Polyalkylene Glycols (PAGs) as High-Performance Base Oil Components in Modern Greases

NLGI 2022

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Agenda



Defining components of a grease Grease production market **Polyalkylene glycols (PAGs)** Chemistry

Performance overview
Lubrication properties
Temperature behavior
Oxidative stability
Material and oil compatibility
Interaction with water
Corrosion protection performance
Formulation examples



About Clariant



BUSINESS SEGMENTS

Care Chemicals	Catalysis	Natural Resources
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Crop Solutions Industrial & Home Care Personal Care Active Ingredients Food Ingredients	Industrial Lubricants Paints and Coatings Construction Aviation	EO/PO Derivatives Softener Quats Sodium Laureth Sulfates

A grease is a solid or semisolid lubricant made by thickening base oils with gelling agents

Greases can be more than lubricants; they are often also used as sealants, corrosion inhibitors, shock absorbers and noise suppressants.



Typical key components of greases and their concentrations:

BASE OIL

(65–95%)

Mineral oils, esters, polyalkylene glycols (PAGs), polyalphaolefins (PAOs), silicone base oils (silicones), polyphenylethers (PPEs), perfluoroalkylethers (PFAEs)

THICKENER

(5–35%) Soaps, bentonite, silica, polyurea, polytetrafluorethylene (PTFE)

ADDITIVES

(0–10%)

- Corrosion inhibitors
- Antioxidants
- EP/AW additives
- Lubricity improvers
- Solid lubricants (e.g., graphite, PTFE)

Around 6% of all greases are based on synthetic oils and lithium types dominate the thickener market

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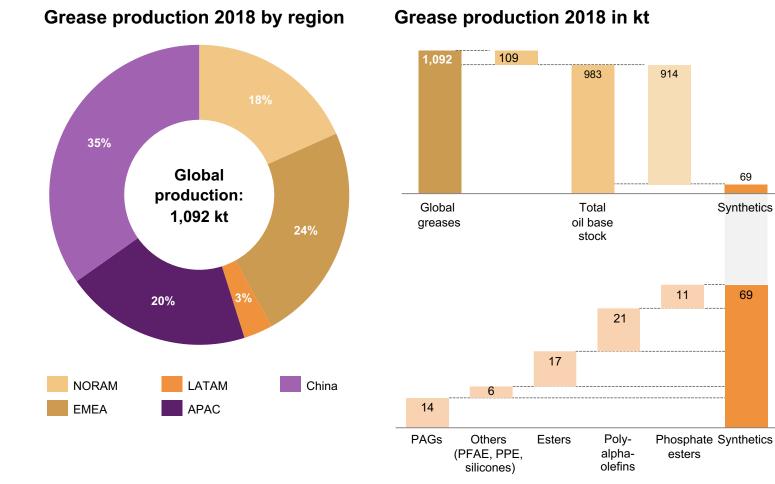
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esters

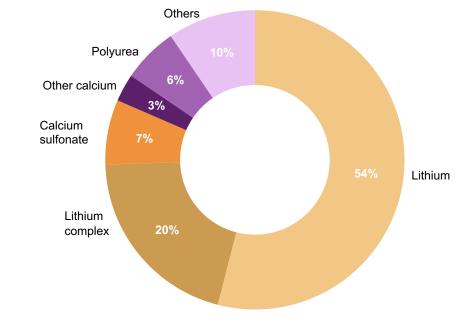
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Synthetics

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Sources: Internal Market estimation; NLGI Regional Production Split; NLGI Grease Production Survey 2017



Greases by thickener type 2017

Lithium types: With 2.3% CAGR, complexes cannibalize simple types (-1.8% CAGR) and other greases

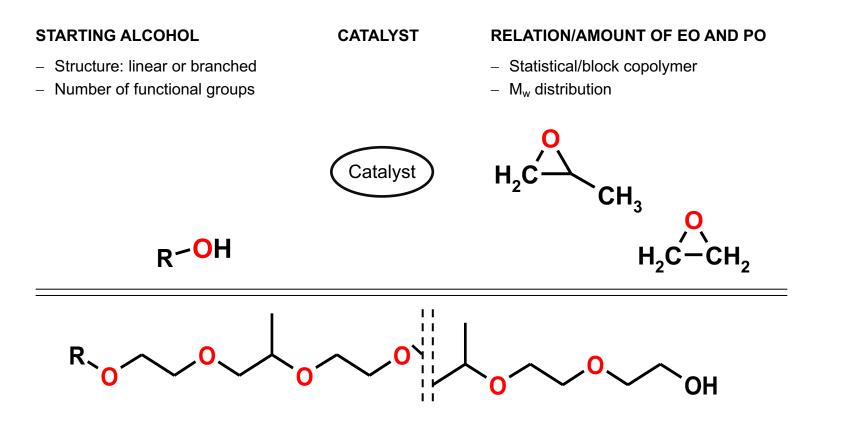
Calcium types: With 12% CAGR, sulfonates are taking shares from lithium complexes

Polyurea: Global CAGR 2.2%, with highest share in Japan, long-life applications, and expected push from e-cars and wind power

Others: incl. calcium complex, bentonite, silica < 1% share

Polyalkylene Glycols (PAGs): Synthetic polymers with highly adjustable properties

By varying factors like starting alcohol, amounts of ethylene and propylene oxide as well as catalysts used, PAG base oils can be precisely tailored to various requirements.



ADJUSTABLE PROPERTIES

- Viscositv
- Viscosity index
- Polarity/water solubility
- Freezing point
- Thermal properties
- Hydrodynamic friction
- Lubrication
- Various other properties



CATEGOR	Y PROPERTIES	RELEVANCY
	Flash point [°C]	Manufacturing, use
High-temperature behavior	Thermo-oxidative stability (5% loss [°C]/middle point [°C]/R [%])	Manufacturing, use; faster wear due to deposits; safety, environmental hazards
	Material compatibility	Insufficient compatibility can cause premature failure of components
Oil/oil and material compatibility	→ Oil/oil miscibility	Switch to other grease type can cause failure in performance; potential use as additive
Low-temperature behavior	→Pour point [°C]	Low-temperature applications
Lubrication properties	Viscosity index →	Temperature behavior, lubrication film thickness, load-carrying capacity
	SRV (friction coefficient µ, wear scar)	Lubrication, extreme pressure (EP)
	Biodegradability and ecotox/LuSC list	Fitness for use in EU Ecolabel greases
Sustainability/compliance	→Labeling	Reduced human toxicity and ecotoxicological hazards
	Food approved (NSF)	Fitness for use in H1 food-approved greases

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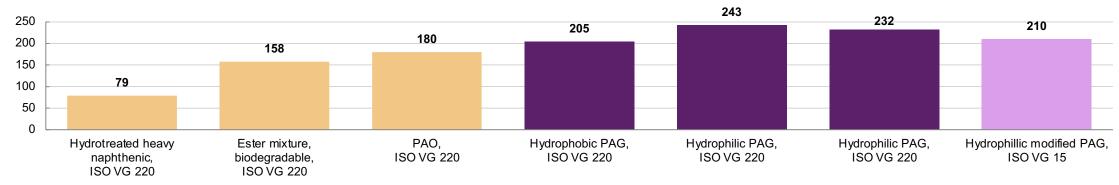
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Performance: good – medium – poor

		HYDROPHOBIC PAGe	5	HYDROPHILIC PAGs		HYDROPHILIC PAG modified	
CATEGORY	PROPERTIES	LOW VISCOUS	HIGH VISCOUS	LOW VISCOUS	HIGH VISCOUS	LOW VISCOUS	RELEVANCY
High-temperature	Flash point [°C]	190–217	227–240	210–231	250	199	Manufacturing, use
behavior →	Thermo-oxidative stability (5% loss [°C]/middle point [°C]/R [%])	184/227/0 No residues	202/249/0 No residues	184/226/0,3 No residues	221/270/0 No residues	176/222/0 No residues	Manufacturing, use; faster wear due to deposits; safety, environmental hazards
Oil/oil and material	Material compatibility	Nitrile butadiene rubb	er (NBR), 2-component e	epoxy-based primers, ethy	rlene propylene diene mo	onomer rubber (EPDM)	Insufficient compatibility can cause premature failure of components
compatibility →	Oil/oil miscibility FULLY SOLUBLE	LN, MN, LP, LV, LEP, LET	LN, LV, LEP	LV, LET	-	LN, LV, LEP, LET	Switch to other grease type can cause failure in performance;
	PARTLY SOLUBLE	MP, LS	MN, LP, MP, LS	LEP	LET*	MN	potential use as additive
	INSOLUBLE	HS	HS, LET	LN, MN, LP, MP, LS, HS	LN, MN, LP, MP, LV, LS, HS, LEP	LP, MP, LS, HS	
Low-temperature behavior →	Pour point [°C]	-66 – -50	-45 – -36	-66 – -50	-50 – -35	-72	Low-temperature applications
Lubrication properties →	Viscosity index	132–185	191–270	170–208	208–298	210	Temperature behavior, lubrication film thickness, load-carrying capacity
	SRV (friction coefficient μ , wear scar)	0.140/0.784 mm Smooth run	0.126/0.584 mm Smooth run	-	0.107/0.560 mm Smooth run	0.122/0.636 mm Non-smooth run	Lubrication, extreme pressure (EP)
Sustainability/ compliance →	Biodegradability and ecotox/LuSC list	YES/NO	NO/NO	YES/YES	YES/YES	NO/NO	Fitness for use in EU Ecolabel greases
	Labeling	NONE	NONE	NONE (except > B 11/100)	NONE	NONE	Reduced human toxicity and ecotoxicological hazards
	Food approved (NSF)	NO	YES	NO	YES	NO	Fitness for use in H1 food-approved greases

Lubrication properties – High viscosity indexes for high load tolerance

The viscosity index (VI) characterizes the viscosity-temperature behavior of lubricants



Viscosity Index ASTM D 2270



Applicability of greases over a wide range of temperatures



Polyalkylene glycols typically have very high VIs, enabling high load-carrying capacities

Lubrication properties – Superior results in friction and wear tests



SRV tests (*Schwingungs-Reibverschleiß*) are tribological tests measuring the friction and wear resulting from the oscillation of two specimens in loaded contact.

Common test for assessing lubrication properties according to standards ASTM D6425-11/DIN 51834-2:2010

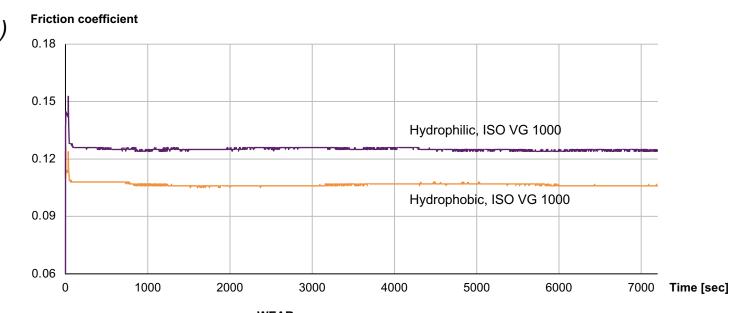
TEST PARAMETERS temperature: 50 °C, frequency: 50 Hz, stroke: 1,0 mm, duration: 120 min, load: 300 N



Closely simulates the operating conditions of lubricants



The Polyalkylene glycols achieve excellent results: low friction coefficients and wear scars



POLARITY	CHEMISTRY	SCAR* W _k [mm]	µ* _{min}	µ* _{max}	µ* ₁₅	µ* ₃₀	µ* ₉₀	µ* ₁₂₀	FAILURE* (μ > 0.3)
Hydrophobic	ISO VG 15 PAG	0.784	0.110	0.192	0.130	0.139	0.144	0.146	_
	ISO VG 1000 PAG	0.584	0.125	0.128	0.127	0.127	0.127	0.126	
	PAO	_	0.151	> 0.3					yes: 16 sec
	PAO	-	0.113	> 0.3	_	_	_	_	yes: 78 sec
Hydrophilic	ISO VG 15, modified PAG	0.636	0.100	0.198	0.110	0.119	0.128	0.135	
*average of 2 runs	ISO VG 1000 PAG	0.560	0.105	0.125	0.107	0.107	0.108	0.108	-

High-temperature behavior – Flash points that allow safe processing



Flash point (according to Cleveland Open Cup, DIN ISO 2592 – replaced DIN 51376):

The lowest temperature at which vapors above a volatile substance can form an ignitable mixture in air



Maximum safe temperature at which fluid can be processed during grease manufacturing

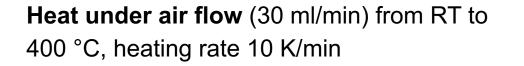


Most PAGs have a flash point above 200 °C, making them suitable for the manufacturing of complex soap grease

POLARITY	ISO VG	FLASH POINT [°C]
Hydrophobic	15	190
	32 – 46	~ 220
	100 – 1000	~ 240
Hydrophilic	22	210
	15	199
	46 – 220	~ 250
	220 – 1000	~ 250
	460 Star structure	270

FLACU DOINT

High-temperature behavior – Thermo-oxidative stability and burn-off





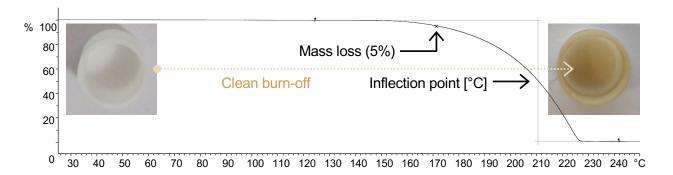
Maximum temperature at which fluid can be processed during grease manufacturing or used



Most PAGs have high inflection points and undergo a mass loss of 5% above 200 °C

All PAGs burn off cleanly

Thermo-oxidative resistance increases with ethylene oxide (EO) content and molecular weight – it can also be greatly improved by adding an antioxidant package



ISO VG	MASS LOSS (5%)	INFLECTION POINT (°C)	RESIDUES (%)
15, hydrophilic	184	227	0
15, hydrophobic + 3% additive package	201	258	0
220, hydrophilic	210	259	0
220, hydrophobic	202	249	0



excellent pour points

Low-temperature behavior – Pour points well below -50 °C possible

Pour point (according to DIN ISO 3016): The lowest temperature at which an oil still flows

<u>)</u> [[][Relevant for low-temperature	POLARITY	ISO VG	POUR POINT [°C]
٥ť	applications	Hydrophobic	15	-66*
			32 – 46	-50
-	The pour point drops with increasing		100 – 320	-40
$\langle \Theta'$	propylene oxide (PO) content and decreasing molecular weight		460 – 1000	-36
		Hydrophilic	22 – 100	-50
	Hydrophobic PAGs reach		15, modified	-72*
	pour points well below -50 °C		150 – 220	-45
			220 – 1000	~ -35
	The lower viscosity hydrophobic PAGs and hydrophilic PAGs have			

*according to ASTM D 7346-14

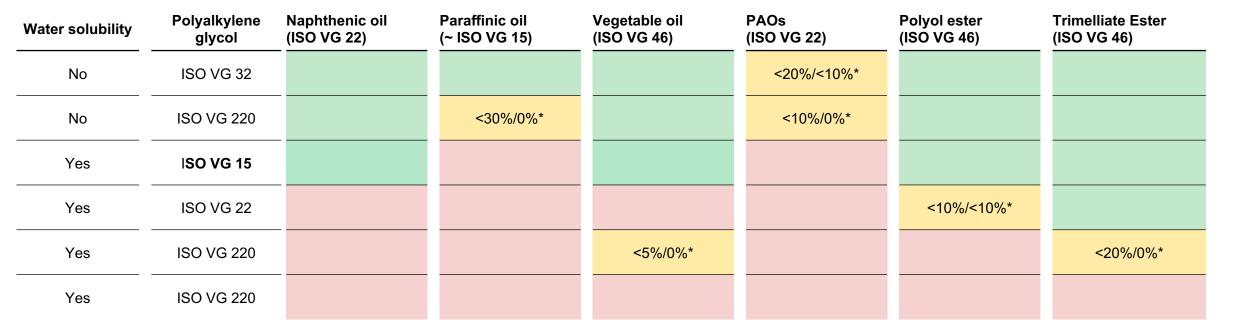
Material and seal compatibility



EPDM compatibility (in analogy to ISO 4925): Average of two measurements, EPDM RM 69, 100 °C, 7 d

	Prevents premature failure of materials	ISO VG	ISO VG 46, hydrophilic PAG	ISO VG 15, hydrophilic PAG, modified	Requirements ISO 4925
$\langle \bar{\otimes} \rangle$		Relative change in volume	+0.9% 🗸	+6.0% 🗸	min. 0% max. 10%
·	elastomer NBR 28/SX, and 2-component epoxy-based primers M 20 and P22	Change in hardness IRHD	-3 🗸	-6 🗸	min15 max. 0%

Miscibility with other oils – Depends on polyalkylene glycol structure



*Basestock in polyalkylene glycol/polyalkylene glycol in basestock



Switch to other grease type can cause failure in performance; only soluble oils can be used as additives



The miscibility of selected PAGs at RT depends on their molecular weight and hydrophobicity

Solubility: • completely soluble

Due to its special architecture, ISO VG 15 is also soluble in some hydrophobic media: low-viscous naphthenic oils, polyol esters and vegetable esters

soluble at certain concentrations

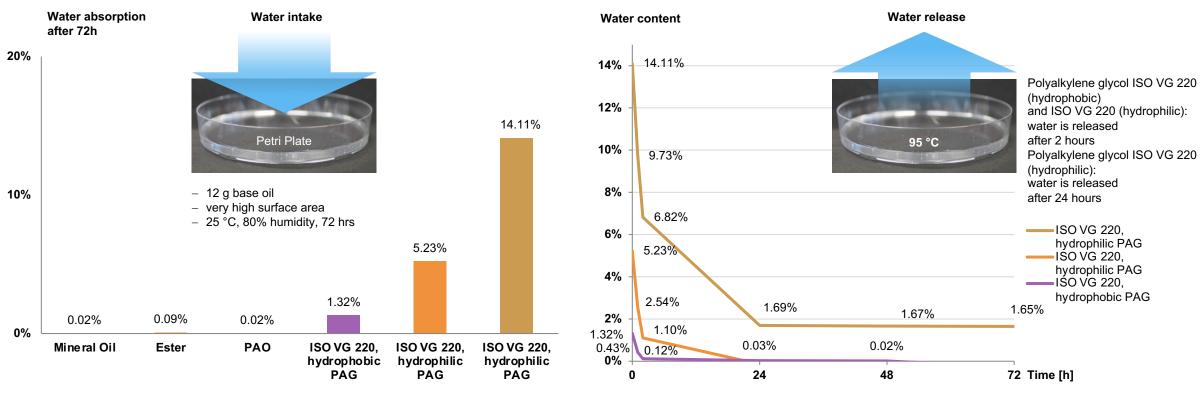


insoluble

WATER ABSORPTION OF PAGs

Hydrophilic PAGs Reversible physisorb water

Polyalkylene glycols absorb water from high humidity environments but tend to release it again

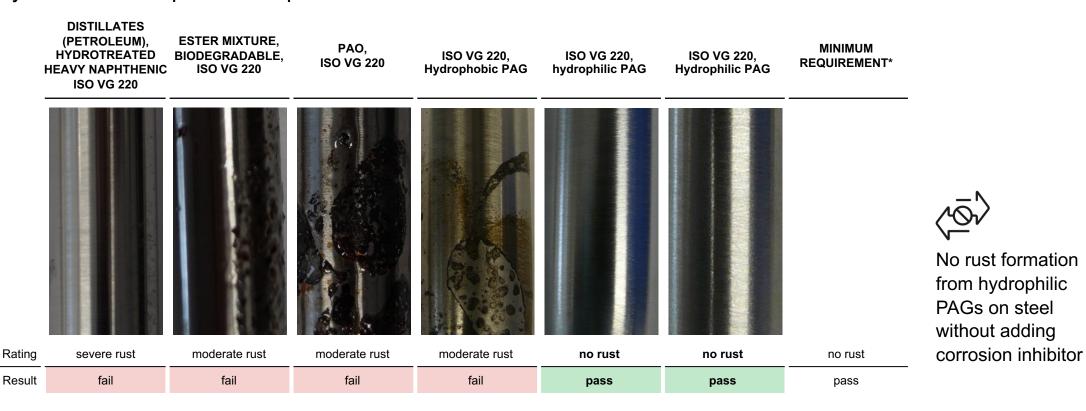


WATER RELEASE OF PAGs

Rust prevention on steel



Rust prevention test (according to DIN ISO 7120 A / ASTM D 665 A): Cylindrical steel specimen is put in stirred 300 ml oil/10 ml water mixture at 60 °C for 24 h



* Minimum requirement according to DIN 51517-3 CLP for gear oils

Formulation 1: Grease based on polyalkylene glycol and soap thickener

- Prepare 10% lithium hydroxide solution by dissolving monohydrated lithium hydroxide in water
- Pour 33% of PAG in GUEDU* mixer at RT
- Add 12-hydroxystearic acid and calcium hydroxide under stirring
- Heat to 75 $^\circ\text{C}$
- At 75 °C, slowly add lithium hydroxidewater solution under stirring
- Heat to 150–155 °C, add remaining PAG
- Heat to 160–165 °C and maintain at this temperature for 1 hour
- After one hour at 163 °C, turn off heat and let formulation cool down under stirring
- At 60 °C, remove formulation from GUEDU mixer and mill using a tricylinder system
- After milling, deaerate formulation in GUEDU mixer under agitation and vacuum

	Formula composition					
	NLGI 1	NLGI 2	NLGI 3			
Ingredient		Content [wt %]				
Polyalkylene glycol ISO VG 15, hydrophilic, modified	88.89	83.54	82.36			
Hydrostearic acid	9.84	14.57	15.62			
Monohydrated lithium hydroxide	0.80	1.18	1.27			
Calcium hydroxide	0.47	0.70	0.75			



Less softening and wear: Li/Ca polyalkylene glycol grease vs. Li/Ca PAO grease

BENEFITS OF THE PAG-BASED GREASE

- (>)High viscosity index of the oil
- Higher mechanical stability after worked penetration test: Li/Ca PAO grease goes from normal to soft consistency, devolving from NLGI class 2 (265–295) to class 1 (310–340)
- Smaller wear scar \geq



			Li/Ca polyalkylene (PAG) glycol grease	Li/Ca Polyalphaolefin (PAO) grease
	Properties	Method	Result	Result
OIL	Kinematic viscosity (40 °C) [mm/s ²]	ASTM D 7042	13	19
	Kinematic viscosity (100 °C) [mm/s ²]	ASTM D 7042	3.6	3.8
	Viscosity index	ASTM D 2270	210	127
	Pour point [°C]	ASTM D 7346-14	-72	-75
GREASE	NLGI grade	ASTM D 217	2	2
	Consistency (1/10 mm)	ASTM D 217	275	281
	Increase		+5.8%	+14.6%
	Worked penetration – 100,000 strokes (1/10 mm)	ASTM D 217	291	322
lycol	Drop point [°C]	ASTM D 566	186	190
	Oil separation (168 hours, 40 °C) [%]	ASTM D 1742	-2.3	-6.7
	Wear scar (400 N,1200 rpm,1 hour) [mm] *no temperature control	ASTM D 2266*	0.54	0.73

Polyalkylene glycol properties compared – Resulting pros and cons

	HYDROPHOBIC PAGs		HYDROPHILIC PAGs	HYDROPHILIC PAG MODIFIED	
	Low viscous	High viscous	Low viscous	High viscous	Low viscous
PROS	Excellent pour points	High thermo-oxidative stability	Biodegradable, suited for	High thermo-oxidative stability	Excellent pour point
	Water repellent, soluble in hydrophobic media	Suited for formulating H1 food-approved greases	formulating eco-labeled greases Excellent pour points	Excellent lubricity	Miscible both in water and some hydrophobic media
	Biodegradable, suited for	Water repellent		Biodegradable, suited for formulating eco-labeled greases	High viscosity index relative to molecular weight
	formulating eco-labeled greases	Good lubricity	Sood lubricity		
CONS	Low thermo-oxidative stability	Not suited for formulating	Low thermo-oxidative stability	Tendency for water physisorption	Low thermo-oxidative stability
Ę	Low lubrication properties	eco-labeled greases	Tendency for water physisorption	Absolutely immiscible with hydrophobic media	Low lubrication properties
	Not suited for formulating H1 food-approved greases		Absolutely immiscible with hydrophobic media	nydrophobic media	Not suited for formulating H1 food-approved greases
			Not suited for formulating H1 food-approved greases		

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what is precious to you?

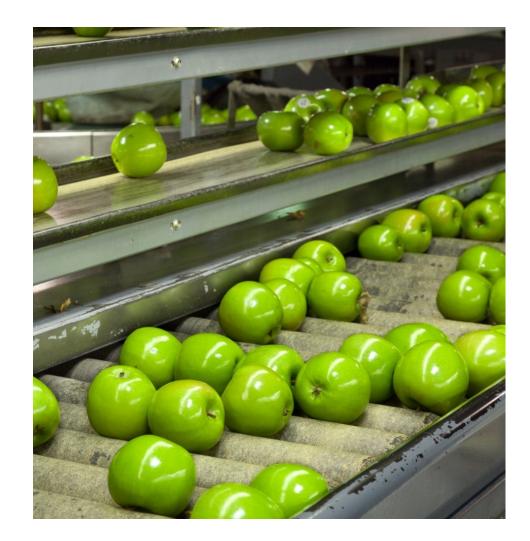
Fit for food – The Polyalkylene glycols are suited for H1 food grade greases

FDA 21CFR 178.3570¹ lists substances as lubricants with incidental food contact:

- Restriction for PAGs is a minimum molecular weight of 1,500 (appr. ISO VG 100)
- Addition to food may not exceed 10 parts per million

NSF's white book² confirms compliance with 178.3570 as lubricants with incidental food contact (H1) or lubricant ingredient with incidental food contact (HX-1)

Nearly all PAGs with ISO VG 100 or higher are NSF-approved and can be used for H1 food grease formulations



¹ http://www.accessdata.fda.gov/scripts/cdrh/cfdocs/cfcfr/CFRSearch.cfm?fr=178.3570

² http://info.nsf.org/usda/psnclistings.asp

Properties of soap thickeners for greases

	SOAP							
	SIMPLE				MIXED	COMPLEX		
Thickener	Ca	Na	Li	AI	Mixture of simple	Ca	Li	AI
Chemical structure		ОН	l _{o: u}	•	soaps			л. Ч.
Manufacturing process*	Chemical (typical temp	peratures ~ 120–160 °C)				Chemical (typical temperature ~ 200 °C)		
Suitable base oils	Mineral oil, PAO, PAG, silicone, esters, vegetable oils					Mineral oil, PAO, PAG, silicone, esters, vegetable oils		
Typical dropping point**	95–105 °C	160–180 °C	175–205 °C	110 °C		≥ 230 °C	≥ 250 °C	≥ 250 °C
Wet conditions	Yes	No	Yes	Medium		Yes	Yes	Yes
Special characteristics	Cost-effective, biodegradable, good water resistance	High dropping points but poor water tolerance	Very good structural properties	Reversible structure if drop point is exceeded		Very good structural properties, water tolerance	Very good walk penetration, good high-temperature characteristics	No hardening at higher temperatures
Cost	Low	Low	High	Medium/high		Medium/high	High	Medium/high
Typical applications	Simple applications and EALs	Electrical applications, gear grease	Multipurpose	Food, special applications, slide bearings, gears		Multipurpose	Multipurpose	Food, steel

Ca = calcium, Na = sodium, Li = lithium, Al = aluminum, PAO = polyalphaolefin, PAG = polyglycol, EALs = environmentally acceptable lubricants *W. J. Bart et al., Schmierfette: Zusammensetzung, Eigenschaften, Prüfung und Anwendung, Band 500, Expert Verlag, 2000 **High-Temperature Grease Guide (https://www.machinerylubrication.com/Read/340/high-temperature-grease); R. T. Vanderbilt Co.

Properties of non-soap thickeners for greases

	NON-SOAP							
	ORGANIC		INORGANIC		CA SULFONATES			
Thickener	Polyurea	PTFE	Silica gel	Bentonite	Ca sulfonates			
Chemical structure			H0	NA D	(Giasson, et al. (17); Authier and Herman (23)).			
Manufacturing process*	Chemical (addition reaction, 180–240 °C)	Physical (RT to < 100 °C)	Physical (RT to < 100 °C)	Physical (RT to < 100 °C)	Chemical (up to 140 °C)			
Suitable base oils	Mineral oil, PAO, PAG, silicone, esters, vegetable oils	PFAE	Mineral oil, PAO, PAG, silicone, esters, vegetable oils					
Typical dropping point**	≥ 245 °C	N/A	N/A		≥ 260 °C			
Wet conditions	Yes	Yes	Yes	Yes	Yes			
Special characteristics	Very high dropping points, excellent mechanical properties but tendency to harden		Good oxidation resistance, thermal resistance, chemically inert, flat temperature/consistency-gradient	Not suited for high bearing point velocities, moderate corrosion protection, not compatible with other types of greases	Inherent extreme pressure (EP) properties, excellent salt fog performance, good rust performance			
Cost	Very high	Very high	High	Medium/high	High			
Typical applications	Bearing grease, low-noise grease, sealed-for-life applications	For chemical inertness	Food, automotive	Food, high-temperature applications	Food, automotive, marine environments			

Ca = calcium, PAO = polyalphaolefin, PAG = polyglycol, PFAE = perfluoroalkylether, RT = room temperature

*W. J. Bart et al., Schmierfette: Zusammensetzung, Eigenschaften, Prüfung und Anwendung, Band 500, Expert Verlag, 2000

**High-Temperature Grease Guide (https://www.machinerylubrication.com/Read/340/high-temperature-grease); R. T. Vanderbilt Co.

Compatibility with internal coatings

Flender test (400 g synthetic oil at 130 °C, 7 days; addition of 5% water, 90 °C, 5 hours)

	INTERNAL COATING	BLISTERING DIN EN ISO 4628-2	PENDULUM HARDNESS RATIO DIN EN ISO 1522	CROSS CUT TEST [GT] OIL LOADED AREA DIN EN ISO 2409	
Requirement	M 20, P 22	≤ 1	>0.50	≤ 1	
Product					FINAL RATING
Hydrophobic** ISO VG 220	M 20	0*	1.07*	1**	Compatible
	P 22	0*	0.83*	0**	Excellently compatible
Hydrophilic** SO VG 220	M 20	0*	1.04*	0**	Excellently compatible
	P 22	0*	0.76*	0**	Excellently compatible
Hydrophilic** SO VG 220	M 20	0*	0.98*	0**	Excellently compatible
	P 22	0*	0.55*	0**	Excellently compatible



PAGs are compatible with Primer M 20 and P 22

^{x1/130} Cross-cut Hydrophilic, ISO VG 220 M20

2//³⁹ Cross-cut Hydrophilic, ISO VG 220 P22

> Back to material compatibility

* Average of two runs, for every tested oil each single run fulfills requirements, tests were conducted by Mäder Aqualack AG

** Contains additive package

Compatibility with sealings

Elastomer compatibility test (according to DIN ISO 1817, NBR 28/SX, 100 °C, 7 d)

	RELATIVE CHANGE IN VOLUME (ISO 2781)	CHANGE IN SHORE A HARDNESS (ISO 48, METHOD M)	DECREASE IN TENSILE STRENGTH (ISO 37)	DECREASE IN ELONGATION AT BREAK (ISO 37)
Minimum requirement CLP oil	max. 0/+10%	max10/+5	max. 30%	max. 40%
Product				
Hydrophobic* ISO VG 220	0.3%	1	5%	19%
Hydrophilic* ISO VG 220	3.9%	-5	5%	10%
Hydrophilic* ISO VG 220	4.3%	-3	0%	10%



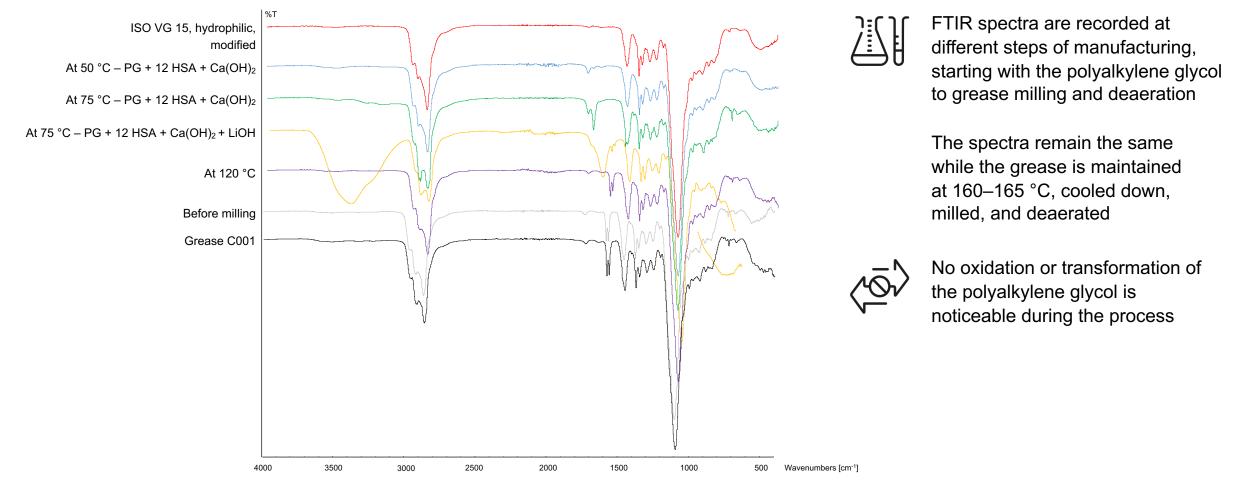
PAGs are compatible with NBR (acrylonitrile butadiene rubber)



Back to material compatibility

Source: Freudenberg Forschungsdienste SE & Co KG

Grease manufacturing steps monitored via FTIR: Li/Ca mixed grease based on an ISO VG 15 polyalkylene glycol



Formulation 2: Food-approved grease based on polyalkylene glycol ISO VG 220 (hydrophobic), silica thickener and **PTFE as additive**

- Pour 50% of ISO VG 220 (hydrophobic) in GUEDU* at RT
- Add silica (and PTFE) under stirring
- When silica (and PTFE) are fully mixed with the oil and no powder is observed, add remaining ISO VG 220 (hydrophobic)
- Stir for 30 minutes
- Remove product from GUEDU and mill using a tricylinder system
- After milling, return product to GUEDU and deaerate under agitation and vacuum

	Formula composition				Formula composition	
	polyalkylene glycol + Silica	polyalkylene glycol + Silica + PTFE			polyalkylene glycol + Silica	polyalkylene glycol + Silica + PTFE
Ingredient	Content (wt%)		Properties	Method	Result	Result
ISO VG 220 (hydrophobic)	80.0	75.5	NLGI grade	ASTM D 217	2	2
HDK 18 (pyrogenic silica)	20.0	22.0	Consistency (1/10 mm)	ASTM D 217	285	283
PTFE	_	2.5	Worked penetration – 100,000 strokes (1/10 mm)	ASTM D 217	285	
			Oil separation (168 hours, 40 °C) [%]	ASTM D 1742	0.82	
			Water wash-out		5 1	2.6

(4 g, 600 rpm, 79 °C) [%]





ASTM D 1264

5.1

2.6

Selection of base oils for study

BASE OIL	MINERAL OIL	ESTER	POLYALPHAOLEFIN (PAO)		Polyalkylene glycols	
Composition	Hydrotreated naphthenic oils: 40% (viscosity 110 mm²/s) 60% (viscosity 400 mm²/s)	Biodegradable esters: 66% (viscosity 145 mm²/s) 33% (viscosity 500 mm²/s)	17.5%	PAO (viscosity 31 mm²/s)	ISO VG 220 (hydrophobic)	
			71% Metallocene PAO (mPAO) (viscosity 614 mm²/s)		ISO VG 220 (hydrophilic)	
			12.5%	Alkylated Naphthalene (viscosity 29 mm ² /s)	ISO VG 220 (hydrophilic)	
Why?	Group III mineral oils are not available at required viscosity	Three esters from different suppliers were tested in terms	PAO was selected because of its high viscosity index		Polyalkylene glycols from existing range were used	
		of lubrication performance Mixture with best lubrication performance was selected	PAO is necessary to adjust viscosity Alkylated naphthalene was added to improve additive solubility		No formulation work necessary	
Viscosity	ISO VG 220	ISO VG 220	ISO VG	220	ISO VG 220	