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# Test Methods and Specification Criteria for Mineral Filler Used in HMA

**PROGRAM (NCHRP) Project Number 9-45**

**NCHRP Webinar**

**19 May, 2010**

**NCHRP 9-45**



# Research Team

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# Objectives of the Study

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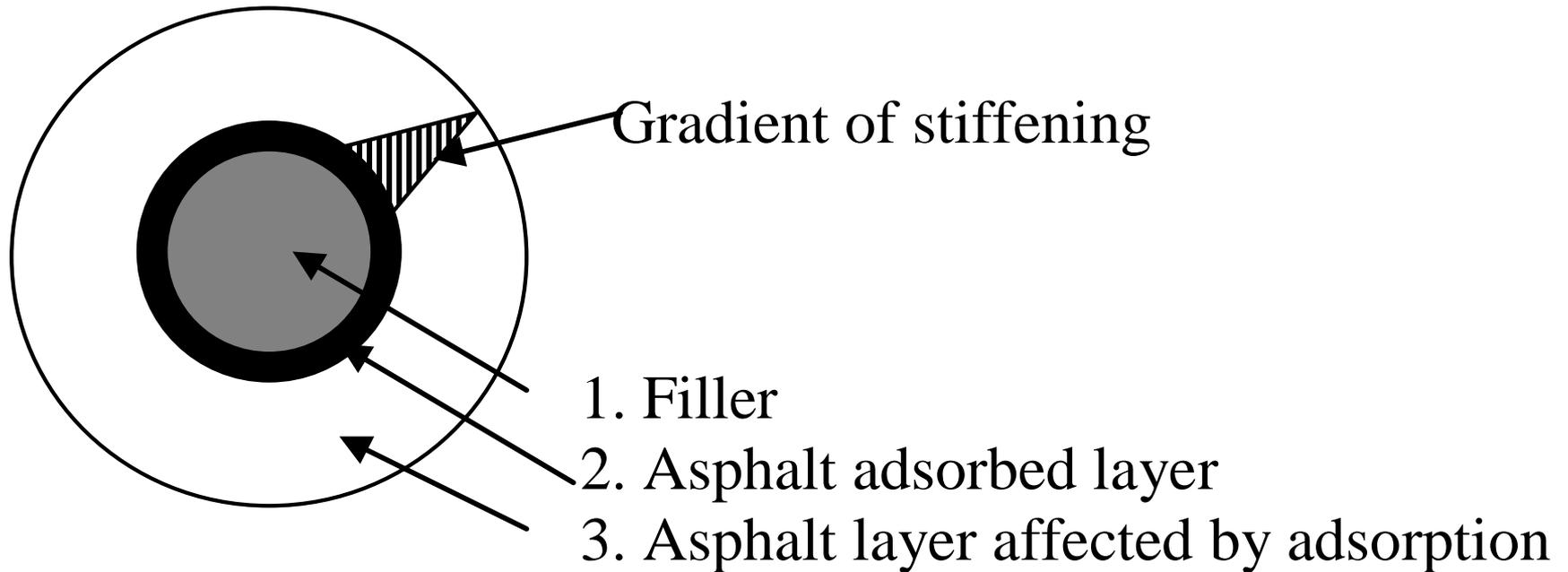
- (1) Identify or develop test methods for mineral filler that characterize its mechanical and chemical effects on the performance of :**
  - (a) mastics and**
  - (b) hot mix asphalt (HMA)**
  
- (2) Recommend specification criteria for mineral filler that optimize HMA performance**

# Project Tasks

- **Task 1: Conduct a review of the worldwide literature**
- **Task 2: Prepare an updated work plan- Select tests that show potential**
- **Task 3: Interim report - Experimental plan for testing**
- **Task 4: Conduct testing of mastics and Mixtures**
- **Task 5: Develop preliminary specifications**
- **Task 6: Assess sensitivity of practice to specifications**
- **Task 7: Final recommendations**

# Basic Concept of Study

## Fillers' Interaction with Binders( Tunnicliff in 1960)



# Literature Review Results

## Important Filler Properties - Geometry

- Similar to fine and coarse aggregates, fillers' geometry can be defined by four measurements: **size, shape, angularity, and texture.**
- Four properties are important:
  - **Two primary**
    - **Fractional Voids (FV)**
    - **Size Distribution and Surface Area**
  - **Two Secondary**
    - **Absorption**
    - **Specific Gravity**

# Literature Review Results

## Important Filler Properties - Composition

- There appears to be **a small number of chemical compounds** that affect asphalt-filler interactions.
- Two main properties are important:
  - **Reactivity**
    - Calcium compound.
    - Water solubility.
  - **Harmful fines**
    - Active Clay Content.
    - Organic Content.

# Task 2- Filler Tests Considered in Work Plan

## Part I- Geometry/ Physical Properties

1. Physical Property	Test Protocol	Remarks
• <u>Fractional voids</u>	Rigden Voids EN 1097-4	No development is necessary.
• <u>Size Distribution</u>	Laser Diffraction ASTM D4464	Type of surfactant and time of agitation will be studied.
• Specific Gravity	AASHTO T-133 & CoreLok	Both methods will be used to evaluate the possibility of measuring apparent and bulk SG
• Absorption	Bitumen Number (EN13179-2) & Laser Diffraction with Time	Not a standard method and some development is needed.
• Shape and Texture	Microscopy using AIMS or UIAIA software	Not a standard method and some development is needed.



# Task 2 – Filler Tests Proposed for Work Plan

## Part II- Composition/Chemical Properties

2. Chemical Property	Test Protocol	Remarks
<ul style="list-style-type: none"> <li>• <u>Calcium Compounds</u> <b>CaO</b></li> </ul>	X-ray Fluorescence, EN 196-21, EN 495-2 ASTM D3042 MN/DOT	Initial testing is needed to determine the appropriate test.
<ul style="list-style-type: none"> <li>• Water Solubility</li> </ul>	EN 1744-1:1998	No development is necessary.
<ul style="list-style-type: none"> <li>• <u>Methylene Blue / <b>MBV</b></u> Plasticity Index</li> </ul>	AASHTO TP57 AASHTO T90	Limited development for TP 57 will be needed.
<ul style="list-style-type: none"> <li>• Organic Content- Loss on Ignition</li> </ul>	EN 1744-1:1998 C17, AASHTO 267-86	Need to determine testing temperature.

# Results of SHAs Survey

## Included 8 questions

1. Does your agency have specifications / guidelines for ***natural*** mineral fillers?

Yes (**4 of 54**)      No (**50 of 54**)

2. Does your agency have specifications / guidelines for ***imported*** mineral fillers in any type of HMA?

Yes (**44 of 54**)      No (**10 of 54**)

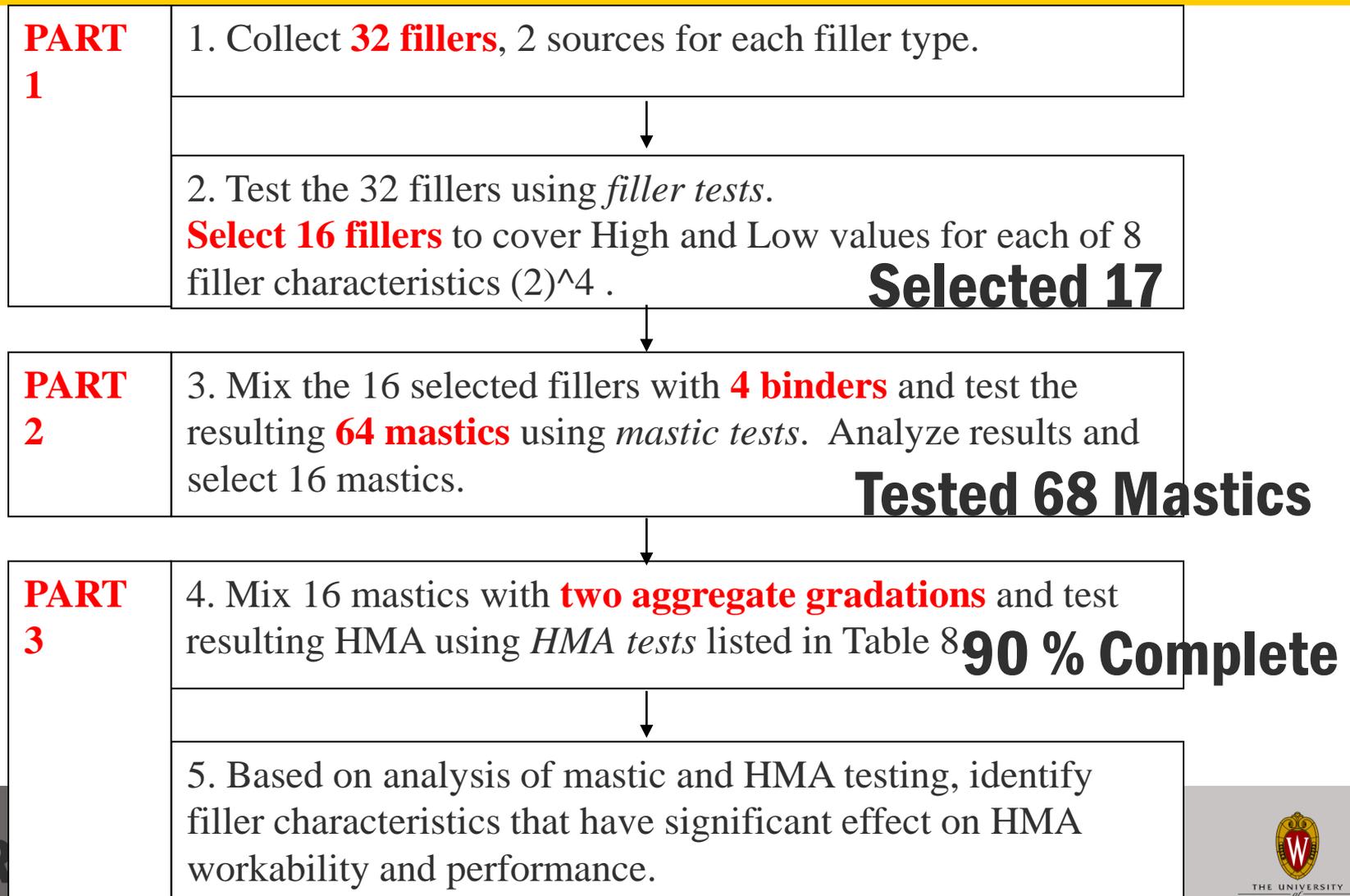
- If yes, what are the gradation limits you specify for imported fillers?
- ASTM D242 or AASHTO M17 (23 of 44), Miscellaneous (21 of 44)

3. Other than gradation, are there other characteristics of mineral filler that you believe have an important affect on HMA performance?

Yes (**27 of 39**)      No (**12 of 39**)

- (1) Plasticity (**12 of 27**)
- (2) Stripping of Mastics or HMA (**5 of 27**)
- (3) Loss on ignition (**4 of 27**)
- (4) Mastics Property (**2 of 27**)
- (5) Bar Linear Shrinkage (**2 of 27**)
- (6) Clay content (**2 of 27**)

# Overall Testing Plan



# Fillers Selected

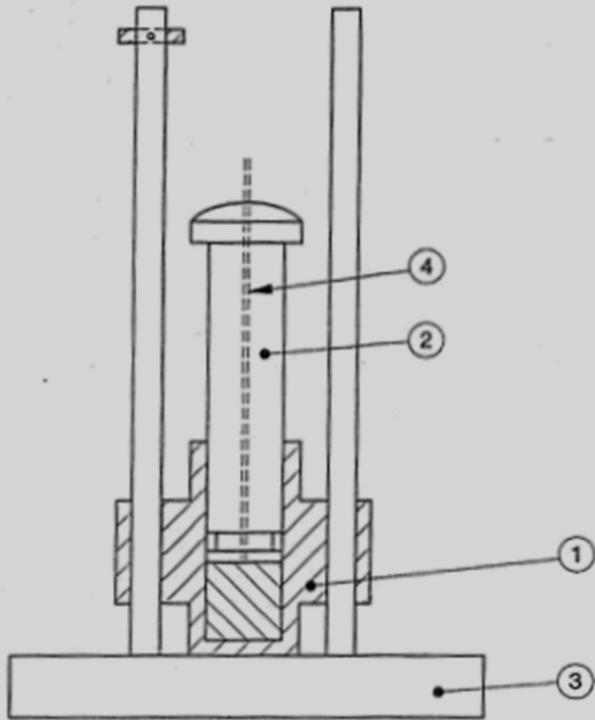
- **Soft and Hard**
  - **LA = 35**
- **Many from 2 sources**

No.	Code	Filler Type
1	LH1	Hard Limestone
2,3	LS1, LS2	Soft Limestone
4,5	DH1, DH2	Hard Dolomite
6,7	DS1, DS2	Soft Dolomite
8, 9	GH1, GH2	Hard Granite
10	GHB1	Hard Granite
11,12	GS1, GS2	Soft Granite
13,14	BH1,BH2	Hard Basalt
15, 16	BV1	Vesicular Basalt
16, 17	GRQ1, GRQ2	Siliceous Gravel Quartzite
18, 19	FAC1, FAC2	Fly Ash Type C
20	FAF1	Fly Ash Type F
21, 22	FAN1, FAN2	Fly Ash Non Spec
23, 24	HL1, HL2	Hydrated Lime
25	CM1	Cement
26, 27	FS1, FS2	Steel Furnace Slag
28	CBC1	Carbon Black Coarse
29	CBF1	Carbon Black Fine
30	CA1	Hard Caliches
31	CA2	Soft Caliches
32	AN1	Andesite

# Range in Filler Properties

	<b>RV</b>	<b>FM</b>	<b>CaO</b>	<b>MBV</b>
<b>Fillers Max</b>	<b>49.1</b>	<b>6.3</b>	<b>50.3</b>	<b>31.6</b>
<b>Fillers Min</b>	<b>26.2</b>	<b>3.0</b>	<b>1.0</b>	<b>0.0</b>

# ***RIGDEN VOIDS (BS 812, EN 1097-4)***



1. Dropping block
2. Plunger
3. Base plate
4. Tare boring



# Asphalt Binders for Producing Mastics

- **PG 64-22 with low asphaltenes**
  - (from a light crude source)
- **PG 64-22 with high asphaltenes**
  - (from a heavy crude source)
- **Binder (a) modified with PPA to a PG 76-22**
- **Binder (a) modified with SBS to a PG 76-22**

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# Mastic Testing and Results

# Mastic Properties Measured

## 1. Workability measure:

Viscosity: 135 C at 1 – 50 RPM

## 2. Rutting:

Jnr, %Recovery, 58 C, 64 C, at 0.1, 3.2, 10kPa

## 3. Fatigue”

G\* .Sinδ, and Time Sweep, 25 C and 10Hz

## 4. Low Temp Cracking:

S, m, at -12 C, Cracking temperature (ABCD)

## 5. Moisture Damage

Pull Off Test, 24hr moisture conditioning at 60 C

# Statistical analysis for Relating Mastic to Filler Properties

## Sample Analysis: Mastic Viscosity

- Forward step wise analysis is conducted
- The combination with the highest adjusted R<sup>2</sup> is used
- The analysis includes the individual parameters and their 2<sup>nd</sup> order interaction

Vars	R-Sq	R-Sq(adj)	Cp	S	RV	FM	CaO	MBV
1	63.8	63.2	13.9	1433.6				
1	43.6	42.7	57.4	1789.8	X			
2	67.6	66.6	7.7	1365.9				
2	67.1	66.1	8.7	1376.6				
3	70.8	69.5	2.8	1306.9			X	
3	70.4	69.0	3.7	1316.1		X		
4	71.6	69.7	3.2	1300.7			X	X
4	71.5	69.7	3.3	1301.2		X	X	X
5	72.3	70.0	3.7	1294.8		X	X	X
5	72.0	69.7	4.3	1301.6		X	X	X
6	72.6	69.9	5.1	1298.4		X	X	X
6	72.5	69.8	5.1	1298.6	X	X	X	X
7	73.3	70.2	5.5	1291.4		X	X	X
7	73.2	70.1	5.6	1293.0		X	X	X
8	74.5	71.1	4.8	1271.9	X	X	X	X
8	74.4	71.0	5.0	1274.4	X	X	X	X
9	75.0	71.1	5.9	1271.8	X	X	X	X
9	74.9	71.1	5.9	1272.3	X	X	X	X
10	75.4	71.0	7.0	1272.6	X	X	X	X
10	75.3	70.9	7.2	1274.8	X	X	X	X
11	75.5	70.7	8.7	1279.8	X	X	X	X
11	75.5	70.7	8.8	1281.0	X	X	X	X

B B  
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R B B a a a R F B a  
RV, FM, CaO, MBV  
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# Example of the Regression Results

The regression equation is

$$\text{Viscosity} = -3238 + 1403 \text{ FM} - 21.3 \text{ RVxFM} - 4.64 \text{ FMxCaO} + 0.269 \text{ Bind x RV} \\ - 1.09 \text{ Bind x FM}$$

Predictor	Coef	SE Coef	T	P
Constant	-3238	2008	-1.61	0.112
FM	1403.5	826.8	1.70	0.095
RVxFM	-21.31	12.01	-1.77	0.081
FMxCaO	-4.643	2.110	-2.20	0.031
Bind x RV	0.26878	0.05242	5.13	0.000
Bind x FM	-1.0895	0.4371	-2.49	0.015

S = 1308.34    R-Sq = 71.7%    R-Sq(adj) = 69.4%

# Statistical Analysis :

## *Important Factors for the TOTAL Results (68 Mastics)*

	1- Viscosity	Relative Viscosity	2- Rutting	Relative Rutting	3- Fatigue	Relative Fatigue	4- Moisture Damage	Relative Moisture Damage	5- Low Temperature Stiffness	Relative Stiffness	Number of Times
R <sup>2</sup> (adj) % - Binder only	42.7	NA	46.2	NA	38.8	NA	1.4	NA	32.5	NA	Total Results
R <sup>2</sup> (adj) % -with others	71.1	53.5	67.1	25.4	49.9	52.0	3.7	55.3	65.0	58.9	
<b>Binder</b>		- *	X	-*		-*	X	-*		-*	2
<b>RV</b>	X	X		X	X	X				X	<u>6</u>
<b>FM</b>	X	X				X		X	X		<u>5</u>
<b>CaO</b>			X						X		2
<b>MBV</b>	X	X	X			X					4
<b>RVxFM</b>	X	X			X	X			X		<u>5</u>
<b>RVxCaO</b>				X	X			X			3
<b>RVxMBV</b>		X	X								2
<b>FMxCaO</b>	X	X			X			X		X	<u>5</u>
<b>FMxMBV</b>									X	X	2
<b>CaOxMBV</b>				X							1
<b>Binder x RV</b>	X	X	X		X	X	X		X		<u>7</u>
<b>Binder x FM</b>	X							X		X	3
<b>Binder x Cao</b>				X					X	X	3
<b>Binder x MBV</b>					X				X	X	3

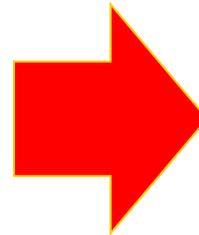
# Study objective (1): Important Filler Properties

- **Primary filler properties**

- **Rigden Voids: RV**
- **Fineness Modulus: FM**

- **Interactions:**

- **RV x FM**
- **FM x CaO**
- **Binder x RV**



- ***RV***
- ***FM***
- ***CaO***

# Check for Inter-correlations

*The 4 filler properties are independent*

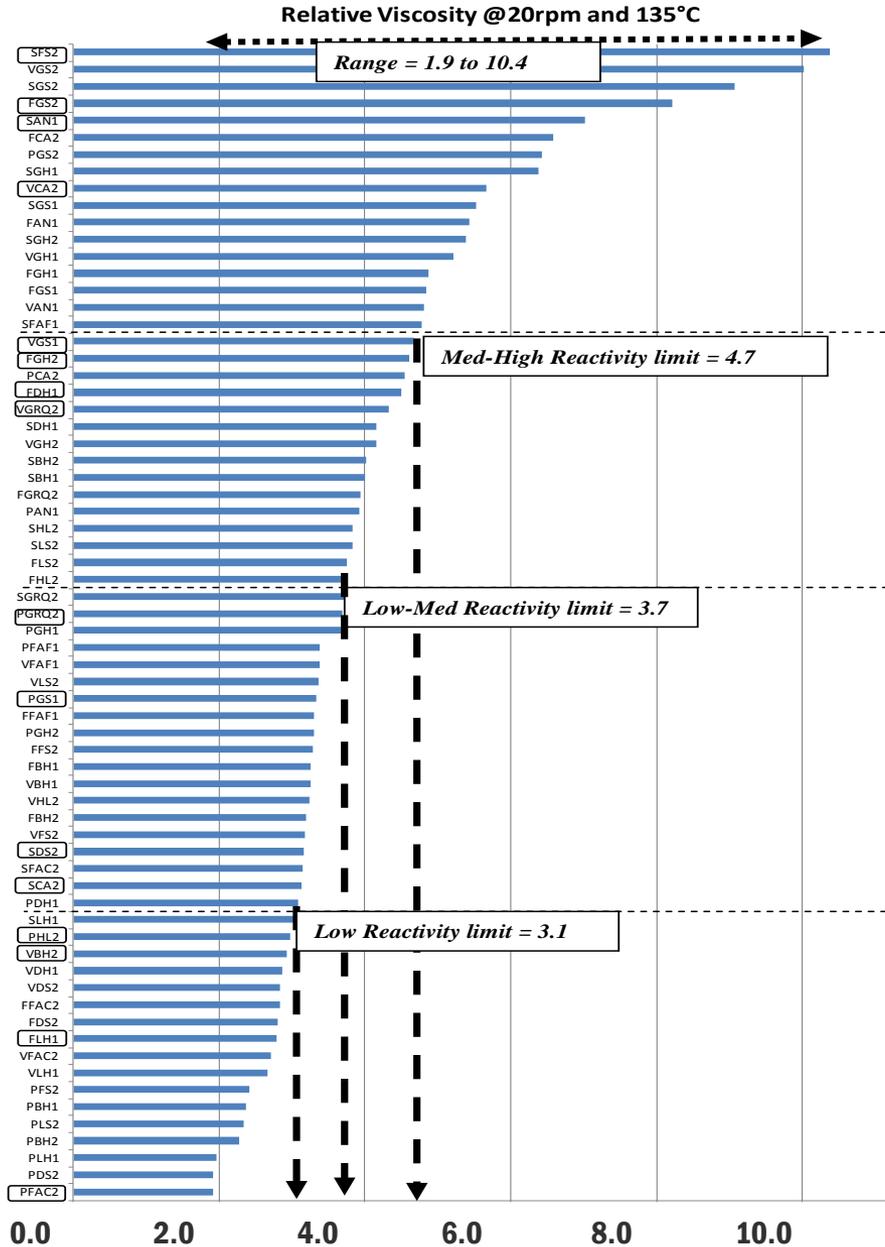
	<i>Rigden Voids (%)</i>	<i>FM</i>	<i>CaO%</i>	<i>MBV</i>
<i>Rigden Voids (%)</i>	1			
<i>FM</i>	0.066	1		
<i>CaO%</i>	-0.078	0.067	1	
<i>MBV</i>	0.279	0.036	-0.396	1

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# Examples of Mastic Results

**Mastic responses are highly filler  
dependent for all 4 binders**

# Mastics Relative Viscosity - Mixture Constructability



**Relative Viscosity Range = 1.9- 10.4**

**Legend:**

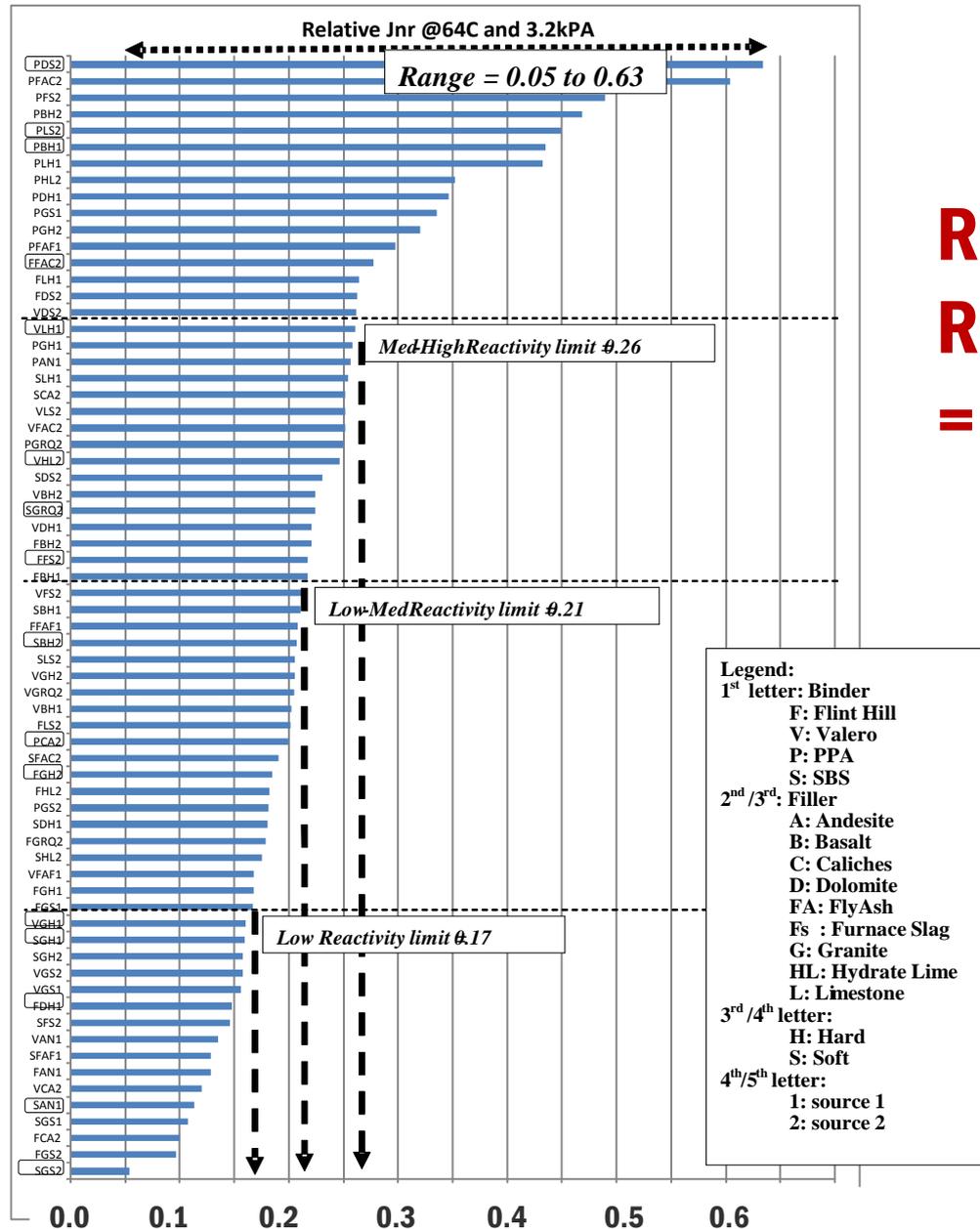
**1<sup>st</sup> letter: Binder**  
 F: Flint Hill  
 V: Valero  
 P: PPA  
 S: SBS

**2<sup>nd</sup> /3<sup>rd</sup>: Filler**  
 A: Andesite  
 B: Basalt  
 C: Caliches  
 D: Dolomite  
 FA: FlyAsh  
 Fs : Furnace Slag  
 G: Granite  
 HL: Hydrate Lime  
 L: Limestone

**3<sup>rd</sup> /4<sup>th</sup> letter:**  
 H: Hard  
 S: Soft

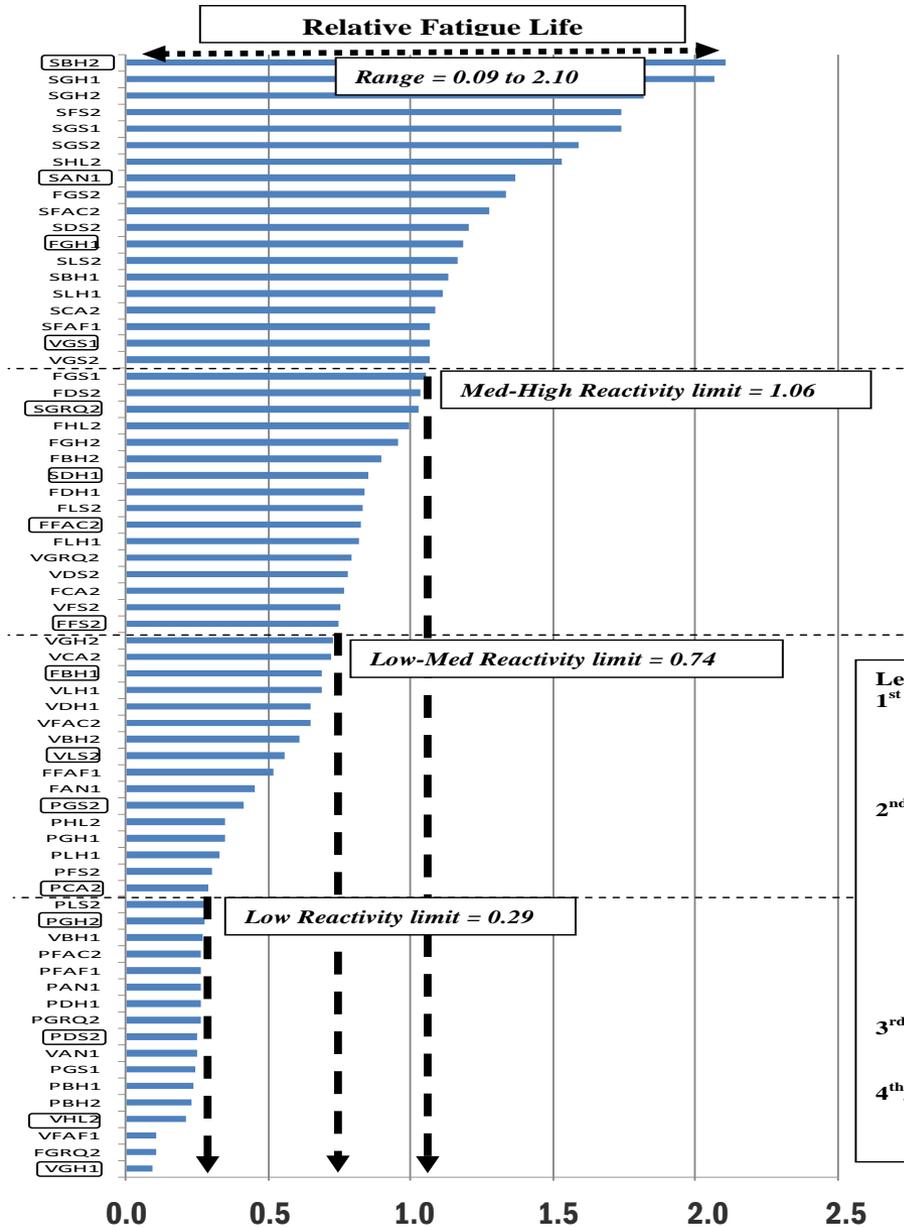
**4<sup>th</sup> /5<sup>th</sup> letter:**  
 1: source 1  
 2: source 2

# Mastics Jnr - Mixture Rutting



**Relative Jnr  
Range  
= 0.05 to 0.63**

# Mastics Fatigue - Mixture Fatigue



**Relative Nf  
Range  
= 0.09 to 2.10**

**Legend:**

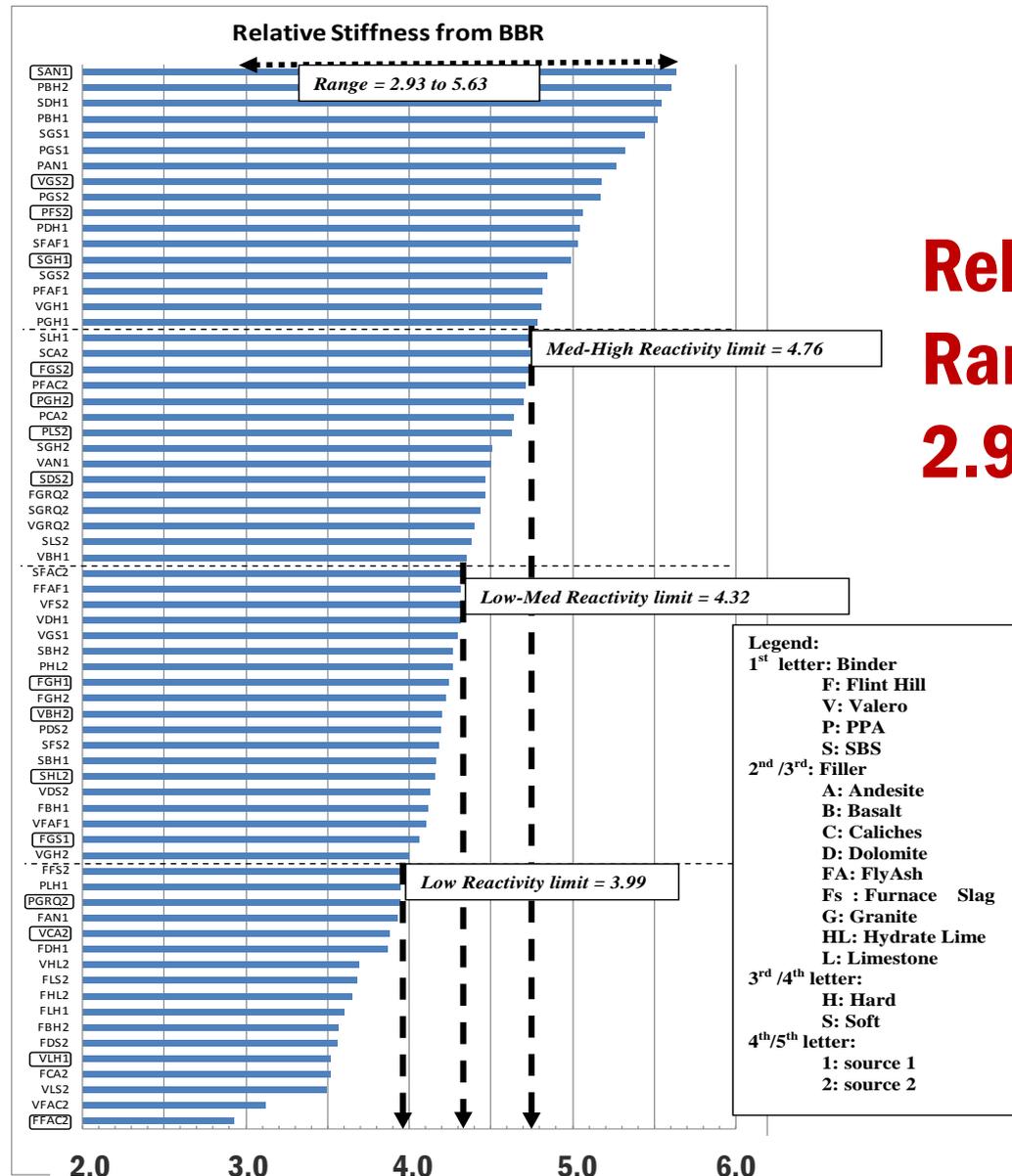
**1<sup>st</sup> letter: Binder**  
 F: Flint Hill  
 V: Valero  
 P: PPA  
 S: SBS

**2<sup>nd</sup> /3<sup>rd</sup>: Filler**  
 A: Andesite  
 B: Basalt  
 C: Caliches  
 D: Dolomite  
 FA: FlyAsh  
 Fs : Furnace Slag  
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 HL: Hydrate Lime  
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**3<sup>rd</sup> /4<sup>th</sup> letter:**  
 H: Hard  
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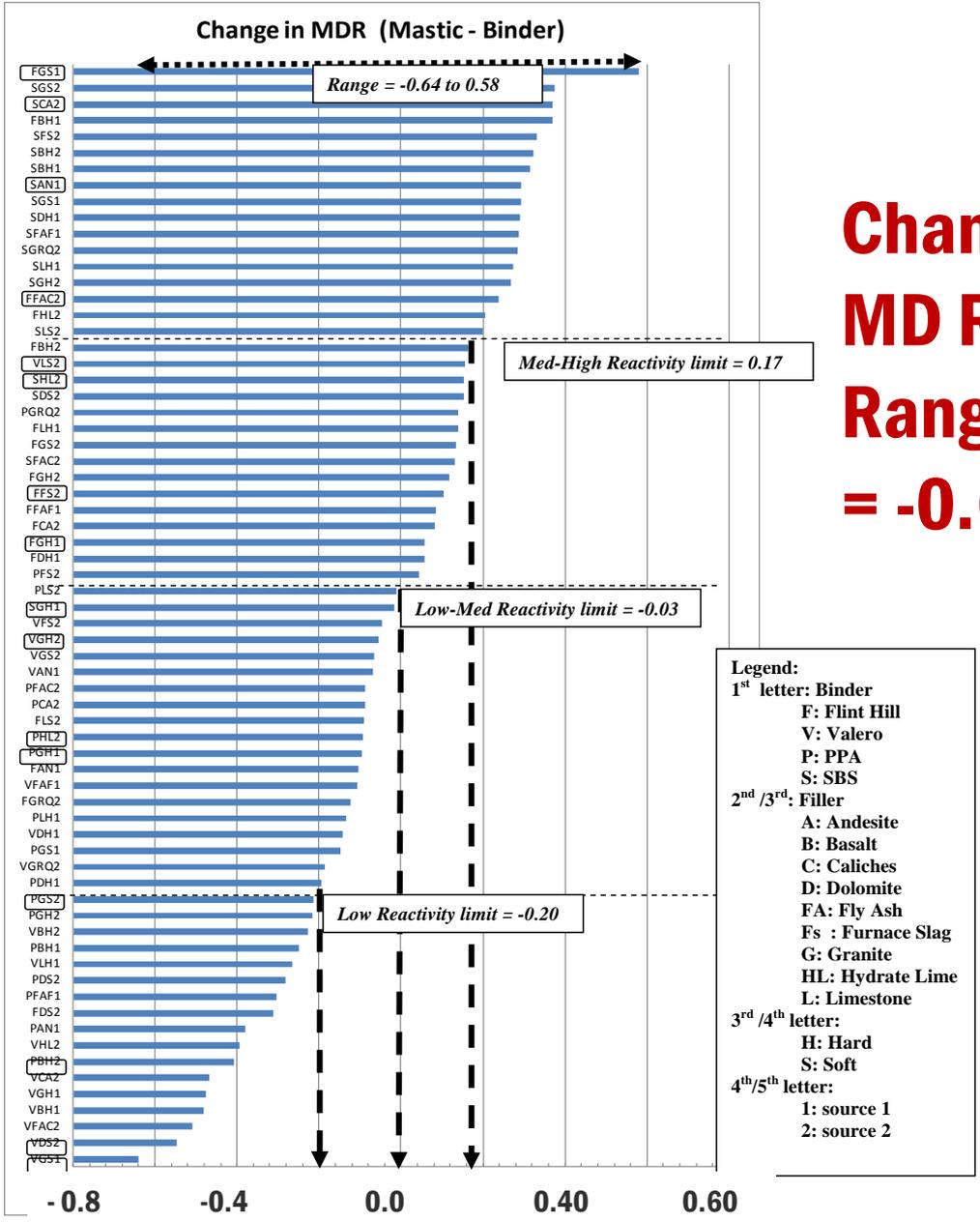
**4<sup>th</sup> /5<sup>th</sup> letter:**  
 1: source 1  
 2: source 2

# Mastics Low Temp S960) - Mixture Low Temperature



**Relative S(60)**  
**Range=**  
**2.93 - 5.63**

# Mastic Moisture Damage



**Change in MD Ratio Range = -0.64 to +0.58**

***Moisture Damage of Mastics***  
***PNEUMATIC ADHESION TENSILE TESTING INSTRUMENT***  
***(PATTI) (ASTM D 3409-93(2002))***



# Statistical Analysis :

## *Important Factors for the **SELECTED** Mastics (16 Mastics)*

	1- Viscosity	Relative Viscosity	2- Rutting	Relative Rutting	3- Fatigue	Relative Fatigue	4- Moisture Damage	Relative Moisture Damage	5- Low Temperature Stiffness	Relative Stiffness	Number of Times
<b>R<sup>2</sup>(adj) % - Binder only</b>	32.5	NA	34.1	NA	38.7	NA	0.0	NA	23.9	NA	Selected
<b>R<sup>2</sup> (adj) % -with others</b>	51.4	40.2	67.4	70.6	30.6	16.2	0.0	21.5	78.0	73.6	
<b>Binder</b>		- *	X	-*		-*	x	-*		-*	2
<b>RV</b>	X	X		X	X	X				X	<b>6</b>
<b>FM</b>	X	X				X		X	X		<b>5</b>
<b>CaO</b>			X						X		2
<b>MBV</b>	X	X	X			X					4
<b>RVxFM</b>	X	X			X	X			X		<b>5</b>
<b>RVxCaO</b>				X	X			X			3
<b>RVxMBV</b>		X	X								2
<b>FMxCaO</b>	X	X			X			X		X	<b>5</b>
<b>FMxMBV</b>									X	X	2
<b>CaOxMBV</b>				X							1
<b>Binder x RV</b>	X	X	X		X	X	X		X		<b>7</b>
<b>Binder x FM</b>	X							X		X	3
<b>Binder x Cao</b>				X					X	X	3
<b>Binder x MBV</b>					x				X	X	3

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# Mixture Testing Results

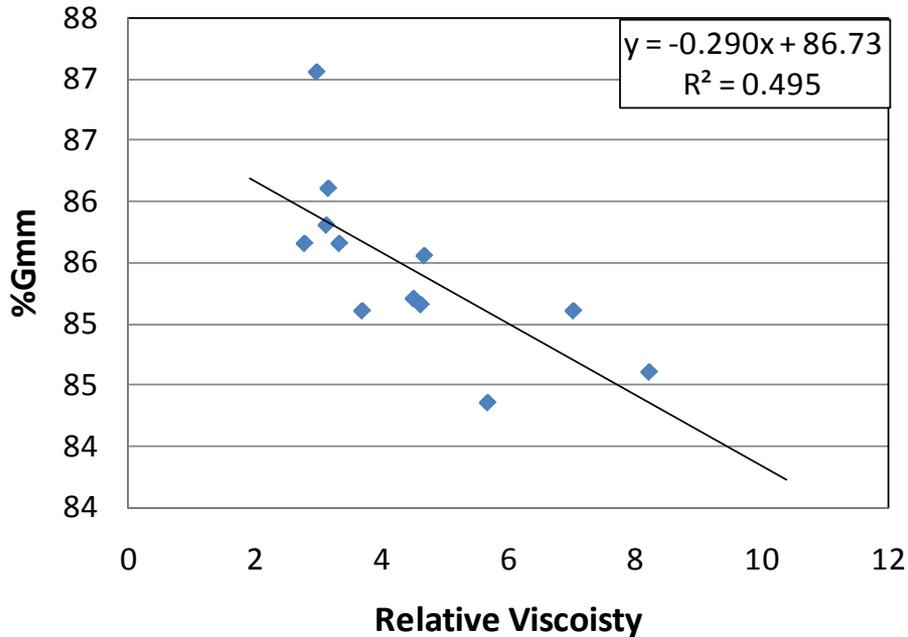
# Mixture Constructability

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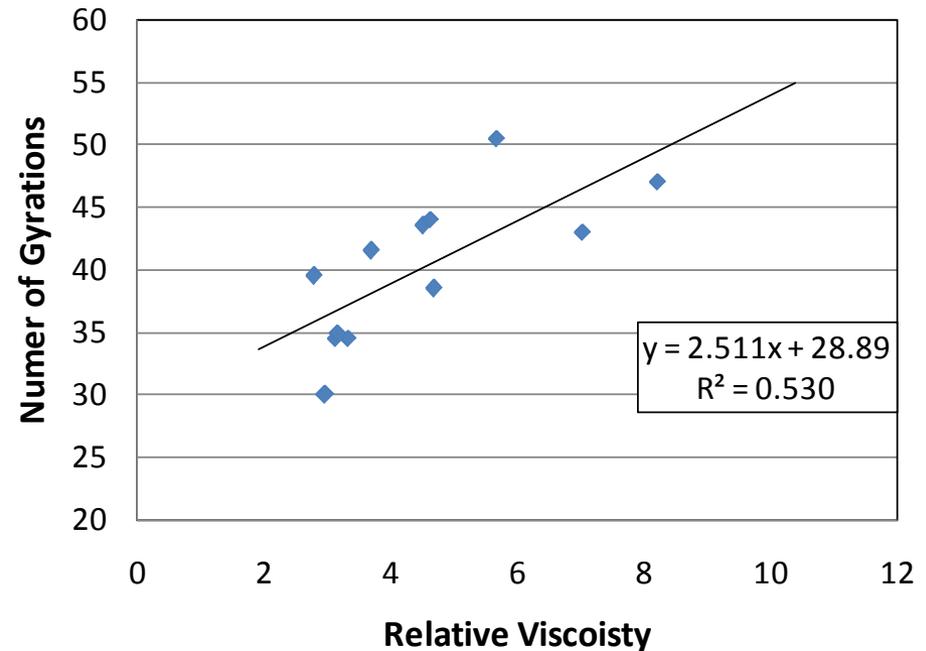
- **Measured the number of gyrations required to reach 92%Gmm**
- **Measured the Density at 8 Gyrations**

# Correlation with the Relative Viscosity after Removing the Outliers

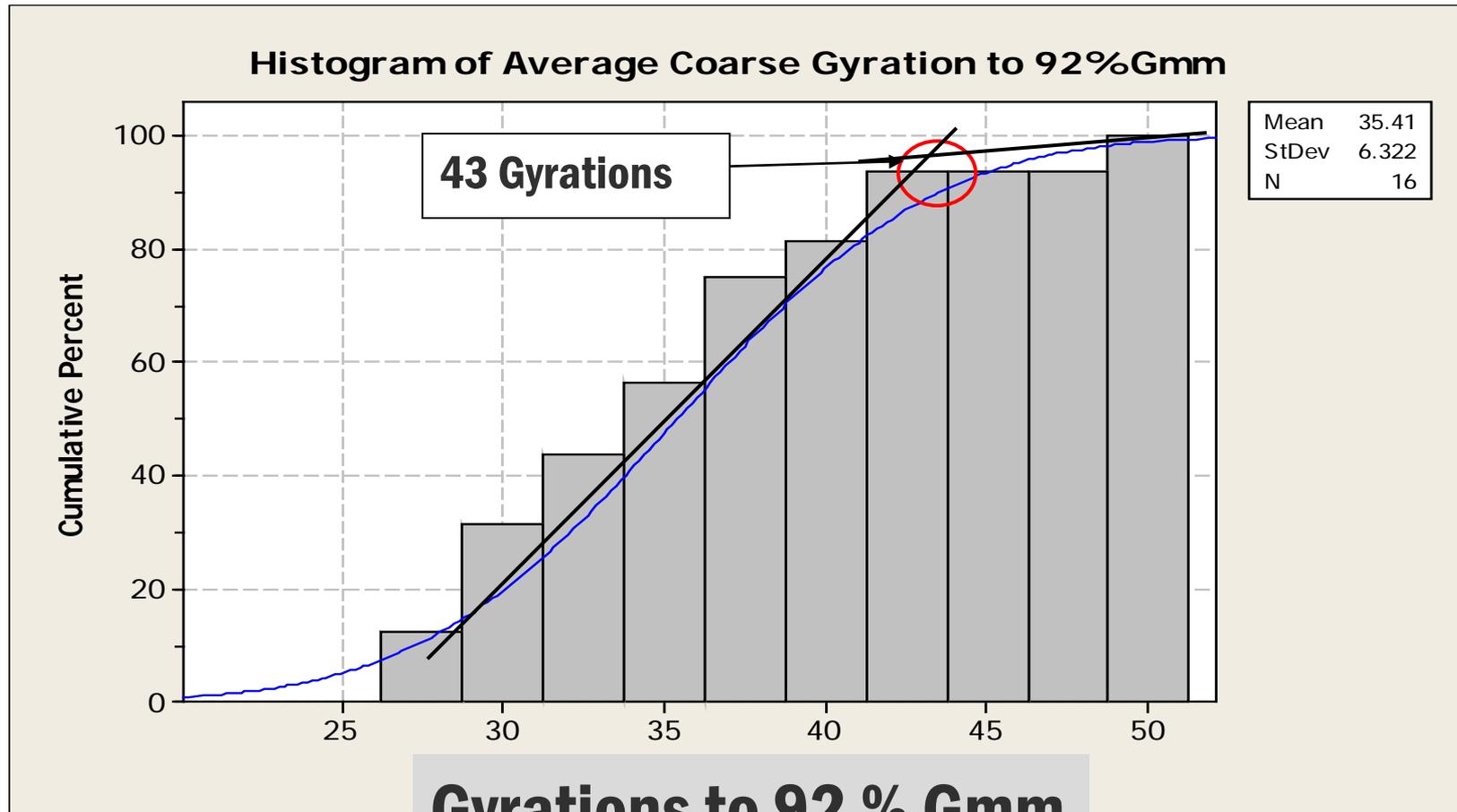
%Gmm at 8 Gyration



Number of Gyration to 92%Gmm

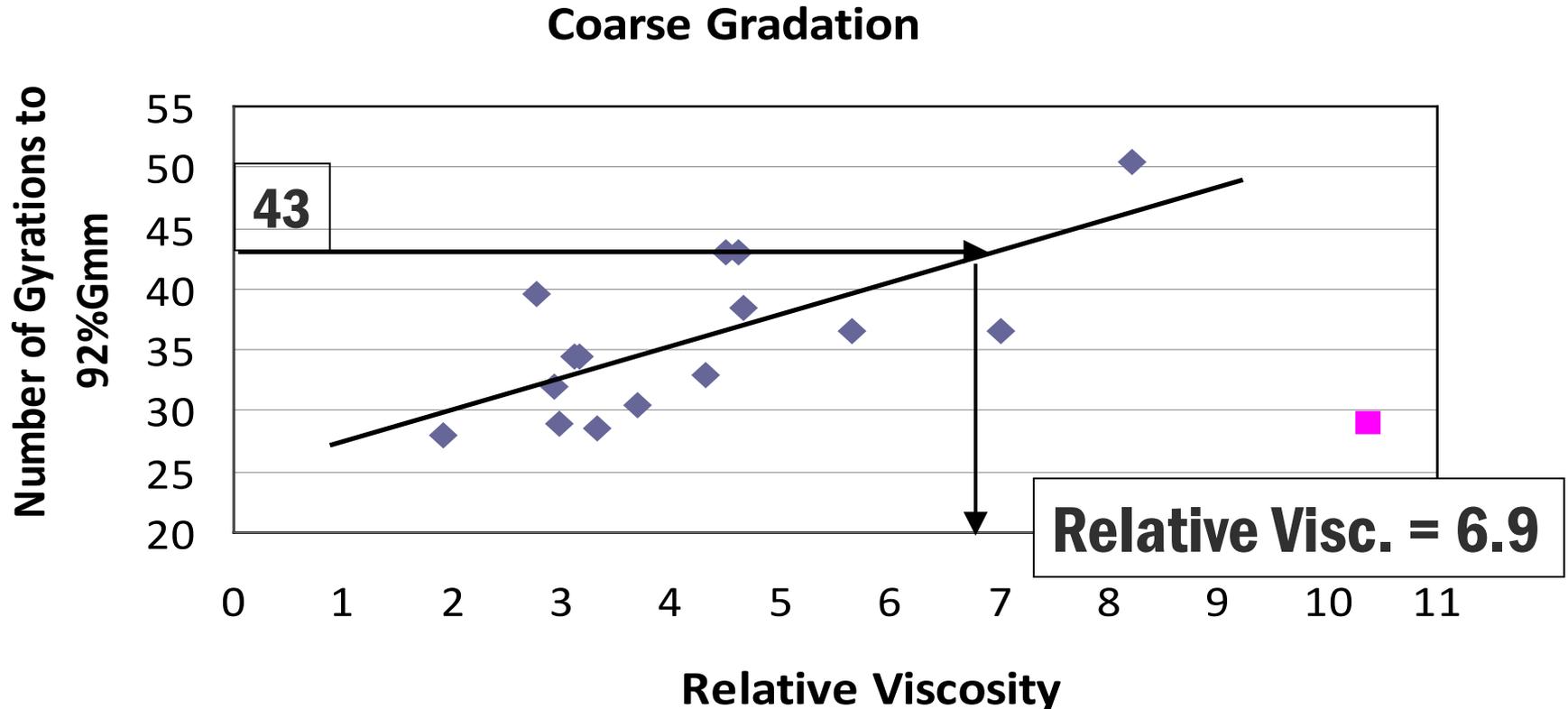


# Identify Limits to avoid Extreme Filler Effects - Inflection Points



# Workability:

## Correlation with Mastic Relative Viscosity



# Workability:

## Derive Filler Limits from Rel Mastic Viscosity

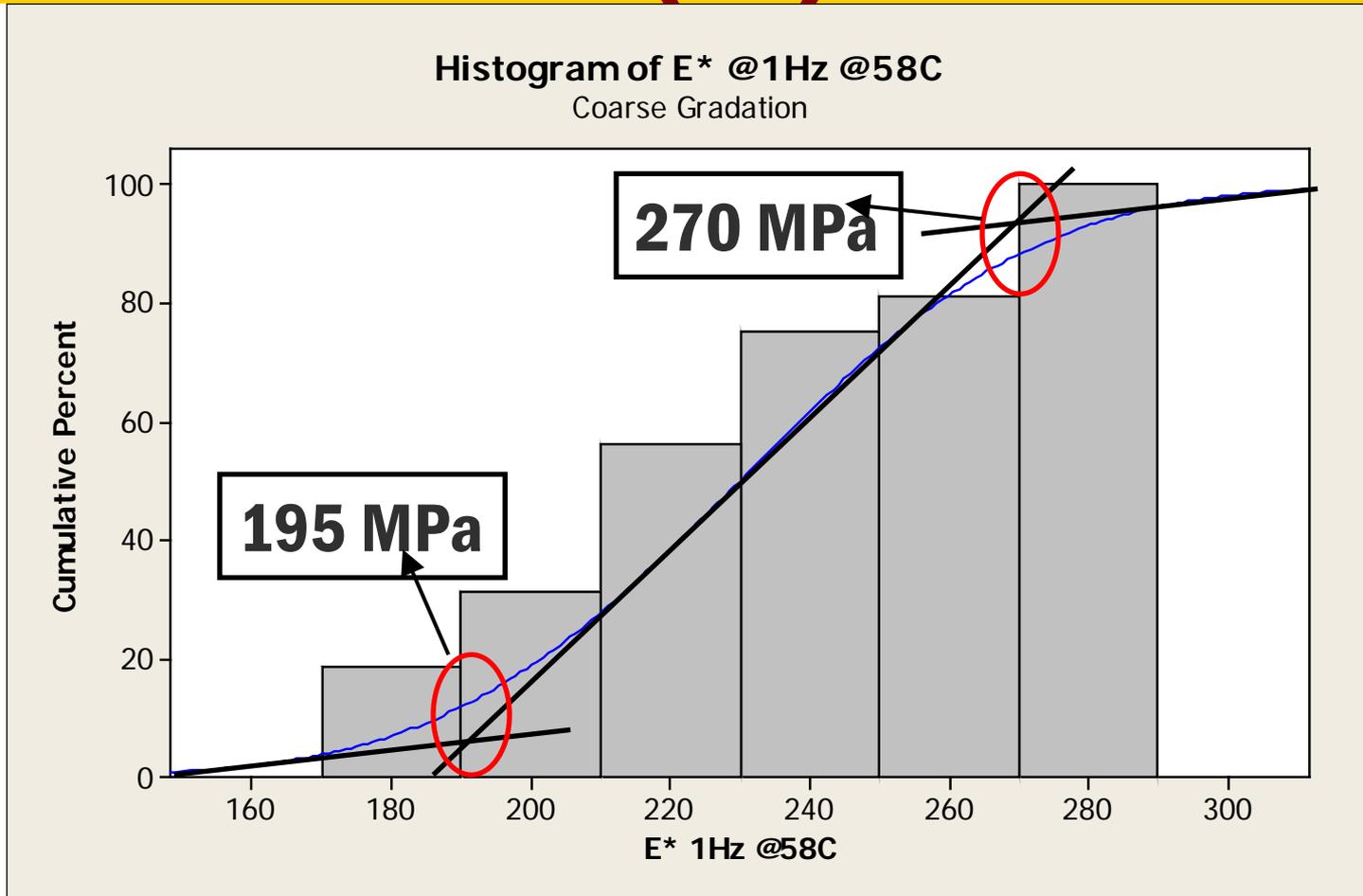
$$\text{Relative Viscosity} = - 11.3 + 0.472 \text{ Rig.Voids} + 2.28 \text{ FM} - 0.0719 \text{ Rig.Void} \times \text{FM} - 0.0238 \text{ CaO}$$

Mix Gradation	Mastic Max R.Visc	Max. Rig Void	Max. FM	Min. Rig. Vx FM
Coarse	6.9	42.2	5.4	128.5
Fine	7.0	42.4	5.5	127.2

- Although CaO is a significant factor, the limits calculated from the equation are beyond the population range

# Inflection points

## Dynamic Modulus ( $E^*$ ) - 58°C



# Flow Number: Jnr to Filler Regression Analysis

**Mastic Jnr = 1.41 + 0.162 Binder Jnr + 0.00847 CaO - 0.0331 Rig.Voids**

	Mix FN	Mastic Jnr	CaO	Rig. Voids
Lower	400	1.6	5.6	26.2
Upper	1650	0.6	45.1	42.2

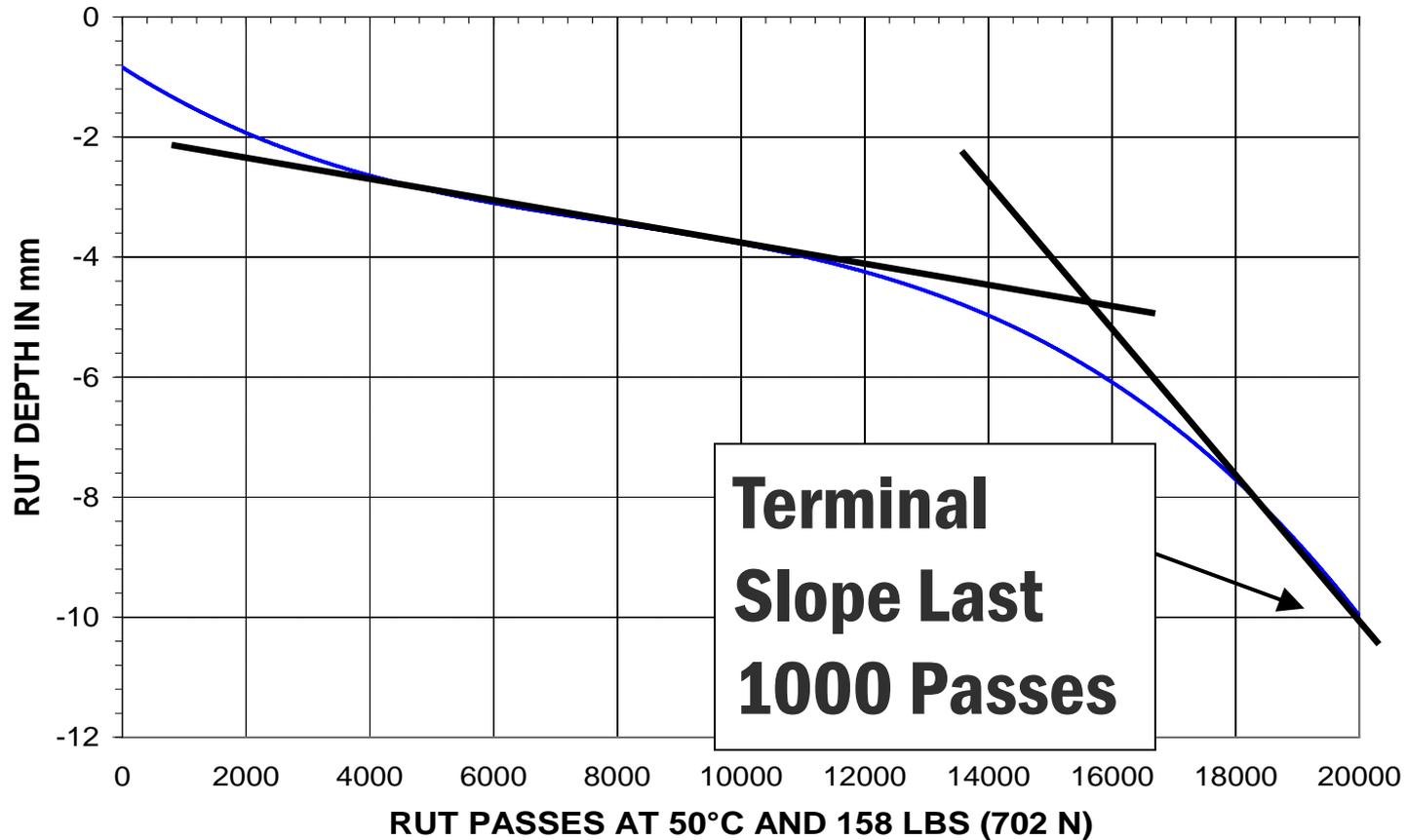
# Low Temperature

- **Strong correlation with BBR outputs**
- **Limits for Mastic properties**

	Mixture Strength	Mastic Relative Stiffness	mastic m-value	Rel. m-value
Lower	3.8	NA	0.25	0.77
Upper	4.9	5.5	0.31	1.04

# Moisture Damage

VALERO + GS1 FILLER 50°C



# Summary of Inflection Points Limits (Extreme effects)

	Rig. Voids (%)		Fineness		CaO (%)	
	Lower	Upper	Lower	Upper	Lower	Upper
<b>Workability</b>		42.9		3.9		
<b>E* @58C</b>	33.1	49.1			1	40.6
<b>E* @25C</b>	31.0	49				
<b>Flow Number/Jnr</b>	26.2	42.2			5.6	45.1
<b>Low Temperature</b>	31.1	44.5				
<b><i>Proposed Limits</i></b>	<b><i>31.0</i></b>	<b><i>43.0</i></b>	<b><i>NA</i></b>	<b><i>4.0</i></b>	<b><i>NA</i></b>	<b><i>45.1</i></b>

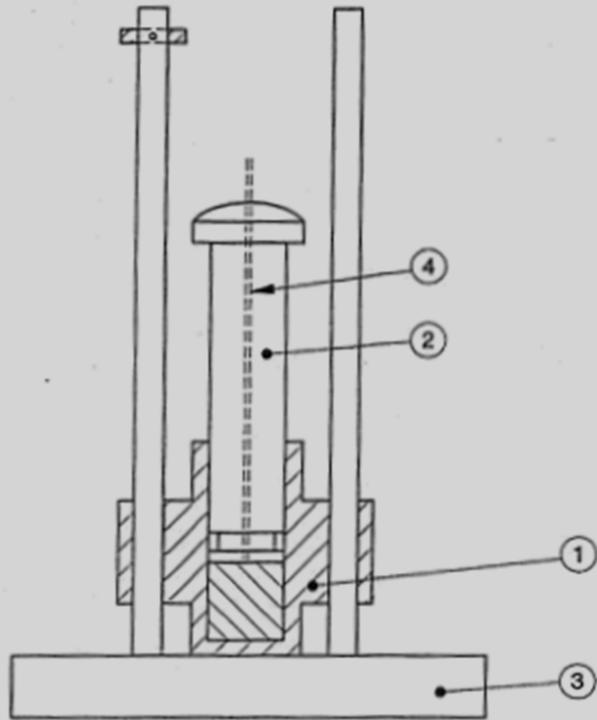
# Interim Mixture Testing Conclusions

- Mixture workability is sensitive to **Rigden Voids** and **size distribution** of the fillers.
- Mixture rutting is sensitive to the **Rigden Voids** and the **chemical activity** of the filler.
- Low temperature strength is sensitive to **Rigden Voids**.
- Results from moisture damage and fatigue are inconclusive at this time.
  - Binder interaction with filler is significant.

# Filler tests to be further investigated in task 5

<b>Test</b>	<b>Cost</b>	<b>Sample Preparation</b>	<b>Running Test</b>	<b>Training required</b>
<b>Rigden Voids</b>	<b>Low (\$1000)</b>	<b>Simple</b>	<b>Simple</b>	<b>Minimal</b>
<b>Laser Diffraction for size distribution</b>	<b>High (~\$ 35 K)</b>	<b>Moderate</b>	<b>Simple</b>	<b>Moderate</b>
<b>X-Ray Florescence for CaO Content</b>	<b>High (\$50K-\$150K)</b>	<b>Complicated</b>	<b>Simple</b>	<b>Moderate</b>

# ***RIGDEN VOIDS (BS 812, EN 1097-4)***



1. Dropping block
2. Plunger
3. Base plate
4. Tare boring



# Next Steps

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- **Investigate other tests' variability**
- **Search for surrogates to minimize cost and complexity.**
- **Further evaluate the proposed filler limits.**

# Acknowledgments

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- **Project Panel**
- **Dr. Ed Harrigan**
- **Suppliers of fillers and binders**

