

Performance of High TBN Sulficylates

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Applications of Detergents

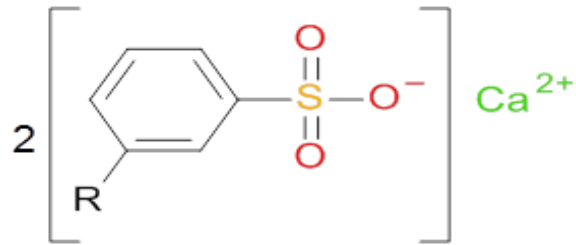
- Detergents are widely used in
 - Automotive engine oils
 - Marine engine oils
 - Metalworking fluids and manufacturing industries



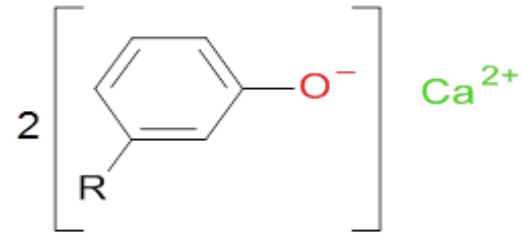
Functions of Detergents in Oils

- Neutralization-Alkalinity reserve
- High temperature detergency and cleanliness
- Rust/corrosion protection
- Friction modification/anti-wear
- Dispersancy
- Oxidation stability

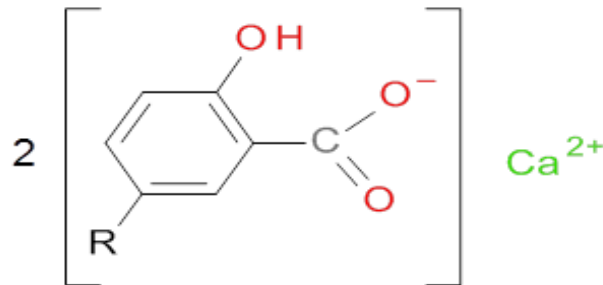
Typical Detergents



Sulfonates



Phenates



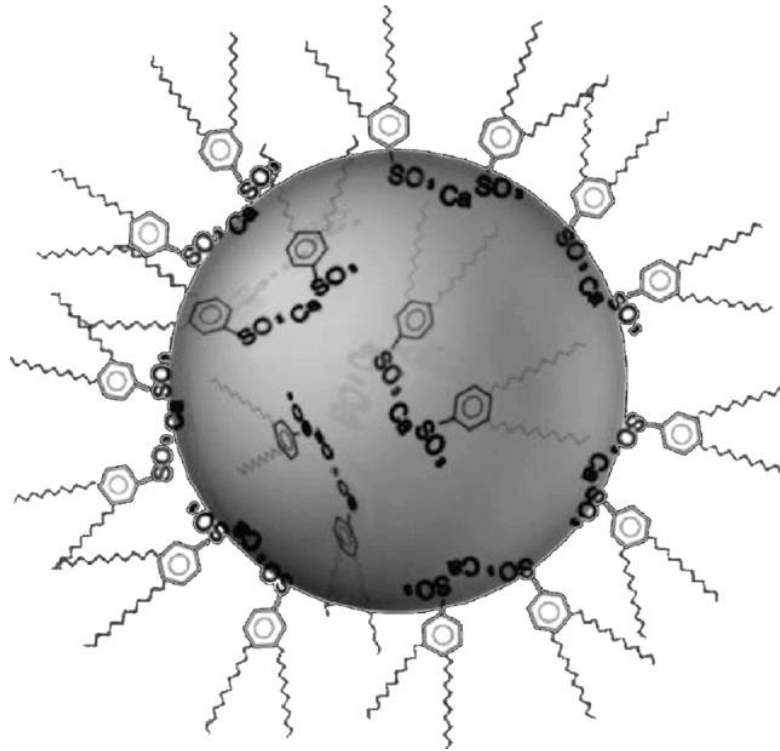
Salicylates

R= alkyl group

Note: (1) Metal can vary, e.g. Mg, Na and Ba; (2) Overbased detergents contain metal carbonate

Model of an Overbased Detergent

The model consists of a spherical core of metal carbonate surrounded by a monolayer of surfactant. The core radius is 1 – 10 nm and the surfactant layer is 1-5 nm thick.



What is Sulficylate?

- **Sulficylate** is a reaction product of overbased sulfonate and salicylate
 - It is not a simple blend of sulfonate and salicylate
 - The new concept and process are associated with US patent 7009072 (R. Muir, 2003)
- High TBN sulficylates have been developed with TBNs up to 450

Test Materials

- New overbased Ca sulfcylates (220 TBN, 350 TBN and 450 TBN)
- Traditional overbased sulfonate, salicylate, and phenate
- Blend of overbased sulfonate and salicylate
- Commercial add-pack of sulfonate and phenate

Typical Properties

	RD 220	RD 310	RD 311	C300	S270	C300/ S270	P250	S/P (MCL)
Additive	Sulficylate	Sulficylate	Sulficylate	Sulfonate	Salicylate	Sulfonate/ salicylate	Phenate	Sulfonate/ phenate
TBN, mg KOH/g	220	350	450	302	270	285	250	302
Vis. @100 °C	43	84	175	75	41	50	281	180
Ca, %	7.7	12.9	17.1	12.0	9.6	10.3	11.4	12.1
S, %	0.83	1.35	1.1	1.9	0.2	1.88	0.95	2.95
Sulfonate soap, %	5-15	15-25	10-20	28		11		10-20
Salicylate soap,%	35-45	20-30	25-35		30	18		
Phenate soap,%							40-50	20-30

Detergency - Panel Coker Test

(Panel: Al, Panel T: 315 °C, Oil Sump T: 100 °C, Splash/bake: 15/45s, 4 hr)

	TBN mgKOH/g	Deposit (mg)/ 3.5% in VP 500 oil	Deposit (mg)/ 4.5 TBN in VP 500 oil
RD 220 (Sulficylate)	220	8	38
RD 310 (Sulficylate)	350	40	43
RD 311 (Sulficylate)	450	32	42
C 300 (Sulfonate)	302	51	30
S 270 (Salicylate)	270	11	110
C300/S270 (Sulfonate/Salicylate)	285	86	116
P250 (Phenate)	250	60	173
Sulfonate/Phenate	302	55	63

Example – Panel Coker Test (at 4.5 TBN)



Sulficylate/ 220 TBN 38 mg	Sulficylate/ 350 TBN 43 mg	Sulficylate/ 450 TBN 42 mg	Sulfonate 30 mg	Salicylate 110 mg	Sulfonate/ salicylate 116 mg	Phenate 173 mg	Sulfonate/ phenate 63 mg
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- All sulficylates have better detergency than commercial sulfonate/phenate blend, sulfonate/salicylate blend and individual phenate and salicylate

Rust Test (ASTM D665B in Sea Water)

	Results @ 0.1% in VP 100 oil
RD 220 (Sulficylate)	Pass (no spots)
RD 310 (Sulficylate)	Pass (no spots)
RD 311 (Sulficylate)	Pass (no spots)
C300 (Sulfonate)	Fail (2 spots, light rust)
S270 (Salicylate)	Fail (over 20 spots)
C300/S270 (Sulfonate/Salicylate)	Fail (18 spots)
P250 (Phenate)	Fail (heavy rust)
Sulfonate/Phenate	Fail (heavy rust)

Rust Test

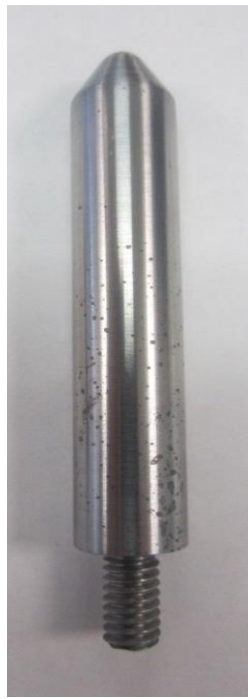
(> 20 times better than sulfonate/phenate package)



RD311
0.1%
Pass



Sulfonate /
Phenate
0.1%
Fail



Sulfonate /
Phenate
0.5%
Fail



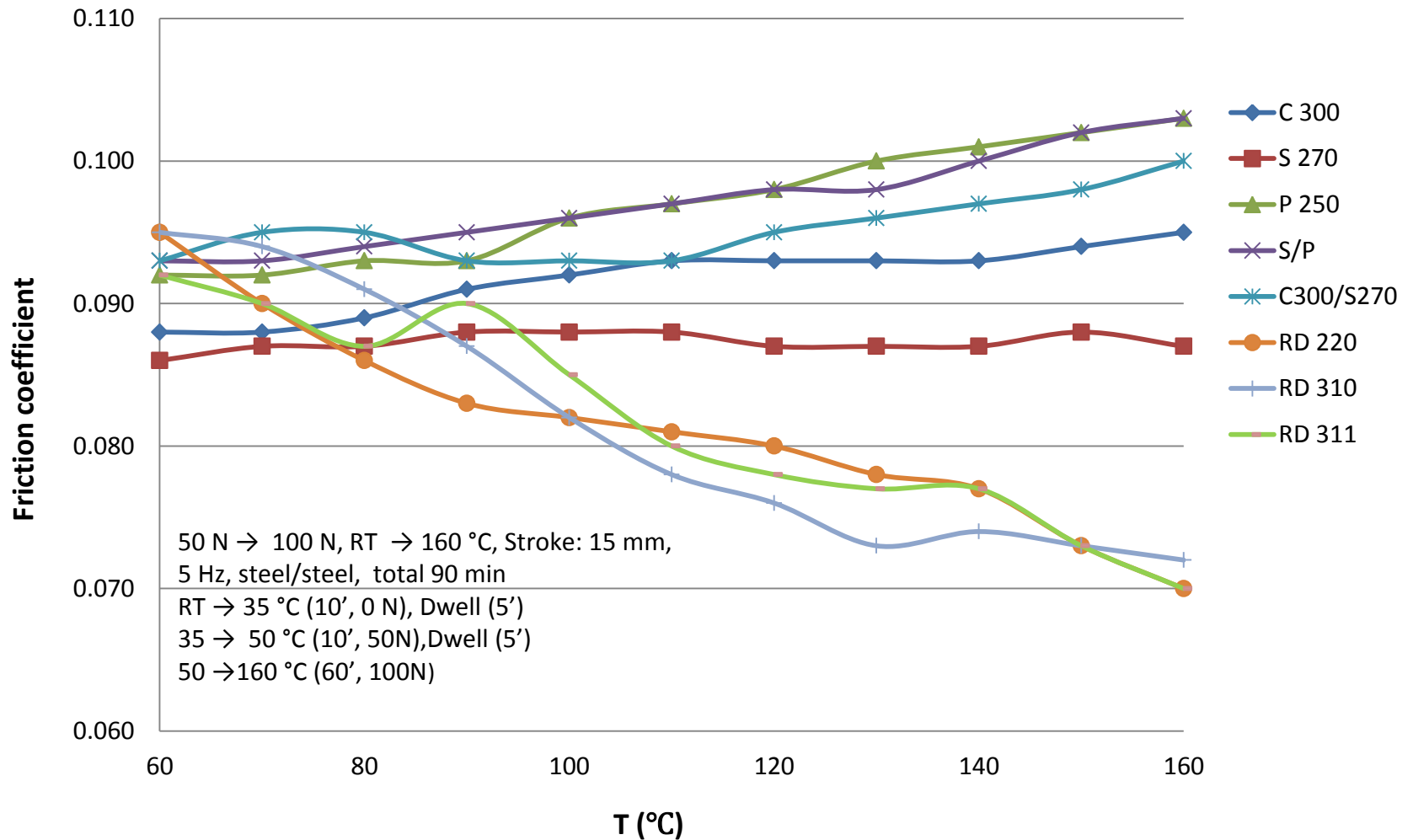
Sulfonate /
Phenate
1.0%
Fail



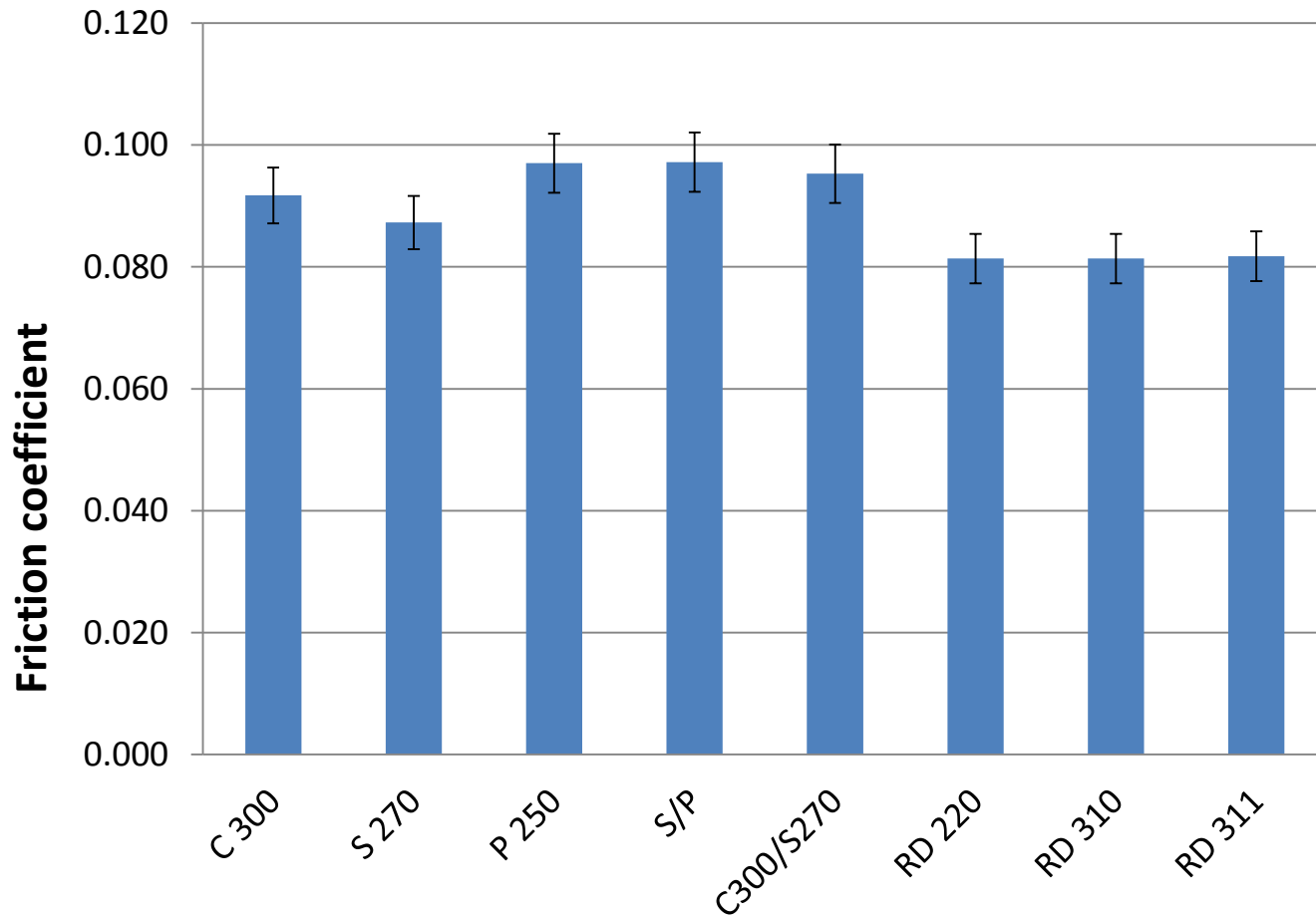
Sulfonate /
Phenate
2.0%
Fail



Friction – Effect of Temperature (Cameron Plint – 4.5 TBN in finished PCMO)

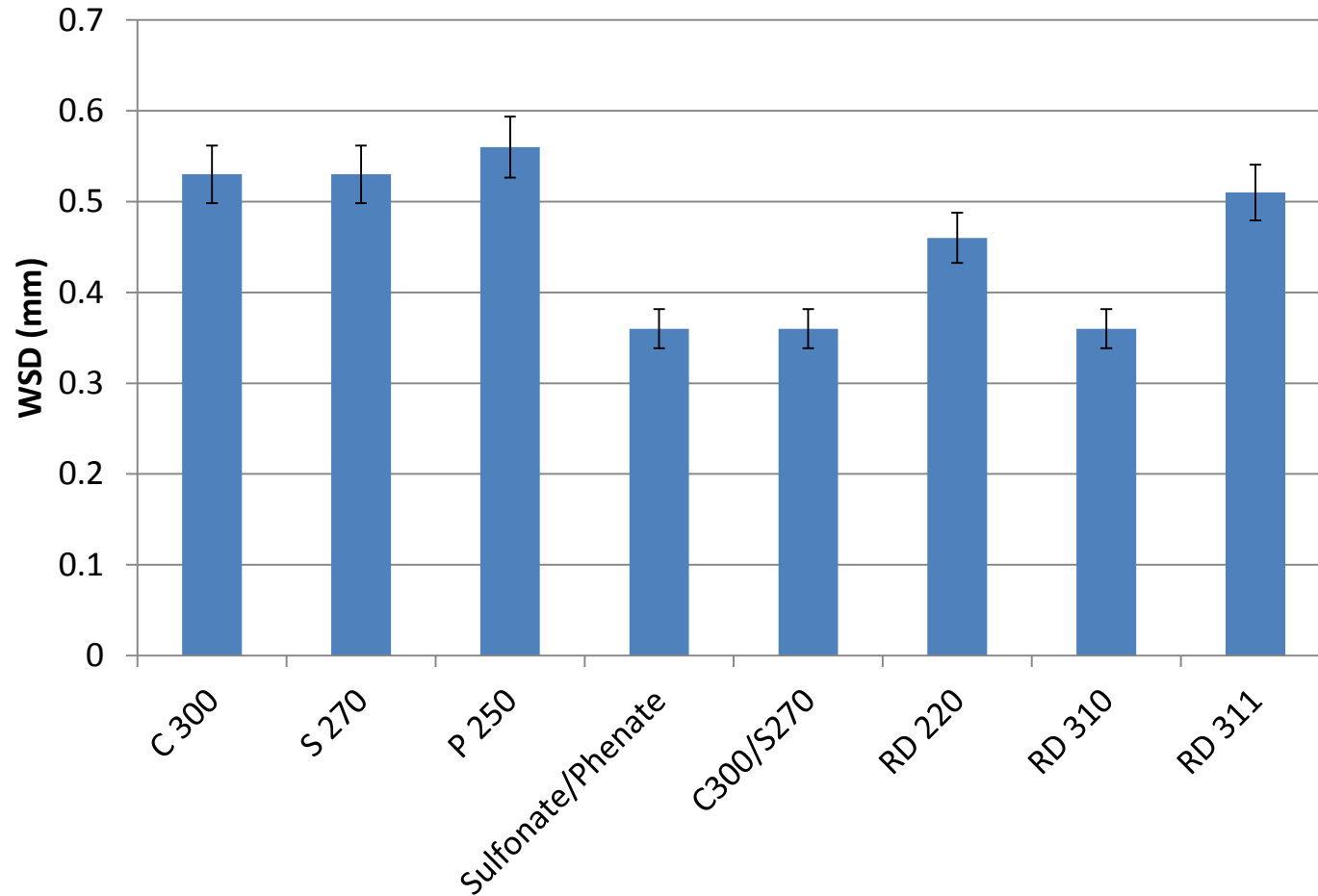


Average Friction Coefficient (Cameron Plint Test)



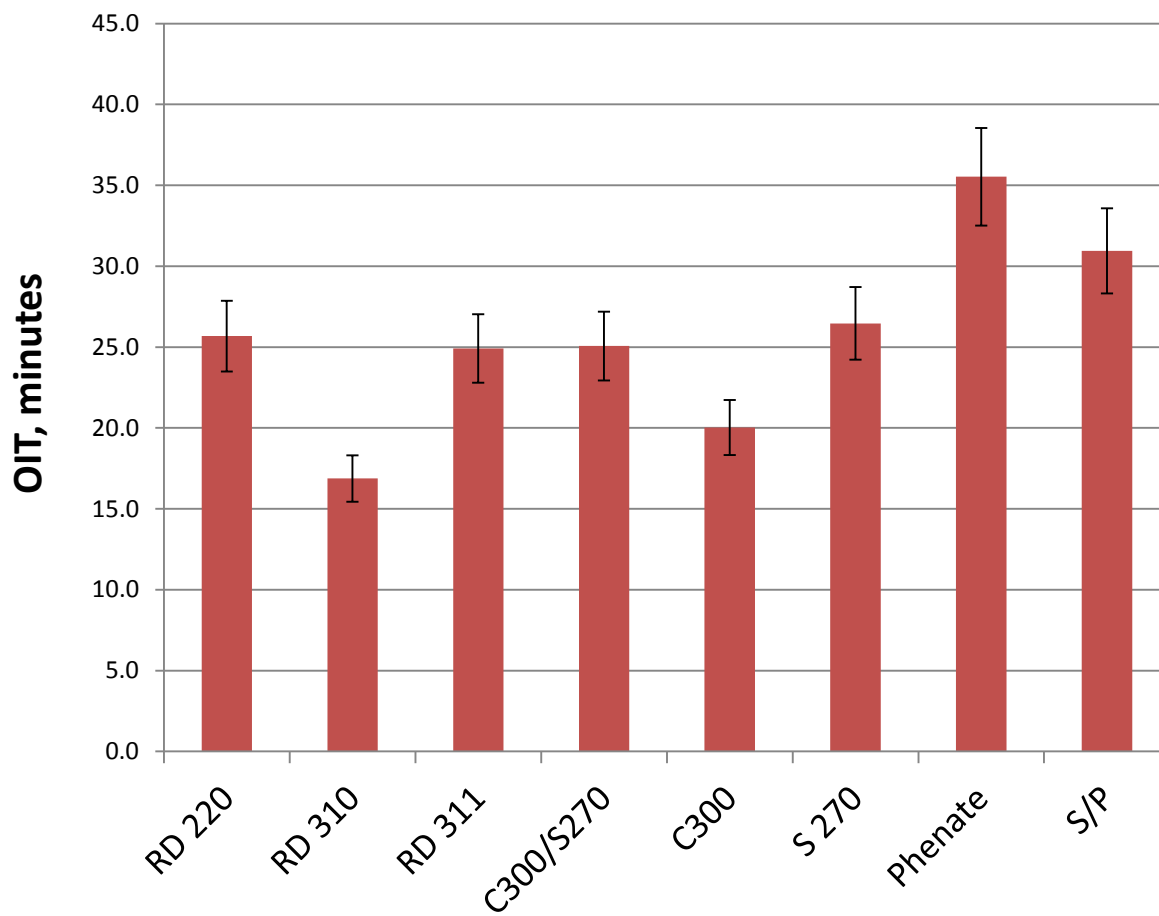
Wear Performance

(4 Ball, ASTM D4172 – 1200 rpm, 40 kg, 75 °C, 4.5 TBN in VP 500 oil)



Oxidation Stability

(PDSC, all @ 4.5 TBN in finished PCMO)



Temp:

195 °C

O₂ Pressure:

500 psi

O₂ Flow:

100 mL/min

Aluminum Pan:

Open

Sample:

3mg

Catalyst:

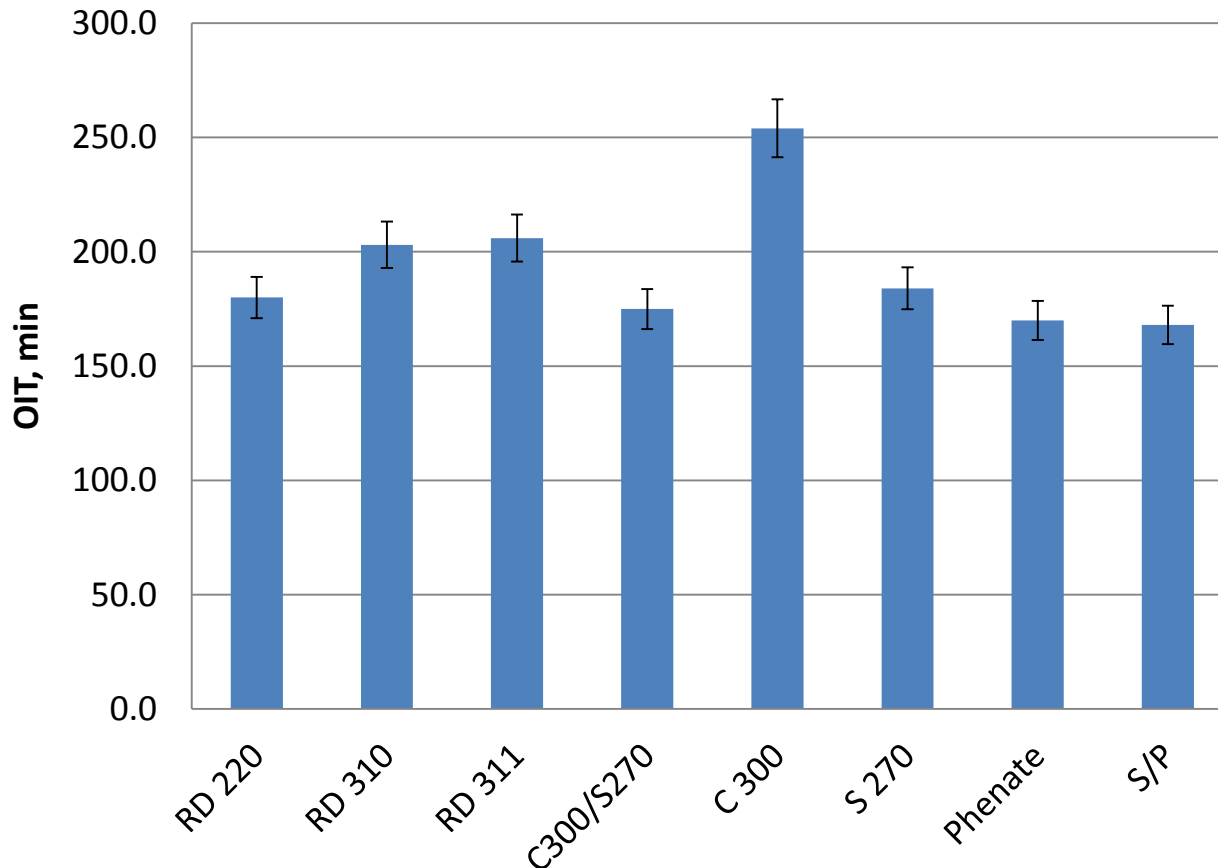
50 ppm Fe

OIT:

Onset of peak

Oxidation Stability

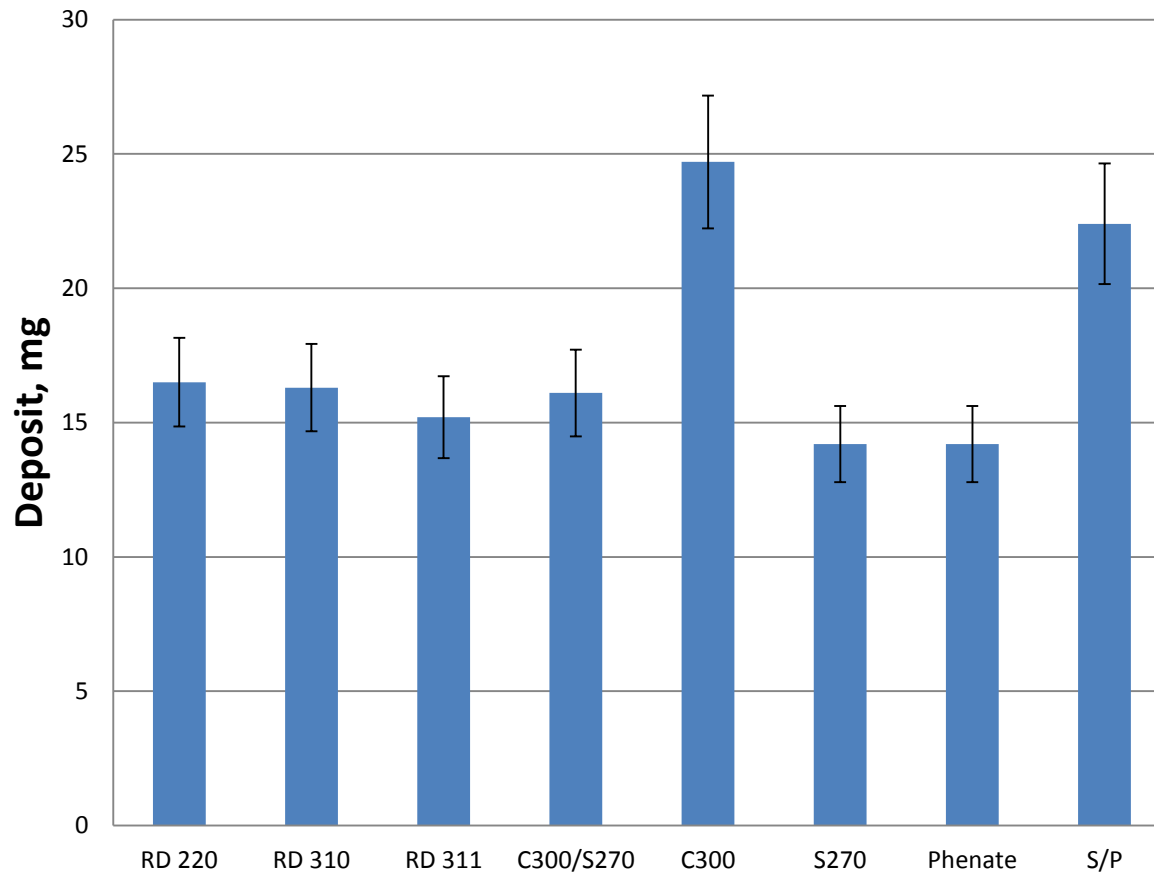
(RPVOT, ASTM D2272 – all @ 4.5 TBN in finished PCMO)



Temp:
150 °C
Starting O₂ P:
90 psi
Catalysts:
H₂O & Cu Wire
Sample:
50 g
OIT at
Drop of 25 psi

Thermo-Oxidative Deposit

(TEOST ASTM D7097 – all @ 4.5 TBN in finished PCMO)



Dry Air:

10 mL/min

Oil Sample: **8.5 g**

Organo Metallic

Catalyst:

1 % of Test Oil

(3:2:1 of Fe:Pb:Sn)

Oil Flow Rate:

0.25 g/min

Temp. (rod):

285 °C

Time:

24 hours

Measurement:

Deposits on rod

Sulficylate vs. Sulfonate/Salicylate

- Stability in base oil
 - Test condition: 70 TBN, SAE 50 oil, 60 °C

Sample	Sediment, Vol. %	
	3 weeks	8 weeks
Blend of Sulfonate/Salicylate	4.0 (Fail)	4.0 (Fail)
3 sulficylates (RD 220, RD 310 and RD 311)	0	Trace (< 0.05)

➤ Synthesized sulficylates are very stable in base oil!

Summary

- New sulficylate detergents were successfully developed with TBNs up to 450
- Sulficylates show excellent stability in base oil while the blend of sulfonate and salicylate is not stable in base oil
- Sulficylates are superior to the traditional detergents or their blends in detergency, rust control, and friction reduction at high temperature
- Sulficylates have excellent performance in anti-wear, oxidation stability and deposit control
- Sulficylates are multi-functional additives

Thanks!

Questions?

