High Performance Grease Development with Functional Products

Functional Products Inc.

September 2021

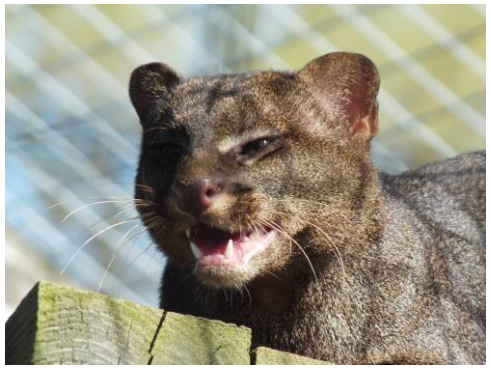
NLGI 2021 Tucson

Safety Moment

- Exits

- Desert hazards
  - Hydrate!
  - Sunscreen
  - Be mindful of wildlife on trails



Outline

- Introduction to Functional
- Grease Polymer Technology
- Portfolio Overview (Shortlist of Top Picks)
- Latest Developments
- V-4700 Project

Functional Products Inc.

- Founded in 1985
- Focus on customer driven solutions
- Full service technical support lab
- ISO 9001 with Design
- **Let us do the search and you do the research**

- Expansion completed 2020
  - Facility doubled
  - 2x lab and technical staff
  - Expanded test capabilities

Market Position

- Full Service Additive supplier and unique basestock supplier
- Over 40 types of tackifiers- food grade, biodegradable, high temperature, high shear, low temperature, textile, chain, anti-mist
- Ability to develop new performance packages with both mineral and bio-based compatibility in condensed timelines.
- Over 160 Polymer Products used in industrial lubricant, grease and specialty technologies
- New Product lines:
  - PMAs (Methacrylate) polymers for gear oil and hydraulic fluid
  - Functional Products now offers Paratac (2017) branded tackifiers and DOW OCPs (2020)
  - Biobased Packages
  - Grease components
  - Fluid and Viscosity Modifiers for esters, PAOs, and experimental oils

Lead Contacts

- David DeVore, President since 2004.
  - Co-Author ELGI Best Paper Award "Polymers Additives in Mineral and Vegetable based Grease"
  - BA Yale, Economics and History

- Gavin Duckworth, Vice President National Accounts
  - 23 years experience in polymers in lubricants focusing on methacrylates
  - US Patent – "Process for preparing low sulfur dispersant polymers".

Functional Products is a technically driven firm, we seek to answer why.

Experienced Technical Staff

- **Erik Willett**, PhD, Polymer Science, University of Akron; BS Chemistry University of Connecticut. Best Paper 2018 NLGI.
- **Jacob Scherger**, PhD Polymer Science, University of Akron; BS Physics, Miami University - Expertise in surface science of polymers. Published in top-tier scientific journals, Langmuir and Macromolecules.
- **Dan Vargo**, MS Chemistry, Case Western Reserve University.

  27 years synthesis chemistry with the Lubrizol Corp. Technical Editor TLT.
- **Bill Tuszynski**, Special Projects and Grease Specialist, PhD Chemistry Cornell University. 30+ years in lubricants and chemical industry
- **Jim Cordek**, BS Chemistry. 30+ years experience, Lubrizol and Dow.
- **Kathy Monda**, Quality and Laboratory Manager, Product registration and Reach Compliance officer

FPI as a Development Partner

- Design/Build/Test
  - Novel and high performance formulations
  - Market general or highly customized additives to complete the project
- “Black box” samples or under NDA
- Aggressive turnaround on projects and MOQs to startup
- Grease Lab and Fluids Lab services

Grease Lab

- Grease Lab
  - Top-treating finished greases with additives
  - Coordinating inside/outside lab testing to validate additives
  - 1 gallon open kettle system
    - Lithium, calcium, silica, polyurea preforms for now
    - 8x 400-gram batches per day output

- Further extended by partnerships with:
  - Falex, Koehler, Savant, Clark, Alcor

Routine Grease Testing

Area	Tests	GC-LB	HPM	HPM WR	HPM CR	HPM HL	HPM LT
Consistency, Worked Cone	D1403 (1/4-scale), D217 (100Kx)	X	X	X	X	X	X
Roll Stability	D1831 (2hrs@RT, 50hrs@80C)		X	X	X	X	X
Dropping Point	D566 (old), D2265 (new)	X					
Low Temperature Rheology	Brookfield viscometer to -60C		X				XX
Low Temperature Torque	D1478 (-20 or -30C)		X	X	X	X	XX
Kesternich Mobility	DIN 51805 (-30C)						X
Oxidative Stability RPVOT	D942 (100hrs@100C)		X	X	X	X	X
Oil Separation, Conical	D6184 (30hrs@100C)		X	X	X	X	X
Oil Separation, Storage	D1742 (24hrs@25C)	X	X	X	X	X	X
Water Washout	D1264 (1hr@79C)	X	X	XX	X	X	X
Water Sprayoff	D4049 (5min@40C)			X			
Roll Stability with Water	D8022 (2hrs@RT)			X			
4-Ball Wear Scar	D2264	X	X	X	X	XX	X
4-Ball EP and Load Wear Index	D2596	X	X	X	X	XX	X
Copper Corrosion	D4048 (24hrs@100C)		X	X	X	X	X
Elastomer Compatibility	D4289 (168hrs @ 125C)	X	X	X	X	X	X

"XX" = improved requirements for this category

Fluids Lab Capabilities

- High performance synthetic lubricant formulation and additive dev.
  - Lower cost, higher performance 75W- automotive gear
  - HVI hydraulics (VI 140, 180, 220+)
  - Environmentally acceptable lubricants
  - NSF H1 incidental food contact

- 1-10 gallon lab pilots
- 1-2 drum production pilots

Routine Fluids Testing

Area	Tests	Industrial Fluids	R&O, HF, Turbine	Industrial Gear	Auto Gear	Crank Case
Kinematic Viscosity	D445 (capillary)	X	X	X	X	X
Viscosity Index	D2270	X	X	X	X	X
Pour Point	D97 (manual tilt)	X	X	X	X	X
Brookfield Viscosity	D2983				X	
Cold Cranking Simulator	D5293					X
TP-1 MRV	D4684					X
Ductless Siphon Tackiness	Internal	X		X		
HTHS	D4683 (150C)					X
Diesel Injector (K-O) Shear	D6278 (30-250 cycles)					X
Sonic Shear	D2603/D5621	X	X			
20hr KRL Shear	CEC L-45-A-99		X	X	X	
4-Ball Wear	D4172 (15 or 40kg@75C)	X	X	X	X	X
4-Ball EP and LWI	D2783	X	X	X	X	
Falex Pin & Vee EP	D3233A	X		X	X	
Copper Strip Corrosion	D130 (3-24hrs@100-120C)	X	X	X	X	X
Turbine Oil Rust	D665A (water), D665B (sea)	X	X	X	X	X
RPVOT Oxidative Stability	D2272	X	X			
TOST	D943		X			
Demulsibility	D1401, D2711, Steam Emulsion	X	X	X	X	X
Foam	D892 (I/II/III), Flender	X	X	X	X	X
Air Release	D3427		X			
Elastomer Compatibility	Various	X	X	X	X	X

Recent Conference Research

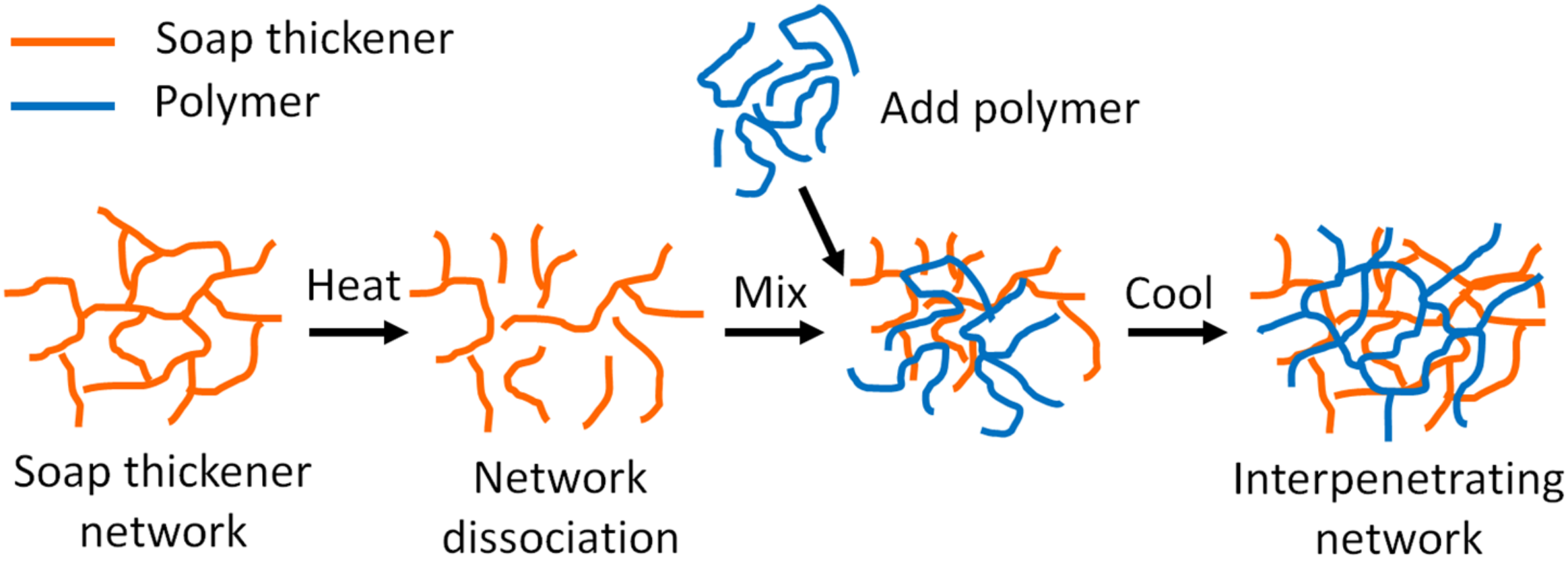
- Formulating water resistance specialty greases
- Shear stability of grease with grease polymers
- Water-based viscosity modifiers for lubricants
- Low temperature behavior of viscosity modifiers (NLGI Award)
- Measuring tackiness objectively (joint paper w/ Falex)
- Durability of tackifiers at high temperature vs. their polymer chemistry
- Quantifying tackiness in grease with probe tack
- Tailoring grease adhesiveness with VMs and tackifiers
- Biobased viscosity modifiers for EAL lubricants and grease (ELGI Award)

Grease Polymer Technology

- 10+ years R&D into ‘polymer-modified greases’
  - NLGI and ELGI author award winners

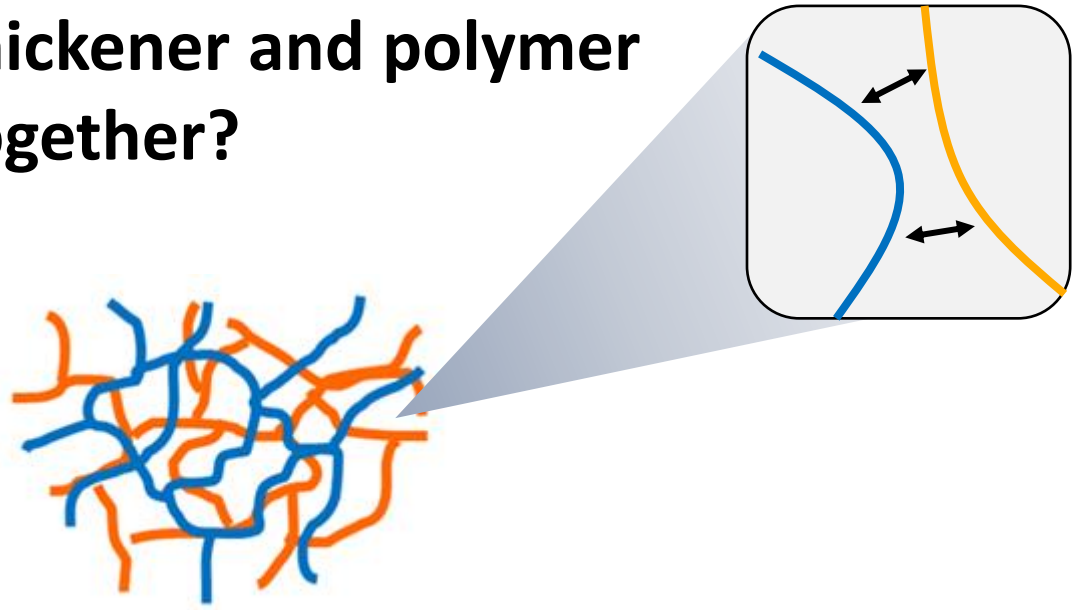
- Why polymers?
  - **G**reater Adhesion
  - **R**educed Bleeding
  - **E**levated Tackiness
  - **A**dded Yield
  - **S**uperior Shear Resistance
  - **E**nhanced Water Resistance

Interpenetrating Network Theory



Three Grease Polymer Types

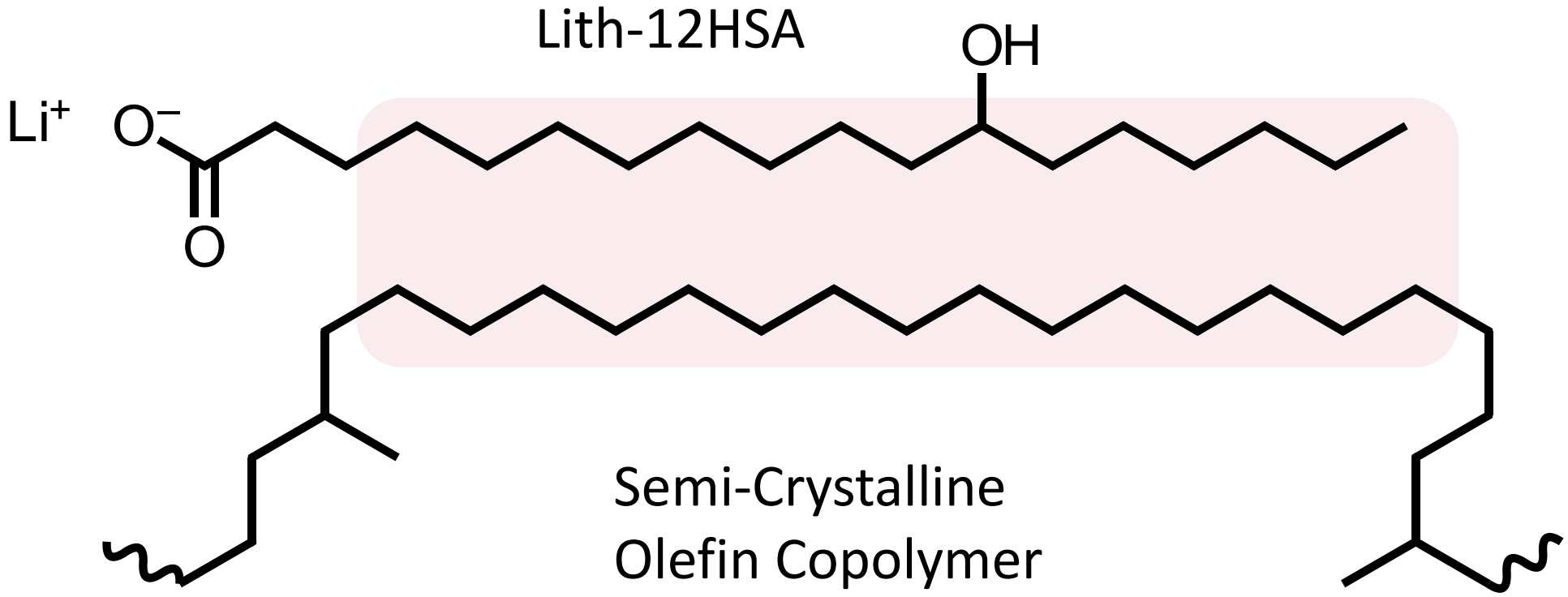
What's holding the thickener and polymer together?



Three types of like-like interactions

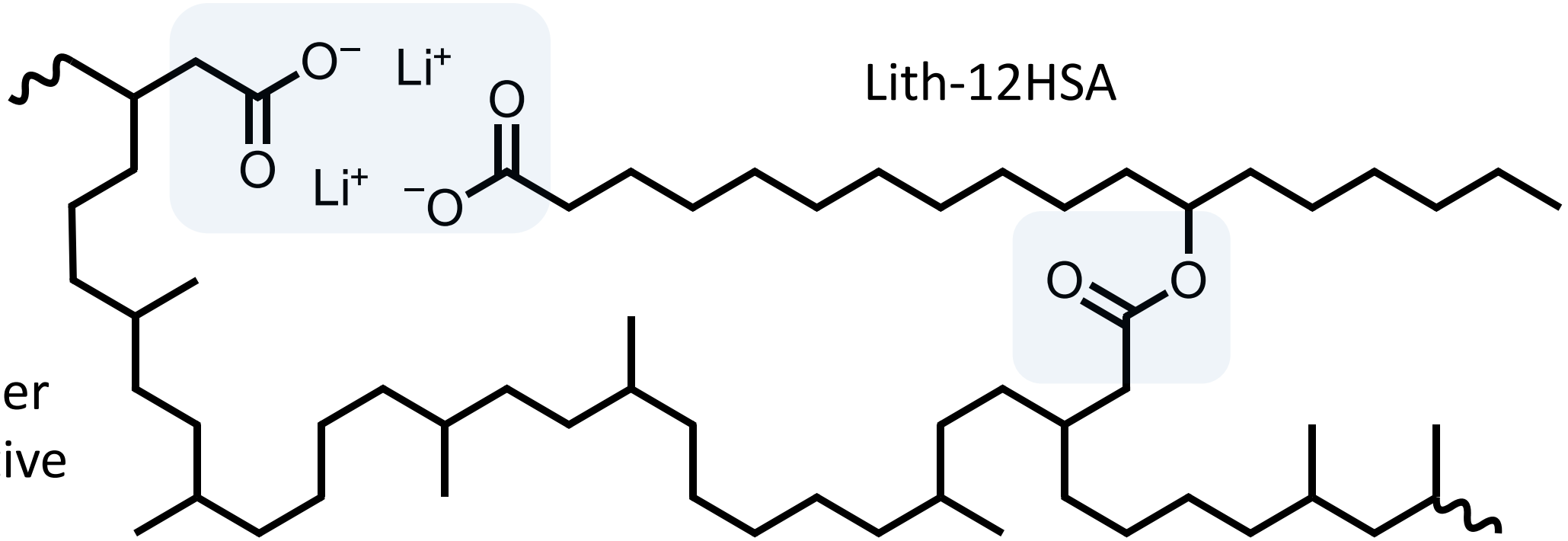
1. Temperature Sensitive

- Crystalline “waxy” interactions



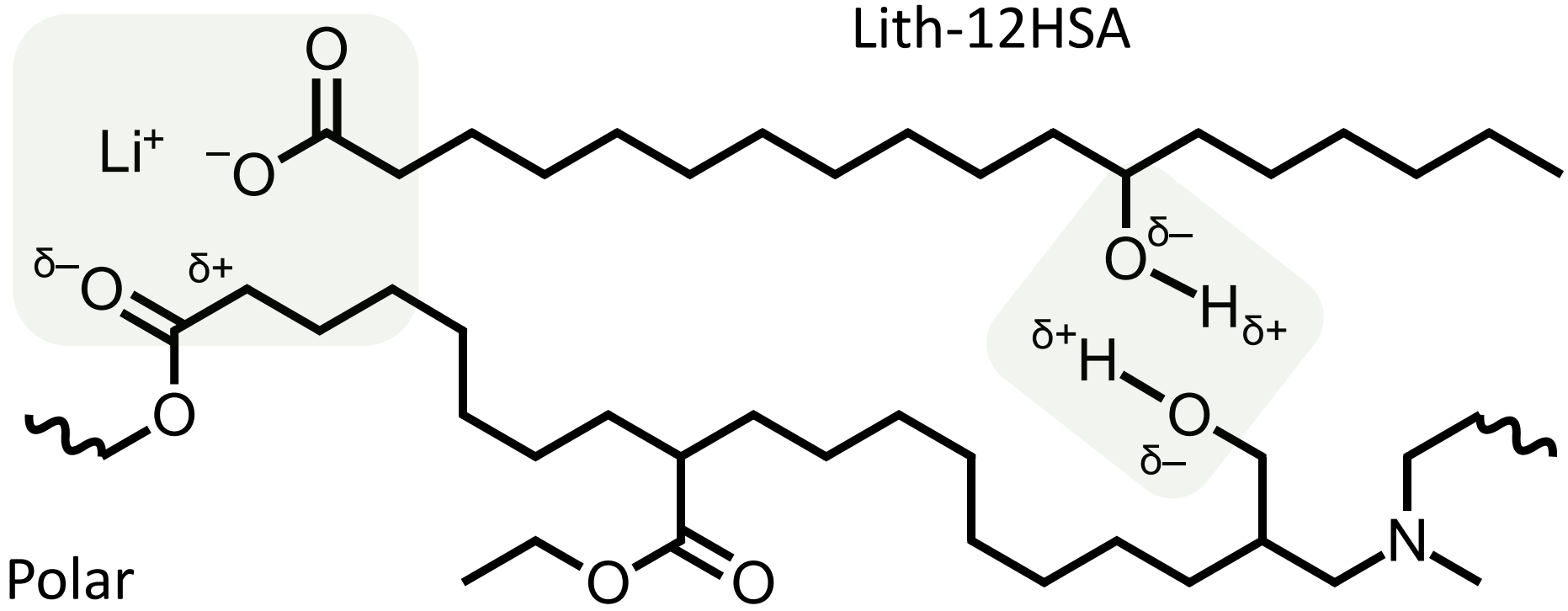
2. Reactive

- Reactive grafting to –OH sites (like a boron complex) or coordination between metal carboxylates (like a diacid)



3. Hydrogen Bonding

- Coordination between high electron density of C=O, C-N, C-O and any metal soap ions



Soapless Grease Modification

- Clay
  - Highly polar surface
  - Dispersant effect by reactive and hydrogen bonding grease polymers
- Silica and PTFE
  - Inert surfaces
  - Viscoelastic modification of surrounding base fluid (tackifier)
- Polyethylene / polypropylene greases
  - Modification with ethylene-propylene viscosity modifiers

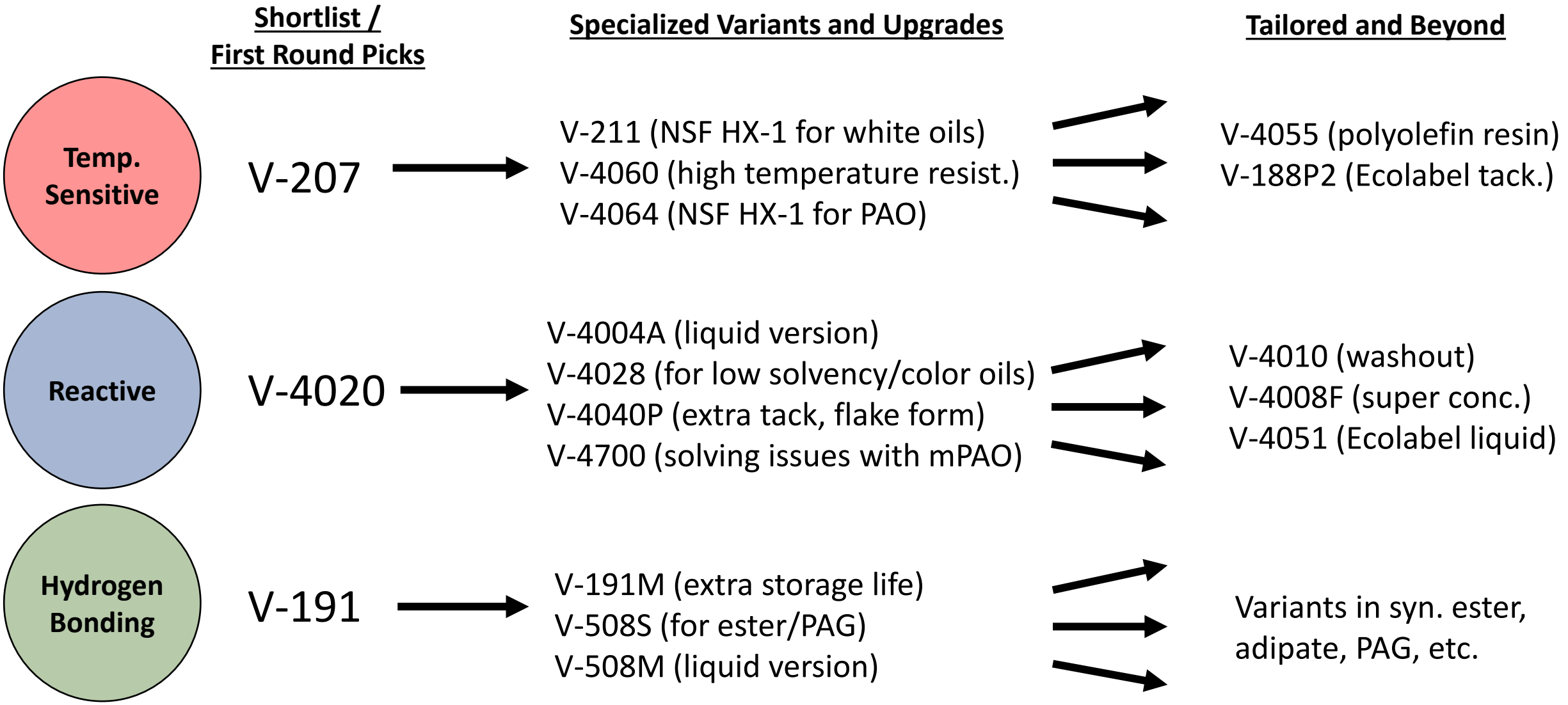
Polymer Additives for Grease

- “Every grease is its own animal”
  - Various advantages and disadvantages from:
    - Thickener type
    - Base oil (type, viscosity, blend ratios)
    - Production method (open kettle, continuous)

- Functional provides a deep portfolio of options suit the specific needs of a specific grease formulation

- And experience on selection and usage of any polymer in grease

A Deep Portfolio



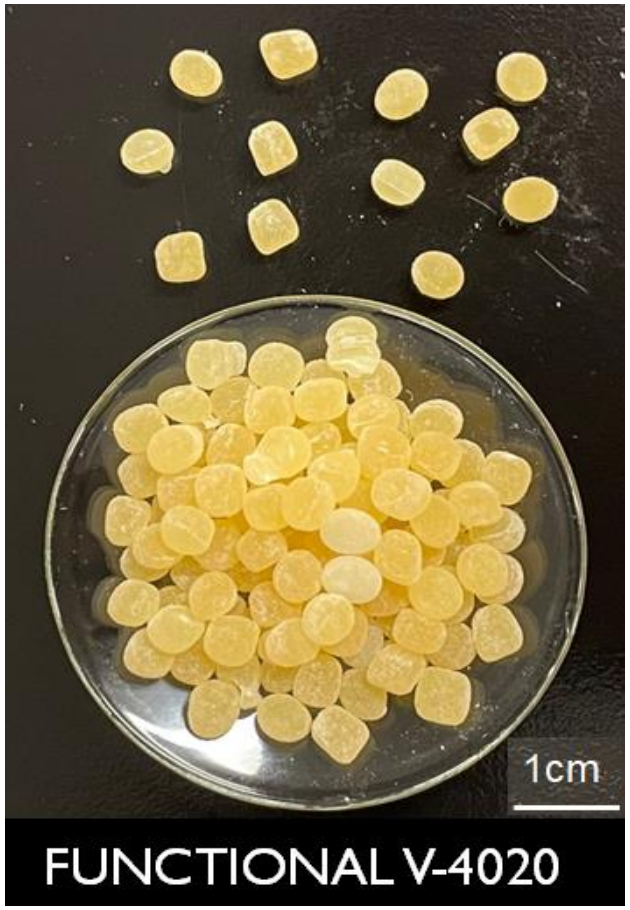
Shortlist of Top Grease Additives by Need

Situation (Thickener or Base Oil Type)	Product	Form	Typical wt%	Temp.	Notes
Conventional Industrial - Standard soap-based greases in paraffinic and naphthenic petroleum oil					
Lithium and Lithium Complex Grease	**V-4020**	**Pellet**	**0.25 -0.5%**	**C/R**	Multifunctional reactive pellet; low tack
Calcium or Aluminum Grease	**V-207**	**Flake**	**0.5 -1%**	**A**	Adds water resistance and moderate tack, cuts oil bleed
Inorganic / Particle Grease	**V-191**	**Liquid**	**0.5 -1%**	**A**	Good synergy with clay, silica, or filled greases; tacky
Synthetic Industrial - Synthetic basestocks with high amounts of mPAO or PB thickener can prevent conventional additive from working properly					
mPAO- or PB-based Grease	**V-4700**	**Liquid**	**2 -10%**	**A**	For soap-based greases in high viscosity synthetic oils
Food Grade Grease - Low solvency PAO, white oil, or H1 Group III base stocks					
H1 Food Grade – White Oil, Group III	**V-211**	**Flake**	**0.5 -1%**	**B**	Optimal for medium solvency H1 base oils
H1 Food Grade – PAO	**V-4064**	**Pellet**	**2 -3%**	**C**	Optimal for low solvency PAO H1 base oils
Environmentally Acceptable Grease - using biodegradable synthetic esters, hydrocarbons, or vegetable oils					
Biodegradable Synthetic (Li/Ca/Al)	**V-4051**	**Liquid**	**1.5 -4%**	**A**	For soap-based biodegradable low solvency base oils
Biobased Vegetable Oil Based	**V-508S**	**Pellet**	**3.2% = ISO150**	**B**	Use as a shear stable VM to build veg oil to high ISO VG

From Functional Products "Basic Grease Polymer Guide"

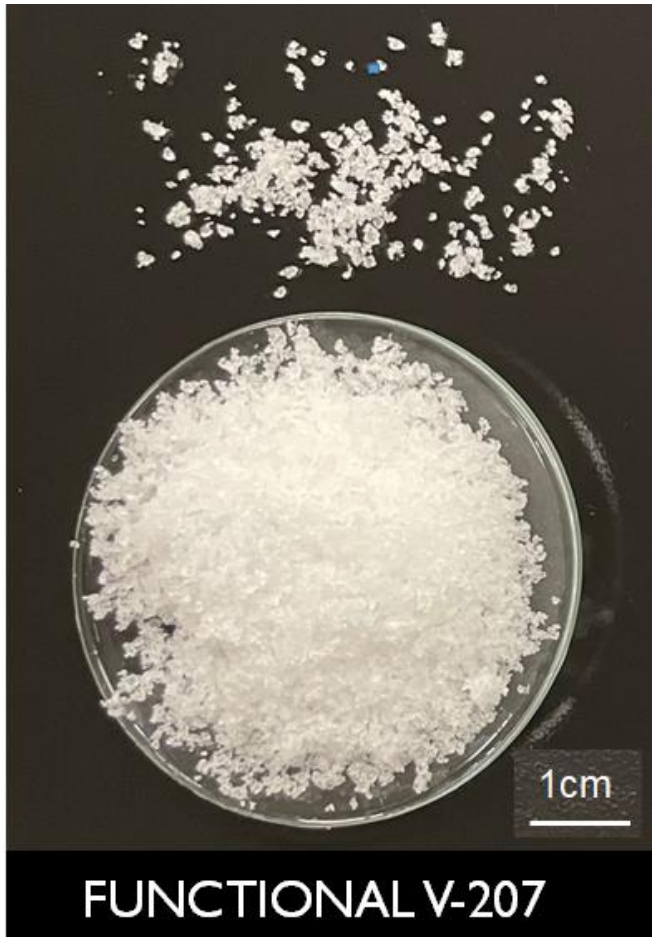
Li/LiX Options

- **FUNCTIONAL V-4020**
  - Reactive: pellet form
  - 0.2 – 0.3wt% typical treat in Li/LiX; 0.5wt% in Ca
  - Added during the reaction; also any time later
  - Improves water resistance, oil bleed, shear stability
    - Minimal tackiness or adhesiveness added



Ca/Al Options

- **FUNCTIONAL V-207**
  - Temperature sensitive: flake form
  - 0.5 – 1.0wt% treat
  - Greatly improves water resistance and oil bleed
    - Adds tack



Particle Greases

- **FUNCTIONAL V-191**
  - Hydrogen bonding: emulsion form
  - 0.5 – 1.0wt% treat
  - Provides improved water resistance
    - Helps disperse the particle
    - Provides tack and viscoelastic reinforcement

High Viscosity Synthetic Greases

- **FUNCTIONAL V-4700**
  - Reactive: high viscosity liquid form
  - 2 – 10wt% treat depending on severity of case
  - Able to disperse into high % mPAO
    - Other grease polymers are insoluble
  - Significant water resistance improvement

NSF HX-1 Options

- **FUNCTIONAL V-211**
  - Temperature sensitive: flake form
  - 0.5wt% treat
  - Prefers medium solvency oils: white oil, Group III, some ester or AN
  - *Excellent* in industrial paraffinic grease

- **FUNCTIONAL V-4064**
  - Temperature sensitive: flake form
  - Prefers low solvency, low viscosity oils: PAO + PB or EPO
  - 1wt% - Improves oil bleed and mechanical stability
  - 2-3wt% - Water resistance improvement

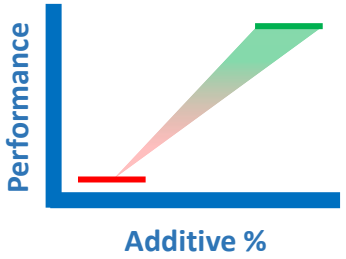
EAL Options

- **FUNCTIONAL V-4051**
  - Reactive: liquid form
  - Biodegradable and European Ecolabel-listed
  - Performs like a liquid version of V-4020

- **FUNCTIONAL V-508S**
  - Hydrogen bonding: pellet form
  - 10wt% max in Ecolabel LuSC
  - Works as a viscosity modifier to build ISO 100+ veg oil, ester, PAG
  - Improves yield, water resistance in bio grease

Simple Project Workflow

Identify Current Performance and Target Goals



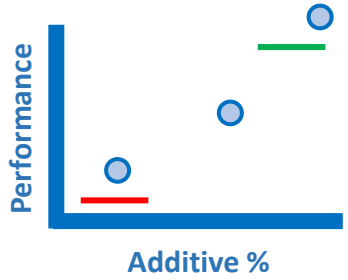
Collect Composition Info (Thickener, Oil)

Li/Ca/Al/Sil/Clay/…
Paraffinic, Naphthenic, PAO, …
ISO VG 68, 220, …
NSF? EAL?

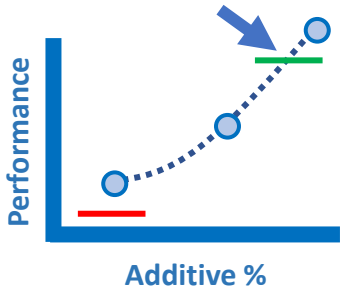
Select Grease Additive from Shortlist Based on Composition

Temperature Sensitive
Reactive
Hydrogen Bonding
Tackifiers
Other

Prepare Greases at Three wt% of Additive and Test



Generate Data Curve and Select Optimal Treat



Complex Projects

- Improving further on performance with additives
  - 2nd or 3rd round with a related but different grease polymer using lessons learned from previous round of testing
- Issues from unique solubility– mPAO, specialty base oils (ester, PAG)
- "Where to start?"
  - Full formulation project from blank slate
  - Determine base oil, ISO VG, NLGI grade, etc.
  - Typical for specialty greases (NSF, EAL, oil field)

Latest Specialty Developments

- 2021 grease conference paper
  - Thursday at 1:45PM, technical session 4

- Viscosity modifiers in grease
  - Tailoring the ISO VG, VI, PP of base fluid to differentiate grease

- Expanded EAL and HX-1 offerings
  - Polymers, packages

- EAL/VGP formulation design

FUNCTIONAL V-188P2 (Ecolabel)

- PAO-based OCP tackifier
  - 60%+ biodegradable, Ecolabel LuSC-listed
  - Intended to support PAO-based biodegradable lubes/grease
    - HEPR hydraulic fluid; PAO grease
    - Also works in petroleum, PAO, OSP, oleates/stearates
- Also useful for textile lubricants, metalworking fluids, sprays

NSF HX-1 submitted and pending

FUNCTIONAL GA-400 (HX-1)

- Food grade gear oil package – 1.1wt% max treat
- 1.1wt% for H1 gear oils – 200 kgf weld load
  - “Light duty” gear oil, sugar mill, grease
- Half treat (0.55wt%) for hydraulic fluids – 160 kgf weld load

GA-400 Performance (HX-1)

Performance in ISO 320 mPAO/PAO Blend		
	0.55wt%	**1.1wt%**
4-Ball EP Weld Load, ASTM D2783	160 kgf	200 kgf
Load Wear Index, ASTM D2783	33.0	40.0
Falex EP Load, ASTM D3233A	760 lbs.	1020 lbs.
4-Ball Wear, ASTM D4172 – 20 kg	0.48 mm	0.31 mm
RPVOT, ASTM D2722	1700	1200
Turbine Oil Rust, ASTM D665A – distilled water	Pass	Pass
Copper Strip Corrosion, ASTM D130 – 3 and 24 hours @ 100°C	1b	1b

- AW/EP can be fortified with CI-427 amine phosphate or Ceramax boron nitride EP

- Requires defoamer/demulsifier
  - 0.1% DF-400 = 0/0 Seq. I/II/II foam D892
  - 1.0% DM-400 = 40/40/0 (30) D1401

FUNCTIONAL VGP HF 46 (ongoing)

FUNCTIONAL VGP HF 46
Lot# 20210414-L

	Test Method	value	unit
lb/gal @ RT		7.57	lb/gal
Spec Gravity @ 60F		0.887	g/mL
API gravity		28.0	
Flashpoint, COC	D92	445F / 229C	C/F
Flame Point, COC	D92	475F / 246C	
KV40, cSt	D445	47.64	cSt
KV100, cSt	D445	10.74	cSt
Viscosity Index	D2270	225	
Pour Point	D97	-39	C
Cloud Point	D97	-27	C
Pour after 72 hr @ -20C	D97 mod	No Gel, Pours	
Demulsibility	D1401	40/40/0 (30)	mL O/W/E (min)
Foam I	D892	10/0	mL/mL
Foam II		30/0	mL/mL
Foam III		20/0	mL/mL
Total Acid Number	**D974**	0.21	Mg
Total Base Number	**D4739**	1.8	mg
Karl Fischer Water	**D6304C**	320	ppm H2O
ICP Elements	**D5185**	541	Ca
		1380	S
		7	Si
		26	B
XRF Elements		508	Ca
		1490	S

	Method	value	unit
4-ball Wear @ 15kg	D4172	0.478	mm
		0.073	COF
4-ball Wear @ 40kg	D4172	0.466	mm
		0.077	COF
Weld Load (D2783)	D2783	160	Kg
Scar One Stage Before Weld		2.75 mm	@126 kg
Vickers Vane Pump - Wear on Vane	D7403	35.0	mg
Wear on Ring		47.4	mg
Total Wear		82.4	mg
Biobased Content (wt%)		67	%
Biodegradability	D7373	≥ 64	%
RPVOT	D2722	71	minutes
Turbine Oil Rust A & B	D665A & B	Pass	
Copper Corrosion @ 3hrs, 100C	D130	1a	
Dielectric Strength	D877	51	kV

V-4700 Project

- Latest development work?

wt% V-4700	0.0%	1.6%	3.2%	4.0%	5.0%
WSO (D4049)	91.0%	67.6%	42.4%	36.4%	12.8%

Summary

- Functional Products (and services)
  - Design
  - Build
  - Test
- High performance and specialty lubricants and greases
- Market general solutions to highly tailored or even site-specific offerings