



# Applications of Gel Permeation Chromatography (GPC) to Asphalt Binder Characterization

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# THE CO-AUTHORS

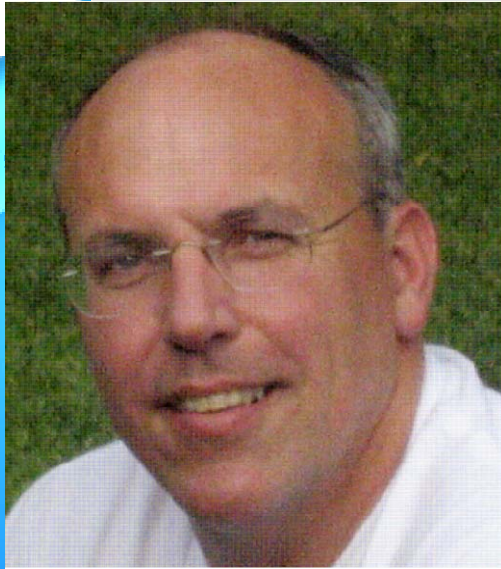


***IOAN NEGULESCU***



***IONELA GLOVER***

# ACKNOWLEDGMENTS



*CHRIS ABADIE*



*BILL KING*



*RAFAEL CUETO*



# Objectives



Develop Experimental Procedures for utilizing Gel Permeation Chromatography (GPC)



Evaluate binder changes during processing sequence using GPC

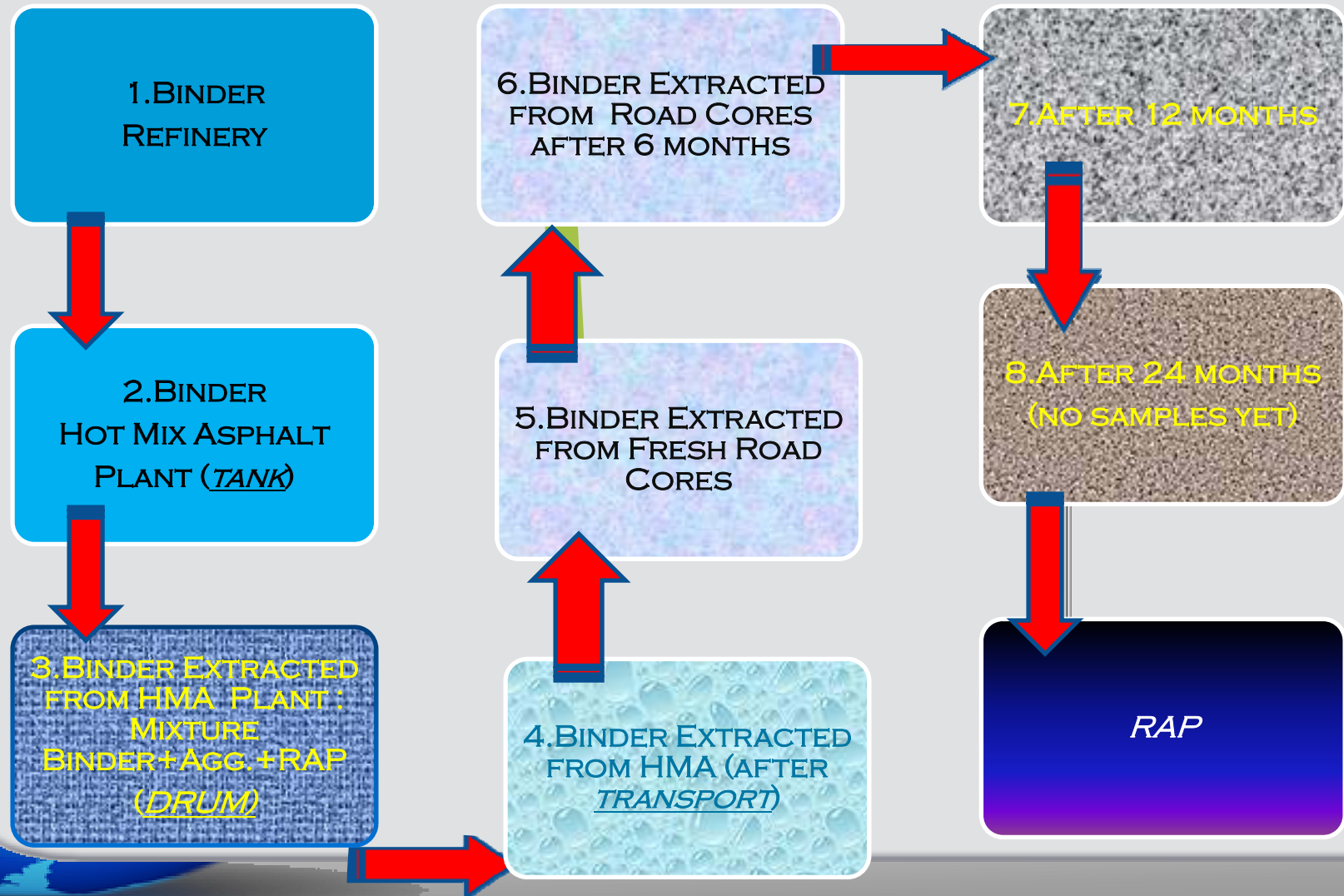


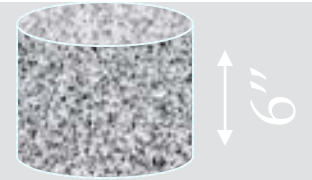
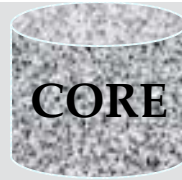
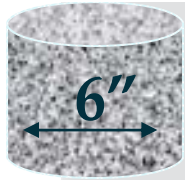
Observe impact of RAP addition during processing



Follow aging process by coring pavements at annual intervals

# SEQUENCE OF BINDER SAMPLES FOR THIS STUDY

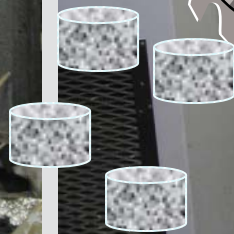
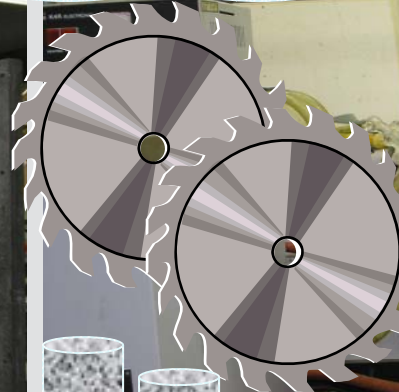
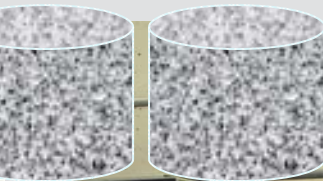




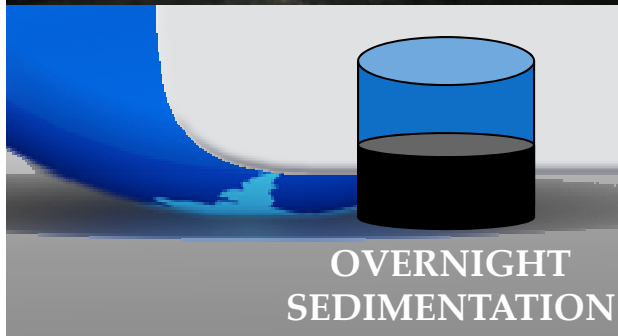
# ASPHALT BINDER EXTRACTION



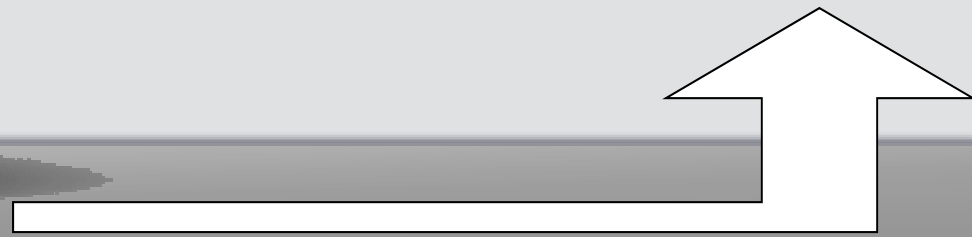
**SOXHLET EXTRACTION**



**ROTARY EVAPORATOR**



**OVERNIGHT  
SEDIMENTATION**



**VACUUM OVEN**

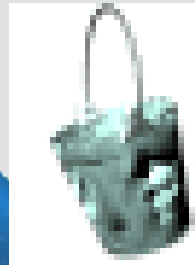
# ALTERNATE EXTRACTION PROCEDURES FOR SMALL SCALE SAMPLES



2. loose mix



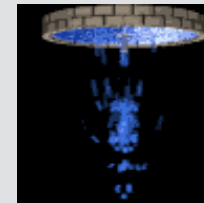
3. warm toluene



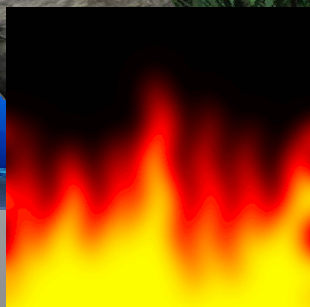
6. THF



1. tin can

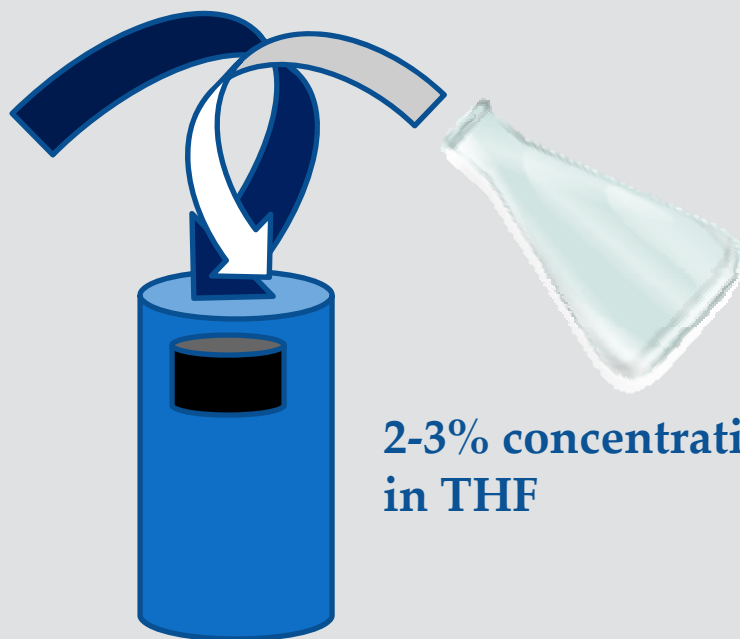


5. decant/evaporate



4. heat

# GPC SAMPLE PREPARATION

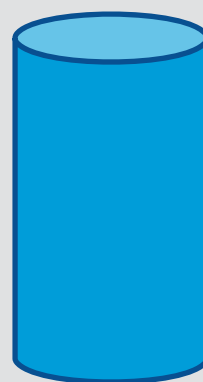


2-3% concentration  
in THF

% insoluble  
by weight



PTFE 45µm  
THF washed filter



1.5 mL vial

3-5 mL  
syringe



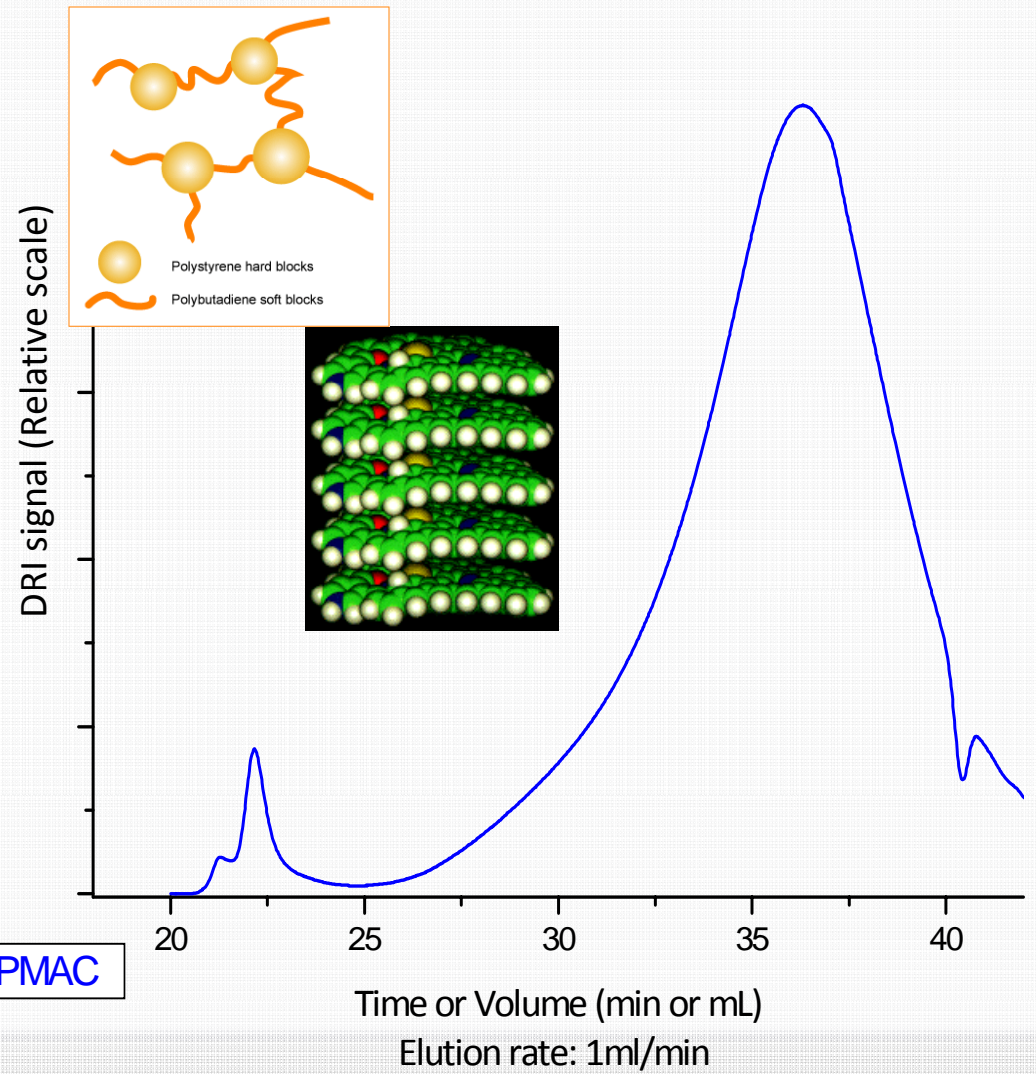
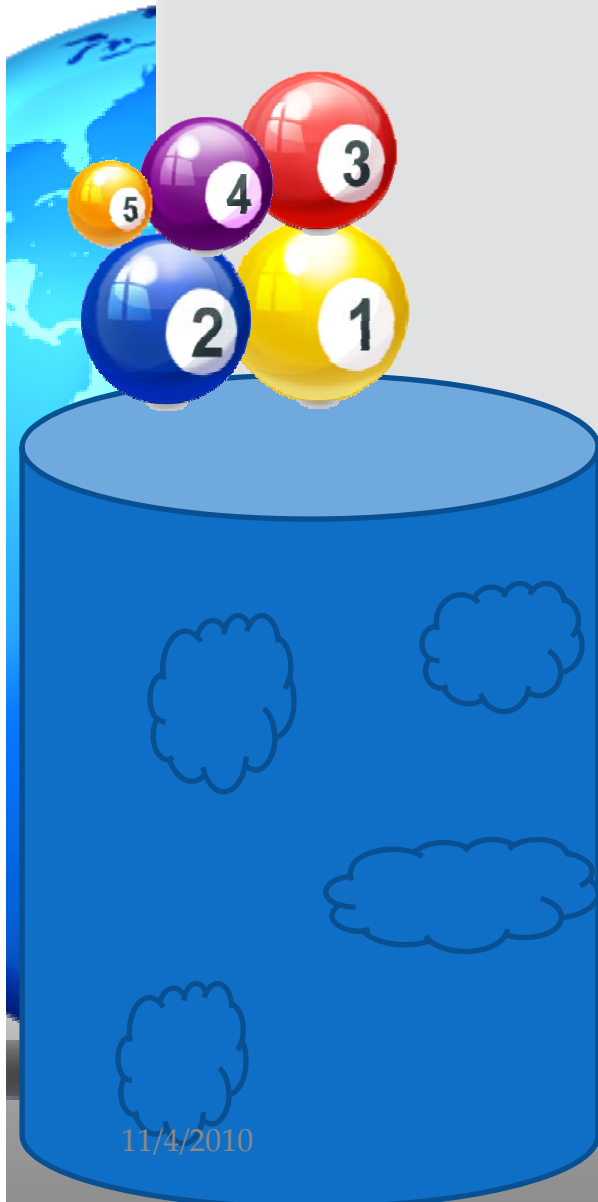
PTFE 45µm  
filter



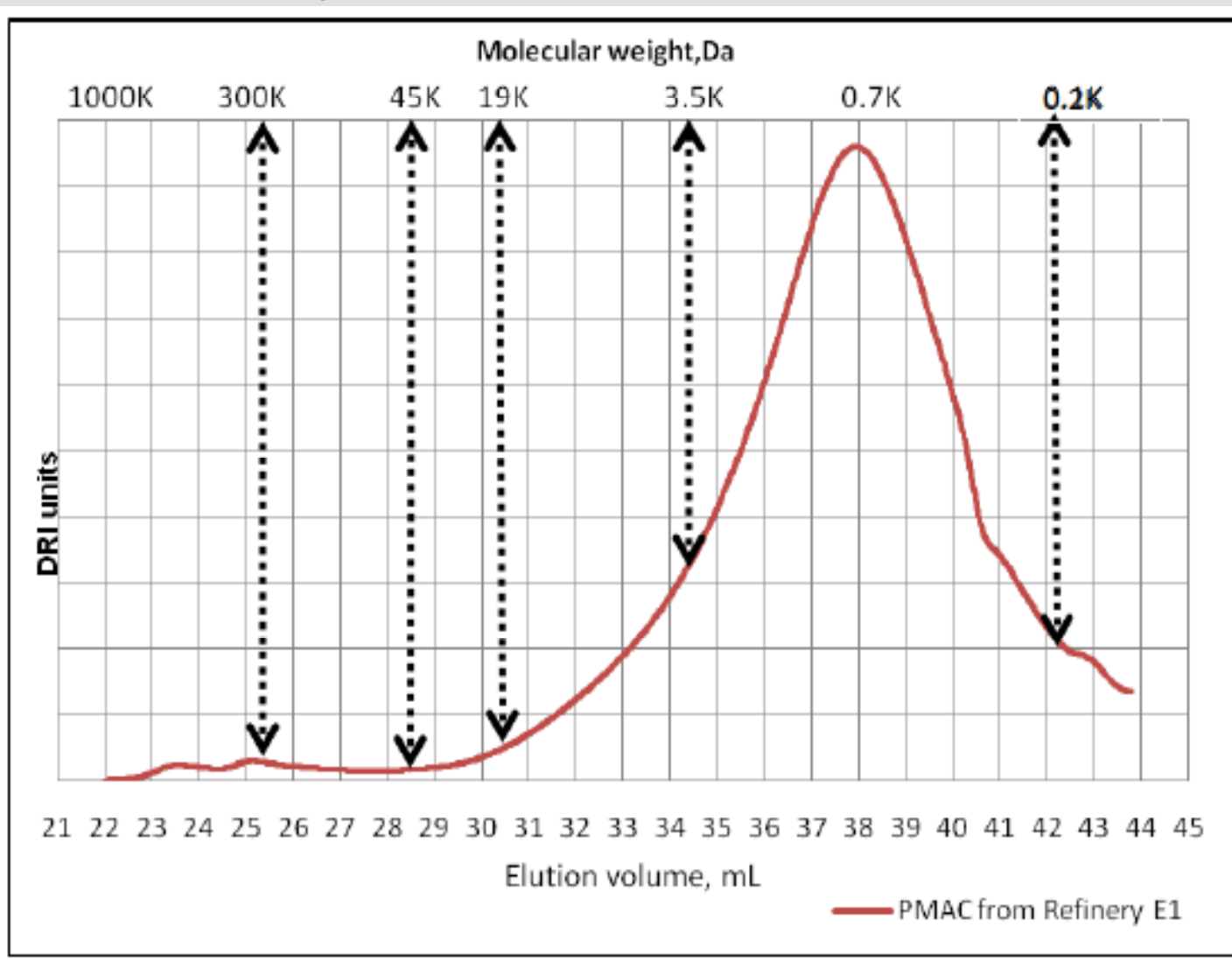
# AGILENT 1100 GPC SYSTEM



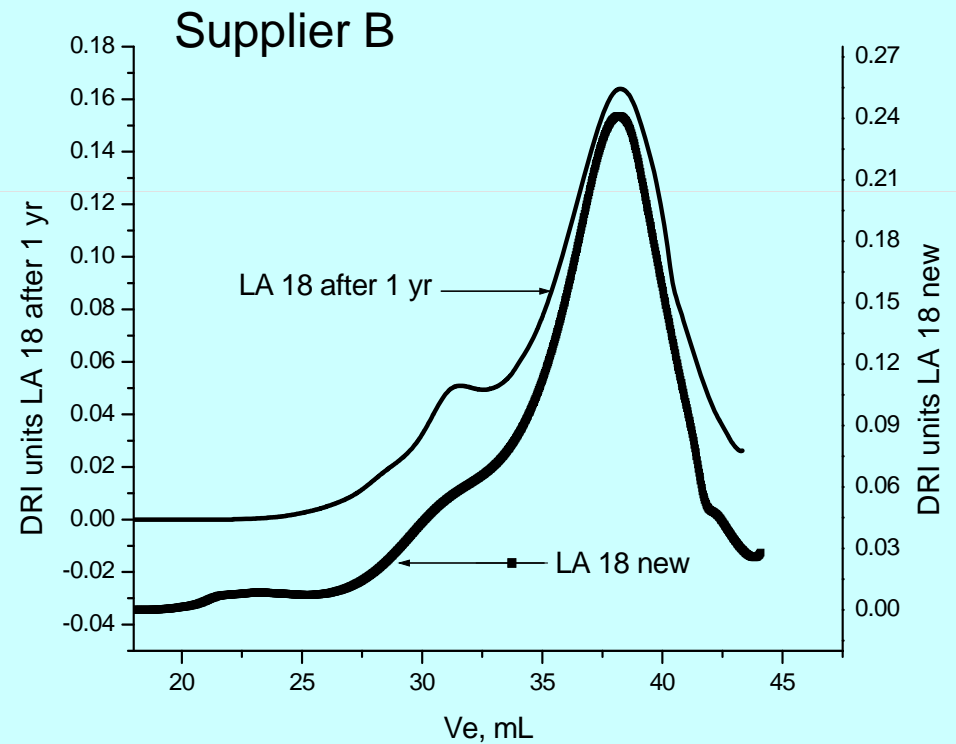
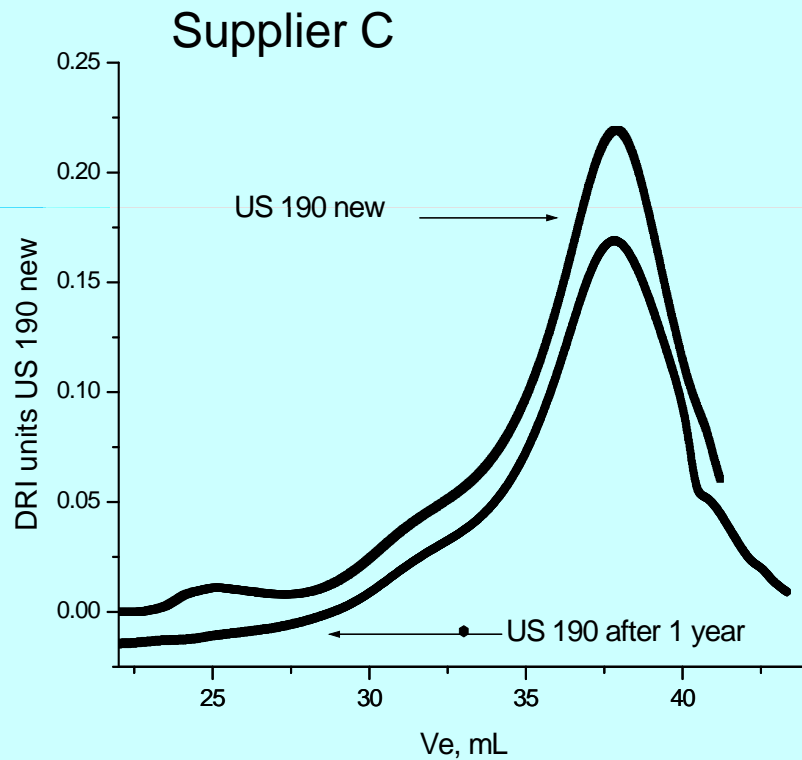
# TYPICAL PMAC GPC CHROMATOGRAM



Regions of a PMAC GPC chromatogram, showing the relationship between elution volume and molecular weights values, based on calibration curve.

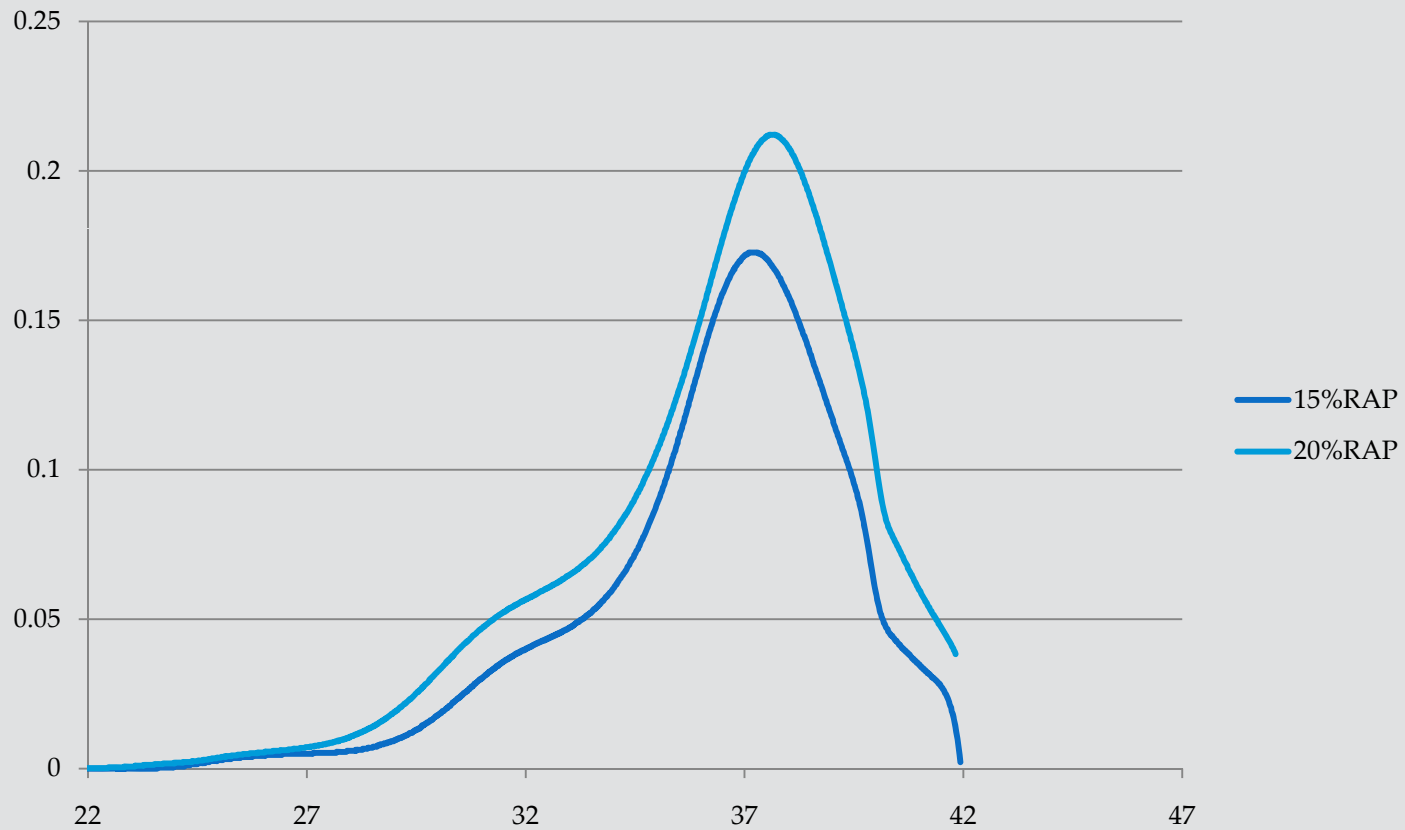


# GPC CHROMATOGRAM OF BINDERS BEFORE AND AFTER 1 YEAR IN THE FIELD



# Effect of RAP conc on GPC

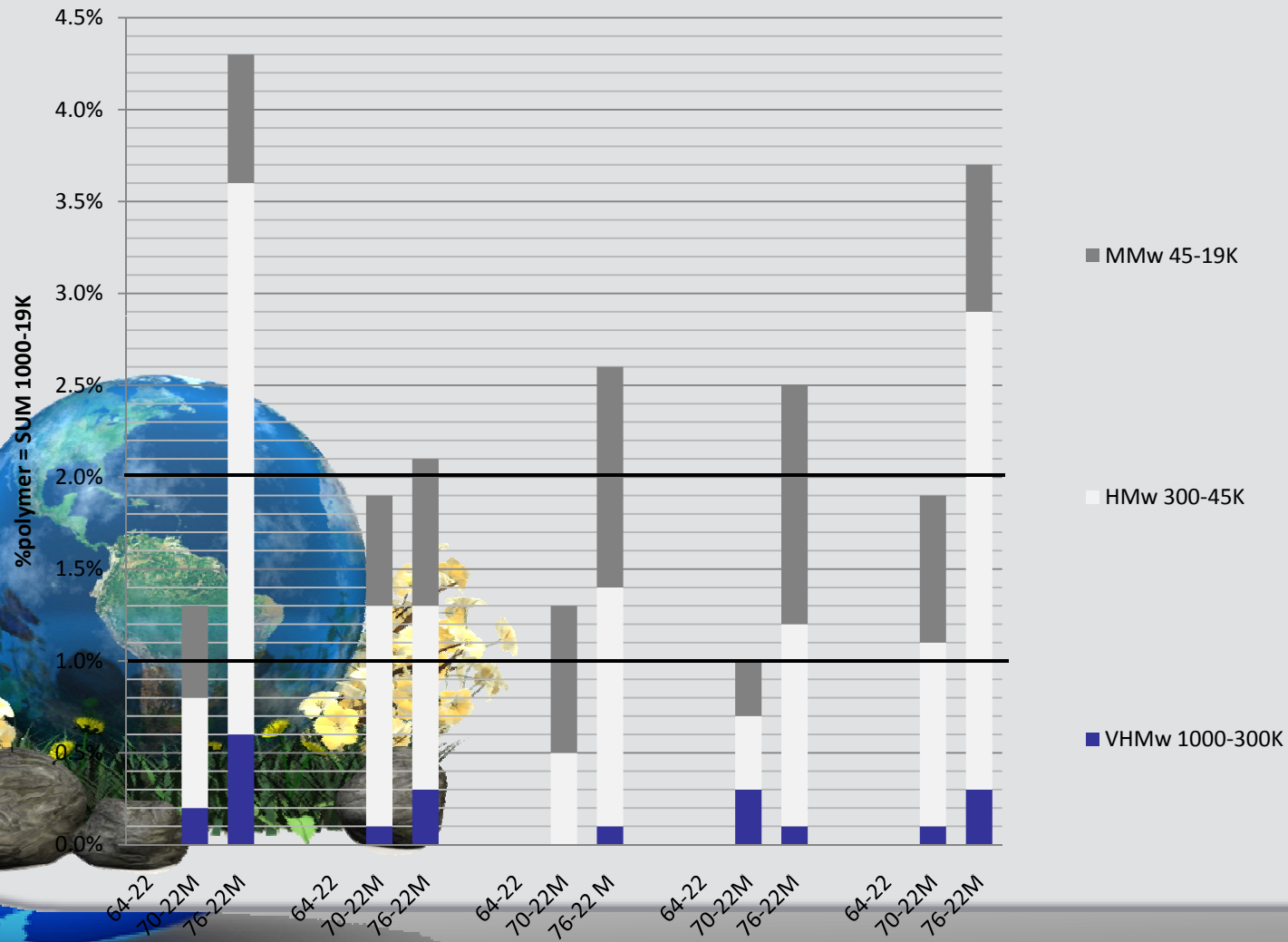
LA 116 15% and 20% RAP



# Distribution of components in original asphalt binders

		VHMw	HMw	MMw	Sum	Asphaltenes	Maltenes
	PG	1000-300K	300-45K	45-19K	1000-19K	19-3K	3-0.2K
A	64-22	0.0%	0.0%	0.0%	0.0%	15%	85%
A	70-22M	0.2%	0.6%	0.5%	1.3%	15%	84%
A	76-22M	0.6%	3.0%	0.7%	4.3%	14%	82%
B	64-22	0.0%	0.0%	0.0%	0.0%	19%	81%
B	70-22M	0.1%	1.2%	0.6%	1.9%	17%	81%
B	76-22M	0.3%	1.0%	0.8%	2.1%	16%	82%
C	64-22	0.0%	0.0%	0.0%	0.0%	16%	84%
C	70-22M	0.0%	0.5%	0.8%	1.3%	17%	82%
C	76-22 M	0.1%	1.3%	1.2%	2.7%	17%	81%

# POLYMER COMPONENT CONTENT OF ORIGINAL ASPHALTS FROM DIFFERENT REFINERIES



# Distribution of components

	VHMw	HMw	MMw	Sum	Asphaltenes	Maltenes
	1000-300K	300-45K	45-19K	1000-19K	19-3.5K	3.5-0.2K
<b>Supplier B2</b>						
Refinery	0.1%	1.0%	1.8%	2.8%	24%	73%
Tank	0.3%	1.1%	1.2%	2.6%	18%	79%
RAP	0.0%	0.5%	2.3%	2.8%	27%	70%
LA 1 after drum	0.1%	1.0%	1.9%	2.9%	24%	73%
LA 1 after truck	0.1%	1.0%	1.9%	2.9%	24%	73%
LA1	0.1%	1.0%	1.8%	2.8%	24%	73%
LA 1/after1 yr	0.1%	1.3%	2.5%	3.9%	21%	75%
LA 1/ after2 yr	0.0%	1.0%	2.0%	3.0%	25%	72%

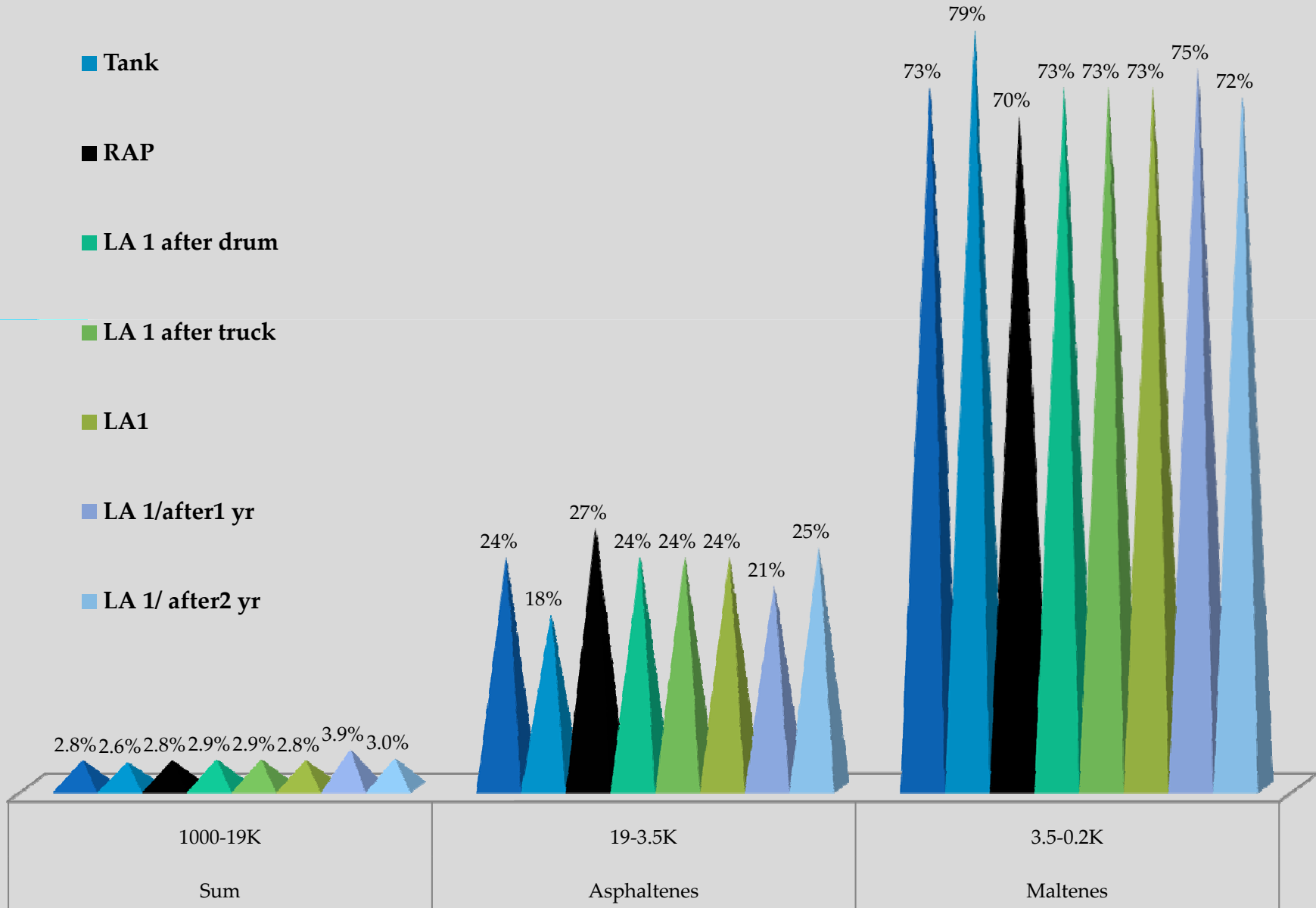


# Typical Binder THF Insolubles

PG Grade or RAP sample	Typical % insoluble THF on 0.45 mμ filter
64-22, original	4%-5%
76-22 original	6-7%
76-22, core new route	10-11%
RAP	7-10%
Source	Atypical
B1 refinery	18%
LA 18 new	23%

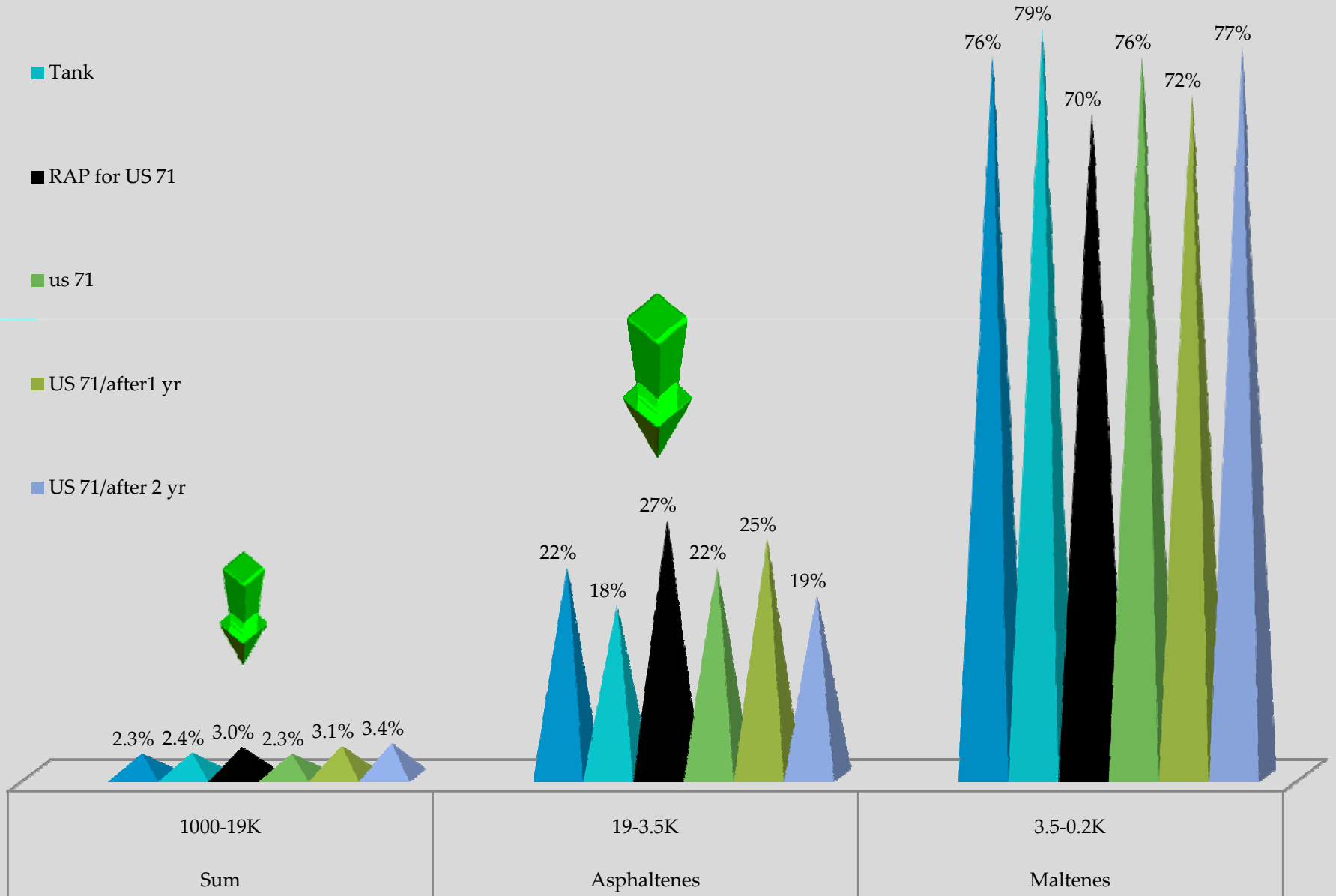
# Supplier B

- Refinery
- Tank
- RAP
- LA 1 after drum
- LA 1 after truck
- LA1
- LA 1/after1 yr
- LA 1/ after2 yr



# Supplier A

- Refinery
- Tank
- RAP for US 71
- us 71
- US 71/after1 yr
- US 71/after 2 yr



# Supplier C

■ Refinery

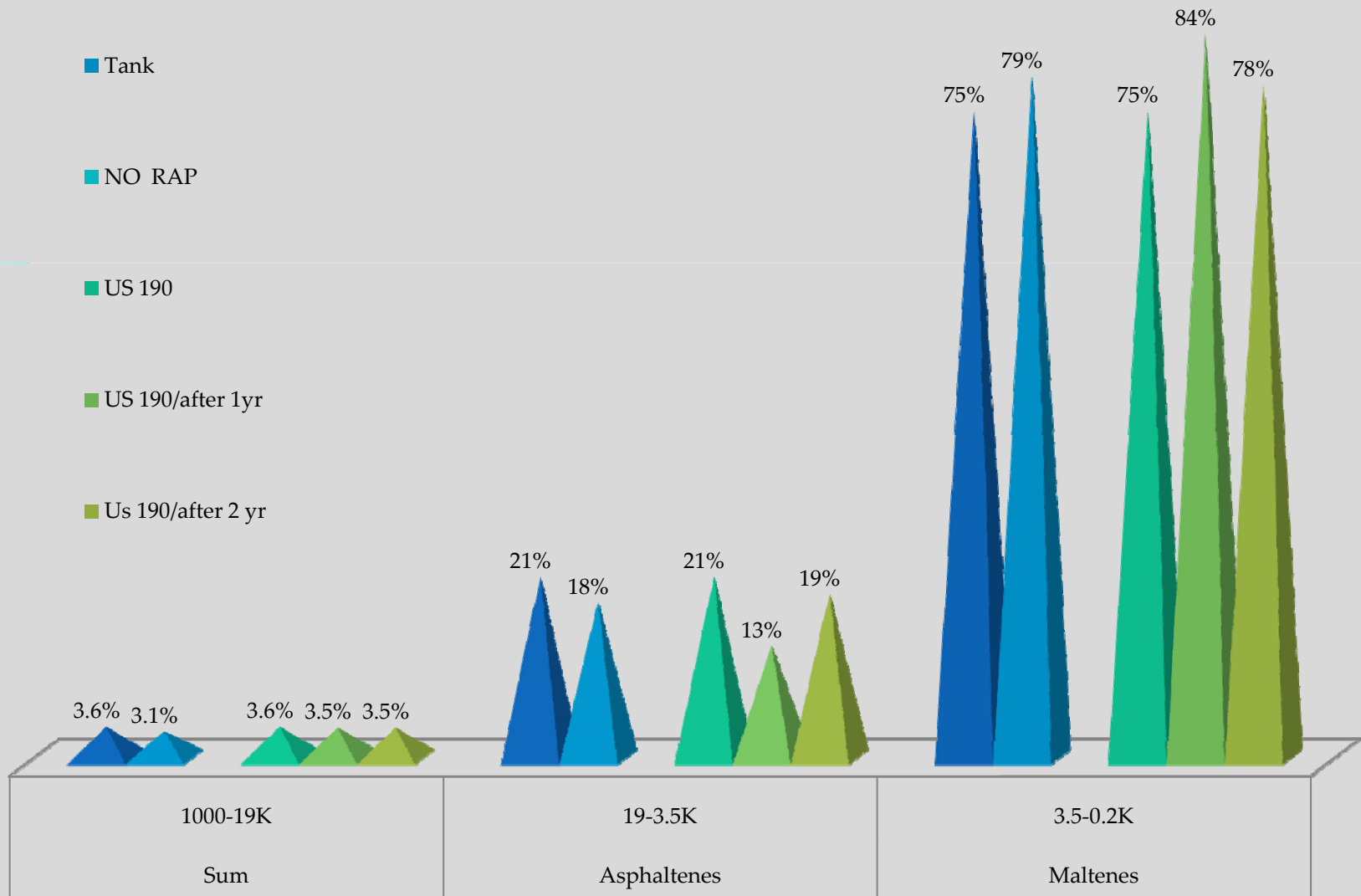
■ Tank

■ NO RAP

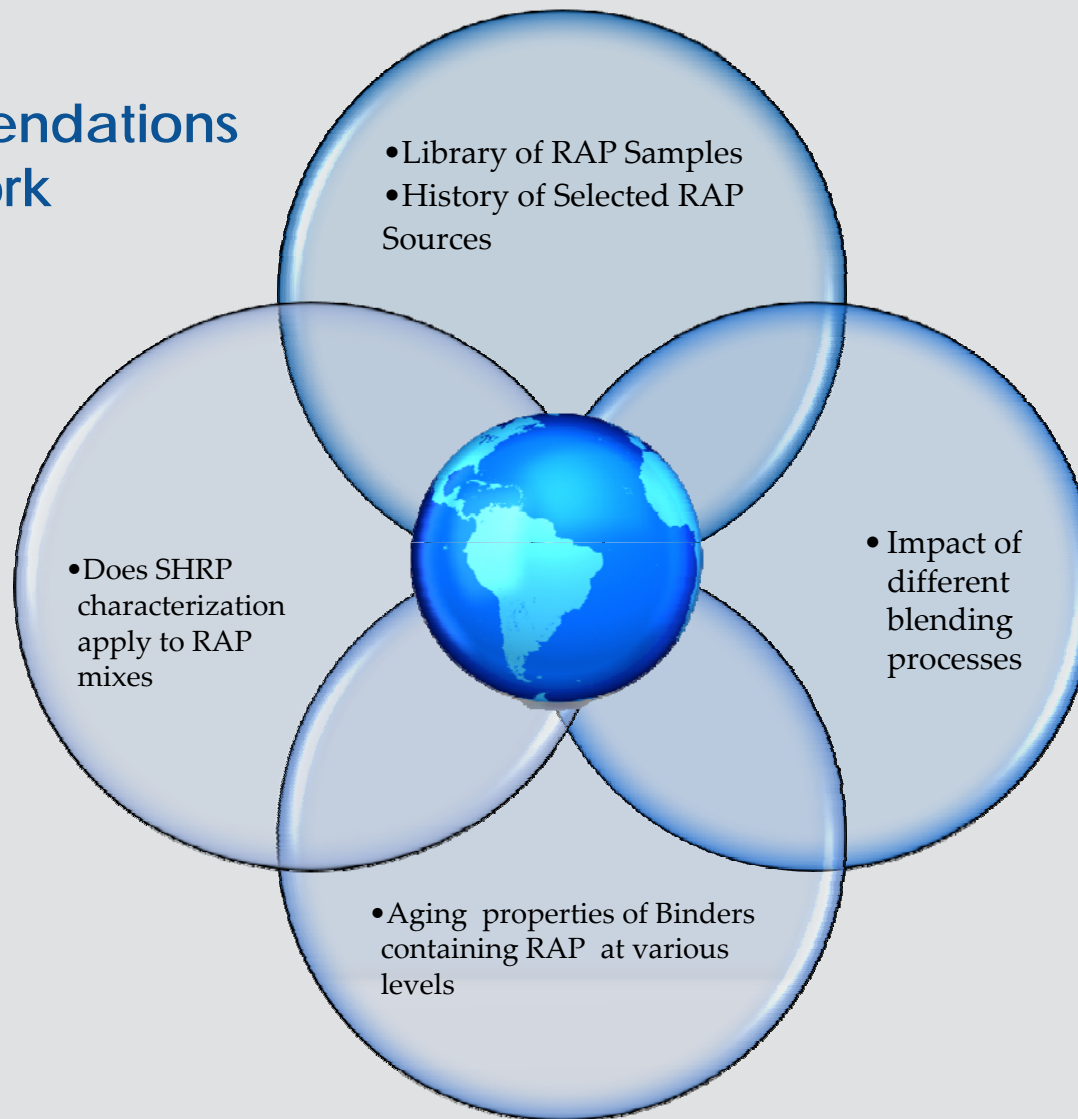
■ US 190

■ US 190/after 1yr

■ Us 190/after 2 yr



## Recommendations Future Work



# RTFO vs. Road Cores

	<b>VHMw</b>	<b>HMw</b>	<b>MMw</b>	<b>Sum</b>	<b>Asphaltenes</b>	<b>Maltenes</b>
	<b>1000-300K</b>	<b>300-45K</b>	<b>45-19K</b>	<b>1000-19K</b>	<b>19-3.5K</b>	<b>3.5-0.2K</b>
Refinery A/ RTFO	0.2%	1.3%	0.9%	2.4%	20%	78%
LA 26 road core	0.2%	1.5%	1.4%	3.1%	25%	72%
Refinery B/ RTFO	0.3%	1.2%	1.3%	2.9%	19%	79%
LA18 road core	0.0%	1.2%	2.3%	3.5%	26%	70%
Refinery D/ RTFO	0.5%	0.8%	0.7%	2.0%	16%	82%
LA 15 road core	0.2%	1.2%	0.9%	2.3%	20%	77%

Old RAP vs  
newer PMAC  
RAP Additives,  
nature and  
content

Aging  
simulation  
related to GPC  
analysis

Concerns

RAP Storage  
time and  
conditions

Percentage  
RAP upper  
limits

# CONCLUSIONS

Experimental data limited by number of RAP samples available. Please send us more samples.

Field samples containing RAP age less than predicted by RTFO laboratory aging.



*Thank You , any Questions ?*

