°C)	Brookfield viscosity (mPa s)		Forced ductility at 10 °C (Nm)	Penetration index
			At 135 °C	At 165 °C		
Unmodified 70/100	81	46	345	101	1.38	−1.1
+4% wax AB	52	85	263	82	3.54	5.1
+4% wax FT	45	89	270	80	4.03	5.2

*Das et al 2012

TESTS		PMB32	PMB32+ 4% WAX
Softening Point	°C	75	93
Penetration	dmm	53	45
Breaking Point Fraass	°C	-14	-11
Elastic recovery at 10 °C	%	72,5	53,4
Brookfield visc at 135 °C @20RPM	mPas	1544	1394
Brookfield visc at 180 °C @50RPM	mPas	258	192

*Butt et al 2009

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Activities in the afternoon

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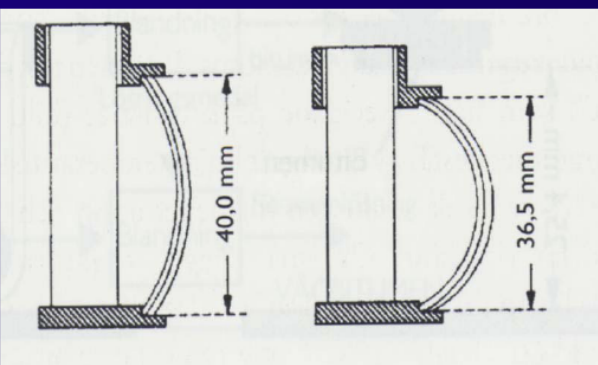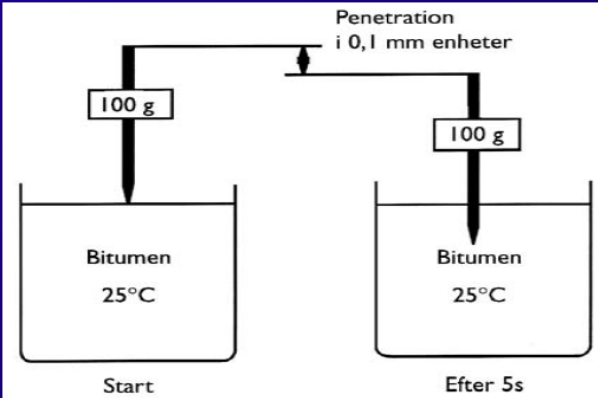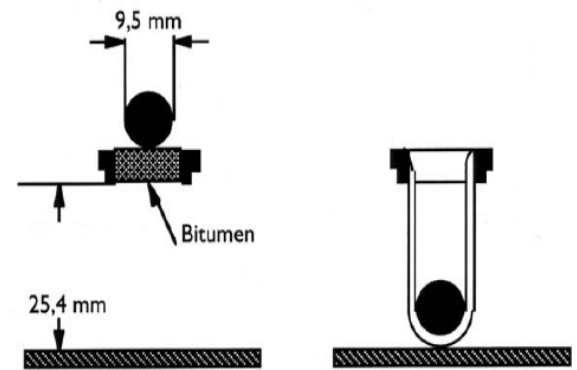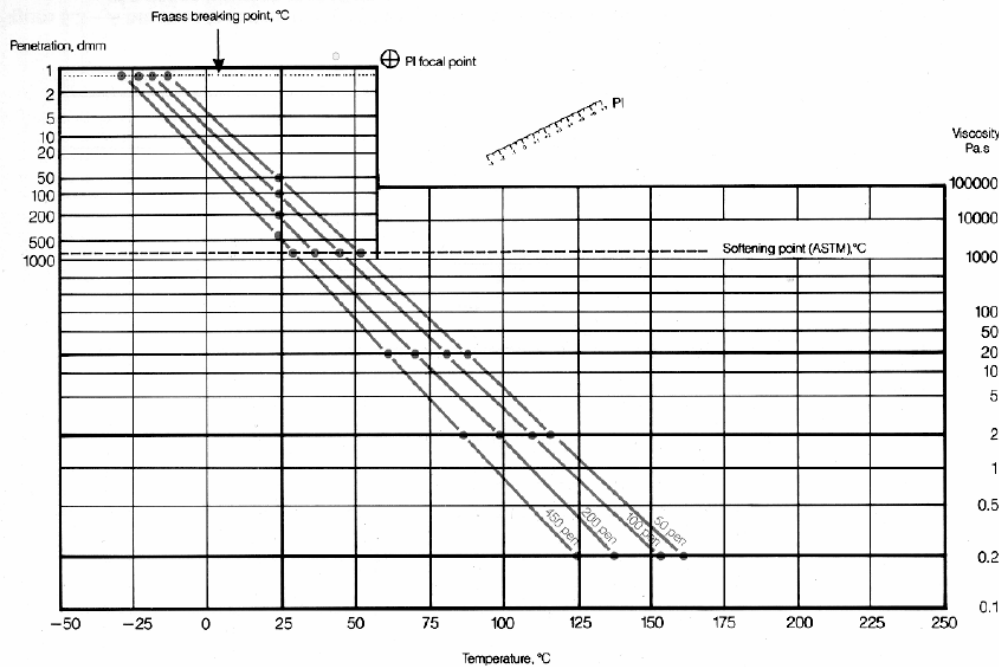
The largest SHRP asphalt research program ($53 million) (FHWA, 1998), had three primary objectives:

- **WHAT?**
Investigate why some pavements perform well, while others do not.

- **HOW?**
Develop tests and specifications for materials that will out-perform and outlast the pavements being constructed today.

- **WHERE?**
Work with highway agencies and industry to have the new specifications put to use.

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The final product of this research program was a new system referred to as "Superpave", which stands for SUperior PERforming asphalt PAVEments.

Superpave, in its final form consists of three basic components:

- *An asphalt binder specification.* This is the PG asphalt binder specification.

- *A design and analysis system based on the volumetric properties of the asphalt mix.* This is the Superpave mix design method.

- *Mix analysis tests and performance prediction models.* This area is not yet complete. Test development and evaluation is on-going.

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Focus on:

Binder Grading Systems

1. Background
2. Methods and Equipment
3. Use and Evaluation

Specifications

Standard Paving Grade –EN 12591 (1999)
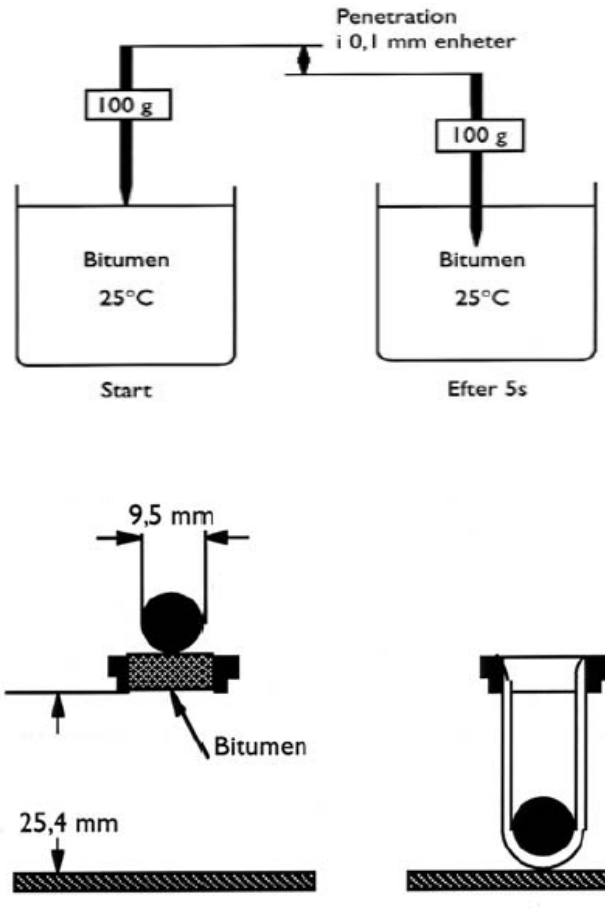
Penetration at 25 °C, 0,1 mm	EN-1426	70-100
Kinematic viscosity at 135 °C, mm²/s	EN-12595	Min. 230
Dynamic viscosity at 60 °C, Pa s	EN-12596	Min. 120
Softening point, °C	EN-1427	43-51
Fraass breaking point, °C	EN-12593	Max. -10
Solubility, %	EN-12592	Min. 99,0
Flash point, °C	EN-22592	Min. 230
Density at 25 °C, kg/m³	EN-ISO-3838	measure
After RTFOT		
Change of mass, %	EN-12607-1	Max. 0,8
Softening point, °C	EN-1427	Min. 45
Retained penetration, %	EN-1426	Min. 46
Increase in softening point, °C	EN-1427	Max. 9

Penetration and Softening Point for CEN-Specifications
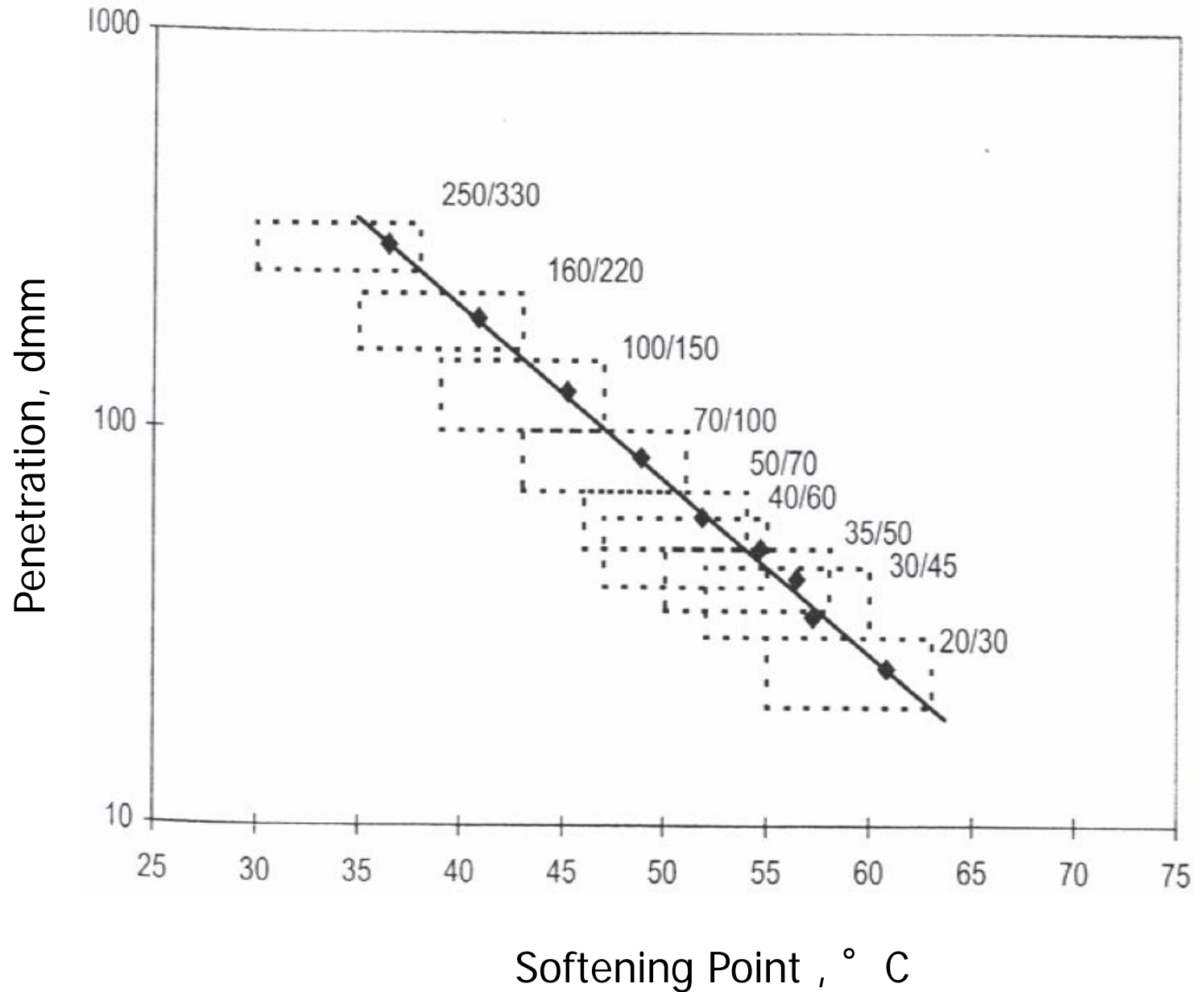
Pmb 100/150 - 75

Pmb	100/150	75
• Polymer modified bitumen	• Penetration	• Softening point

Soft bitumen V **1500**

↑ **Viscosity**

Emulsion BE **60** R 160/220

↑ **Bitumen content %**

↑ **Penetration**

- **General**

- **Equipments**

- **Test methods**

- **Specifications**

Performance Grade

PG 76 - 22

Tmax

- The highest measured 7-day temperature 20 mm under the pavement surface (average7-day max pavement design temp)

Tmin

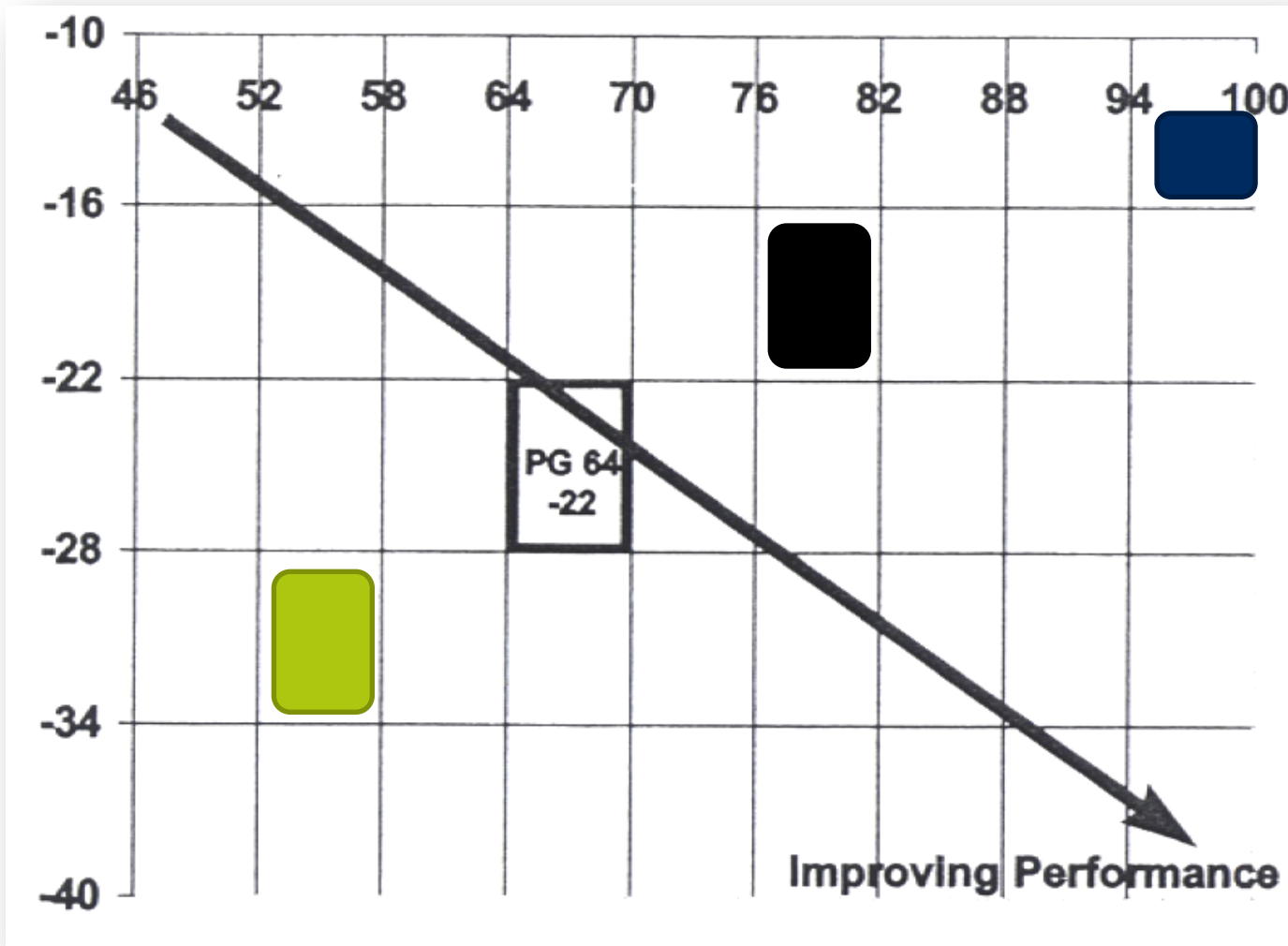- The lowest measured temperature in the pavement surface (min pavement design temp)

Superpave Binder Grades

Performance according to Superpave

Northern Sweden

Southern Sweden

Middle East

Performance Grades (PG)

Table 1: Performance Graded Asphalt Binder Specifications (from AASHTO, 2001)

Performance Grade	PG 46			PG 52							PG 58					PG 64					
	34	40	46	10	16	22	28	34	40	46	16	22	28	34	40	10	16	22	28	34	40
Average 7-day Maximum Pavement Design Temperature, °C[a]	< 46			< 52							< 58					< 64					
Minimum Pavement Design Temperature, °C[a]	-34	-40	-46	-10	-16	-22	-28	-34	-40	-46	-16	-22	-28	-34	-40	-10	-16	-22	-28	-34	-40
ORIGINAL BINDER																					
Flash Point Temp, T 48, Minimum (°C)	230																				
Viscosity, ASTM D 4402:[b] Maximum, 3 Pa*s, Test Temp, °C	135																				
Dynamic Shear, TP 5:[c] G*/sinδ', Minimum, 1.00 kPa Test Temp @ 10 rad/s, °C	46			52							58					64					
ROLLING THIN FILM OVEN RESIDUE (T 240)																					
Mass Loss, Maximum, percent	1.00																				
Dynamic Shear, TP 5: G*/sinδ', Minimum, 2.20 kPa Test Temp @ 10 rad/s, °C	46			52							58					64					
PRESSURE AGING VESSEL RESIDUE (PP 1)																					
PAV Aging Temperature, °C[d]	90			90							100					100					
Dynamic Shear, TP 5: G*/sinδ', Maximum, 5000 kPa Test Temp @ 10 rad/s, °C	10	7	4	25	22	19	16	13	10	7	25	22	19	16	13	31	28	25	22	19	16
Physical Hardening[e]	Report																				
Creep Stiffness, TP 1 Determine the critical cracking temperature as described in PP 42	-24	-30	-36	0	-6	-12	-18	-24	-30	-36	-6	-12	-18	-24	-30	0	-6	-12	-18	-24	-30
Direct Tension, TP 3 Determine the critical cracking temperature as described in PP 42	-24	-30	-36	0	-6	-12	-18	-24	-30	-36	-6	-12	-18	-24	-30	0	-6	-12	-18	-24	-30

Climate conditions →

Rutting →
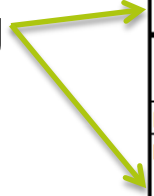
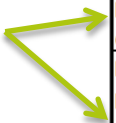Fatigue →

Brittle Fracture →

PAV - Pressure Aging Vessel

Simulates long term aging

RTFO - Rolling Thin Film Oven

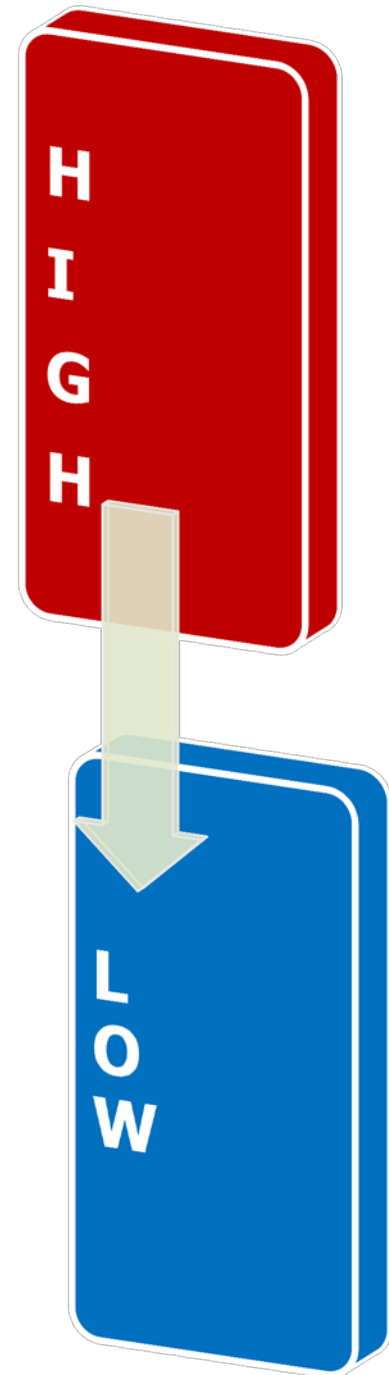Simulates short term aging

RV- Rotational Viscosity

Measures performance at high temperatures

DSR – Dynamic Shear Rheometer

Measures performance at medium and normal temperatures

BBR – Bending Beam Rheometer

Measures performance at low temperatures

HIGH

LOW

Bitumen aging

Oxidation

-Irreversible chemical reaction

-Formation of functionalities containing oxygen

Evaporation

-Loss of volatile components

Exudation
-Loss of oily components due to exudation of bitumen into the mineral aggregate
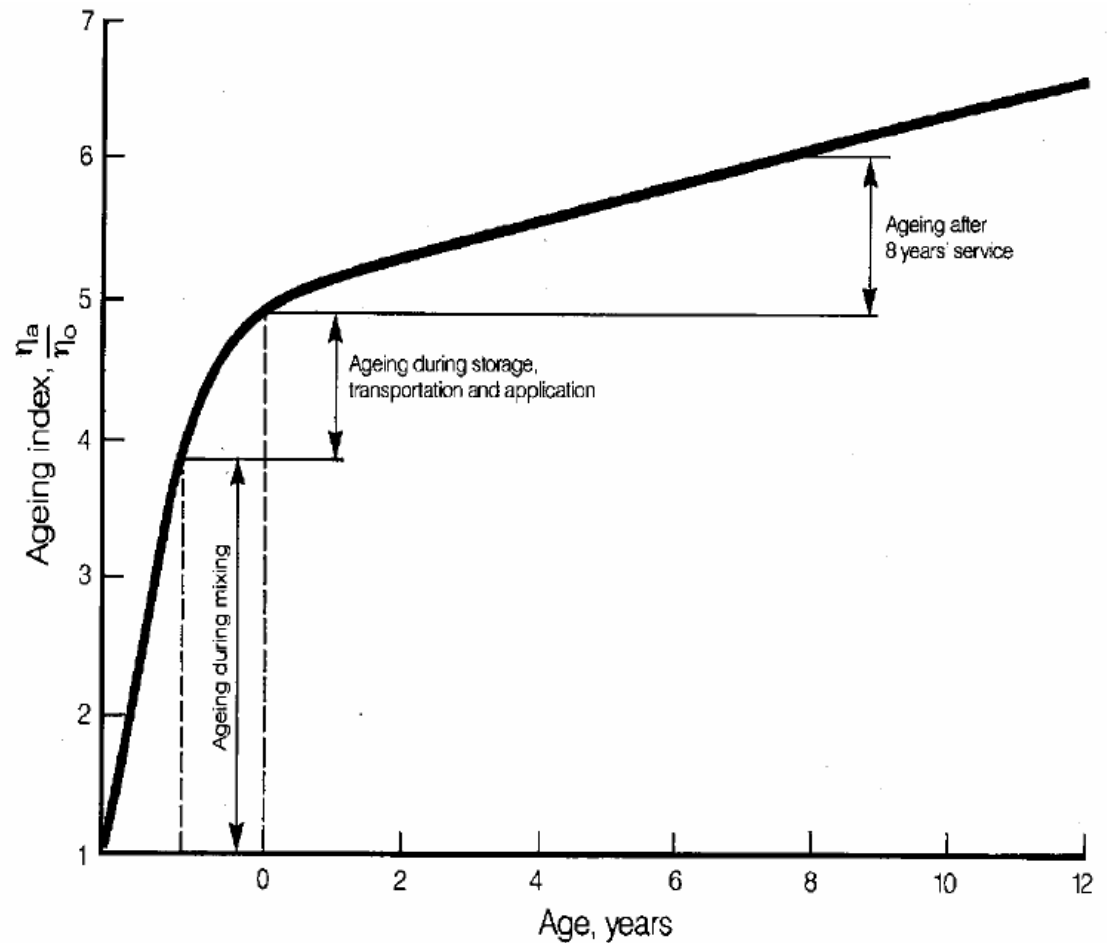
Physical hardening
-Molecular association (steric hardening)

-Low temperature physical hardening

Most during production;

Storage transport and laying;

In the pavement.

Aging

Binder aged in
RTFO (Rolling Thin Film Oven)

Binder aged in
PAV (Pressure Aging Vessel)

RTFO – Rolling Thin Film Oven

- RTFO simulates aging of the binder during mixing and laying of an asphalt mix (short time aging).

- Aging in rotating bottles, 85 min/163° C.

- Weight loss is determined.

- DSR analysis.

- The main part of the aged bitumen sample will then be further aged in PAV.

# PAV - Pressure Aging Vessel

- PAV simulates long time aging on the road. (long term aging).

- Aging under high pressure (2070 KPa) at 90, 100 or 110° C/20 hours.

- DSR (G*sinδ) and BBR (S and m) and possibly DTT.

- Rotational Viscometer (Brookfield) for evaluating the workability at higher temperatures (production, mixing, transport and laying)

- The dynamic viscosity is measured at 135° C **< 3 Pas**
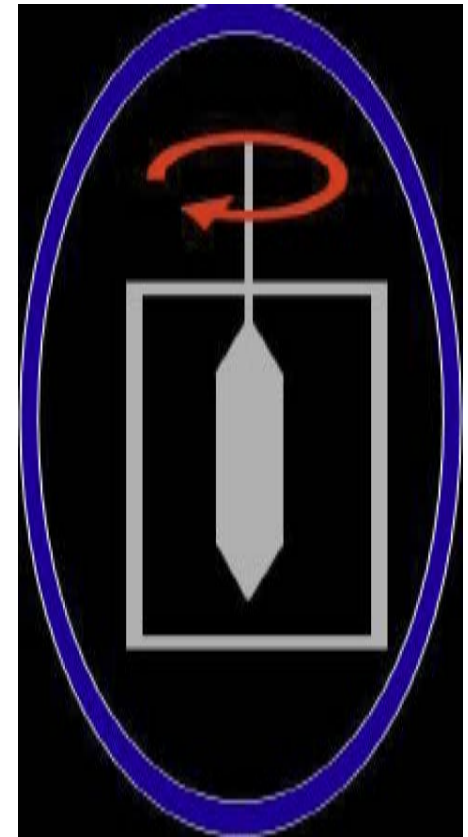
DSR - Dynamic Shear Rheometer

- Shearing between oscillating spindle and fix plate

- Frequency: 10 rad/s (1.57 Herz) at particular temperature.

Measures:

- Complex modulus G*, total shear resistance

- Phase angle δ, relationship between elastic and viscous part.

- Shear strain: 1 - 12%

- Temp range: 4 to 85° C (G* between 0.1 and 10 000 KPa)

Complex modulus *Versus Temperature*

Phase angle, Deg vs. Temperature, °C

PMB32+4% wax (non-aged)
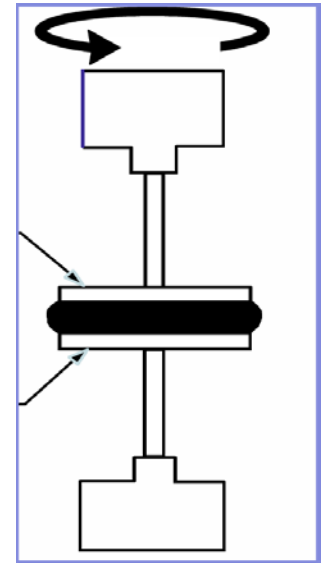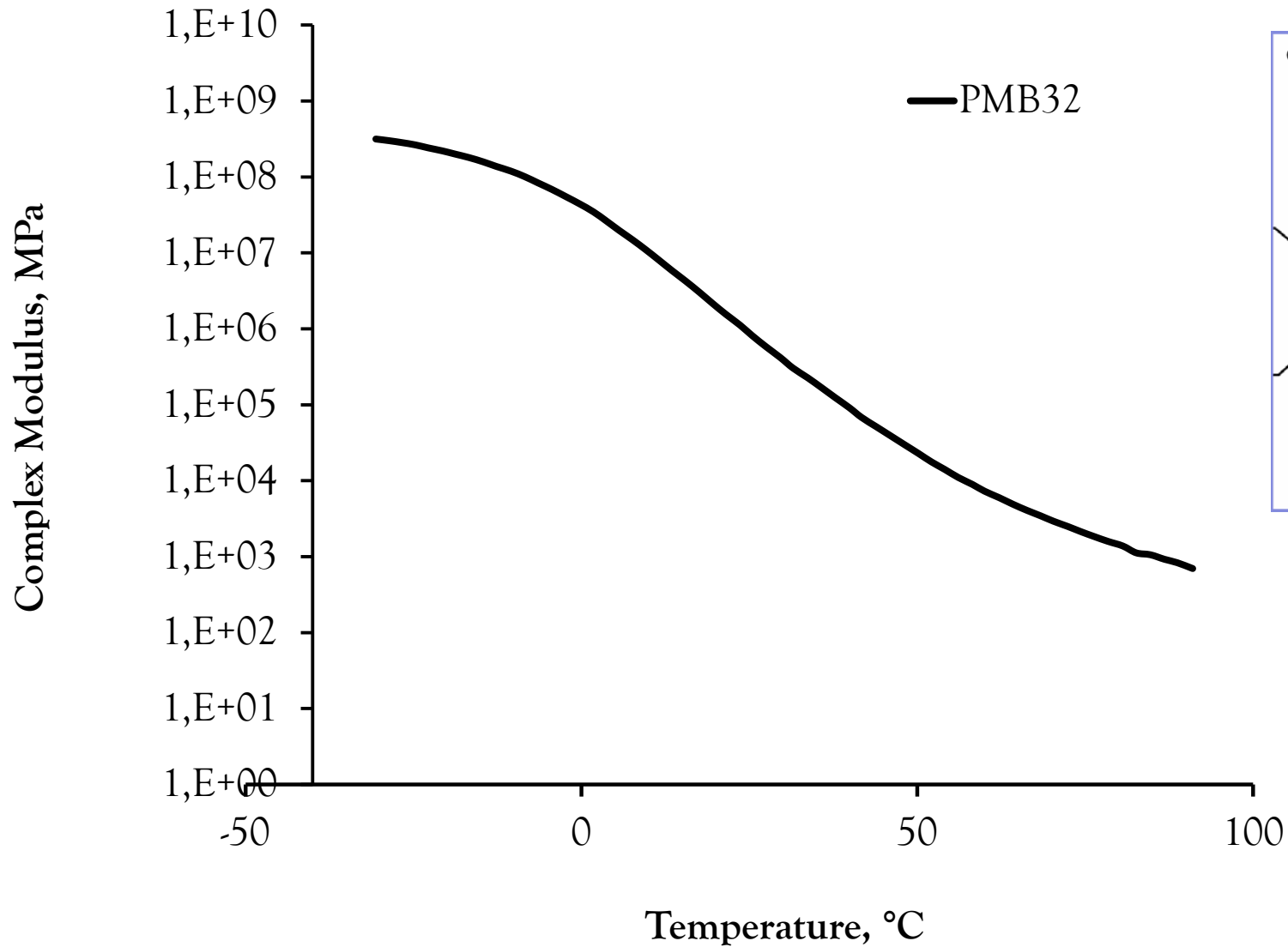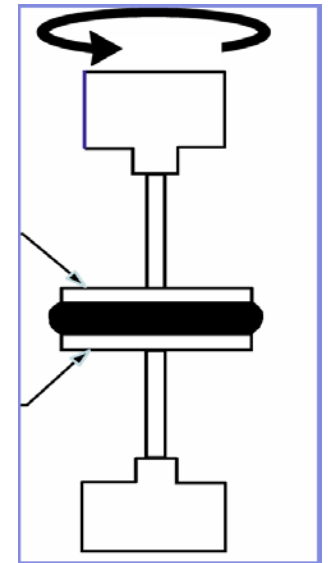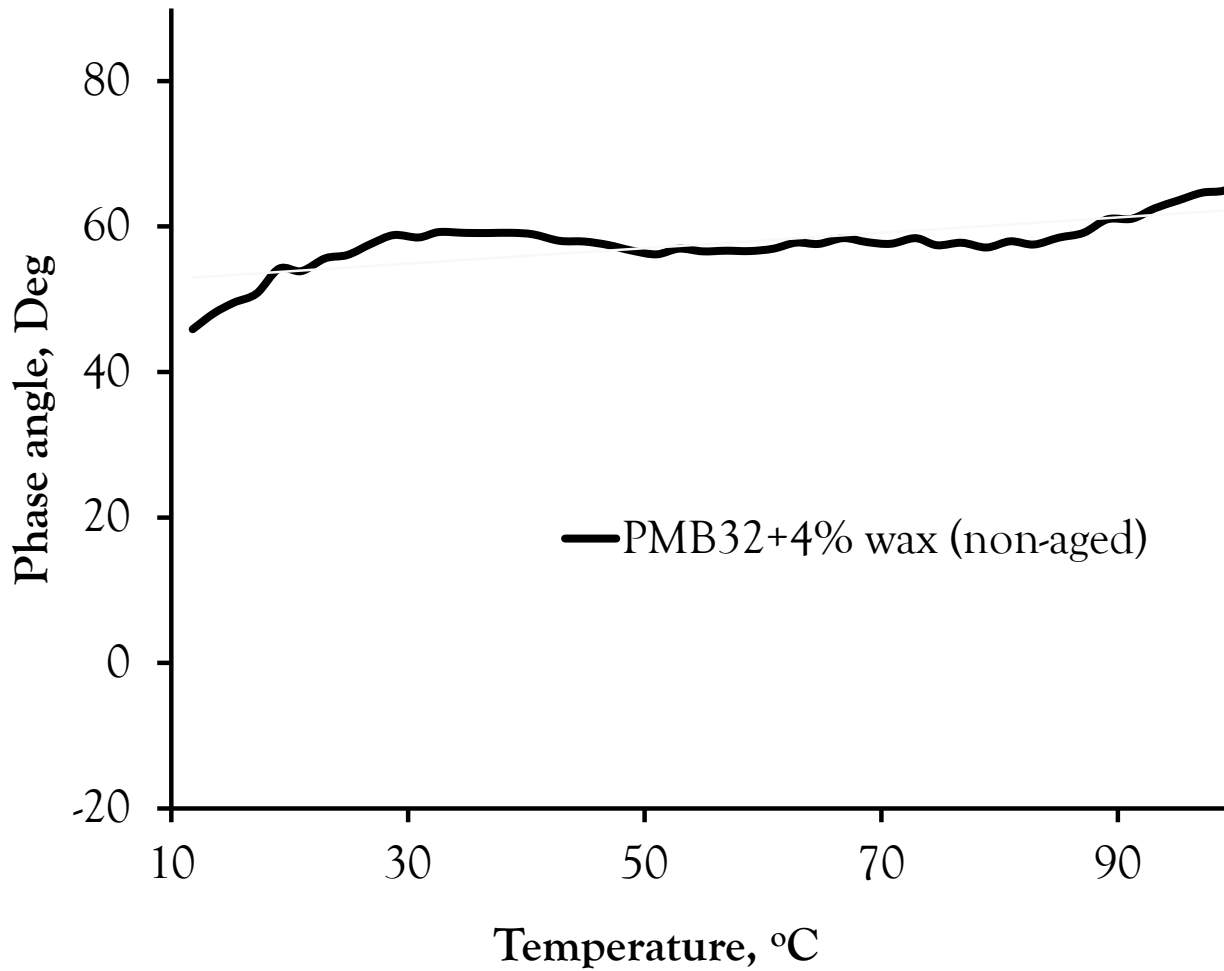
Requirements

Permanent deformation is controlled by:

- $G*/\sin\delta$ for non aged binder > 1.00 KPa

- $G*/\sin\delta$ for RTFO aged binder > 2.20 KPa

Requirements

Fatigue is controlled by:

- $G*\sin\delta$ for RTFOT/PAV aged material = max 5 MPa

Viscous Part

G*

δ

Elastic Part
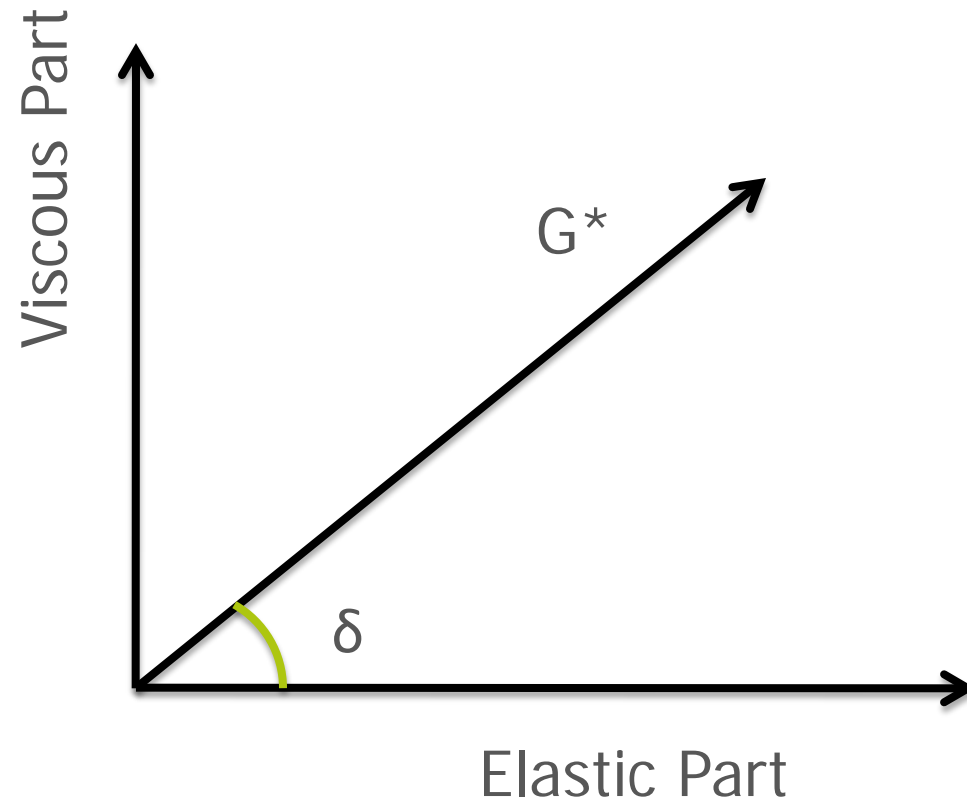
BBR (Bending Beam Rheometer)
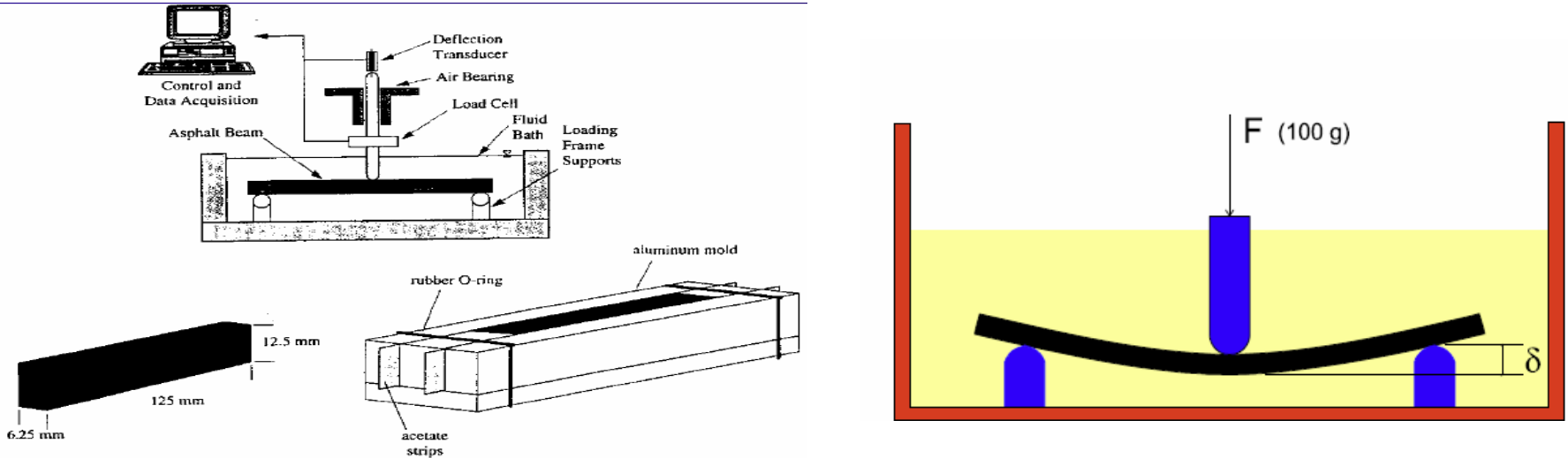
- For testing at low temperatures where bitumen is very stiff.

- A small beam of binder is loaded to simulate stresses built up at falling temperature in a pavement.

- The midpoint deflection of the beam versus time (4 minutes) is measured.

- Flexural creep stiffness S and m-value under creep loading.

- The test is performed at a temperature 10° C higher than the expected lowest pavement temperature.
- The requirement of <300 MPa corresponds to the stiffness after 2 hours loading. Increasing the test temperature leads to a shorter testing time of **60sec.**
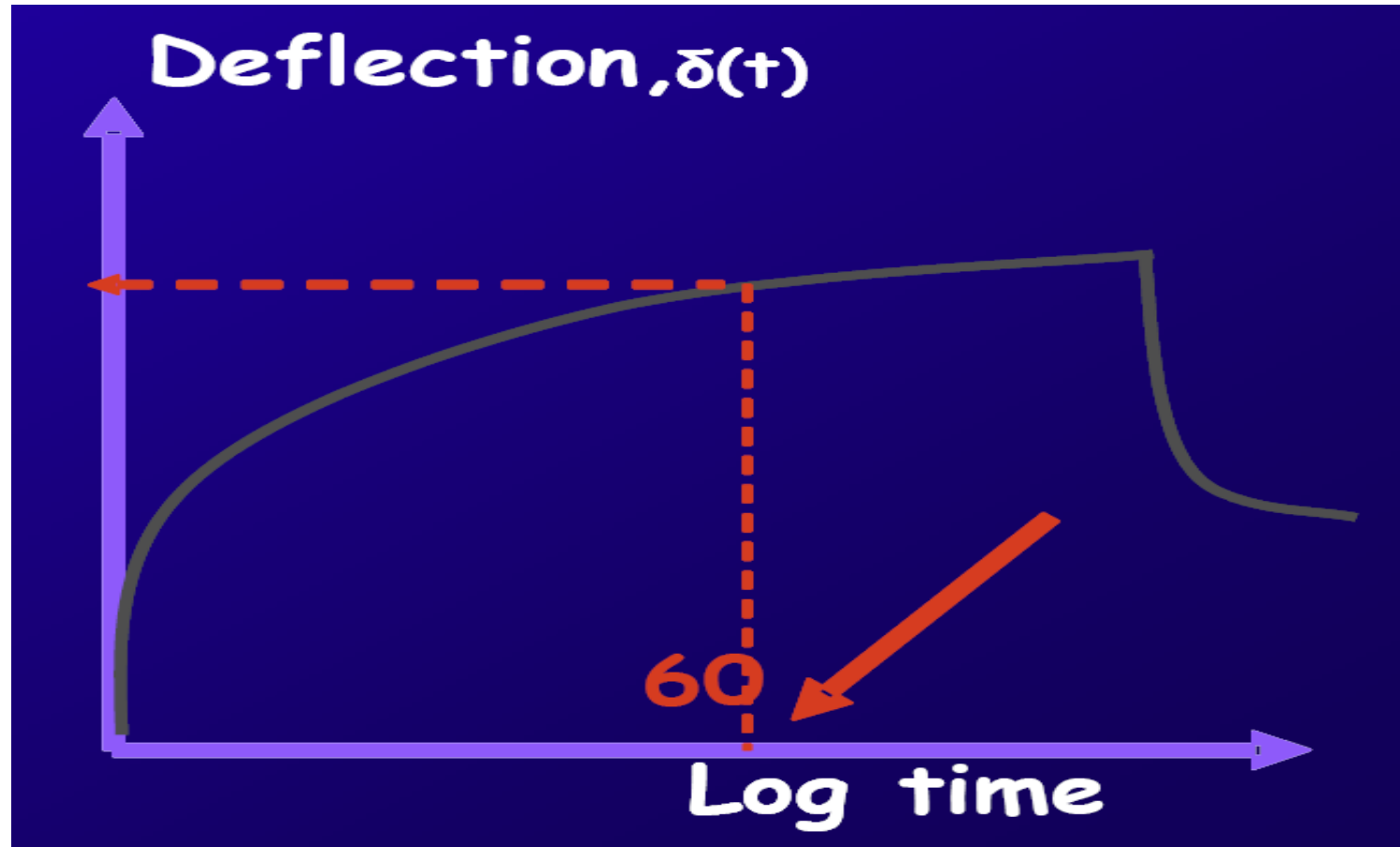
$S(t) = PL^3/4bh^3\delta(t)$

*Equation and registered deflection give the stiffness S at 60 sec.

m-value is the slope of the stiffness and time on the log-log scale.
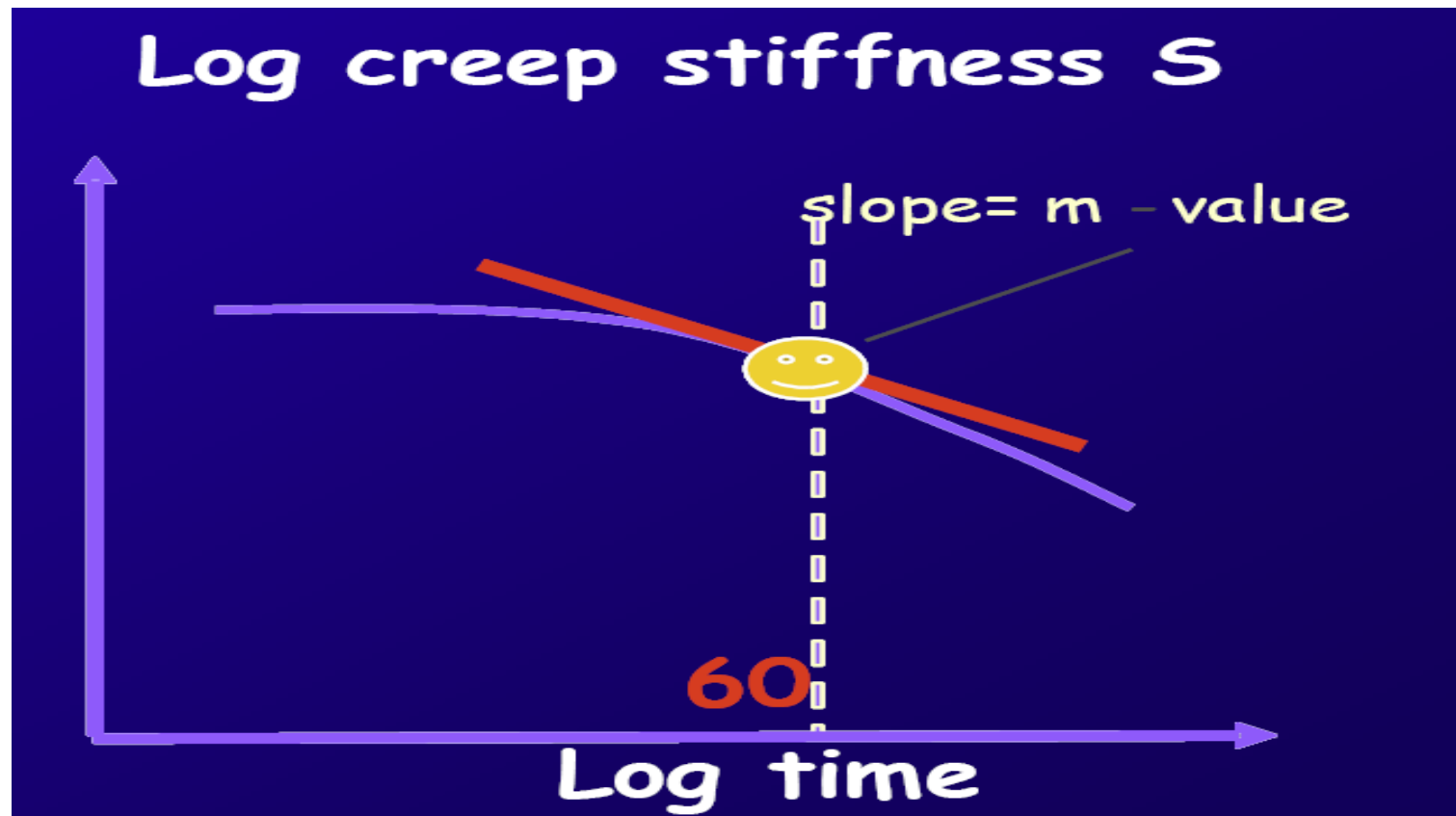
Requirements

Cracking at low temperature is controlled by:

- S for RTFO/PAV aged binder < 300 MPa
- m-value for RTFO/PAV aged binder > 0.300
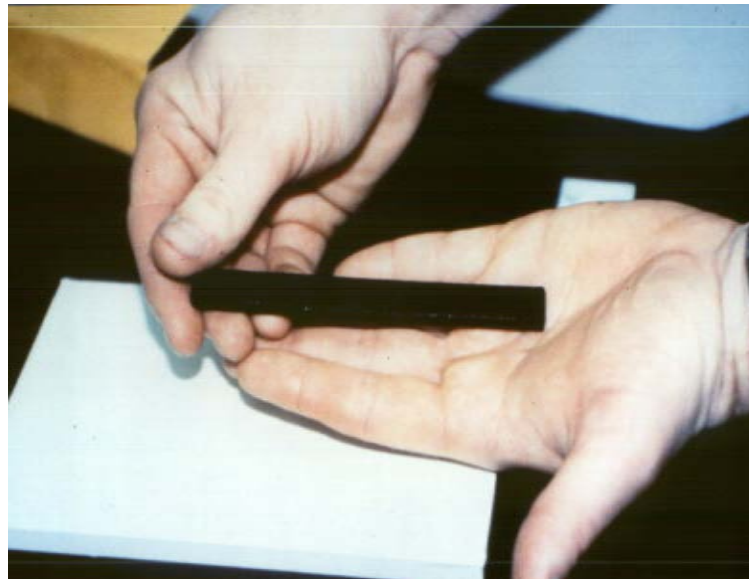
- The current Superpave binder specification does not appear to adequately determine the performance characteristics of modified binders.

- Many Highway agencies have included additional tests to the existing Superpave binder specification to assure a desired modifier is included in the binder.

- In the near future we will have the answers ………Until that time, the Highway agencies will have to use engineering judgement in specifying modified asphalt binders.

Flow Chart for PG-qualities According to Superpave

Tank binder	RV viscosity at 135°C — Viscosity $\mu < 3Pas$	Pumpability
	COC Flash Point Temperature $> 230°C$	
	DSR at T_{max} — $G^*/sin\delta > 1.00$ kPa	Rutting
RTFOT residue	DSR at T_{max} — $G^*/sin\delta > 2.20$ kPa	
	BBR at T_{min} — $S(60s) < 300MPa; m(60) > 0.300$	Thermal cracking
PAV residue	DT at T_{min} — $E_f > 1.0\%$ at T_{min}	
	DSR at $T_{intermediate}$ — $G^*sin\delta < 5.00$ MPa	Fatigue

Established Grade

ali.b
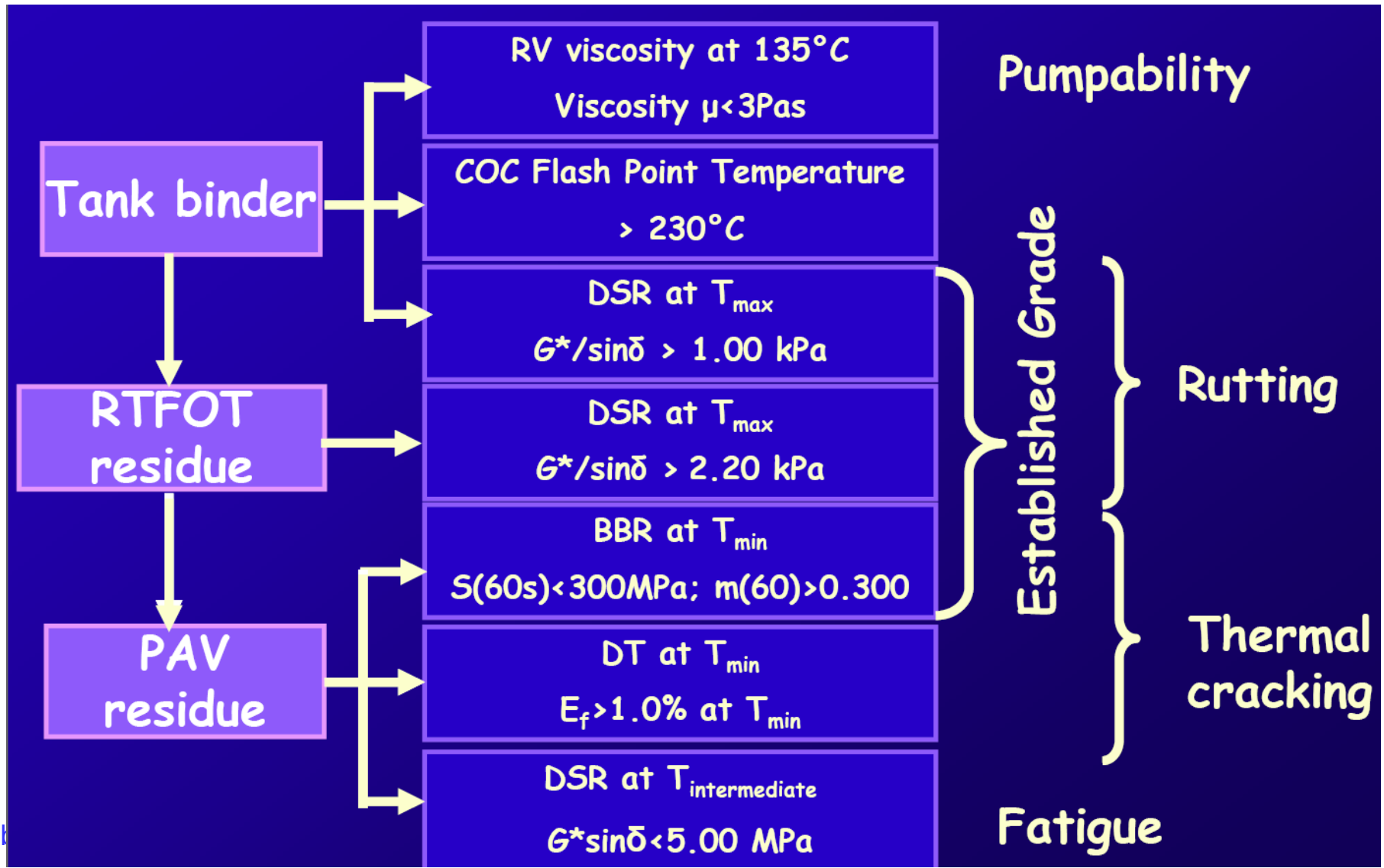
Performance Grades

Avg 7-day Max, °C	PG 46	PG 52	PG 58	PG 64	PG 70	PG 76	PG 82
1-day Min, °C	-34 -40 -46 -28 -34	-10 -16 -22 -28 -34 -40 -46	-16 -22 -28 -34	-40 -10 -16 -22 -28 -34 -40	-10 -16 -22 -28 -34 -40	-10 -16 -22 -28 -34	-10 -16 -22

ORIGINAL

≥ 230 °C	(Flash Point) **FP**						
≤ 3 Pa·s @ 135 °C	(Rotational Viscosity) **RV**						
≥ 1.00 kPa	(Dynamic Shear Rheometer) **DSR** G*/sin δ						
	46	52	58	64	70	76	82

(ROLLING THIN FILM OVEN) RTFO Mass Loss ≤ 1.00 %

≥ 2.20 kPa	(Dynamic Shear Rheometer) **DSR** G*/sin δ						
	46	52	58	64	70	76	82

(PRESSURE AGING VESSEL) PAV

	PG 46	PG 52	PG 58	PG 64	PG 70	PG 76	PG 82
20 Hours, 2.07 MPa	90	90 100	100	100 (110)	100 (110)	110 (110)	
≤ 5000 kPa	(Dynamic Shear Rheometer) **DSR** G* sin δ						
	10 7 4	25 22 19 16 13 10 7	25 22 19 16 13	31 28 25 22 19 16	34 31 28 25 22 19	37 34 31 28 25	40 37 34
S ≤ 300 MPa m ≥ 0.300	(Bending Beam Rheometer) **BBR** "S" Stiffness & "m"-value						
	-24 -30 -36 -18 -24	0 -6 -12 -18 -24 -30 -36	-6 -12 -18 -24 -30	0 -6 -12 -18 -24 -30	0 -6 -12 -18 -24 -30	0 -6 -12 -18 -24	0 -6 -12
Report Value	(Bending Beam Rheometer) **BBR** Physical Hardening						
≥ 1.00 %	(Direct Tension) **DT**						
	-24 -30 -36 -18 -24	0 -6 -12 -18 -24 -30 -36	-6 -12 -18 -24 -30	0 -6 -12 -18 -24 -30	0 -6 -12 -18 -24 -30	0 -6 -12 -18 -24	0 -6 12

Permanent Deformation

Addressed by high temp stiffness;

- G*/sin δ on unaged binder >1.00 KPa

- G*/sin δ on RTFO aged binder >2.20 KPa

Fatigue Cracking

Addressed by intermediate temperature stiffness;

- G*sin δ on RTFO & PAV aged binder <5000 kPa

Low Temperature Cracking

Addressed by low temperature stiffness and m - value;

- S for RTFO/PAV aged binder < 300 Mpa

- m-value for RTFO/PAV aged binder > 0.300

- Conformance testing: Does this binder sample meet all specification requirements of a certain performance grade (such as PG64-28)?

- Classification testing: What performance grade (or grades) does this binder sample meet? A series of tests are performed to classify an unknown binder, using a trial and error process.

- In the Matlab Exercise, you will program a decision tree to do Superpave Performance Grade (PG) Binder classification.

Bitumen conforming to PG 64-28

Binder property / Test	Results	Unit	Requirement
Original			
Flashpoint (Cleveland open cup)	**293**	[°C]	230 min
Viscosity at 135 °C (Brookfield)	**0.3**	[Pas]	3 max
DSR $\left|G*\right|\big/\sin\delta$ at 64 °C	**1.31**	[kPa]	1.00 min
After RTFOT			
Weight loss after RTFOT	**0.32**	[%]	1.00
DSR: $\left|G*\right|\big/\sin\delta$ at 64 °C	**2.63**	[kPa]	2.20 min
After RTFOT + PAV (100 °C)			
DSR: $\left|G*\right|\cdot\sin\delta$ at 22 °C	**4517**	[kPa]	5000 max
BBR at -18 °C Stiffness after 60 sec m-value	**274** **0.346**	[MPa] [ ]	300 max 0.300 min
BBR at -18 °C Stiffness After 24 h m-value	**0.350** **0.300**	[MPa] [ ]	To be reported

ORIGINAL BINDER

Flash point: 340 ° C
Viscosity: 0.42 Pas

Modulus (G*/sinδ) at;

64° C :	**70° C:**
1.61 KPa	0.72 KPa

RTFOT

Change in mass: 0.08 %

Modulus (G*/sinδ) at;

58° C :	**64° C:**
7.28 KPa	3.24 KPa

PAV

Modulus (G*sinδ) at;

25° C :   **22° C:**   **19° C:**   **16° C:**

1943 kPa   2917 kPa   4047 kPa   5442 kPa

BBR

Creep stiffness/m-value at;

-6° C: **-12° C :**   **-18° C:**

46.3 MPa   107 MPa   229 MPa

0.411   0.350   0.305

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PG 64-28

Binder property / Test	Results	Unit	Requirement
Unaged			
DSR $\lvert G*\rvert/\sin\delta$ at 58 °C		[kPa]	1.00 min
DSR $\lvert G*\rvert/\sin\delta$ at 64 °C	1.61	[kPa]	1.00 min
DSR $\lvert G*\rvert/\sin\delta$ at 70 °C	0.72	[kPa]	1.00 min
After RTFOT			
DSR: $\lvert G*\rvert/\sin\delta$ at 58 °C	7.28	[kPa]	2.20 min
DSR: $\lvert G*\rvert/\sin\delta$ at 64 °C	3.24	[kPa]	2.20 min
DSR: $\lvert G*\rvert/\sin\delta$ at 70 °C		[kPa]	2.20 min
After RTFOT + PAV (100 °C)			
DSR: $\lvert G*\rvert\cdot\sin\delta$ at 13 °C		[kPa]	5000 max
DSR: $\lvert G*\rvert\cdot\sin\delta$ at 16 °C	5445	[kPa]	5000 max
DSR: $\lvert G*\rvert\cdot\sin\delta$ at 19 °C	4047	[kPa]	5000 max
DSR: $\lvert G*\rvert\cdot\sin\delta$ at 22°C	2917	[kPa]	5000 max
DSR: $\lvert G*\rvert\cdot\sin\delta$ at 25°C	1943	[kPa]	5000 max
DSR: $\lvert G*\rvert\cdot\sin\delta$ at 28°C		[kPa]	5000 max
DSR: $\lvert G*\rvert\cdot\sin\delta$ at 31°C		[kPa]	5000 max
DSR: $\lvert G*\rvert\cdot\sin\delta$ at 34°C		[kPa]	5000 max
BBR at -6 °C Stiffness after 60 sec m-value	46.3 0.411	[MPa] [ ]	300 max 0.300 min
BBR at -12 °C Stiffness after 60 sec m-value	107 0.350	[MPa] [ ]	300 max 0.300 min
BBR at -18 °C Stiffness after 60 sec m-value	229 0.305	[MPa] [ ]	300 max 0.300 min
BBR at -24 °C Stiffness after 60 sec m-value		[MPa] [ ]	300 max 0.300 min

PG 58 – 22 ???

Measure G*/sinδ on unaged binder at 58°C

↓

G*/sinδ ≥ 1,00 kPa at 58°C ? → **NO** → Binder does not conform to PG58-22

↓ **YES**

Measure G*/sinδ at RTFO aged binder at 58°C

↓

G*/sinδ ≥ 2,20 kPa at 58°C ? → **NO** → Binder does not conform to PG58-22

↓ **YES**

Measure G*sinδ at RTFO/PAV aged binder at 22°C

↓

G*sinδ ≤ 5000 kPa at 22°C ? → **NO** → Binder does not conform to PG58-22

↓ **YES**

Measure creep stiffness at -12°C

PG 58 – 22 ???

Measure creep stiffness at -12°C

↓

S<300MPa and m>0,300 ?

YES → Binder conforms with requirements for a PG58-22

NO ↓

300<S<600MPa and m≥0,300

YES → Measure tensile failure strain at -12°C

NO ↓

Binder does not conform to PG58-22

↓

Tensile failure at elongation≥ 1% ?

YES → Binder conforms to PG58-22

NO ↓

Binder does not conform to PG58-22

Break for Lunch