MATERIALES
Exit The Ordinary

MATERIALES
Introduction to MPTB

ESTEC, November 29th 2023

Who we are

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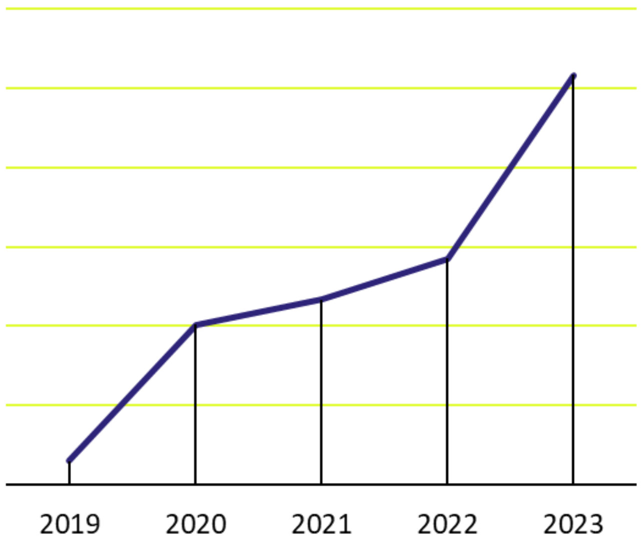
Materiales was founded in 2019 and is located in Hamburg, Germany

Currently we are a team of 9 people with 7 technical specialists



For 2023 a turnover of 1.03 M€ is forecasted

What we do

Consultancy

We are a consultant office, and support our customers as specialists and project managers

Testing

In our lab, we perform material tests, conduct customer specific experiments and develop individual formulations

Research & Development

In 2021 we started with the development of own materials

Our expertise

Our USP is profound expert knowledge at the interface of chemistry, material science and engineering

We understand materials – what they are made of, how they are manufactured and tested, how they perform and when they fail

Our expertise

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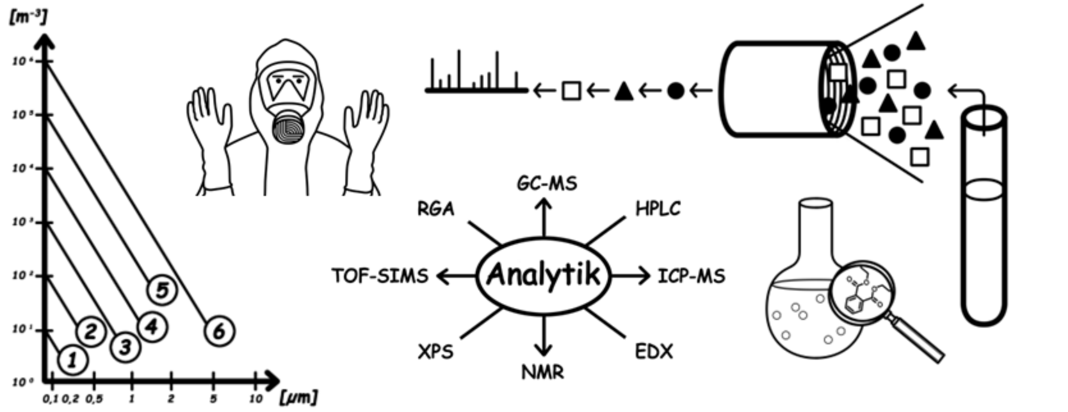
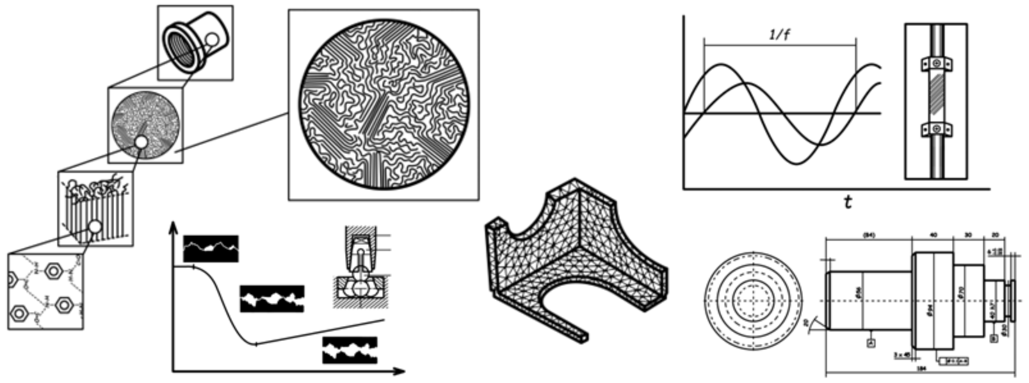
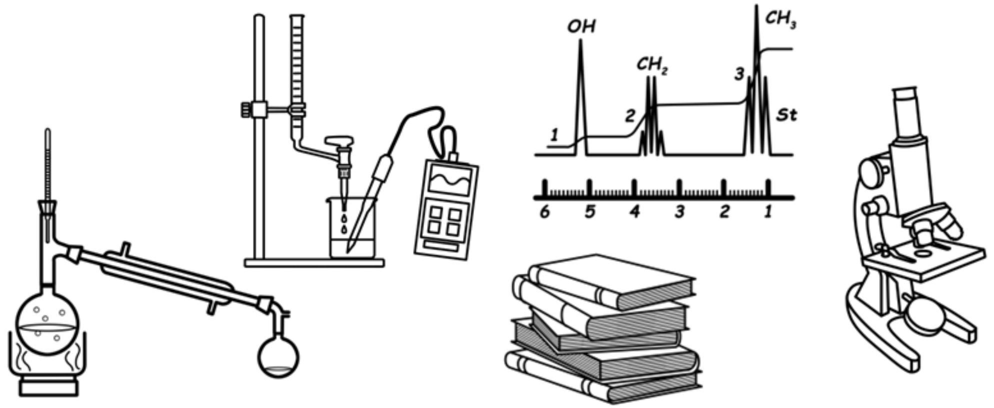


Chemical analytics and contamination control

Management of hazardous chemicals



Material science and engineering



General chemical consulting

Industries we serve

Our expertise is interdisciplinary and across industries. Our main focus is on high performance applications which demand tightly specified materials.

Industrial sectors of recent and current activities:

Semiconductor technology

Cleanroom manufacturing

Space tribology

Mechanical engineering

Chemical industry

Manufacturing

Current internal R&D activities

Lubricants

- Development and commercialization of PFPE-based vacuum lubricant with Long-Term-Storage properties
- Development of innovative (non-PFAS) vacuum lubricants based on Ionic Liquids

Elastomers

- Highly precise dynamic-mechanical material analysis, development of methods for material modeling and FEM simulation
- Development of PFAS-free and cleanroom vacuum compatible elastomers for damping application (proposal at BMBF pending)

Cleanroom materials

- Planning and establishment of a lab unit for assessment of materials cleanroom compatibility

Development of European Grease Lubricants Stabilized for Long-Term Storage

ESA contract No. 4000137211/21/NL/KML/rk

ESTEC, November 29th 2023

Agenda

- Project goals and programme of work
- Definition of requirements
  - Impact of LTS on grease lubricated spacecraft mechanisms
  - Specification of space lubricant requirements
  - Verification of LTS properties
- Formulation development
- Testing of preliminary final formulations
- Outlook

Project goals and programme of work

Project goal was the development of a grease formulation for application in space mechanisms, stabilized for Long-Term-Storage (LTS)

- Stability under cleanroom storage conditions for at least 15 years
- Suitability as vacuum grease for space application
- Demonstration of tribological performance
- „European-based" – all materials shall be from European sources
- Target TRL = 4 (functional laboratory prototype)

Project goals and programme of work

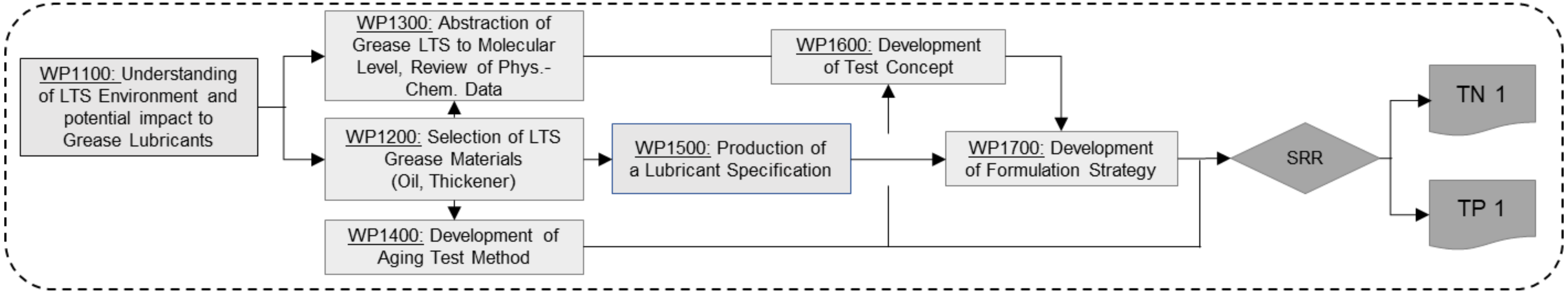
The project was scheduled for a duration of 24 months and conducted together with ESTL as subcontractor

Technical officer from ESTEC was Malgorzata Holynska. Technical support from ESTEC was provided by Theo Henry

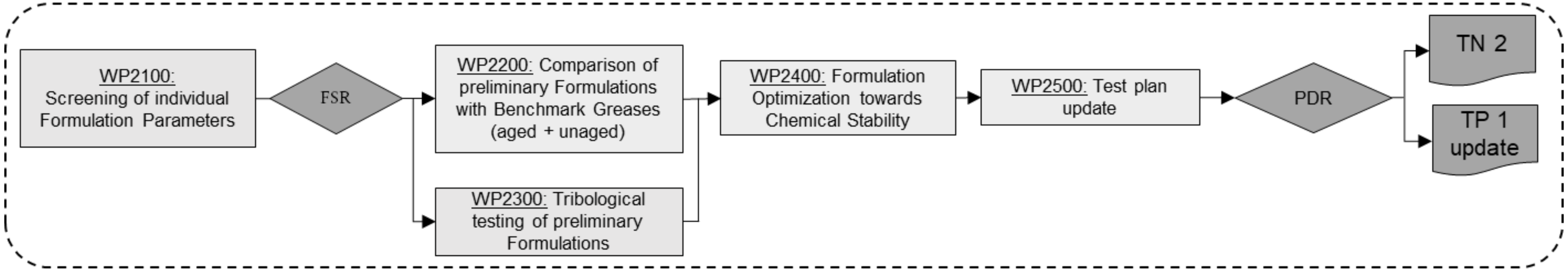
Project goals and programme of work

Project work was structured into three major work packages:

WP1000: Literature/market screening and definition of requirements



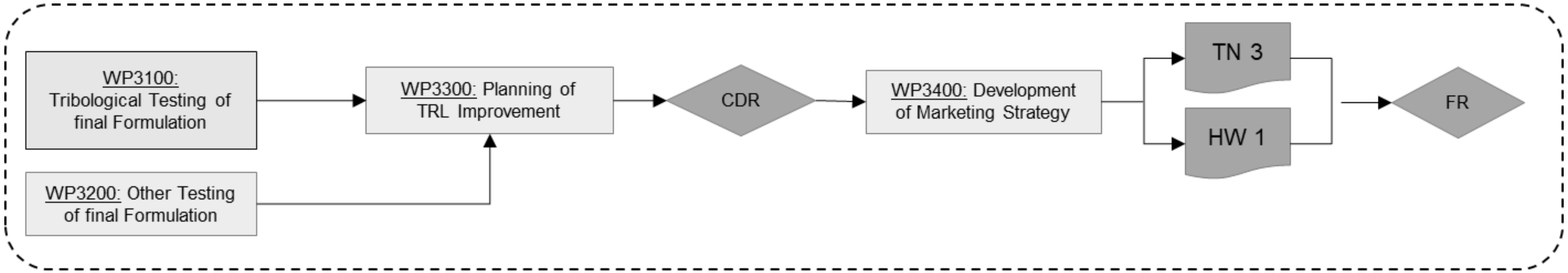
WP2000: Development and testing of preliminary formulations and choice of final formulation



Project goals and programme of work

Project work was structured into three major work packages:

WP3000: Full testing of final formulation



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Definition of requirements

Impact of LTS on grease lubricated spacecraft mechanisms

What is LTS?

- Possibility of storage under defined conditions over long time intervals
- Retainment of lubricant functionality after storage (tribological properties, outgassing)

Why is LTS important?

- Extended storage periods of lubricated components on-ground and during missions
- MTG: AIT time of 5 years, 8.5 years of orbit operation, 17.5 years of storage for recurring models[1]

Which lubricants do show proven LTS properties?

- So far, LTS properties could only be demonstrated for Braycote 601 EF[2,3]

1 Senese S et al.; "Meteosat Third Generation (MTG) Mechanisms: Impact on Instrument Performances"; 18th European Space Mechanisms and Tribology Symposium 2019
2 NASA; "Braycote Grease Retains Tribological Properties for 17 Years in Controlled Storage"; NASA Engineering and Safety Center Technical Bulletin No 07-01 2007
3 T. Henry et al.; "Investigation of Long-Term Storage of Space Materials for Future Constellation Missions: Study of Braycote 601 EF Lubricant"; IOP Conference Series: Materials Science and Engineering 2023

Impact of LTS on grease lubricated spacecraft mechanisms

Different phenomena can influence lubricant functionality in a space mechanism

- Oxidation
- Evaporation
- Chemical degradation
- Lubricant creep
- Phenolic cage losses
- Separation of greases

Impact of LTS on grease lubricated spacecraft mechanisms

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The described phenomena were assessed with regard to the likelihood to occur during LTS and the severity of an occurrence (1 = not likely, not severe to 5 = very likely, very severe).

Physical delamination/oil separation has been identified as the highest risk during LTS

Issue	Likelihood	Severity	Risk Rating
Oxidation (PFPE)	1	2	2
Oxidation (MAC)	1	2	2
Evaporation (PFPE)	1	4	4
Evaporation (MAC)	2	4	8
Chemical Degradation (PFPE)	2	3	6
Chemical Degradation (MAC)	1	3	3
Thermal Degradation (PFPE)	1	3	3
Thermal Degradation (MAC)	1	3	3
Lubricant Creep (PFPE)	2	3	6
Lubricant Creep (MAC)	1	3	3
Phenolic Cage Loss	3	2	6
Separation of Greases	5	2	10

Based on these initial considerations it was decided to develop the LTS grease based on PFPE oil

Verification of LTS properties

Since LTS is a long-term property, an approach was required for verification. Due to the selection of PFPE as base oil, only oil separation was expected as considerable risk. Therefore, an approach for the accelerated measurement of oil separation had to be developed.

- Accelerated aging in material science and engineering is often conducted by using time-temperature relation according to Arrhenius. „Rule of thumb“: Temperature increase by 10 °C increases aging by factor 2-3. Only valid, if a single process occurs at a time

$$A = A_0 \cdot e^{-\frac{Q}{RT}}$$

A = reaction rate
Q = activation energy
R = ideal gas constant
T = temperature

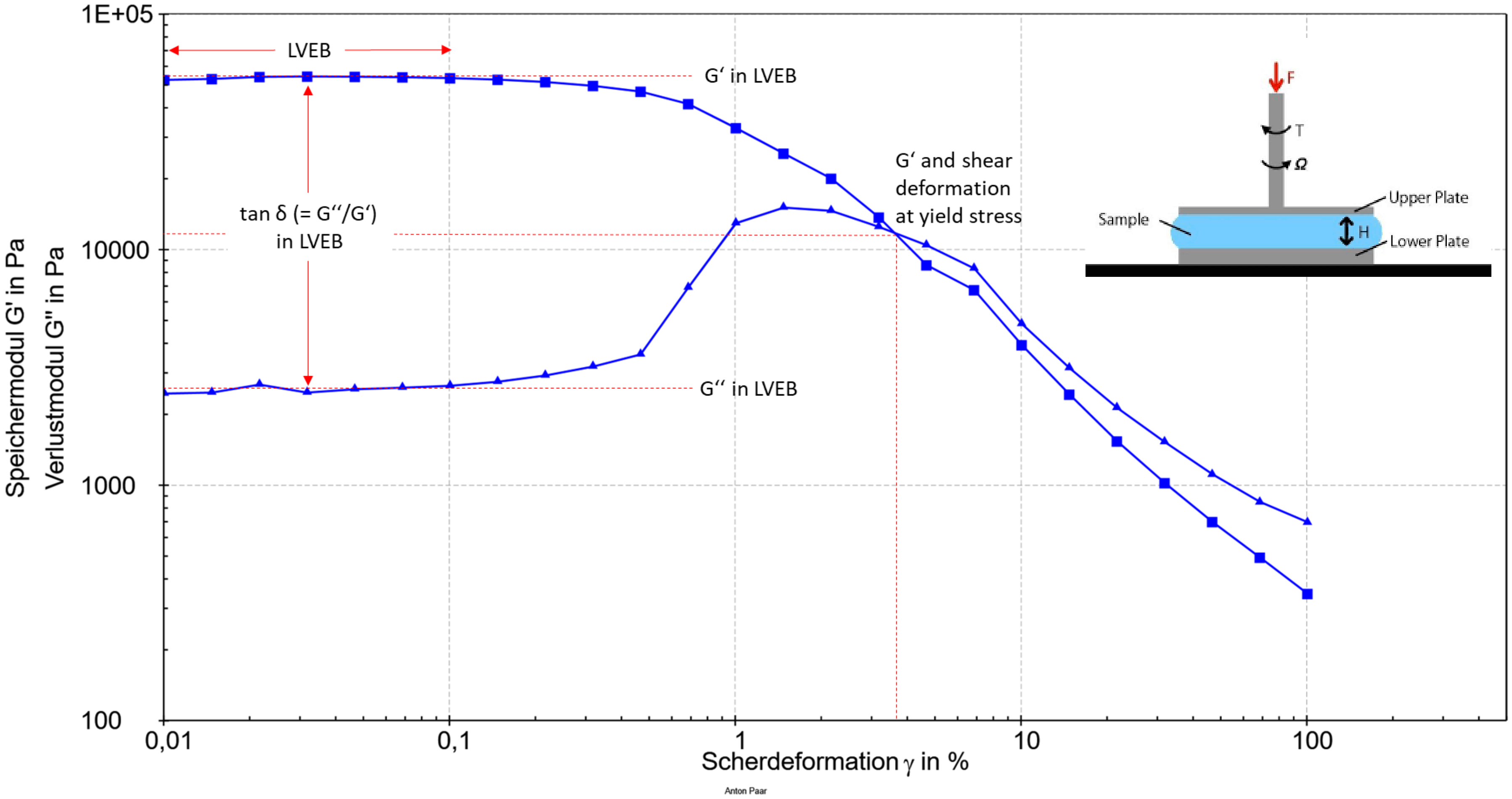
- Oil separation is driven by diffusion. If no further degradation processes occur, the Arrhenius approach can be applicable for an assessment of LTS by oil separation testing at high temperature

Verification of LTS properties

- Grease is regarded as porous filler network of particles that are filled with oil. If filler network gets destroyed by high temperatures, this is a competitive process to the pure diffusion
- Also: Oils show temperature dependent behavior of viscosity (indicated by viscosity index). It is expected that this is an additional impact that increases diffusion
- Accelerated aging for 22 days at 100 °C is „equivalent“ to 15 years at room temperature according to the Arrhenius „rule of thumb“. Due to an eventual impact to the filler network and the effect of viscosity index, it would actually be a worst case scenario with regard to oil separation

LTS properties were assessed by aging samples for 22 days at 100 °C. Samples were analyzed before aging. Aged samples were rehomogenized and analyzed again. Retainment of properties indicated adequate LTS behavior

Verification of LTS properties - Rheological amplitude sweep



Specification of space lubricant requirements

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A technical specification was produced to define requirements of the formulation to be developed in terms of suitability as a space lubricant. Therefore, guidelines recently produced by ESTL were applied[7]

Test No.	Test Category	Method Preferred	Description	Batch Verification	Lot Acceptance (Verification)	Validation	Qualification
1	BASE OIL TESTS	ASTM D4052	Density /specific gravity	X			
2		ASTM D97	Pour point	X			
3		ASTM D445	Viscosity	X			
4		ASTM D2270	Viscosity index	X			
5		FEDSTD79D 3009.3	Particle count	X	X		
6		--	Molecular weight distribution	X			
7		ESA-DOK	Vapour pressure	X		X	
8	GENERAL LUBRICANT TEST REQUIREMENTS	--	Component and process	X			
9		REACH Reg 1907/2006	Health and safety	X			
10		--	Life and storage	X			
11		--	Solvent and chemical compatibility	X			
12		--	Appearance	X	X		
13		--	FTIR	X	X		
14		ASTM D972	Evaporation	X	X		
15		ECSS-Q-ST-70-02	Outgassing	X			
16		ESA-DOK	Vapour pressure			X	
17		--	Other application properties			X	
18		PRA-ESTL-ED-0017	Spiral orbit tribometry	X		X	
19		ECSS-E-ST-33-01C	Component level tests			X	
20	TESTS APPLICABLE TO GREASE ONLY	ASTM D217	Grease penetration	X	X		
21		ASTM D2265	Dropping point	X			
22		ASTM D-6184	Oil separation	X			
23		FEDSTD791D 3005.4	Particulate contamination	X	X		
24		--	Particulate additive information	X			
MECHANISM, INSTRUMENT OR SYSTEM LEVEL QUALIFICATION TESTS							
25		ECSS-E-ST-33-01C	Mechanism qualification tests				X

7 A. Kent et al., "Guidelines for Qualification of New Fluid Lubricants", Conference paper ESMATS 2023.
8 ESA-ESTL-TM-0375 01: Guidelines for Qualification of New Fluid Lubricants , ESTL 2023

Specification of space lubricant requirements

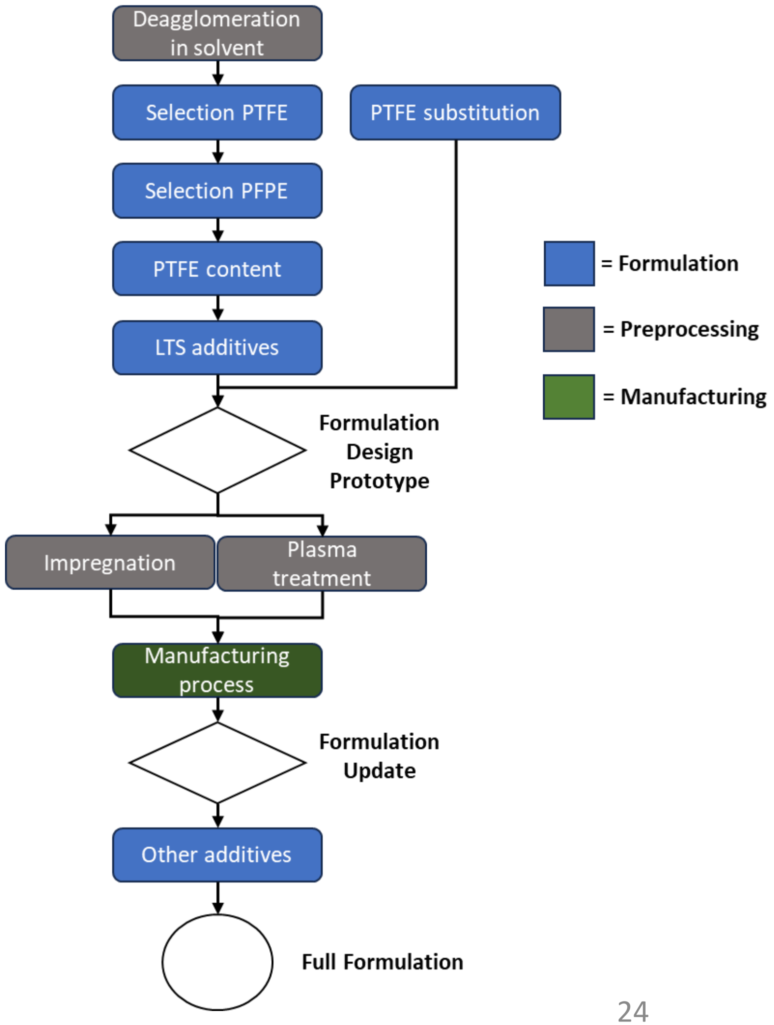
Technical datasheets (TDS) from commercial greases were reviewed, and a typical range of values was defined or limits were determined by standards (e.g. outgassing). Values were more guideline than strict limit, since even TDS values of commercial greases could differ from individual batch certificates (COA).

Parameter	Value/Units
Composition	Known components (or component chemistries) with existing evidence for success shall be preferred (e.g., PFPE & MAC oils, PTFE thickeners)
Apparent Viscosity (Grease)	ca. 40Pa.s @ 20°C 10 s^{-1} ca. 5 Pa.s @ 20°C 100 s^{-1}
Kinematic Viscosity (Base Oil)	100 – 400cSt @ 20°C ; 80 – 200cSt @ 40°C ; 20 – 70cSt @ 100°C
Viscosity Index (Base Oil)	>130
Oil separation	< 10%
Pour Point (Base Oil)	<-50°C
	<-70°C
Evaporation ASTM D972	<1% wt.
Particle Contamination	compulsory: <1000 particles (25 - 74 microns) ; 0 particles 75 micron or larger ideally: <150 particles (25 - 74 microns) ; 0 particles 35 micron or larger
FTIR	chemical spectrum from 4000 to 700 cm^{-1}
Operational temperature range	compulsory: -40 to +100°C, ideally: -60 to +120°C
Max temperature	Thermally stable to 20°C above maximum operating temperature, ideally stable to 300 °C
Thermal cycling	No change in properties after thermal cycling
Outgassing properties (TML, RML, CVCM)	compulsory. TML (%) <1 ; RML (%) <1 ; CVCM (%) < 0.1 ideally: CVCM (%) <0.02
Evaporative losses / vapour pressure (base oil)	compulsory: 4 x 10^{-11} mbar @ 20°C ; 5 x 10^{-8} mbar @ 100°C ideally: 4 x 10^{-13} mbar @ 20°C ; 5 x 10^{-9} mbar @ 100°C
Tribological performance – SOT friction	Comparable to existing grease lubricants 0.1 - 0.15 under standard SOT test conditions (2.25GPa Peak, vacuum)

Formulation development

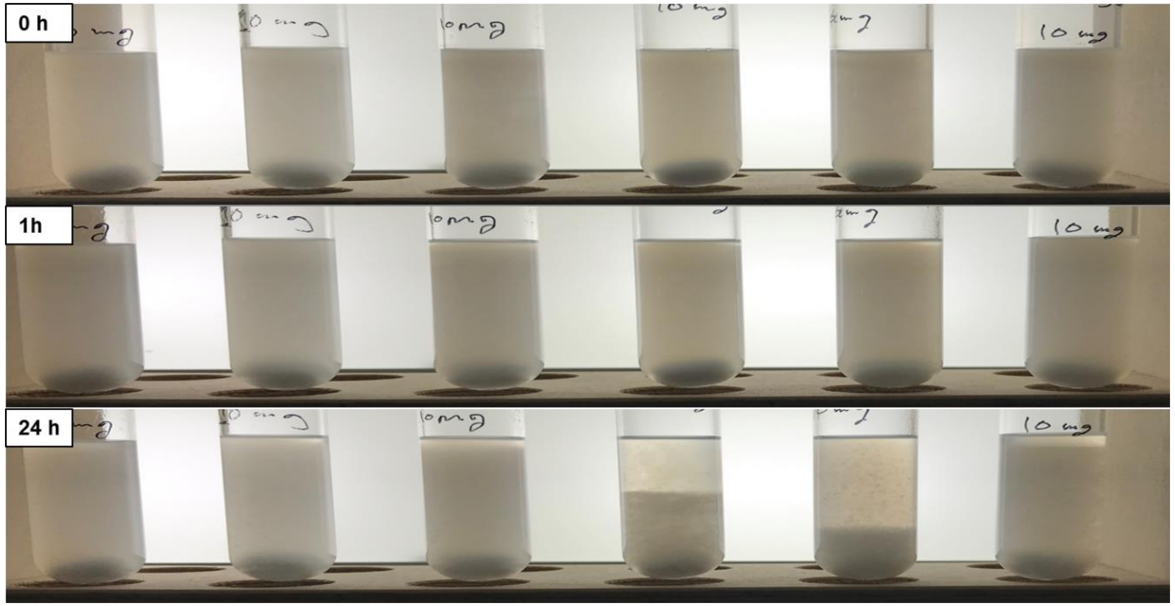
Formulation development

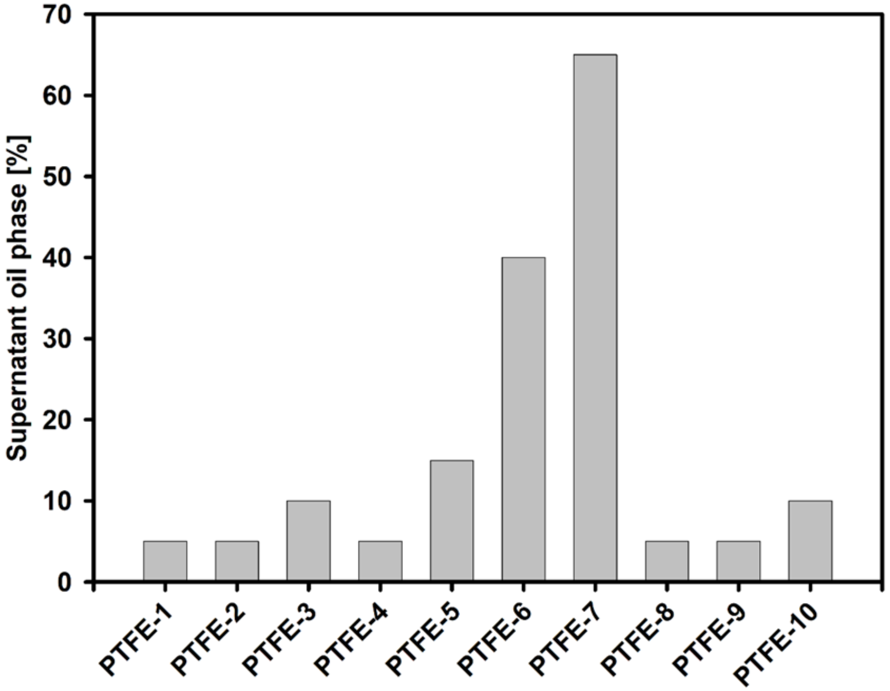
- A concept was worked out for the screening of individual parameters and a sequential formulation development
- Baseline desicion: The grease shall be from PFPE base oil and from PTFE thickener. Further additives depend on test results
- Formulation components
  - Base oil / Base oil mixture
  - Thickener
  - „LTS additives“ – components that reduce oil separation
  - „Other additives“ – to increase resistance against degradation
- Main methods for assessment
  - Oil separation test according to ASTM D 6184 (204 °C/30 h or 100 °C/22 d)
  - Rheological analysis (amplitude sweep and measurement of viscosity)
  - Thermogravimetric analysis



Formulation development - PTFE type

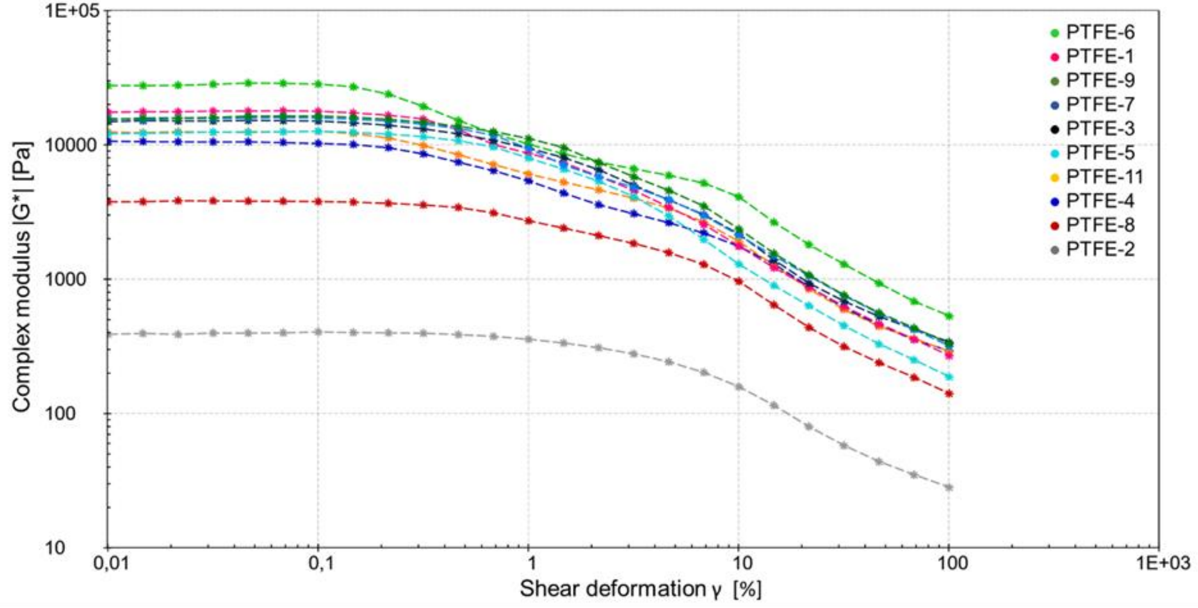
- Various PTFE micropowders were supplied
- First assessment: „Snowglobe“ test for dispersibility and stability in dispersion. Ultrasound assisted dispersion of small powder amount in PFPE oil (Fomblin M15). Observation of settling behavior

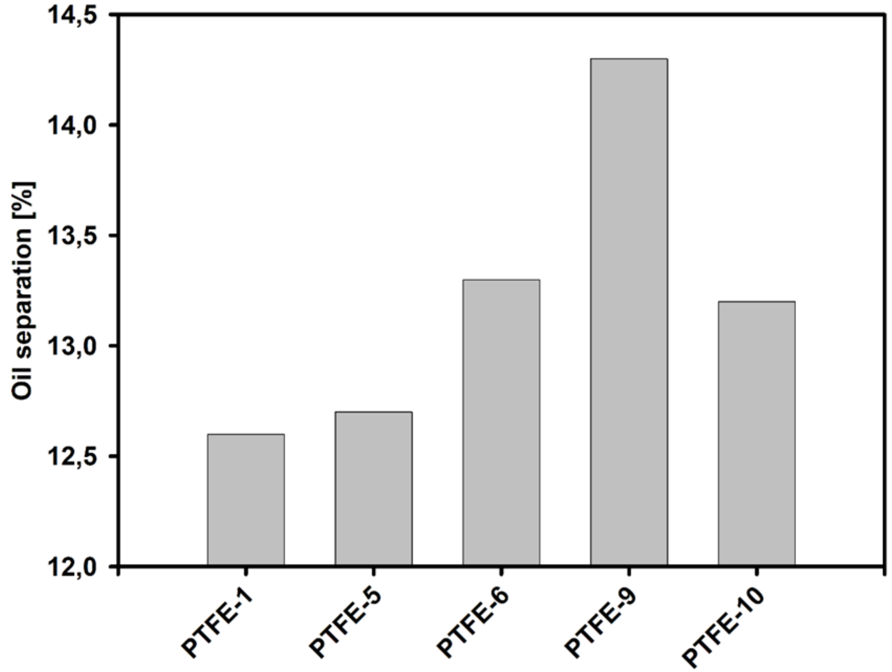




Formulation development - PTFE type

- Second assessment: Rolling of greases (25% solid content), rheological amplitude sweep and oil separation test at 204 °C/30 h ; base oil was Fomblin M15
- Not all powders yielded stable greases
- Best in class: PTFE-1





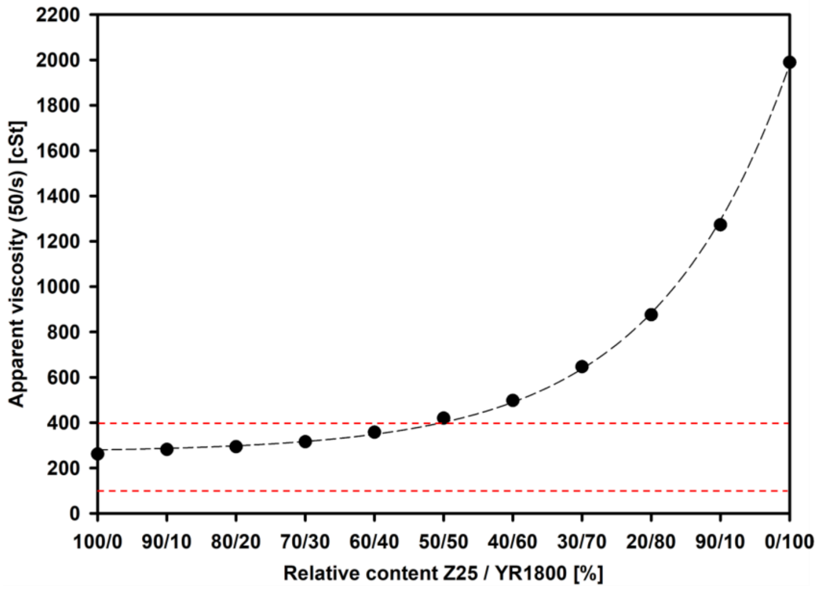
Formulation development - PFPE oil

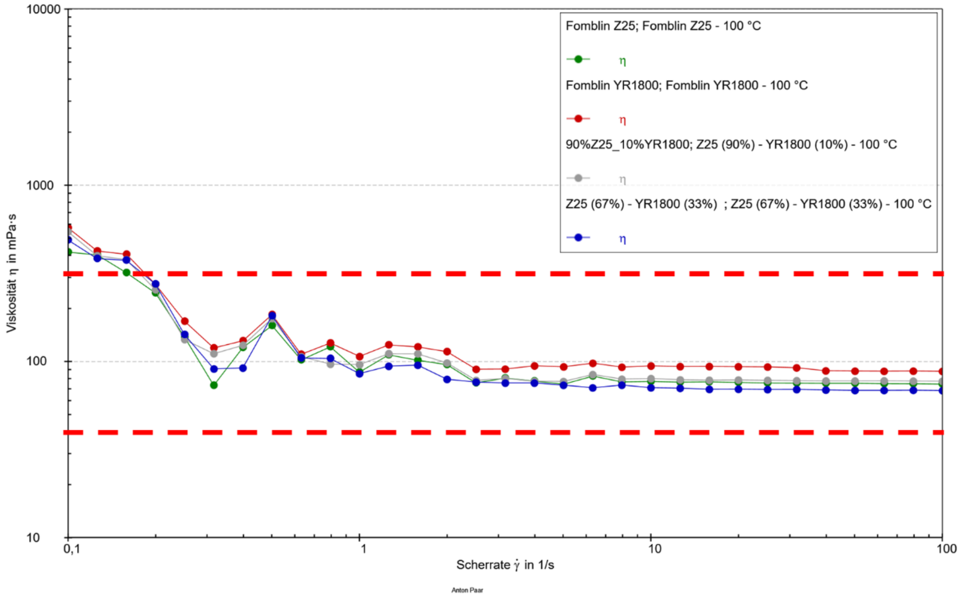
- Supplier data for ca. 80 PFPE oils was reviewed on basis of the developed specification. Finally, 4 oils were chosen for the development
  - Base oil/major oil component: Fomblin Z25 (space heritage), Fomblin M15 (low cost, only considered for testing)
  - (Minor) oil blend components: Fomblin YR1800 (viscosity increase), Krytox 1525 (surface tension close to PTFE)

PFPE oil	Fomblin M15	Fomblin Z25	Fomblin YR1800	Krytox 1525	Grease specification
Supplier	Solvay	Solvay	Solvay	Castrol	European
Headquarters	Belgium	Belgium	Belgium	USA	European
Kinematic viscosity	150 cSt @ 20°C 85 cSt @ 40°C 22 cSt @ 100°C	223 cSt @ 20°C 133 cSt @ 40°C 41 cSt @ 100°C	1850 cSt @ 20°C 510 cSt @ 40°C 47 cSt @ 100°C	250 cSt @ 20°C 52 cSt @ 40°C 10.6 cSt @ 100°C	100 – 400 cSt @ 20 °C 80 – 200 cSt @ 40 °C 20 – 70 cSt @ 100 °C
Viscosity index	286	350	148	115	>130
Vapor loss	0.8 % @149°C/ 22h	0.4 % @204°C/ 22h	0.5 % @204°C/ 22h	0,6 % @149 °C/ 22h	< 1% @ 125 °C / 24 h
Surface tension	24 dyn/cm @ 20°C	25 dyn/cm @ 20°C	24 dyn/cm @20°C	19 dyn/cm @ 20°C	
Remarks	Low cost alternative to Z25 – mostly used for screening (WP2100)	Widely used oil that meets all requirements	Use as additive to increase viscosity	Use as additive to reduce surface tension	Additional requirement: Pour point < - 50 °C

Formulation development - PFPE oil

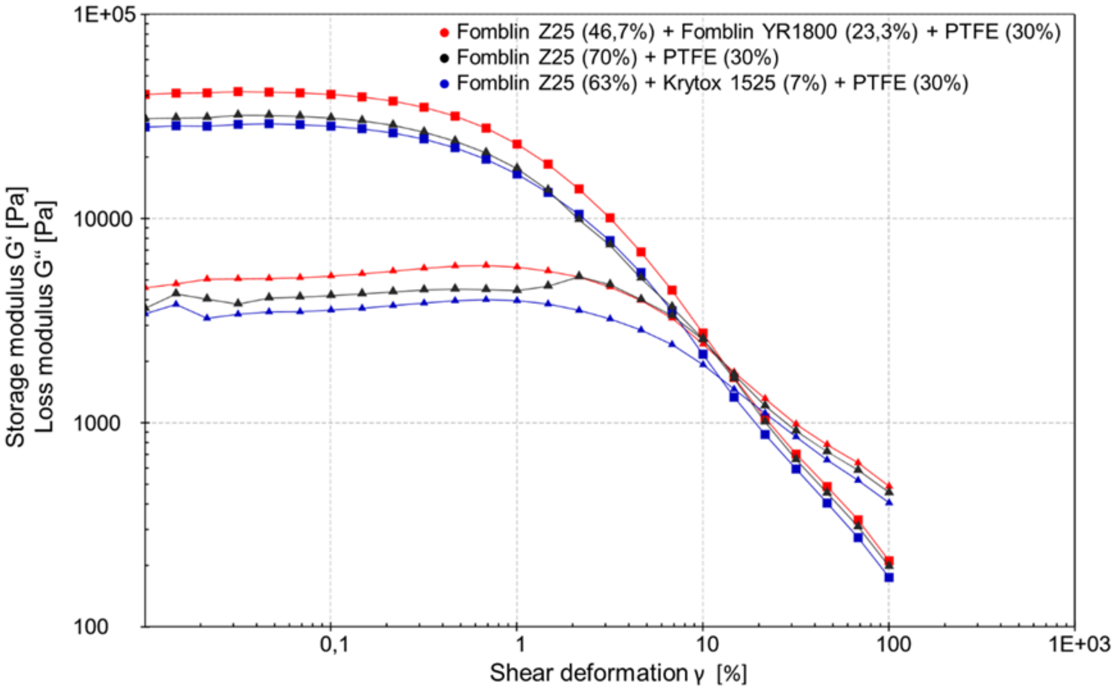
- Basic considerations of blending: 1) Oil shall have maximum possible viscosity due to less mobility/ diffusion ; 2) Oil surface tension shall be as close as possible to PTFE for high compatibility – lower tendency to separate
- Viscosity of blends from Fomblin Z25 and Fomblin YR1800 at 20 °C was analyzed. A ratio of 67:33 (Z25:YR1800) was found to be well within specification limits.

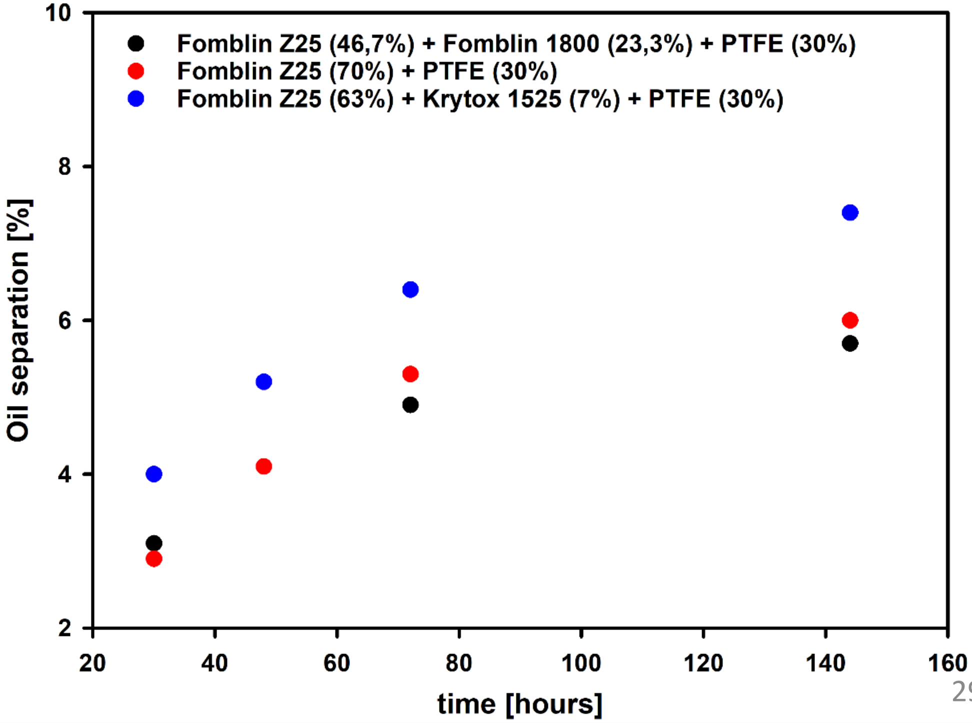




Formulation development - PFPE oil

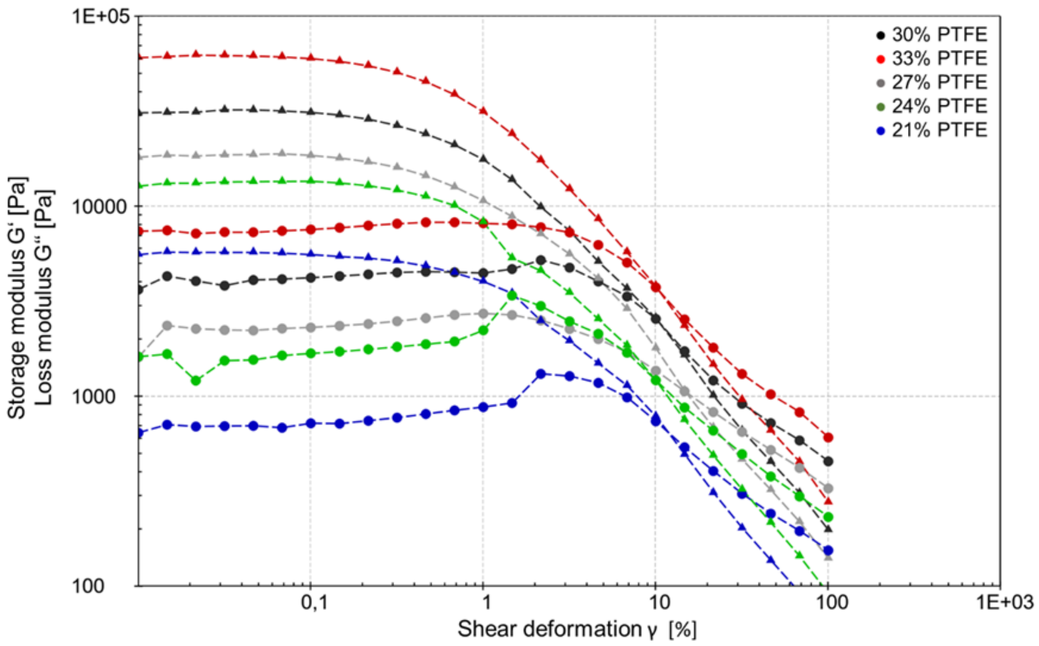
- Greases with 30% PTFE content were produced with oil blends. Rheological amplitude sweeps and oil separation (100 °C, 22 d) were performed
- No beneficial effect of Krytox 1525 was found. The blend from Z25/YR1800 performed better than pure Z25. Decision to proceed with base oil Z25/YR1800 67:33

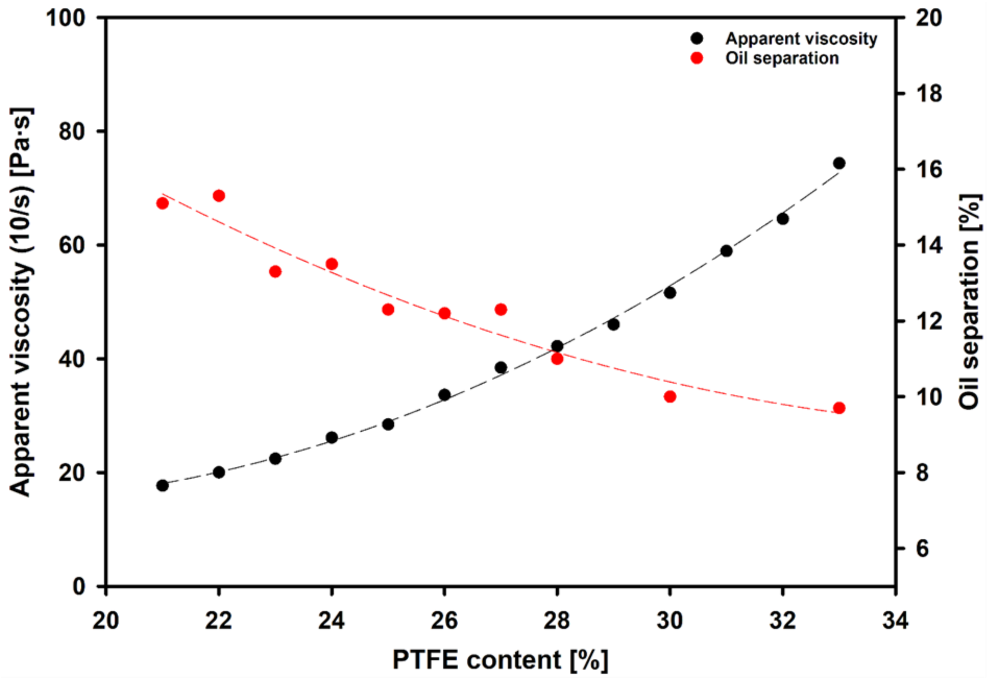




Formulation development - PTFE content

- Greases with Fomblin Z25 at different PTFE contents were produced. Analysis with rheological amplitude sweeps and oil separation tests (204 °C, 30 h)
- At PTFE concentrations >30% grease viscosity was higher than found for Braycote 601 EF. Decision to proceed with PTFE at concentration of 30%

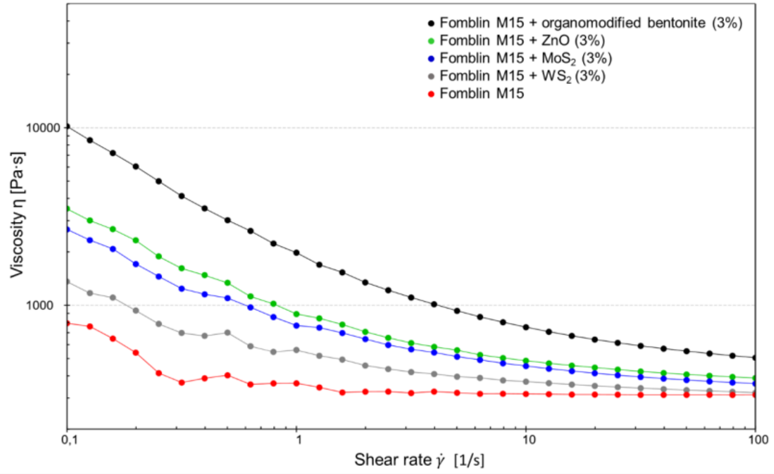


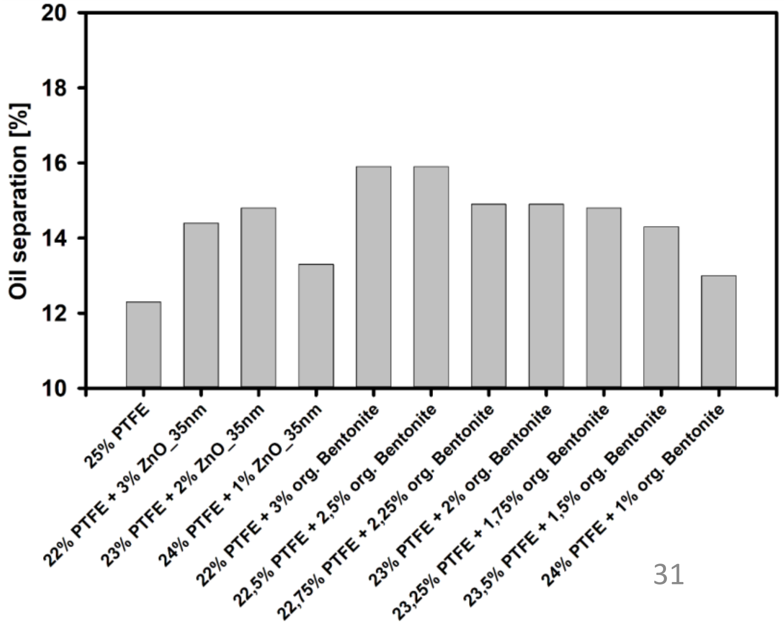


Formulation development - LTS additives

- Different inorganic nanoparticles and an organomodified bentonite were tested for their potential to further reduce oil separation
- Most nanoparticles did not lead to thickening effect, only ZnO and organomodified bentonite increased oil viscosity at moderate concentration of 3%
- It was found that a partial substitution of PTFE against LTS additive increased oil separation

Decision not to proceed with „LTS additives“ for improvement of LTS – organomodified bentonite with positive impact on tribology

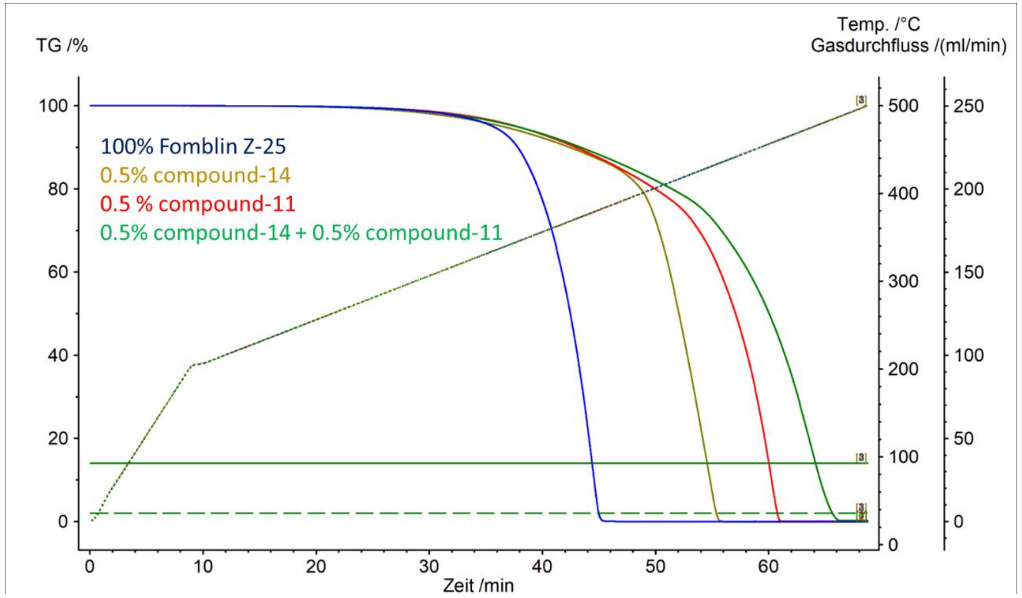




Formulation development - Other additives

- Different compounds were tested for their potential to stabilize PFPE against degradation. Assessment was via thermogravimetry in alignment with a previous paper[8]
- It was found that typical additive stabilizers lead to an earlier beginning of composition at elevated temperature, but delay the process in the following
- Combination of antioxidant compound-11 and metal scavenger compound-14 showed synergetic effect
- Both compounds with no or high melting point – no adverse impact on outgassing expected

Decision to include additives for improvement of chemical stability.



8 Lu, W. et al; "Catalytic Decomposition of Perfluoropolyether Lubricants" ; IEEE Transactions on Magnetics 2003

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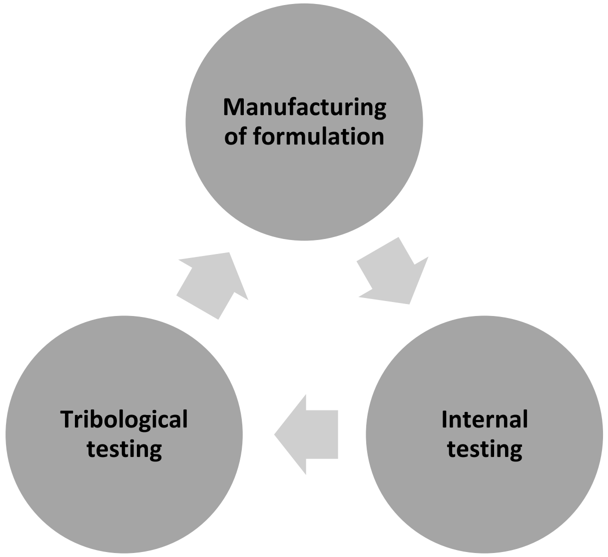
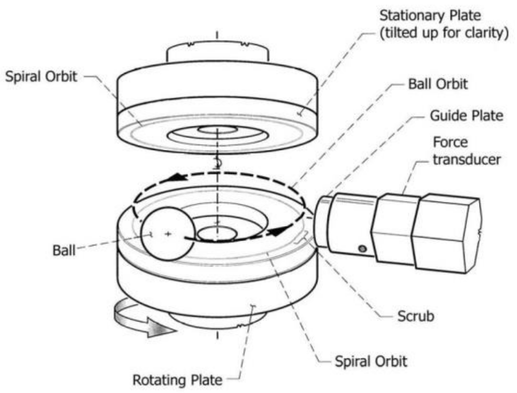
Testing of preliminary and final formulations

Preliminary formulations

- Development of final formulation in an iterative cycle for gradual improvement of properties
- Internal testing:
  - Artificial aging and testing of properties before/after
  - Oil separation
  - Rheology: Amplitudes sweep, viscosity and viscosity index
  - Thermogravimetry: Water content, thermal stability
- External testing:
  - Tribological SOT testing at ESTL in vacuum
  - Selected cases: Outgassing at DLR





Preliminary formulations

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Grease manufacturing consisted of the following basic steps

- Dispersion of non-thickening additives in the PFPE base oil by ultrasonication
- Homogenization of PFPE and thickening components/additives
- Rolling on ointment mill

Preliminary formulations

Grease	Tribology and Outgassing				Oil Separation		Rheology						TGA					
	SOT (orbits/μg)	Outgassing (%)			100 °C / 22 d	204 °C /30h	Amplitude sweep (25 °C)				Viscosity (20 °C)							
		TML	RML	CVCM			LVEB		Yield point		10/s [Pas]	100/s [Pas]	Water content [%]	T (2% mass loss N_2) [°C]	T (2% mass loss air) [°C]	Mass loss (55 min. N_2) [%]	Mass loss (190 min air) [%]	Residual mass (N_2) [%]
							tan δ	G' [Pa]	γ [%]	G' [Pa]								
A (unaged)	81	-	-	-	12.3	12.5	0.13	14382	13.0	952	27.93	5.19	0.03	325.2	307.7	74.82	82.24	-0.26
A (aged)	-	-	-	-	13.2	13.0	0.136	13766	9.8	1279	29.54	5.45	< 0.01	334.3	339.2	74.23	79.48	0.19
B (unaged)	103	-	-	-	8.6	10.6	0.134	38800	13.5	1619	52.36	8.86	< 0.01	326.7	313.6	70.14	76.90	0.36
B (aged)	-	-	-	-	8.4	10.8	0.14	28109	12.0	1761	48.99	8.69	< 0.01	330.5	344.9	66.96	75.98	0.11
C (unaged)	112	-	-	-	9.1	11.2	0.117	27922	4.3	2987	39.26	6.59	< 0.01	306.7	313.7	76.99	96.28	1.99
C (aged)	-	-	-	-	10.5	12.1	0.133	31216	9.9	1426	40.71	6.82	< 0.01	310.8	317.4	76.64	98.38	1.82
D (unaged)	133,5	-	-	-	7.8	10.4	0.124	34790	9.2	1818	45.53	7.41	< 0.01	323.3	304.6	73.28	98.50	1.58
D (aged)	-	-	-	-	9.4	11.3	0.13	30093	9.9	1708	44.74	7.30	< 0.01	326.5	317.3	74.61	93.33	1.19
E (unaged)	142	-	-	-	8.3	10.9	0.127	52277	7.0	2722	49.97	8.25	< 0.01	334.4	324.3	73.45	96.18	1.58
E (aged)	-	-	-	-	9.6	11.1	0.138	27864	3.0	4526	50.43	8.39	< 0.01	317.3	318.9	74.98	95.20	1.01
F (unaged)	104	-	-	-	8.3	9.4	0.131	47513	0.9	10301	62.45	9.46	< 0.01	309.6	330.5	63.25	77.74	1.72
F (aged)	-	-	-	-	9.0	8.8	0.131	51858	12.5	1580	54.56	8.87	< 0.01	318.6	323.5	66.21	78.51	0.98
G (unaged)	137	0.22	0.17	0.07	8.4	10.9	0.121	35922	1.8	6303	45.81	7.46	< 0.01	328.2	319.4	68.03	83.64	2.63
G (aged)	198	-	-	-	9.5	11.7	0.134	36851	10.9	1560	46.56	7.55	< 0.01	325.1	317.9	71.44	83.77	2.50
H (unaged)	133,5	-	-	-	9.0	11.3	0.122	35408	10.8	1751	51.37	7.99	< 0.01	325.5	331.2	72.20	84.02	2.12
H (aged)	-	-	-	-	9.3	11.7	0.13	41779	12.3	1484	47.51	7.57	< 0.01	318.1	318.1	73.23	86.95	1.80
I (unaged)	94	-	-	-	8.0	10.1	0.12	47963	14.7	2142	73.55	10.42	< 0.01	328.3	332.6	69.13	74.04	4.88
I (aged)	-	-	-	-	8.1	9.9	0.13	48962	18.3	1578	74.40	10.49	< 0.01	331.0	320.4	69.37	75.29	5.12
J (unaged)	264	0.16	0,12	0.03	7.1	8.0	0.133	46827	9.48	1984	53.64	9.05	0.03	332.5	312.4	71.46	86.74	1.51
J (aged)	-	-	-	-	8.1	9.5	0.128	50007	8.93	2684	64.20	10.43	< 0.01	317.0	322.5	71.55	87.14	1.72
K (unaged)	241	-	-	-	5.8	7.7	0.131	52496	9.54	2376	63.24	10.38	< 0.01	329.1	335.9	71.01	79.99	0.90
K (aged)	-	-	-	-	7.2	8.6	0.135	41530	11.3	1998	63.40	10.39	< 0.01	338.0	336.0	65.64	79.66	1.58
L (unaged)	-	0.15	0.12	0.03	6.8	8.1	0.124	52890	8.15	2945	62.17	10.03	< 0.01	338.6	346.5	70.09	79.42	2.17
L (aged)	-	-	-	-	7.4	8.6	0.13	52861	8.68	2679	63.97	10.41	< 0.01	335.3	332.2	70.05	79.55	1.93
B[1] unaged	133 (Ref. ESTL)	-	-	-	6.0	12.4	0.062	21452	14.4	1794	55.44	8.45	< 0.01	369.4	368.5	76.06	86.63	1.75
B[1] aged		-	-	-	7.6	-	-	-	-	-	-	-	< 0.01	374.7	361.0	73.97	85.99	1.51
B[2] unaged		-	-	-	-	11.0	0.048	31545	19.2	1314	60.10	8.73	0.01	351.5	350.9	76.85	87.74	1.36
B[2] aged		-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
BM[3]unaged	-	-	-	-	5.4	12.6	0.066	37412	10.3	4208	89.104	12.41	< 0.01	335.6	335.0	76.21	84.31	1.38
BM[3]aged	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-

[1]Braycote 601 EF, 1st batch, [2]Braycote 601 EF, 2nd batch, [3]Braycote 601 Micronic

Preliminary formulations

Beneficial for tribological lifetime:

- More PTFE
- Use of organomodified bentonite
- Use of additives

Grease	Tribology and Outgassing				Oil Separation		Rheology						TGA					
	SOT (orbits/µg)	Outgassing (%)			100 °C / 22 d	204 °C /30h	Amplitude sweep (25 °C)				Viscosity (20 °C)							
		TML	RML	CVCM			LVEB		Yield point		10/s [Pas]	100/s [Pas]	Water content [%]	T (2% mass loss N_2) [°C]	T (2% mass loss air) [°C]	Mass loss (55 min. N_2) [%]	Mass loss (190 min air) [%]	Residual mass (N_2) [%]
							tan δ	G' [Pa]	γ [%]	G' [Pa]								
A (unaged)	81	-	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	5.19	0.03	325.2	307.7	74.82	82.24	-0.26
A (aged)	-	-	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	5.45	< 0.01	334.3	339.2	74.23	79.48	0.19
B (unaged)	103	-	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	8.86	< 0.01	326.7	313.6	70.14	76.90	0.36
B (aged)	-	-	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	8.69	< 0.01	330.5	344.9	66.96	75.98	0.11
C (unaged)	112	-	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	6.59	< 0.01	306.7	313.7	76.99	96.28	1.99
C (aged)	-	-	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	6.82	< 0.01	310.8	317.4	76.64	98.38	1.82
D (unaged)	133,5	-	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	7.41	< 0.01	323.3	304.6	73.28	98.50	1.58
D (aged)	-	-	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	7.30	< 0.01	326.5	317.3	74.61	93.33	1.19
E (unaged)	142	-	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	8.25	< 0.01	334.4	324.3	73.45	96.18	1.58
E (aged)	-	-	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	8.39	< 0.01	317.3	318.9	74.98	95.20	1.01
F (unaged)	104	-	-	-	8.3	9.4	0.131	47513	0.9	10301	62.45	9.46	< 0.01	309.6	330.5	63.25	77.74	1.72
F (aged)	-	-	-	-	9.0	8.8	0.131	51858	12.5	1580	54.56	8.87	< 0.01	318.6	323.5	66.21	78.51	0.98
G (unaged)	137	0.22	0.17	0.07	8.4	10.9	0.121	35922	1.8	6303	45.81	7.46	< 0.01	328.2	319.4	68.03	83.64	2.63
G (aged)	198	-	-	-	9.5	11.7	0.134	36851	10.9	1560	46.56	7.55	< 0.01	325.1	317.9	71.44	83.77	2.50
H (unaged)	133,5	-	-	-	9.0	11.3	0.122	35408	10.8	1751	51.37	7.99	< 0.01	325.5	331.2	72.20	84.02	2.12
H (aged)	-	-	-	-	9.3	11.7	0.13	41779	12.3	1484	47.51	7.57	< 0.01	318.1	318.1	73.23	86.95	1.80
I (unaged)	94	-	-	-	8.0	10.1	0.12	47963	14.7	2142	73.55	10.42	< 0.01	328.3	332.6	69.13	74.04	4.88
I (aged)	-	-	-	-	8.1	9.9	0.13	48962	18.3	1578	74.40	10.49	< 0.01	331.0	320.4	69.37	75.29	5.12
J (unaged)	264	0.16	0,12	0.03	7.1	8.0	0.133	46827	9.48	1984	53.64	9.05	0.03	332.5	312.4	71.46	86.74	1.51
J (aged)	-	-	-	-	8.1	9.5	0.128	50007	8.93	2684	64.20	10.43	< 0.01	317.0	322.5	71.55	87.14	1.72
K (unaged)	241	-	-	-	5.8	7.7	0.131	52496	9.54	2376	63.24	10.38	< 0.01	329.1	335.9	71.01	79.99	0.90
K (aged)	-	-	-	-	7.2	8.6	0.135	41530	11.3	1998	63.40	10.39	< 0.01	338.0	336.0	65.64	79.66	1.58
L (unaged)	-	0.15	0.12	0.03	6.8	8.1	0.124	52890	8.15	2945	62.17	10.03	< 0.01	338.6	346.5	70.09	79.42	2.17
L (aged)	-	-	-	-	7.4	8.6	0.13	52861	8.68	2679	63.97	10.41	< 0.01	335.3	332.2	70.05	79.55	1.93
B[1] unaged	133 (Ref. ESTL)	-	-	-	6.0	12.4	0.062	21452	14.4	1794	55.44	8.45	< 0.01	369.4	368.5	76.06	86.63	1.75
B[1] aged		-	-	-	7.6	-	-	-	-	-	-	-	< 0.01	374.7	361.0	73.97	85.99	1.51
B[2] unaged		-	-	-	-	11.0	0.048	31545	19.2	1314	60.10	8.73	0.01	351.5	350.9	76.85	87.74	1.36
B[2] aged		-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
BM[3]unaged	-	-	-	-	5.4	12.6	0.066	37412	10.3	4208	89.104	12.41	< 0.01	335.6	335.0	76.21	84.31	1.38
BM[3]aged	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-

[1]Braycote 601 EF, 1st batch, [2]Braycote 601 EF, 2nd batch, [3]Braycote 601 Micronic

Preliminary formulations

Grease	Tribology and Outgassing				Oil Separation		Rheology						TGA					
	SOT (orbits/µg)	Outgassing (%)			100 °C / 22 d	204 °C /30h	Amplitude sweep (25 °C)				Viscosity (20 °C)							
		TML	RML	CVCM			LVEB		Yield point		10/s [Pas]	100/s [Pas]	Water content [%]	T (2% mass loss N₂) [°C]	T (2% mass loss air) [°C]	Mass loss (55 min. N₂) [%]	Mass loss (190 min air) [%]	Residual mass (N₂) [%]
							tan δ	G' [Pa]	γ [%]	G' [Pa]								
A (unaged)	81	-	-	-	12.3	12.5	0.13	14382	13.0	952	27.93	5.19	0.03	325.2	307.7	74.82	82.24	-0.26
A (aged)	-	-	-	-	13.2	13.0	0.136	13766	9.8	1279	29.54	5.45	< 0.01	334.3	339.2	74.23	79.48	0.19
B (unaged)	103	-	-	-	8.6	10.6	0.134	38800	13.5	1619	52.36	8.86	< 0.01	326.7	313.6	70.14	76.90	0.36
B (aged)	-	-	-	-	8.4	10.8	0.14	28109	12.0	1761	48.99	8.69	< 0.01	330.5	344.9	66.96	75.98	0.11
C (unaged)	112	-	-	-	9.1	11.2	0.117	27922	4.3	2987	39.26	6.59	< 0.01	306.7	313.7	76.99	96.28	1.99
C (aged)	-	-	-	-	10.5	12.1	0.133	31216	9.9	1426	40.71	6.82	< 0.01	310.8	317.4	76.64	98.38	1.82
D (unaged)	133,5	-	-	-	7.8	10.4	0.124	34790	9.2	1818	45.53	7.41	< 0.01	323.3	304.6	73.28	98.50	1.58
D (aged)	-	-	-	-	9.4	11.3	0.13	30093	9.9	1708	44.74	7.30	< 0.01	326.5	317.3	74.61	93.33	1.19
E (unaged)	142	-	-	-	8.3	10.9	0.127	52277	7.0	2722	49.97	8.25	< 0.01	334.4	324.3	73.45	96.18	1.58
E (aged)	-	-	-	-	9.6	11.1	0.138	27864	3.0	4526	50.43	8.39	< 0.01	317.3	318.9	74.98	95.20	1.01
F (unaged)	104	-	-	-	8.3	9.4	0.131	47513	0.9	10301	62.45	9.46	< 0.01	309.6	330.5	63.25	77.74	1.72
F (aged)	-	-	-	-	9.0	8.8	0.131	51858	12.5	1580	54.56	8.87	< 0.01	318.6	323.5	66.21	78.51	0.98
G (unaged)	137	0.22	0.17	0.07	8.4	10.9	0.121	35922	1.8	6303	45.81	7.46	< 0.01	328.2	319.4	68.03	83.64	2.63
G (aged)	198	-	-	-	9.5	11.7	0.134	36851	10.9	1560	46.56	7.55	< 0.01	325.1	317.9	71.44	83.77	2.50
H (unaged)	133,5	-	-	-	9.0	11.3	0.122	35408	10.8	1751	51.37	7.99	< 0.01	325.5	331.2	72.20	84.02	2.12
H (aged)	-	-	-	-	9.3	11.7	0.13	41779	12.3	1484	47.51	7.57	< 0.01	318.1	318.1	73.23	86.95	1.80
I (unaged)	94	-	-	-	8.0	10.1	0.12	47963	14.7	2142	73.55	10.42	< 0.01	328.3	332.6	69.13	74.04	4.88
I (aged)	-	-	-	-	8.1	9.9	0.13	48962	18.3	1578	74.40	10.49	< 0.01	331.0	320.4	69.37	75.29	5.12
J (unaged)	264	0.16	0,12	0.03	7.1	8.0	0.133	46827	9.48	1984	53.64	9.05	0.03	332.5	312.4	71.46	86.74	1.51
J (aged)	-	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	64.20	10.43	< 0.01	317.0	322.5	71.55	87.14	1.72
K (unaged)	241	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	63.24	10.38	< 0.01	329.1	335.9	71.01	79.99	0.90
K (aged)	-	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	63.40	10.39	< 0.01	338.0	336.0	65.64	79.66	1.58
L (unaged)	-	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	62.17	10.03	< 0.01	338.6	346.5	70.09	79.42	2.17
L (aged)	-	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	63.97	10.41	< 0.01	335.3	332.2	70.05	79.55	1.93
B[1] unaged	133 (Ref. ESTL)	-	-	-	6.0	12.4	0.062	21452	14.4	1794	55.44	8.45	< 0.01	369.4	368.5	76.06	86.63	1.75
B[1] aged		-	-	-	7.6	-	-	-	-	-	-	-	< 0.01	374.7	361.0	73.97	85.99	1.51
B[2] unaged		-	-	-	-	11.0	0.048	31545	19.2	1314	60.10	8.73	0.01	351.5	350.9	76.85	87.74	1.36
B[2] aged		-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
BM[3]unaged	-	-	-	-	5.4	12.6	0.066	37412	10.3	4208	89.104	12.41	< 0.01	335.6	335.0	76.21	84.31	1.38
BM[3]aged	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-

[1]Braycote 601 EF, 1st batch, [2]Braycote 601 EF, 2nd batch, [3]Braycote 601 Micronic

Improvement of tribological lifetime after aging. Phenomenon is known at ESTL, also for greases being stored at RT for a while --- subsequent impregnation effect?

Preliminary formulations

Grease	Tribology and Outgassing				Oil Separation		Rheology						TGA					
	SOT (orbits/µg)	Outgassing (%)			100 °C / 22 d	204 °C /30h	Amplitude sweep (25 °C)				Viscosity (20 °C)							
		TML	RML	CVCM			LVEB		Yield point		10/s [Pas]	100/s [Pas]	Water content [%]	T (2% mass loss N_2) [°C]	T (2% mass loss air) [°C]	Mass loss (55 min. N_2) [%]	Mass loss (190 min air) [%]	Residual mass (N_2) [%]
							tan δ	G' [Pa]	γ [%]	G' [Pa]								
A (unaged)	81	-	-	-	12.3	12.5	0.13	14382	13.0	952	27.93	5.19	0.03	325.2	307.7	74.82	82.24	-0.26
A (aged)	-	-	-	-	13.2	13.0	0.136	13766	9.8	1279	29.54	5.45	< 0.01	334.3	339.2	74.23	79.48	0.19
B (unaged)	103	-	-	-	8.6	10.6	0.134	38800	13.5	1619	52.36	8.86	< 0.01	326.7	313.6	70.14	76.90	0.36
B (aged)	-	-	-	-	8.4	10.8	0.14	28109	12.0	1761	48.99	8.69	< 0.01	330.5	344.9	66.96	75.98	0.11
C (unaged)	112	-	-	-	9.1	11.2	0.117	27922	4.3	2987	39.26	6.59	< 0.01	306.7	313.7	76.99	96.28	1.99
C (aged)	-	-	-	-	10.5	12.1	0.133	31216	9.9	1426	40.71	6.82	< 0.01	310.8	317.4	76.64	98.38	1.82
D (unaged)	133,5	-	-	-	7.8	10.4	0.124	34790	9.2	1818	45.53	7.41	< 0.01	323.3	304.6	73.28	98.50	1.58
D (aged)	-	-	-	-	9.4	11.3								326.5	317.3	74.61	93.33	1.19
E (unaged)	142	-	-	-	8.3	10.9								334.4	324.3	73.45	96.18	1.58
E (aged)	-	-	-	-	9.6	11.1								317.3	318.9	74.98	95.20	1.01
F (unaged)	104	-	-	-	8.3	9.4								309.6	330.5	63.25	77.74	1.72
F (aged)	-	-	-	-	9.0	8.8								318.6	323.5	66.21	78.51	0.98
G (unaged)	137	0.22	0.17	0.07	8.4	10.9								328.2	319.4	68.03	83.64	2.63
G (aged)	198	-	-	-	9.5	11.7	0.134	36851	10.9	1560	46.56	7.55	< 0.01	325.1	317.9	71.44	83.77	2.50
H (unaged)	133,5	-	-	-	9.0	11.3	0.122	35408	10.8	1751	51.37	7.99	< 0.01	325.5	331.2	72.20	84.02	2.12
H (aged)	-	-	-	-	9.3	11.7	0.13	41779	12.3	1484	47.51	7.57	< 0.01	318.1	318.1	73.23	86.95	1.80
I (unaged)	94	-	-	-	8.0	10.1	0.12	47963	14.7	2142	73.55	10.42	< 0.01	328.3	332.6	69.13	74.04	4.88
I (aged)	-	-	-	-	8.1	9.9	0.13	48962	18.3	1578	74.40	10.49	< 0.01	331.0	320.4	69.37	75.29	5.12
J (unaged)	264	0.16	0,12	0.03	7.1	8.0	0.133	46827	9.48	1984	53.64	9.05	0.03	332.5	312.4	71.46	86.74	1.51
J (aged)	-	-	-	-	8.1	9.5	0.128	50007	8.93	2684	64.20	10.43	< 0.01	317.0	322.5	71.55	87.14	1.72
K (unaged)	241	-	-	-	5.8	7.7	0.131	52496	9.54	2376	63.24	10.38	< 0.01	329.1	335.9	71.01	79.99	0.90
K (aged)	-	-	-	-	7.2	8.6	0.135	41530	11.3	1998	63.40	10.39	< 0.01	338.0	336.0	65.64	79.66	1.58
L (unaged)	-	0.15	0.12	0.03	6.8	8.1	0.124	52890	8.15	2945	62.17	10.03	< 0.01	338.6	346.5	70.09	79.42	2.17
L (aged)	-	-	-	-	7.4	8.6	0.13	52861	8.68	2679	63.97	10.41	< 0.01	335.3	332.2	70.05	79.55	1.93
B[1] unaged	133 (Ref. ESTL)	-	-	-	6.0	12.4	0.062	21452	14.4	1794	55.44	8.45	< 0.01	369.4	368.5	76.06	86.63	1.75
B[1] aged		-	-	-	7.6	-	-	-	-	-	-	-	< 0.01	374.7	361.0	73.97	85.99	1.51
B[2] unaged		-	-	-	-	11.0	0.048	31545	19.2	1314	60.10	8.73	0.01	351.5	350.9	76.85	87.74	1.36
B[2] aged		-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
BM[3]unaged	-	-	-	-	5.4	12.6	0.066	37412	10.3	4208	89.104	12.41	< 0.01	335.6	335.0	76.21	84.31	1.38
BM[3]aged	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-

[1]Braycote 601 EF, 1st batch, [2]Braycote 601 EF, 2nd batch, [3]Braycote 601 Micronic

Compliance with outgassing requirements despite of additive use. Destillation of base oil and bakeout of bentonite before grease rolling might further improve cleanliness

Preliminary formulations

Grease	Tribology and Outgassing				Oil Separation		Rheology						TGA					
	SOT (orbits/µg)	Outgassing (%)			100 °C / 22 d	204 °C /30h	Amplitude sweep (25 °C)				Viscosity (20 °C)							
		TML	RML	CVCM			LVEB		Yield point		10/s [Pas]	100/s [Pas]	Water content [%]	T (2% mass loss N2) [°C]	T (2% mass loss air) [°C]	Mass loss (55 min. N2) [%]	Mass loss (190 min air) [%]	Residual mass (N2) [%]
							tan δ	G' [Pa]	γ [%]	G' [Pa]								
A (unaged)	81	-	-	-	12.3	12.5	0.13	14382	13.0	952	27.93	5.19	0.03	325.2	307.7	74.82	82.24	-0.26
A (aged)	-	-	-	-	13.2	13.0	0.136	13766	9.8	1279	29.54	5.45	< 0.01	334.3	339.2	74.23	79.48	0.19
B (unaged)	103	-	-	-	8.6	10.6	0.134	38800	13.5	1619	52.36	8.86	< 0.01	326.7	313.6	70.14	76.90	0.36
B (aged)	-	-	-	-	8.4	10.8	0.14	28109	12.0	1761	48.99	8.69	< 0.01	330.5	344.9	66.96	75.98	0.11
C (unaged)	112	-	-	-	9.1	11.2	0.117	27922	4.3	2987	39.26	6.59	< 0.01	306.7	313.7	76.99	96.28	1.99
C (aged)	-	-	-	-	10.5	12.1	0.133	31216	9.9	1426	40.71	6.82	< 0.01	310.8	317.4	76.64	98.38	1.82
D (unaged)	133,5	-	-	-	7.8	10.4	0.124	34790	9.2	1818	45.53	7.41	< 0.01	323.3	304.6	73.28	98.50	1.58
D (aged)	-	-	-	-	9.4	11.3	0.13	30093	9.9	1708	44.74	7.30	< 0.01	326.5	317.3	74.61	93.33	1.19
E (unaged)	142	-	-	-	8.3	10.9	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	324.3	73.45	96.18	1.58
E (aged)	-	-	-	-	9.6	11.1	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	318.9	74.98	95.20	1.01
F (unaged)	104	-	-	-	8.3	9.4	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	330.5	63.25	77.74	1.72
F (aged)	-	-	-	-	9.0	8.8	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	323.5	66.21	78.51	0.98
G (unaged)	137	0.22	0.17	0.07	8.4	10.9	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	319.4	68.03	83.64	2.63
G (aged)	198	-	-	-	9.5	11.7	0.134	36851	10.9	1560	46.56	7.55	< 0.01	325.1	317.9	71.44	83.77	2.50
H (unaged)	133,5	-	-	-	9.0	11.3	0.122	35408	10.8	1751	51.37	7.99	< 0.01	325.5	331.2	72.20	84.02	2.12
H (aged)	-	-	-	-	9.3	11.7	0.13	41779	12.3	1484	47.51	7.57	< 0.01	318.1	318.1	73.23	86.95	1.80
I (unaged)	94	-	-	-	8.0	10.1	0.12	47963	14.7	2142	73.55	10.42	< 0.01	328.3	332.6	69.13	74.04	4.88
I (aged)	-	-	-	-	8.1	9.9	0.13	48962	18.3	1578	74.40	10.49	< 0.01	331.0	320.4	69.37	75.29	5.12
J (unaged)	264	0.16	0,12	0.03	7.1	8.0	0.133	46827	9.48	1984	53.64	9.05	0.03	332.5	312.4	71.46	86.74	1.51
J (aged)	-	-	-	-	8.1	9.5	0.128	50007	8.93	2684	64.20	10.43	< 0.01	317.0	322.5	71.55	87.14	1.72
K (unaged)	241	-	-	-	5.8	7.7	0.131	52496	9.54	2376	63.24	10.38	< 0.01	329.1	335.9	71.01	79.99	0.90
K (aged)	-	-	-	-	7.2	8.6	0.135	41530	11.3	1998	63.40	10.39	< 0.01	338.0	336.0	65.64	79.66	1.58
L (unaged)	-	0.15	0.12	0.03	6.8	8.1	0.124	52890	8.15	2945	62.17	10.03	< 0.01	338.6	346.5	70.09	79.42	2.17
L (aged)	-	-	-	-	7.4	8.6	0.13	52861	8.68	2679	63.97	10.41	< 0.01	335.3	332.2	70.05	79.55	1.93
B[1] unaged	133 (Ref. ESTL)	-	-	-	6.0	12.4	0.062	21452	14.4	1794	55.44	8.45	< 0.01	369.4	368.5	76.06	86.63	1.75
B[1] aged		-	-	-	7.6	-	-	-	-	-	-	-	< 0.01	374.7	361.0	73.97	85.99	1.51
B[2] unaged		-	-	-	-	11.0	0.048	31545	19.2	1314	60.10	8.73	0.01	351.5	350.9	76.85	87.74	1.36
B[2] aged		-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
BM[3]unaged	-	-	-	-	5.4	12.6	0.066	37412	10.3	4208	89.104	12.41	< 0.01	335.6	335.0	76.21	84.31	1.38
BM[3]aged	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-

[1]Braycote 601 EF, 1st batch, [2]Braycote 601 EF, 2nd batch, [3]Braycote 601 Micronic

Slight increase of oil separation, also for Braycote 601 EF --- shows need of good rehomogenization before use

Preliminary formulations

Grease	Tribology and Outgassing				Oil Separation		Rheology						TGA					
	SOT (orbits/µg)	Outgassing (%)			100 °C / 22 d	204 °C /30h	Amplitude sweep (25 °C)				Viscosity (20 °C)							
		TML	RML	CVCM			LVEB		Yield point		10/s [Pas]	100/s [Pas]	Water content [%]	T (2% mass loss N₂) [°C]	T (2% mass loss air) [°C]	Mass loss (55 min. N₂) [%]	Mass loss (190 min air) [%]	Residual mass (N₂) [%]
							tan δ	G' [Pa]	γ [%]	G' [Pa]								
A (unaged)	81	-	-	-	12.3	12.5	0.13	14382	13.0	952	27.93	5.19	0.03	325.2	307.7	74.82	82.24	-0.26
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B (aged)	-	-	-	-	8.4	10.8	0.14	28109	12.0	1761	48.99	8.69	< 0.01	330.5	344.9	66.96	75.98	0.11
C (unaged)	112	-	-	-	9.1	11.2	0.117	27922	4.3	2987	39.26	6.59	< 0.01	306.7	313.7	76.99	96.28	1.99
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E (unaged)	142	-	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	324.3	73.45	96.18	1.58
E (aged)	-	-	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	318.9	74.98	95.20	1.01
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G (unaged)	137	0.22	0.17	0.07	8.4	10.9	0.121	35922	1.8	6303	45.81	7.46	< 0.01	328.2	319.4	68.03	83.64	2.63
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B[1] aged		-	-	-	7.6	-	-	-	-	-	-	-	< 0.01	374.7	361.0	73.97	85.99	1.51
B[2] unaged		-	-	-	-	11.0	0.048	31545	19.2	1314	60.10	8.73	0.01	351.5	350.9	76.85	87.74	1.36
B[2] aged		-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
BM[3]unaged	-	-	-	-	5.4	12.6	0.066	37412	10.3	4208	89.104	12.41	< 0.01	335.6	335.0	76.21	84.31	1.38
BM[3]aged	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-

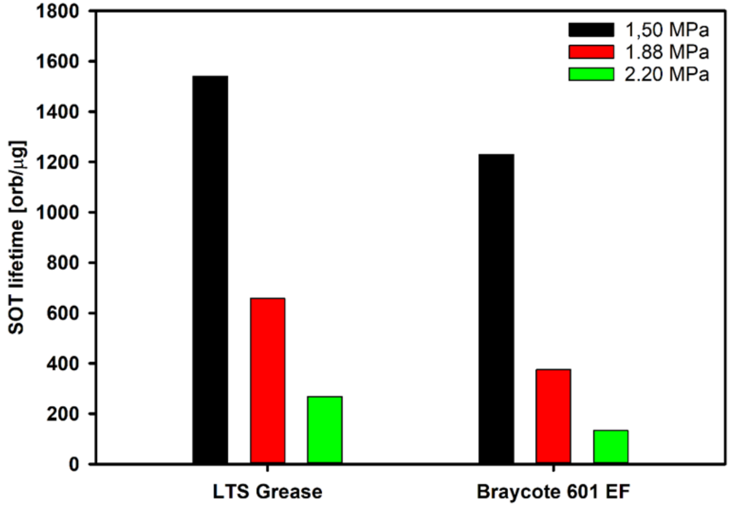
[1]Braycote 601 EF, 1st batch, [2]Braycote 601 EF, 2nd batch, [3]Braycote 601 Micronic

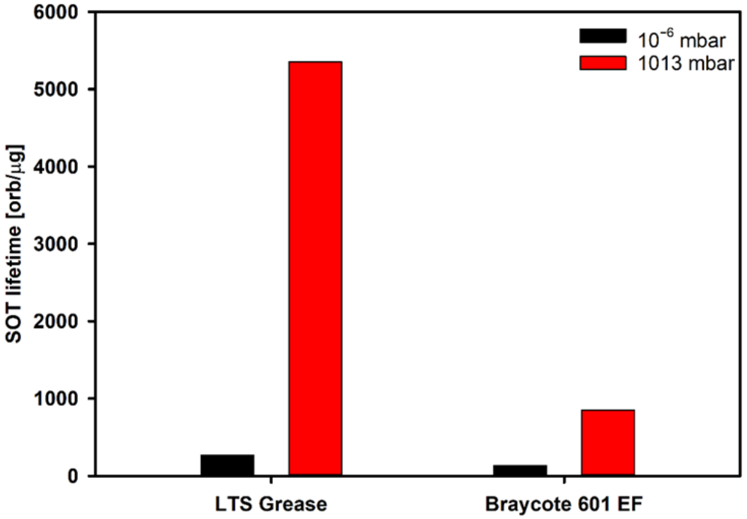
Formulation J was selected as final formulation

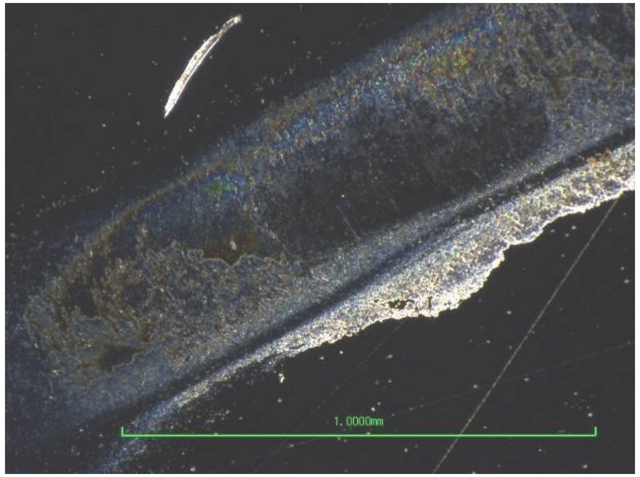
Full testing of final formulation Tribological characterization

- SOT tests at various conditions were performed. The LTS grease was compared with Braycote 601 EF

Test ID	Peak Hertzian Contact Stress [GPa]	Environment/ pressure [mbar]	Temperature [°C]	Average Lifetime [orbits/µg]	Average Steady State CoF
1	2.25	Vacuum / <1.3x10^{-6}	22 ± 3	268.6	0.084
2	1.88	Vacuum / <1.3x10^{-6}	22 ± 3	657.8	0.119
3	1.50	Vacuum / <1.3x10^{-6}	22 ± 3	1540.5	0.119
4	2.25	Vacuum / <1.3x10^{-6}	50 ± 3	207.3	0.108
5	2.25	Vacuum / <1.3x10^{-6}	80 ± 3	156.5	0.092
6	2.25	Cleanroom air moist (55 ± 10% rh) / 101	22 ± 3	5356.9	0.113



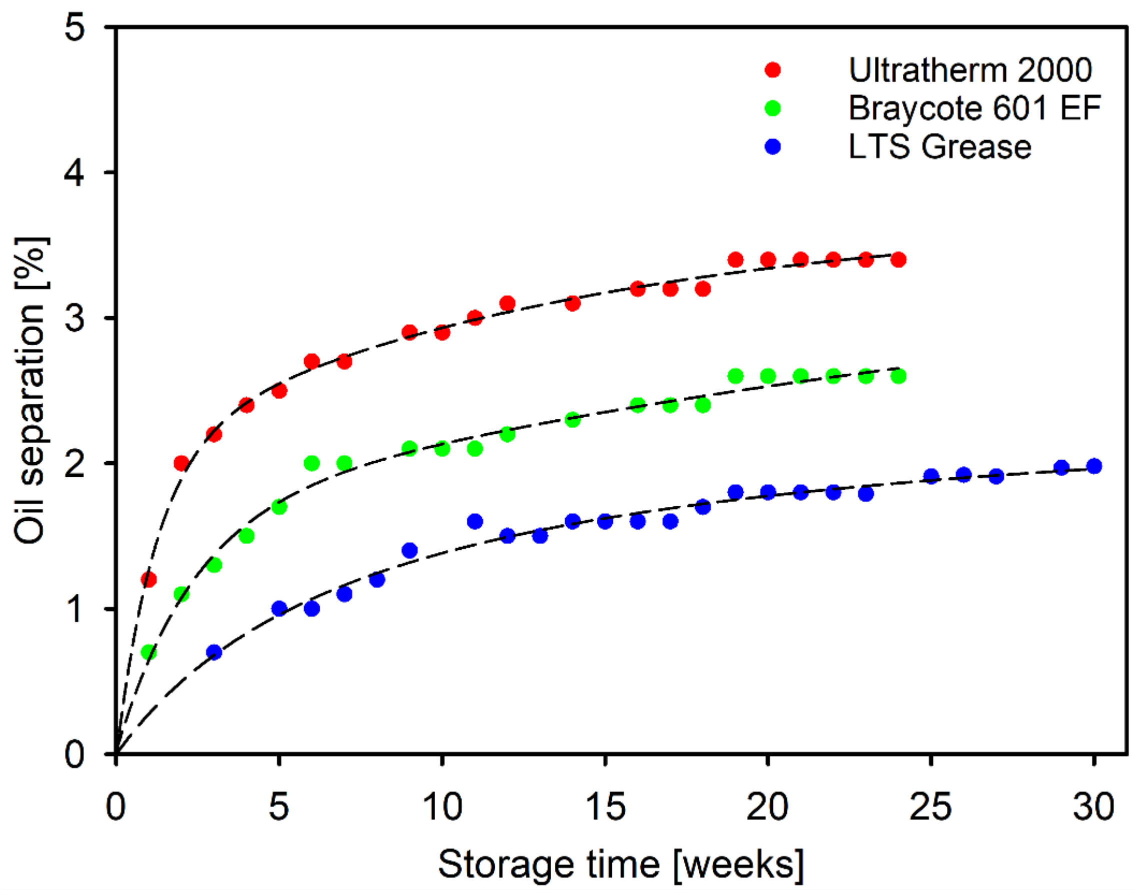




Full testing of final formulation Oil separation

MATERIALES
Exit The Ordinary

- Oil separation tests at room temperature were performed (and are still continued)



Full testing of final formulation Material compatibility

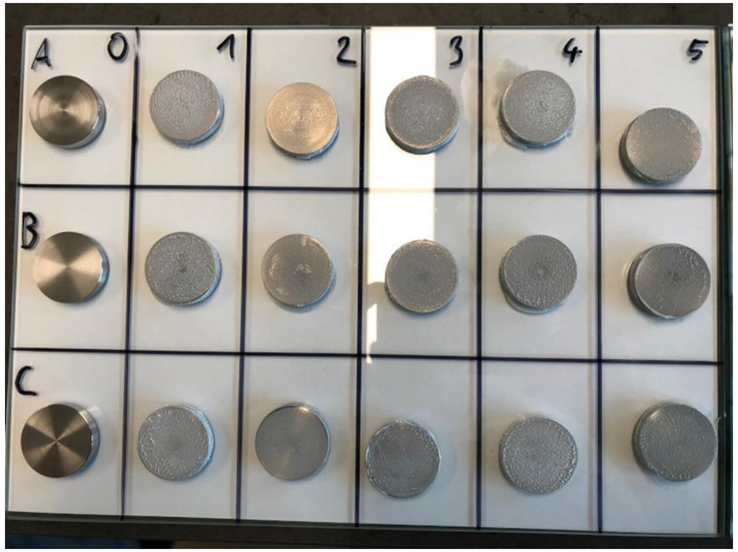
- Corrosion testing was performed with different preliminary formulations (incl. formulation J) and Braycote 601 EF (climate chamber, 2 weeks @ 80 °C/60% rh)

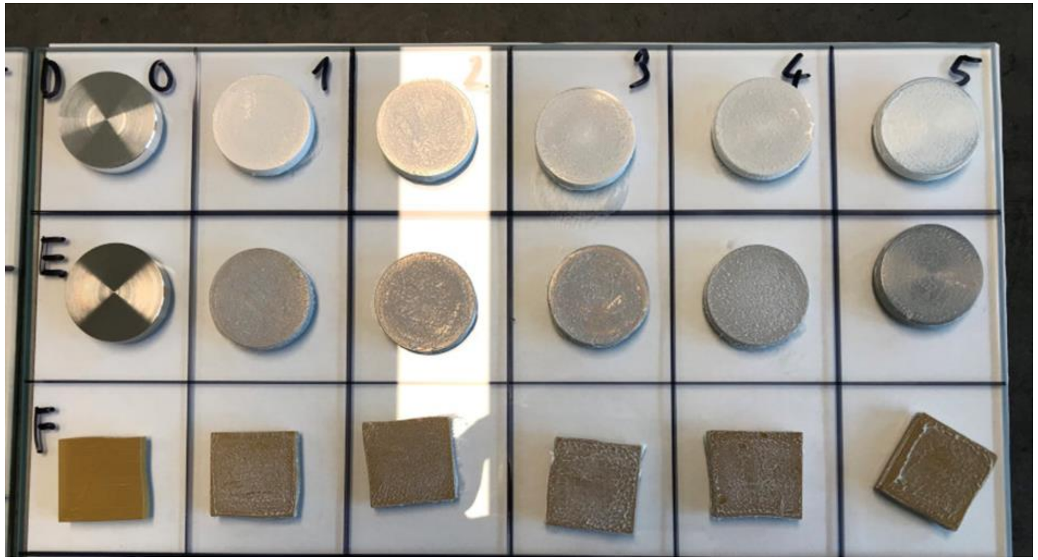
No corrosive impact on metals or swelling of polymer was observed

0	1	2	3	4	5
Blind reference	Formulation J	Formulation K	Formulation L	Formulation G	Braycote 601 EF

A	B	C	D	E	F
SAE 52100 steel	440 C steel	17-4 steel	Al 7075	Ti6AlV4	Polyimide (Tecasint 4011)

before:





Full testing of final formulation
Material compatibility

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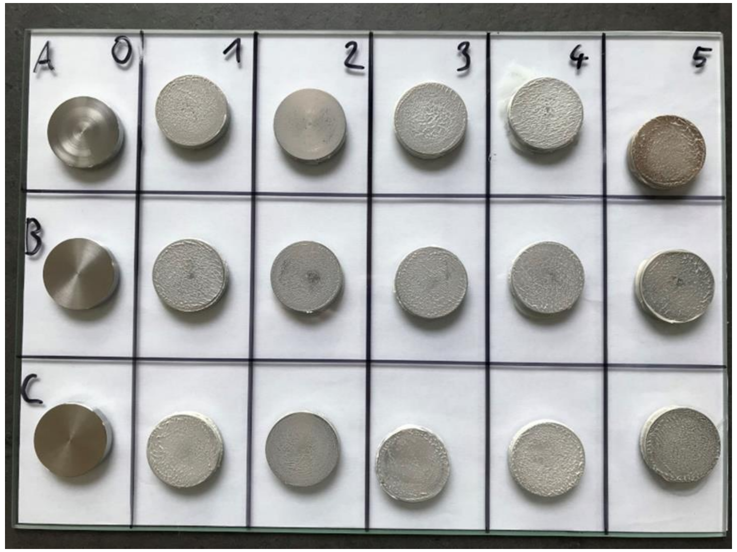
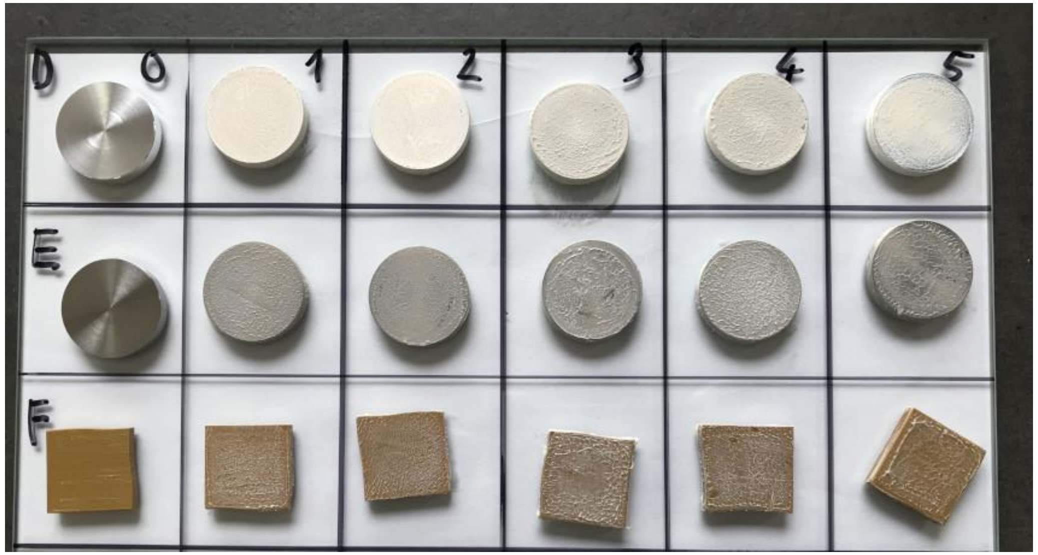
0	1	2	3	4	5
Blind reference	Formulation J	Formulation K	Formulation L	Formulation G	Braycote 601 EF

A	B	C	D	E	F
SAE 52100 steel	440 C steel	17-4 steel	Al 7075	Ti6AlV4	Polyimide (Tecasint 4011)

after:





Full testing of final formulation
Material compatibility

- Impregnation of phenolic resin (Krütex 100P) was performed with base oil. Assessment of mass uptake and visual changes

Saturation of oil uptake was observed – no signs of swelling or incompatibility

Process step	Weight change [%]			
	Sample 1	Sample 2	Sample 3	Average
Vacuum drying	-4.05	-3.96	-4.02	-4.01
1st impregnation	3.39	4.62	3.57	3.86
2nd impregnation	0.31	0.31	0.25	0.29
3rd impregnation	0.05	0.10	0.07	0.07

before:

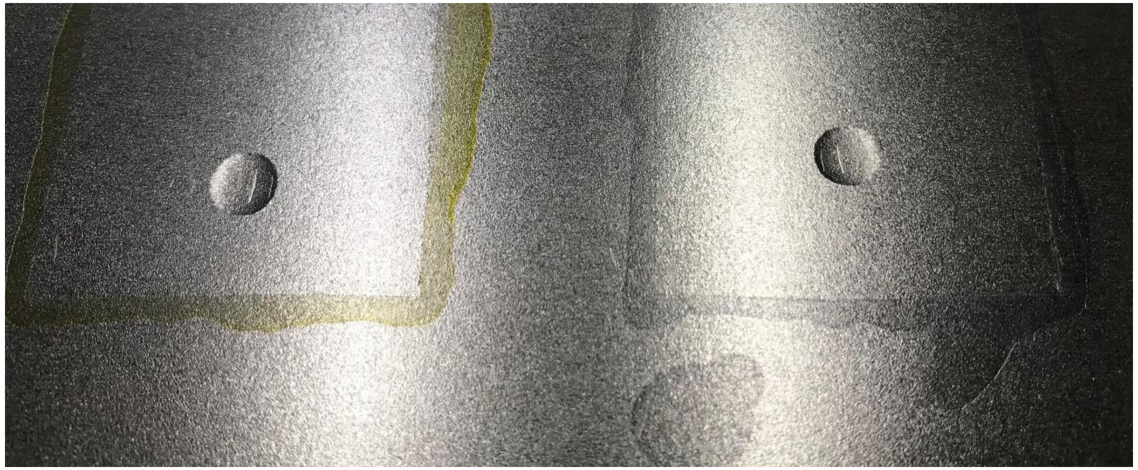
after:

Full testing of final formulation
Material compatibility

- Compatibility with creep barriers (Acota EGC 2708 + Dr Tillwich Antispread E2 Concentrate) was tested on a tilted plate at 100 °C / 24h

Oil remained within the borders of the creep barrier

before



after

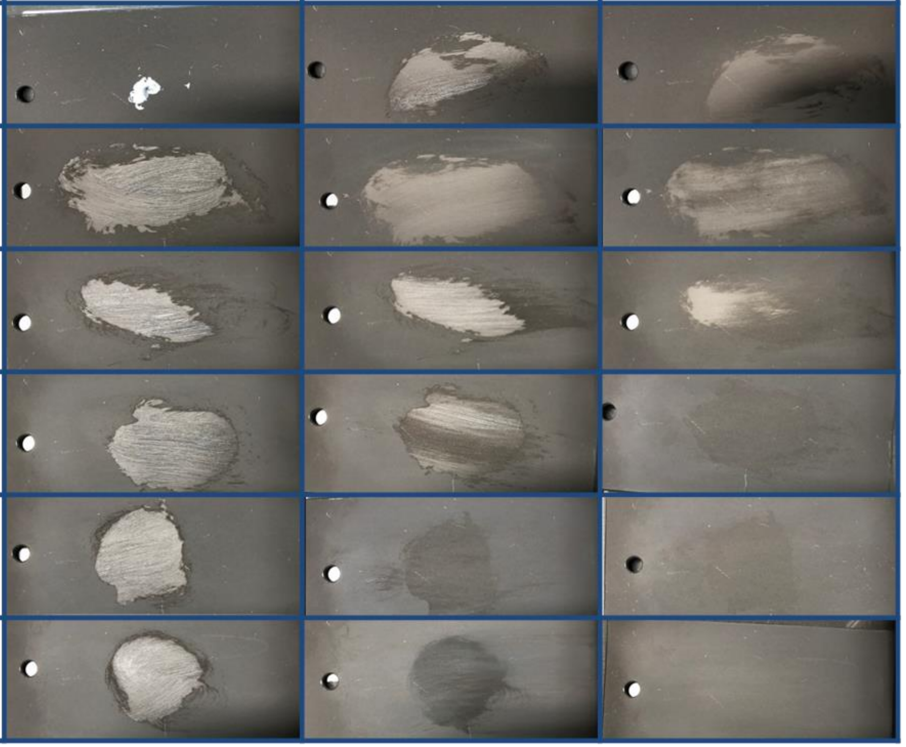
Full testing of final formulation Material compatibility

Testing of solubility and cleanibility

- A small amount of grease was placed on the test surface
- Grease was evenly distributed in a thin layer using dry tissue paper
- A tissue paper with a small amount of solvent was wiped twice over the grease stain with little manual pressure
- A tissue paper with solvent was wiped over the grease stain repeatedly for 15 seconds. Moderate manual pressure was exerted

As expected, only the fluorinated Turmotempoil was capable of fully removing the grease

Solvent	1 – grease distribution	2 – little effort	3 – moderate effort
None			
Water			
Acetone			
Heptane			
Iso-propanol			
Turmotempoil 480			



Full testing of final formulation
Further testing

MATERIALES
Exit The Ordinary

Testing	Standard/ Reference	Success criteria WP1500	Braycote 601EF ('*' = data from TDS)	Formulation J
Density	DIN EN ISO 12185	1-2 g/ml	1.85 g/ml*	1.97 g/ml (calculated)
Thermal stability (TGA)		No degradation until 20°C above maximum operating temperature. Ideally no degradation until 250-300 °C	Mass loss 2% (N_2): 369°C Mass loss 2% (O_2): 368°C	Mass loss 2% (N_2): 333°C Mass loss 2% (O_2): 312°C
Outgassing properties	ECSS-Q-ST-70-02C	TML < 1%; RML < 1%, CVCM < 0.1%	TML* <1% max ; CVCM* <0.1% max	TML: 0.16%; RML: 0.12%; CVCM: 0.03%
Evaporation	ASTM D972	< 1%	2% max*	0.68%
Oil separation	ASTM D6184	< 10%	204°C/30 h: 12.4% 100°C/22 d: 6.0%	204°C/30 h: 8.0 % 100°C/22 d: 7.1 %
Rheology – Viscosity, Viscosity index	DIN 51810-1	Viscosity of base oil: 100 – 400 cSt @ 20°C; 80 – 200 cSt @ 40°C; 20 – 70 cSt @ 100°C. Viscosity index of base oil >130. Viscosity of grease according to spec WP1500: ~40 Pa.s @ 20°C 10 s^{-1} ; ~5 Pa.s @ 20°C 100 s^{-1}.	Viscosity of base oil*: 110-170 cSt (38°C) / 40-50 cSt (99°C) Viscosity index of base oil*: 340 min Viscosity of grease: 10/s: 60.1 Pa·s ; 100/s: 8.7 Pa·s	Viscosity of base oil: 167 cSt (40°C) / 36 cSt (100°C) Viscosity index of base oil: 262 Viscosity of grease: 10/s: 53.6 Pa·s ; 100/s: 9.0 Pa·s
Dropping Point	ASTM D2265	>20°C above maximum operating temperature (specification WP1500)	182°C min*	200°C (ASTM D566)
Pour Point Operational temperature range - lower limit	ASTM D5985	< -50°C, ideally -70°C (specification WP1500)	-73°C max	-71°C (ASTM D5950)
Operational temperature range – upper limit	FAG FE 9 / ECSS-Q-ST-70-02C	+ 120°C (specification WP1500)		125°C (lifetime FAG FE9: 131 h)

* Taken from TDS

Conclusions

- Successful development of a grease with suitability for space application
- Demonstration of LTS properties by artificial aging and comparison with Braycote 601 EF, which is a proven LTS grease
- Tribological performance significantly better than for Braycote 601 EF and other PFPE based greases

Outlook

Outlook – next steps

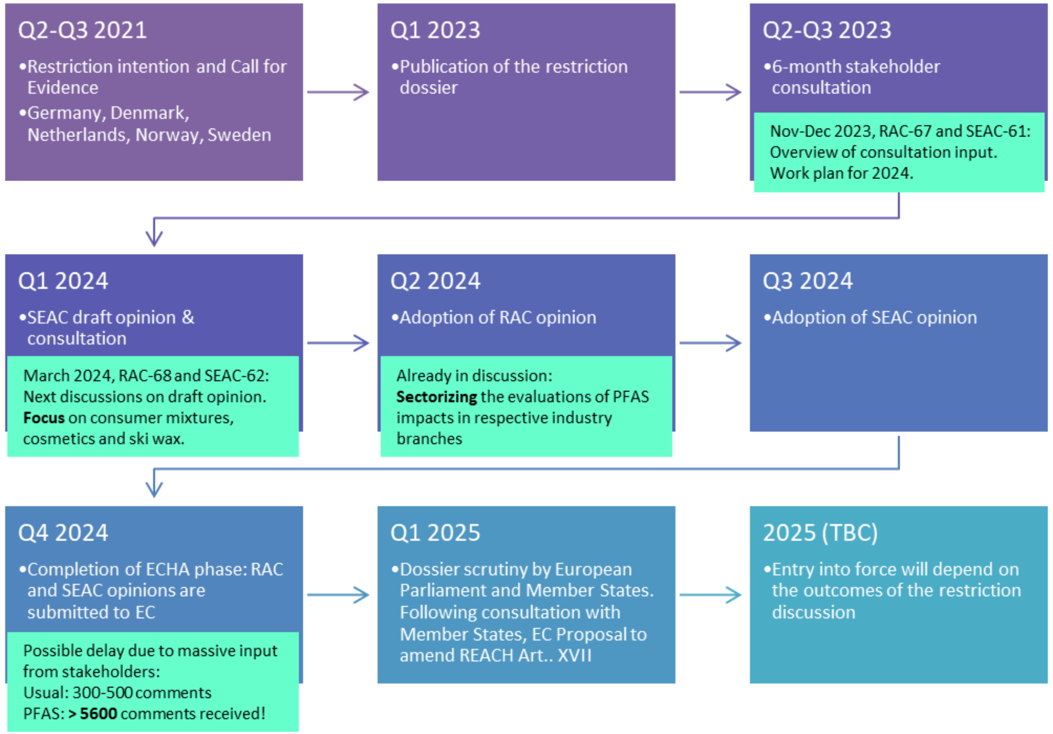
MATERIALES
Exit The Ordinary

The following next steps are planned

- Establishment of scaled-up manufacturing process and quality assurement
- Further LTS qualification by implementation into ESTL real-time LTS activity
- Qualification for space mechanisms (e.g. via GSTP – **partners needed**)
- Development of product lines for terrestrial applications (e.g. semicon industry)
- Preparation of market launch

Outlook - PFAS

- Risk of restrictions for a grease based on PFPE and PTFE
- Many unknowns:
    - What will be restricted?
    - How will it be restricted? Exemptions for certain industries??
    - When will restrictions become effective??
    - How will material suppliers react??
-



Outlook - PFAS

MATERIALES opinion: No reason to stop now

- Content of a future PFAS restriction is unknown
  - Chance for fluoropolymers to be excluded
  - Chance for certain sectors to receive permissions
- Currently, no alternatives exist
  - Properties of PFPE are unmatched
  - Even if PFPE can be replaced by MAC or other oils for certain applications, no other viable options to PTFE for clean applications exist --- this would not only affect space industry (semiconductor manufacturing, high performance optics)

Outlook - PFAS

MATERIALES opinion: No reason to stop now

- PFPE grease availability will decrease, also with exception from PFAS
  - Market participants already withdraw due to unknown future
  - PFPE for vacuum applications is a niche product and dispensable for major lubricant suppliers
  - Formulations that use 3M materials (e.g. Dyneon PTFE → Braycote 601 EF??) will have to be redesigned
  - Potential for small companies like MATERIALES to fill the gap

- Effective date of a future PFAS restriction is unknown
  - PFPE/PTFE vacuum grease should remain available, at least until alternatives are ready
  - Established infrastructure can easily be adapted to other PFAS-free options

Exit The Ordinary

Thanks for Your attention!
Questions?

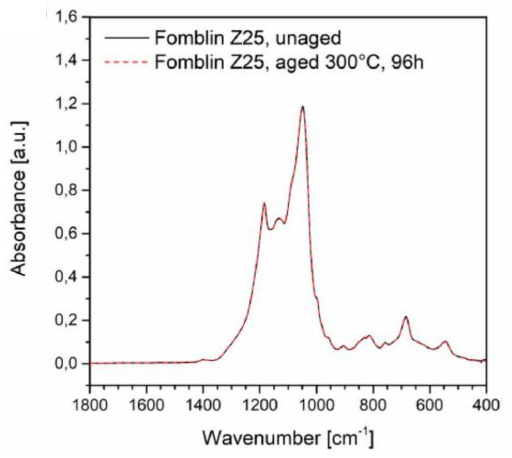
Backup slides

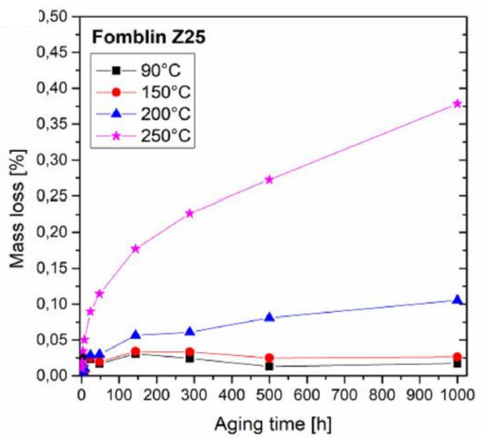
Impact of LTS on grease lubricated spacecraft mechanisms

Different phenomena can influence lubricant functionality in a space mechanism

Oxidation:

- Known for MAC based lubricants at elevated temperatures. Behavior at room temperature over many years is an unknown (note: shelf life Nye2001a ≈ 4 years)
- PFPE based lubricants are known to be very resistant to oxidation due to their chemical structure. Oxidation is only expected to occur at very high temperatures[4]
- Testing of PFPE fluids stored at ESTL for 40+ years under uncontrolled conditions did not show evidence of oxidation[5]





FTIR spectrum and mass loss of Fomblin Z25[4]

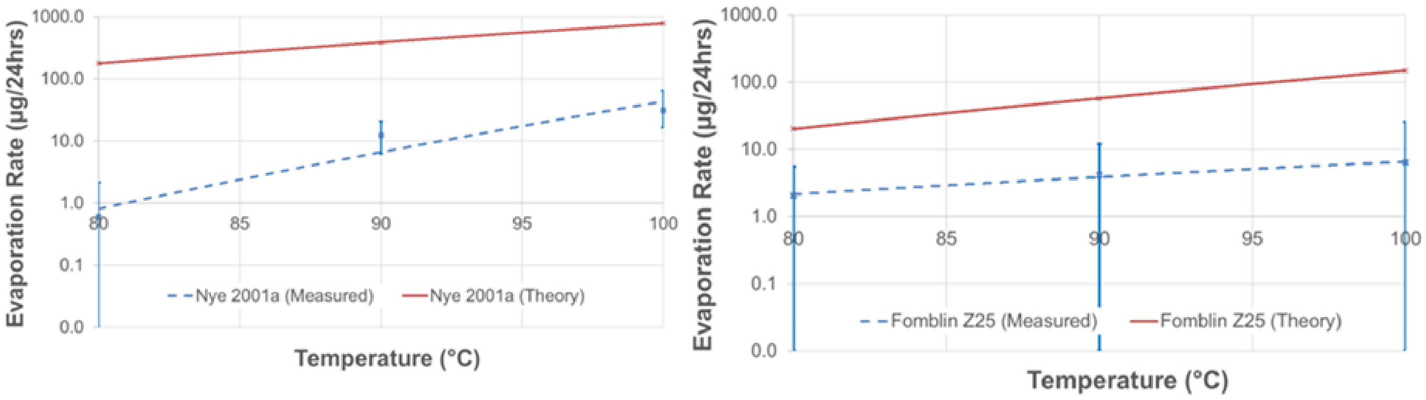
4 A. Wolfberger et al.; "Assessment of the Chemical Degradation of PFPE Lubricants and Greases for Space Applications: Implications for Long-Term On-Ground Storage"; CEAS Space Journal 2021
5 ESA-ESTL-TM-0264 01: "SOT Assessment of Pre-Existing Fluid Samples for LTS"; ESTL 2021

Impact of LTS on grease lubricated spacecraft mechanisms

Different phenomena can influence lubricant functionality in a space mechanism

<u>Evaporation</u>:

- To be considered for long time intervals, in particular at elevated temperature
- Mainly a function of vapor pressure
- Not only the vapor pressure of the base oil, but also of additives is to be considered
- MAC show higher evaporation risk compared to PFPE



Comparison of experimentally measured evaporative loss rates for Nye 2001a (left) and Fomblin Z25 (right) with the predictions of Langmuir theory[6]

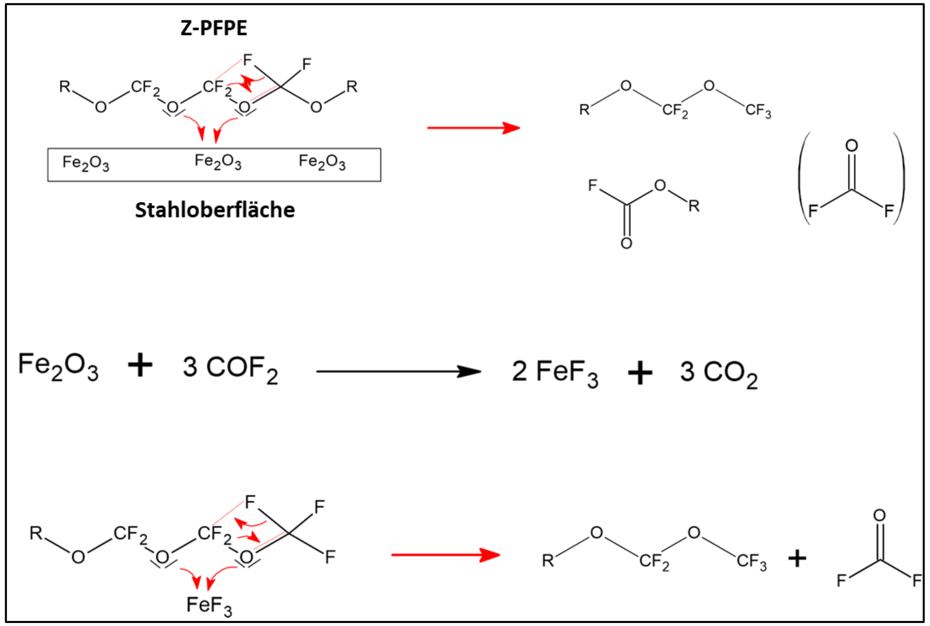
6 ESA-ESTL-TM-0162 01: "An Experimental Assessment of the Evaporation Lives of Space Oils"; ESTL 2018

Impact of LTS on grease lubricated spacecraft mechanisms

Different phenomena can influence lubricant functionality in a space mechanism

<u>Chemical degradation</u>:

- PFPE based lubricants are known to be susceptible to chemical degradation when being in operation and in contact with Lewis acids (e.g. FeF_3)
- No such behavior is known for MAC
- Might theoretically occur during LTS at periods of in-flight inactivity during missions with long cruise phases



Impact of LTS on grease lubricated spacecraft mechanisms

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Different phenomena can influence lubricant functionality in a space mechanism

Lubricant creep:

- Migration of oil can occur over time, when larger amounts of lubricant are applied
- Would lead to viscosity increase in the lubricant due to oil loss
- Impact can be minimized by application of anti-creep coatings with lower surface energy than the fluid

Phenolic cage losses:

- Phenolic ball bearing cages are porous and are typically impregnated with oil prior to assembly
- Oil from lubricant could be taken up during a period of months or years in storage
- This would again lead to oil loss

Impact of LTS on grease lubricated spacecraft mechanisms

Different phenomena can influence lubricant functionality in a space mechanism

Separation of greases:

- Greases are disperse systems, which means they are thermodynamically unstable
- Oil and filler separate over time (even visible after days/weeks), so greases have to be rehomogenized before use
- Greases on lubricated compounds can be rehomogenized by periodical exercising
- Oil separation can lead to oil loss and thus viscosity increase. Also, oil loss can be a contamination risk

Full testing of final formulation
Material compatibility

Testing of solubility and cleanibility

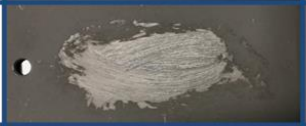
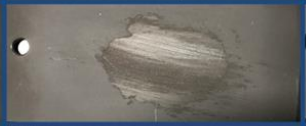
- A small amount of grease was placed on the test surface

- Grease was evenly distributed in a thin layer using dry tissue paper

- A tissue paper with a small amount of solvent was wiped twice over the grease stain with little manual pressure

- A tissue paper with solvent was wiped over the grease stain repeatedly for 15 seconds. Moderate manual pressure was exerted

As expected, only the fluorinated Turmotempoil was capable of fully removing the grease

Solvent	1 – grease distribution	2 – little effort	3 – moderate effort
None			
Water			
Acetone			
Heptane			
Iso-propanol			
Turmotempoil 480			

Full testing of final formulation Tribological characterization

- RGA analyses of formulation J were made and compared to Braycote 601 EF

Detected species formulation J

m/z
28
44
47
51
66
69
85
97
100
104
119
131
169

Typical species Braycote 601 EF

m/z	Degradation Species
28	CO (potentially CO & N_2)
44	CO_2
47	CFO
66	CF_2O
69	CF_3
119	C_2F_5
135	C_2F_5O

Fomblin Z25

Molecular Weight: 17100 g/mol

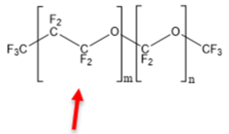
Acetal content: 33-66 %

Fomblin YR1800

Molecular weight: 7300 g/mol

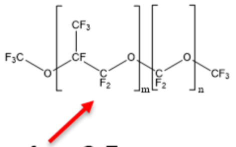
Acetal content: 0-5%

Z-type PFPE

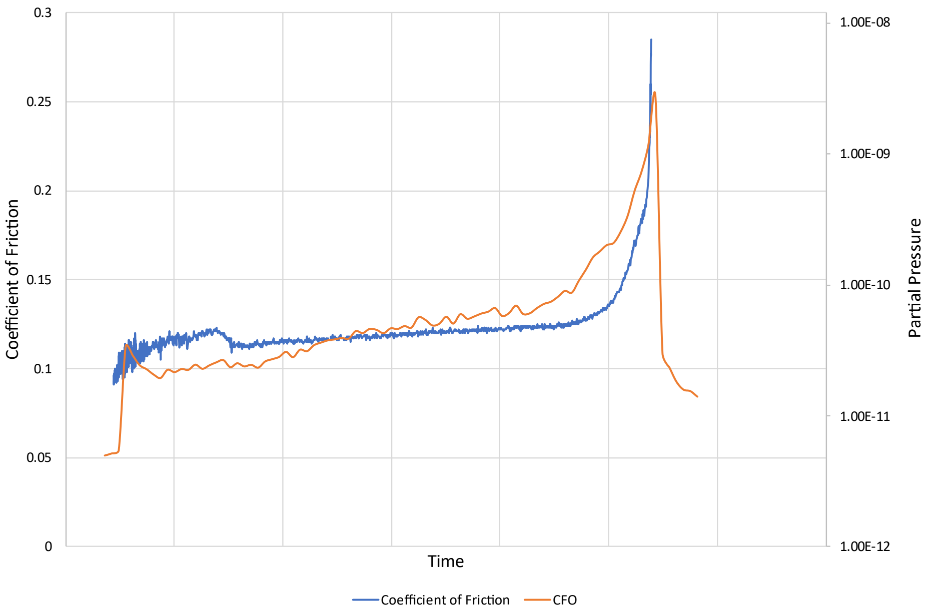


can form C_2F_5O
135 m/z

Y-type PFPE



can form C_3F_7
169 m/z



Full testing of final formulation Impact of humidity

- Aging of preliminary formulation G was performed in a regular oven (80 °C, 0% rh) and in a climate chamber (80°C, 60% rh) for investigation of the impact of humidity on grease properties and LTS

Grease	**Oil Separation**		**Rheology**					
	100 °C / 22 d	204 °C /30h	Amplitude sweep				Viscosity	
			LVEB		Yield point		10/s [Pas]	100/s [Pas]
			tan δ	G' [Pa]	γ [%]	G' [Pa]		
G (unaged)	8.4	10.9	0.121	35922	1.8	6303	45.81	7.46
G (aged, 100 °C, 22 d)	9.5	11.7	0.134	36851	10.9	1560	46.56	7.55
G (aged, 80 °C, 22 d)	8.5	9.8	0.121	45104	9.5	1877	50.10	8.12
G (aged, 80 °C, 60% rel. humidity, 22 d)	7.1	6.1	0.129	39148	10.0	1673	48.04	7.87

Grease	**TGA**					
	Water content [%]	T (2% mass loss N_2) [°C]	T (2% mass loss air) [°C]	Mass loss (55 min N_2) [%]	Mass loss (190 min air) [%]	Residual mass (N_2) [%]
G (unaged)	<0.01	328.2	319.4	68.03	83.64	2.63
G (aged, 100 °C, 22 d)	<0.01	325.1	317.9	71.44	83.77	2.50
G (aged, 80 °C, 22 d)	<0.01	320.5	320.5	73.12	83.51	1.74
G (aged, 80 °C, 60% rel. humidity, 22 d)	0.34	319.0	315.4	65.86	83.21	2.29