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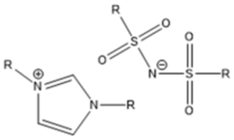
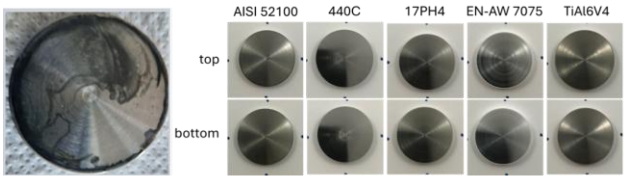


Properties of grease (oil formulation with PTFE)

Parameter	Method	Value
Density		1.37 g/ml
Grease penetration		NLGI class 2
Apparent viscosity	DIN 51810-1	64.7 (10^{-1} s) ; 9.5 (100^{-1} s)
Dropping point	ASTM D566	218 °C
Thermal stability (TGA),air		284 °C
Thermal stability (TGA), N_2		314 °C
Outgassing	ECSS-Q-ST-70-02	TML: 0.24% ; RML: 0.15% CVCM: 0.08% ; WVR: 0.06%
Evaporation	ASTM D972	0.15 (150 °C)
Oil separation (100 °C)	ASTM D6184	30 h: 2.4 % ; 22 d : 3.4 %

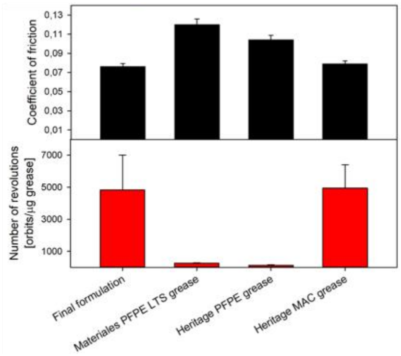
Corrosive properties of plain fluid vs formulated fluid



Properties of oil formulation

Parameter	Method	Value
Density		1.07 g/ml
Pour point	ASTM D 5985	-54 °C
Viscosity	DIN 51810-1	317 mPa·s (20 °C)
Visc. index	ASTM D2270	143
Outgassing	ECSS-Q-ST-70-02	TML: 0.37 % ; RML: 0.21 % ; CVCM: 0.09 % ; WVR: 0.17 %

Tribological properties in SOT test



Highly durable vacuum lubricant formulations based on Ionic Liquids

Executive Summary Report

Early Technology Development

Open Ideas Channel

Affiliation(s): Materiales GmbH (Prime), European Space Tribology Laboratory (Sub), Institute Paul Matzke (Sub)

Activity summary:

The material inherent properties Ionic Liquids display an immense potential for application as lubricant in vacuum environment, in particular due to their low vapor pressures and their performance in tribological systems. However, many promising fluids exert corrosion on metals and alloys. In this activity, space-compliant IL formulations were developed and tested for their compliance with essential requirements for space application. A PFAS-free oil formulation with excellent overall properties could be identified.

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Highly durable vacuum lubricant formulations based on Ionic Liquids –

Executive Summary Report

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Version	Change date	Pages	Change description
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0.2	Draft		
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1.0			Final version
1.1	24.05.25	11	Change in table capture

Table of contents

1. Introduction

1.1. Purpose

This document is the summary report for the project “Highly durable vacuum lubricant formulations based on Ionic Liquids”, filed under ESA contract number 4000141962_23_NL_AS_ov. In this document, the main project findings are summarized concisely.

Note that this is the non-confidential version of the document, with limited information included. All formulation components will be represented by generic names.

1.2. Scope

The essential results of the project “Highly durable vacuum lubricant formulations based on Ionic Liquids” will be included in this document.

1.3. Applicable documents

Reference number	Document name
AD1	ESA contract 4000141962_23_NL_AS_ov
AD2	ESA contract 4000141962_23_NL_AS_ov – TN1 report
AD3	ESA contract 4000141962_23_NL_AS_ov – TN2 report
AD4	ESA contract 4000141962_23_NL_AS_ov – TP1 test plan
AD5	ESTL document DW-004209-01B: “SOT Sample Disk Drawing”
AD6	ESTL document DW-004210-01B: “SOT Guide Plate Drawing”
AD7	ESTL document PRA-ESTL-PR-7508 02: “Grease Lubrication of Components”
AD8	Materiales document VL2_005_03:“Durchführung von TGA Messungen“
AD9	Materiales document VL2_002_01:“Rheologie - Bestimmung von Viskosität und Viskositätsindex von Schmierölen“
AD10	Materiales document VL2_002_02:“Rheologie - Bestimmung der Viskosität von Schmierfetten“

1.4. References

Reference number	Document name
RD1	ESA-ESTL-TM-0174 01A; Ionic Liquids – Review of State of the Art and Benchmarking., ESTL, UK (2016).
RD2	Dörr, N. et al. Five-stage selection procedure of ionic liquids for lubrication of steel-steel contacts in space mechanisms. Tribol. Lett. 67 (2019)
RD3	ESA-ESTL-TM-0178 01; Ionic Liquids – Performance as Space Lubricants., ESTL, UK (2018)
RD4	ESA-ESTL-TM-0331 01; Formulated lubricants based upon ionic liquids, ESTL, UK (2022)
RD5	Keller, A. et al. Practical Evaluation of Ionic Liquids for Application as Lubricants in Cleanrooms and under Vacuum Conditions. *Lubricants* 12, 194 (2024).
RD6	Schüler, F.; Holynska, M.; Henry, T. and Buttery, M. Development of novel lubricant formulations for application in space environment. Poster presentation, ISMSE (2024)
RD7	ESA-ESTL-TM-0375 01; Guidelines for qualification of new fluid lubricants, ESTL, UK (2024).
RD8	https://www.skf.com/de/products/lubrication-management/manual-lubrication-tools/lubricant-analysis-tools/grease-test-ki
RD9	ESA contract: 4000137211_21_NL_KML_rk, Final Report
RD10	Braycote 601 EF Technical Datasheet, Castrol (2020)

1.5. Abbreviations and definitions

Abbreviation	Definition
ASTM	American Society for Testing and Materials
CoF	Coefficient of friction
CVCM	Collected Volatile Condensable Material
ECHA	European chemicals agency
ECSS	European Cooperation for Space Standardization
ESTL	European Space Tribology Laboratory
GEO	Geostationary earth orbit
HSE	Health, Safety and Environmental Constraints
IL	Ionic Liquid
LEO	Lower earth orbit
MAC	Multiple alkylated cyclopentane
MSDS	Material Safety Data Sheet
NLGI	National lubricating grease institute
PFAS	Perfluoro- and polyfluoroalkyl substances
PFPE	Perfluoropolyether
PTFE	Polytetrafluoroethylene
REACH	Registration, Evaluation, Authorisation and Restriction of Chemicals
RML	Recovered Mass Loss
SOT	Spiral orbit tribometer
TGA	Thermogravimetric analysis
TML	Total Mass Loss
TRL	Technology readiness level
UHMW-PE	Ultra-High-Molecular-Weight Polyethylene
WVR	Water Vapor Regained

2. Ionic Liquids for use in space applications

The extreme conditions in space demand innovative, highly advanced and well-understood technical solutions to ensure mission success. Lubrication plays a crucial role in ensuring the functionality and longevity of equipment. In general, traditional lubricants do not meet the requirements for space application as they display significant evaporation leading to functionality loss and contamination. Additional fundamental lubricant requirements such as a functionality over wide temperature ranges, chemically inertness to withstand radiation and aggressive species, and a compatibility with a wide range of spacecraft materials further narrow down the range of applicable materials.

The majority of fluids used for space applications is based either on perfluoropolyether (PFPE) or multiple alkylated cyclopentanes (MAC). The unique properties of Ionic Liquids (IL) have been extensively documented, and their exceptional tribological performance has been showcased in numerous technical articles. As molecular salts with negligible vapor pressures, ILs are highly attractive compounds for vacuum applications. Surprisingly, relatively few studies of the use of ionic fluids as lubricants under test conditions applicable to spacecrafts were published so far [RD1]. Pioneer work has been performed by Advanced Aerospace Composites GmbH (AAC) and The European Space Tribology Laboratory (ESTL) recently (RD2 – RD4]. Until today, no ILs that comply with all requirements for space application have been reported. The corrosive impact of many tribologically advantageous ILs on metallic materials is a known and major obstacle. The understanding of the relationship between chemical structure and macroscopic properties of organic matter is one key towards tailor-made ILs [RD5].

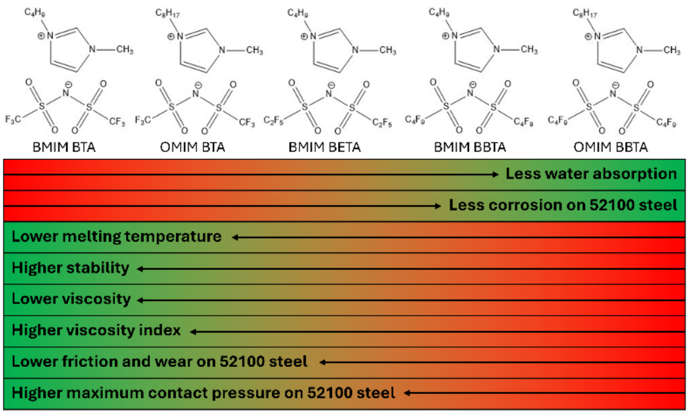

Figure 1: Impact of chemical structure on selected properties of imidazolium based ILs [RD6]

Therefore, and based on the development work that has been conducted at Materiales since 2021, in this activity (ESA contract 4000141962_23_NL_AS_ov) Ionic Liquid base oil formulations were developed on the basis of a dedicated lubricant specification, which was derived from a recently published guideline for qualification, validation and verification of lubricants for space [RD7]. An electrochemical corrosion test approach was developed as a fast assessment method to support formulation development. After the screening of individual parameters, full formulations of lubricant greases and a manufacturing procedure were defined. A final formulation was selected for comprehensive testing, including analyses of thermal properties, viscosity, outgassing, tribological performance in vacuum and material compatibility.

3. Formulation development

Based on the results of earlier studies at Materiales, and an investigation of PFAS-free ILs during this project, three fluids that could meet all requirements from an initial test program were found.

- IL7
- IL22
- IL29

IL29 is a fluid that does not belong to the group of PFAS, which will be restricted in the future. Both other fluids are PFAS by definition due to their chemical structure. Selected results from initial qualification in the Materiales laboratory can be found in Table 1. Analyses for viscosity were performed rheologically, corrosion tests were performed in a climate chamber and thermo-oxidative properties were assessed by oven storage at elevated temperatures over up to 16 weeks and monitoring of mass loss. For comparison, reference values for Fomblin Z25, a PFPE oil qualified for space applications, are listed. The mass uptake of IL29 in the oven, indicated by a negative mass loss, might have occurred to oxidation of impurities that were in the raw product before bakeout.

Table 1: Selected results from initial IL qualification at Materiales

Compound	Mass loss in oven	Viscosity	Viscosity index	Corrosion on 1.3505 steel
IL7	0.8% (200 °C, 16 wks)	52 mPa·s	205	-
IL22	0.05% (100 °C, 16 wks) ; 0.9% (150 °C, 6 wks)	317 mPa·s	114	0/+
IL29	-0.54% (150 °C, 8 wks)	317 mPa·s	143	0
Fomblin Z25	0.9% (200 °C, 15 wks)	223 mPa·s	317	+

IL7 displayed excellent thermal properties and a low viscosity at high viscosity index, but it behaved very corrosive to 1.3505 carbon steel. IL22 and IL29 were limited in thermal and thermo-oxidative stability, but yielded the best overall results. In initial tribological qualification in air, performed at Tribology Centre of Competence of Mannheim university (CCT), all ILs performed significantly better than Fomblin Z25. Tests according to ASTM D7421-19 and DIN 51834-2 were conducted.

Different pretreatments for the ILs were tested. It was found that a washing with calcium hydroxide powder reduces the corrosive impact of IL7, which could not be confirmed for both other fluids. Also, purity of the supplied fluids and the impact of bakeout on outgassing according to ECSS-Q-ST-70-02C were investigated. It was found that the procedures at Materiales were sufficient to meet the requirements of the standard, but results were not fully satisfying for a future application (see Table 2). The initial procedure (standard bakeout) was performed over a duration of 24 hours at 100 °C and a pressure of ca. 10^{-1}-10^{-2} mbar. In an improved procedure, the fluids were dried for 48 hours at 120 °C and a pressure of ca. 1-3·10^{-3} mbar). It is planned to perform the bakeout at high vacuum of 10^{-5}-10^{-6} mbar and a temperature of 120 °C in the future. In cooperation with the supplier of the fluids, unreacted raw material was identified as the outgassing matter. It is therefore expected to achieve extremely low outgassing rates after its efficient removal.

Table 2: Results from outgassing tests according to ECSS-Q-ST-70-0C

IL	Process	TML	RML	CVCM	WVR
		[%]	[%]	[%]	[%]
IL7	Standard Bakeout	0.70	0.17	0.08	0.53
IL7	Improved Bakeout	0.64	0.10	0.07	0.53
IL22	Standard Bakeout	0.36	0.24	0.09	0.12
IL22	Improved Bakeout	0.27	0.15	0.05	0.12
IL29	Standard Bakeout	0.53	0.39	0.14	0.14
IL29	Improved Bakeout	0.29	0.15	0.09	0.13

Three types of formulation components were investigated for the base oils: Corrosion inhibitors, antioxidants and thickeners. For each category, a screening with candidate components were performed. The selection of corrosion inhibitors and antioxidants was based on the results of prior work at Materiales. For the preselection of additives, only compounds with a low outgassing risk were considered. Since outgassing data is typically not available for such chemicals, preferably solid compounds with high melting points were selected.

The role of corrosion inhibitors was investigated systematically by use of an electrochemical corrosion test and conventional corrosion testing in the climate chamber. Tests were performed with 1.3505 carbon steel, which is a typical bearing material and also a “worst case material” since it is more susceptible to corrosion in comparison with stainless steels and other alloys. Among the three tested ILs, IL7 by far the most corrosive compound. IL22 displays only small signs corrosion, and IL29 showed moderate but clearly visible corrosion. From the different investigated anticorrosion additives, a material that was synthesized inhouse displayed the best corrosion protection. The addition of this compound had a positive impact on all IL base liquids, as it can be seen in Figure 2, Figure 3 and Figure 4.

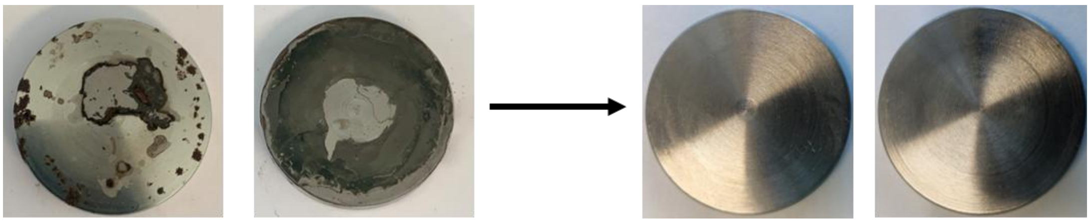

Figure 2: Corrosion tests with IL7 without (left) and with (right) corrosion inhibitor

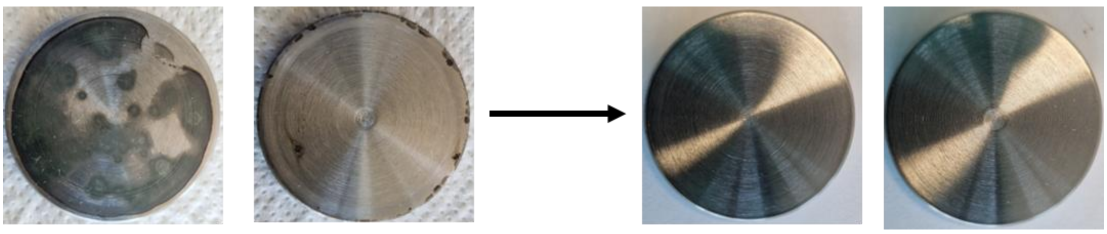

Figure 3: Corrosion tests with IL29 without (left) and with (right) corrosion inhibitor

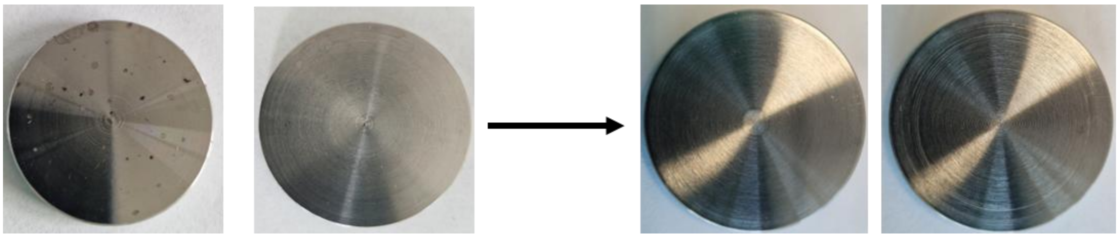

Figure 4: Corrosion tests with IL22 without (left) and with (right) corrosion inhibitor

Antioxidants were primarily investigated for IL22 and IL29, since the chemical structure of IL7 is extremely stable already and comparable to PFPE (see Table 1). Various additives were tested by mixing them with the ILs, followed by thermogravimetric analysis. A positive impact by the antioxidant was indicated by a delay in thermal degradation. Different additive concentrations were tested in order to identify optimal amounts (see Figure 5). The ideal concentration for IL22 was at 2% addition, while for IL29 identical results were received for concentrations between 1-2 %.

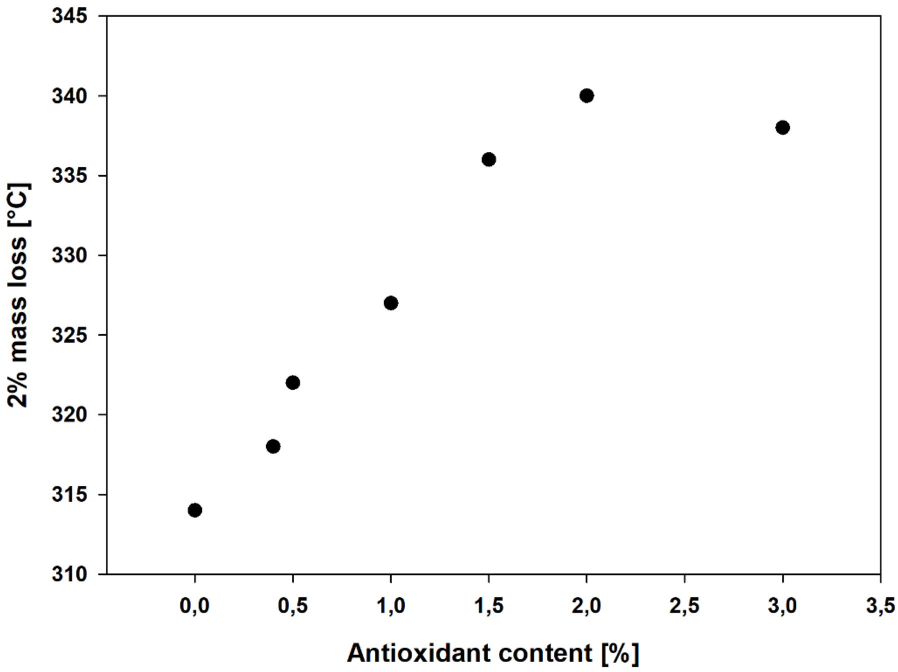

Figure 5: Depiction of TGA decomposition (2% mass loss) of IL22 at different concentrations of antioxidant

For the manufacturing of greases, four different thickeners were tested: Bentonite, organomodified Bentonite, a PFAS-free thickener and polytetrafluoroethylene (PTFE). PTFE is a known and established thickener technology, while the other three candidate were all PFAS-free. The best results from preliminary testing were achieved for “PFAS-free thickener” and PTFE. The “PFAS-free thickener” thickened greases displayed an extremely high compatibility between oil and solid, resulting in very little oil separation. However, the grease consistency and flow behavior were unexpected. PTFE thickened greases behaved as known from other base oils.

Different analyses, among them tribological tests on the spiral orbit tribometer (SOT) were performed with preliminary fully formulated greases. Results were compared with results from earlier test campaigns. Remarkably, the greases based on the non-PFAS fluid IL29 displayed the best performance among the investigated formulations, but were also better than for all heritage lubricants. It needs to be noted that only two tests were performed per formulation, and that the variation of results was higher than normally seen with SOT tests.

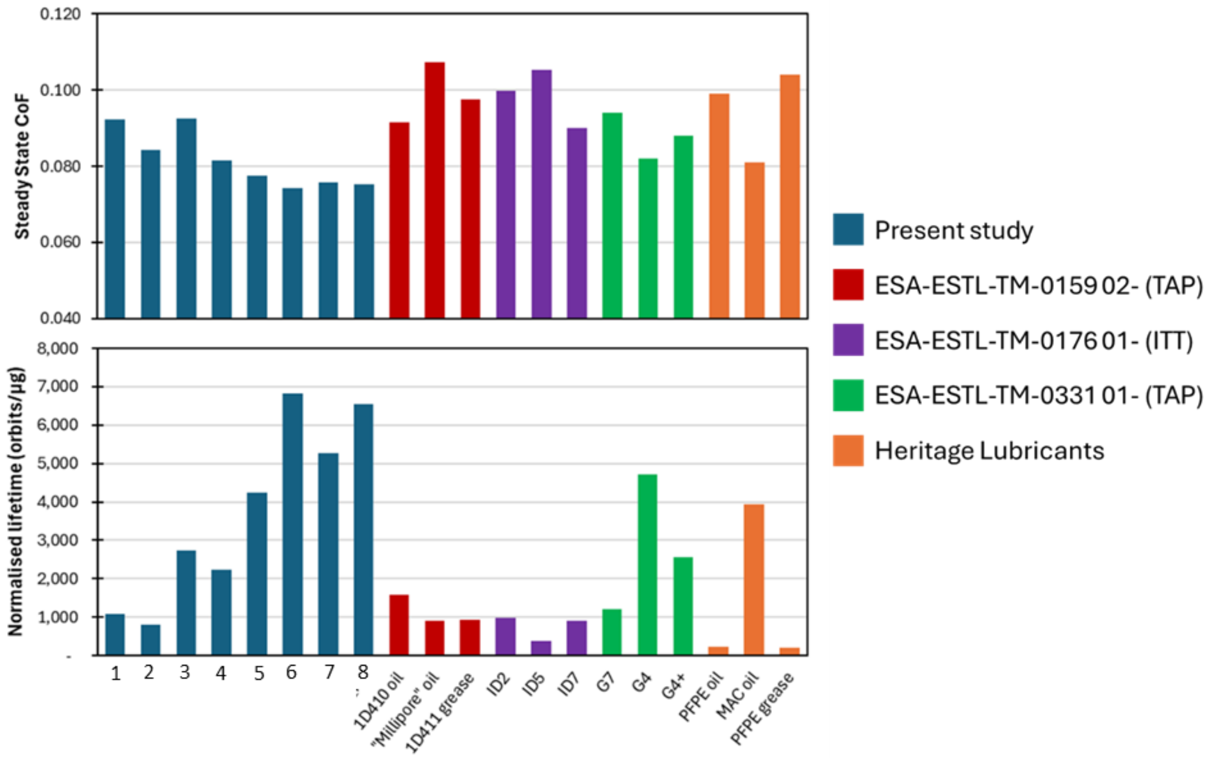

Figure 6: SOT tests with preliminary greases (in blue) and comparison with earlier tests with ILs and with heritage lubricants. Sample codes: 1,2 = IL7-based, 3,4 = IL22-based, 5-8 = IL29-based. 1,3,5,7 contains PTFE, 2,4,6,8 contains "PFAS-free thickener"

It was decided to select IL29 as the base oil for a final formulation, since it shows the following advantages over the other tested liquids:

- No PFAS
- Tribological performance
- Moderate corrosion that could be mitigated with additives

PTFE was chosen as thickener for the final formulation. While "PFAS-free thickener" displayed positive results in testing and it is a PFAS-free compound, different aspects remained unclear. The choice was based on the consideration to use of a known and understood thickener technology, since it most likely provides the clearest information about the IL base oil formulation.

4. Final testing

The final formulation was tested comprehensively. Results were compared to the lubrication specification defined in the initial phase of the project, and also compared to the heritage PFPE/PTFE grease Braycote 601 EF. Results are displayed in Table 3 and Table 4.

Table 3: Test results for the final base oil formulation

Parameter	Method	Value	Specification requirement	Reference values for Braycote 601 EF [RD9+RD10]
Density	Weight of 1 ml – no standard applied	1.07 g/ml	No success criteria defined	1.85 g/ml
Pour point	ASTM D 5985	-54 °C	< -30 °C, ideally -50 °C	-72 °C
Viscosity	[AD9], DIN 51810-1	317 mPa·s	50 – 500cSt @ 20°C 40 – 400cSt @ 40°C 10 – 100cSt @ 100°C Viscosity index > 90	250 mPa·s
Viscosity index	ASTM D2270	143		350
Outgassing	ECSS-Q-ST-70-02	TML: 0.37 % RML: 0.21 % CVCM: 0.09 % WVR: 0.17 %	TML (%) <1 RML (%) <1 CVCM (%) < 0.1 Ideally: CVCM (%) < 0.02	TML: 0.06% [a] RML: 0.05% [a] CVCM: 0.01% [a]

[a] Source: www.spacematdb.com; average data from multiple batches

Table 4: Test results for the final grease formulation

Parameter	Method	Value	Specification requirement	Reference values for Braycote 601 EF [RD9+RD10]
Density	Calculation from raw materials	1.37 g/ml	No success criteria defined	1.85 g/ml
Grease penetration	SKF method [RD8]	NLGI class 2	No success criteria defined	NLGI class 2
Apparent viscosity	[AD10]	64.7 (10^{-1} s) 9.5 (100^{-1} s)	No success criteria defined	57.8 (10^{-1} s) 8.6 (100^{-1} s) (average from 2 batches)
Dropping point	ASTM D566	218 °C	>20 °C above max. operating temperature	182 °C
Thermal stability (TGA), 2% mass loss (air)		284 °C	No degradation < 250 °C	360 °C (average from 2 batches)
Thermal stability (TGA), 2% mass loss (nitrogen)		314 °C	No success criteria defined	360 °C (average from 2 batches)
Outgassing	ECSS-Q-ST-70-02	TML: 0.24% RML: 0.15% CVCM: 0.08% WVR: 0.06%	TML (%) <1 RML (%) <1 CVCM (%) < 0.1 Ideally: CVCM (%) < 0.02	TML: 0.19% [a] RML: 0.16% [a] CVCM: 0.05% [a]
Evaporation	ASTM D972	0.15% (150 °C)	< 1% (100 °C)	2% max (200 °C)
Oil separation (100 °C)	ASTM D6184	30 h: 2.4 % 22 d : 3.4 %	< 8% after 22 d (100 °C)	30 h: 3.0 % 22 d: 6.0 %

Corrosion tests were performed with the different alloys AISI 52100, 440C, 17PH4, EN-AW 7075 and TiAl6V4. Therefore, samples were stored in a climate chamber at 80 °C and 60% relative humidity for a period of 2 weeks time, which is an accelerated test scenario for storage at ambient temperature for a period of 15-20 years. As shown in Figure 7, no signs of corrosion were observed in these tests. Additionally the polyimide Tecasint 4011 was investigated for compatibility. An incompatibility with organic material would typically be visible by significant weight changes due to swelling. No weight changes were observed.

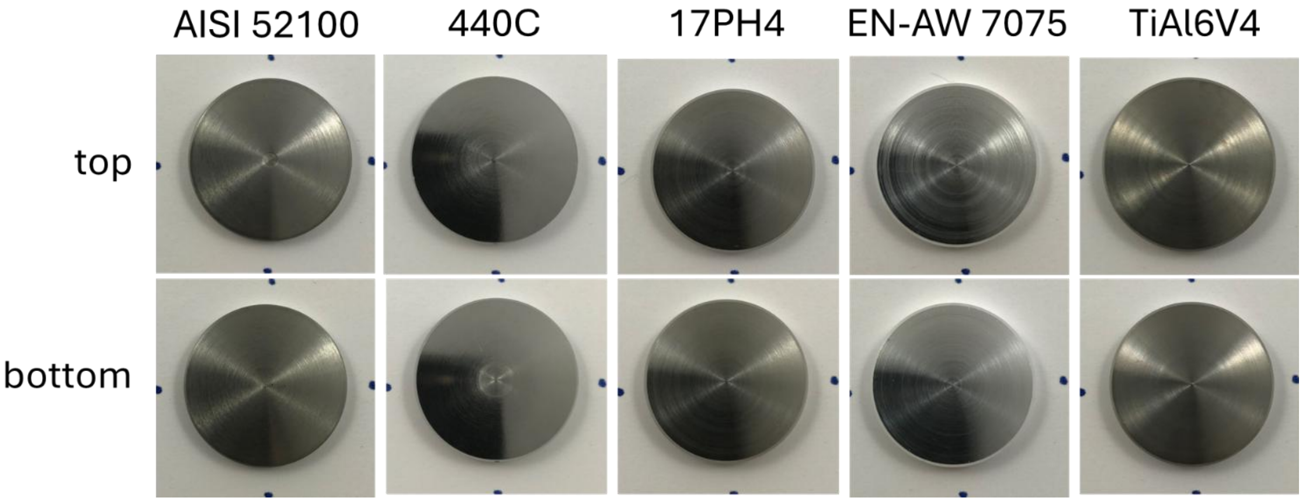

Figure 7: Visual inspection of alloys after corrosion testing

The compatibility with cleaning solvents was assessed in a simple test setup, where grease was applied on a black and porous surface, and where wiping with a solvent drained cloth was performed. As it can be seen in Figure 8, iso-propanol and acetone are feasible cleaning fluids.

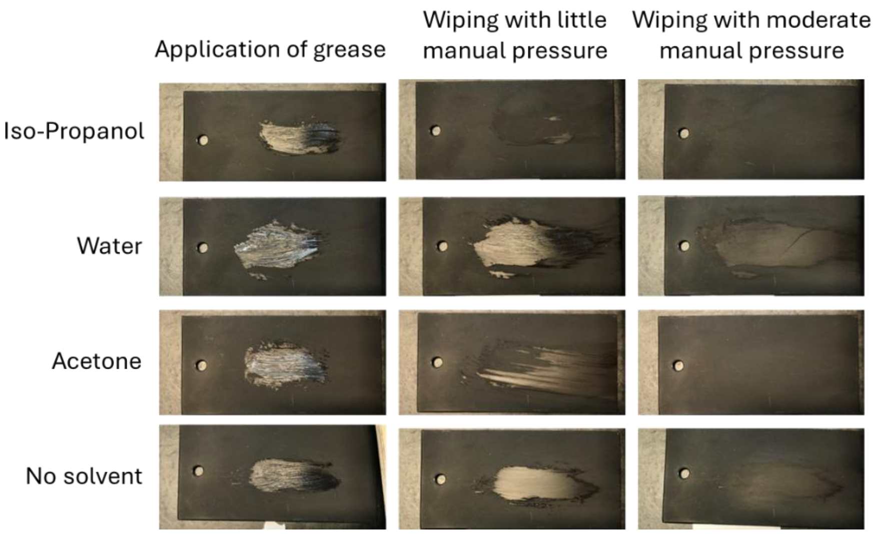

Figure 8: Results from cleaning tests with different solvents

The tribological evaluation was performed through SOT testing at ESTL. Tribological tests in vacuum and air atmosphere were conducted with the SOT tribometer at ESTL. Results were consistent with the tests conducted with this formulation during WP 2000. Additionally, tests at 120 °C temperature were performed. SOT tests were conducted under identical parameters consistent with standard ESTL test conditions, to facilitate comparison between presently investigated fluids and with existing datasets.

- Test samples – All 440C steel contacts. Flats polished to ≤0.05 µm R_a. Balls 12.7 mm grade 5
- Peak Hertzian contact stress –2.25 GPa (1.50 GPa Mean)
- Ball rotation speed – 100 RPM
- Temperature – RT (22 ±3°C), 120 °C
- Environment – Laboratory air (55 ±10% RH), high vacuum (≤5 x 10^{-6} mbar)
- Failure criteria - Friction coefficient increased to ≥0.28

Together with the test data from preliminary formulations, more representative data for testing at room temperature under high vacuum and in air could be obtaines. The comparison of the results from high vacuum testing with existing heritage lubricants is depicted in Figure 9. The final formulation displays significantly longer SOT lifetimes in comparison to PFPE based lubricants, and a comparable behavior to heritage MAC grease. Among the compared lubricants, the coefficients of friction were lowest.

The tests at 120 °C led to a decrease in lifetime, as this is typically observed for other fluid lubricant. It is still worthwhile to note that the SOT lifetime was 4-5 times higher under these conditions as this is found for the thermally very stable PFPE-based greases.

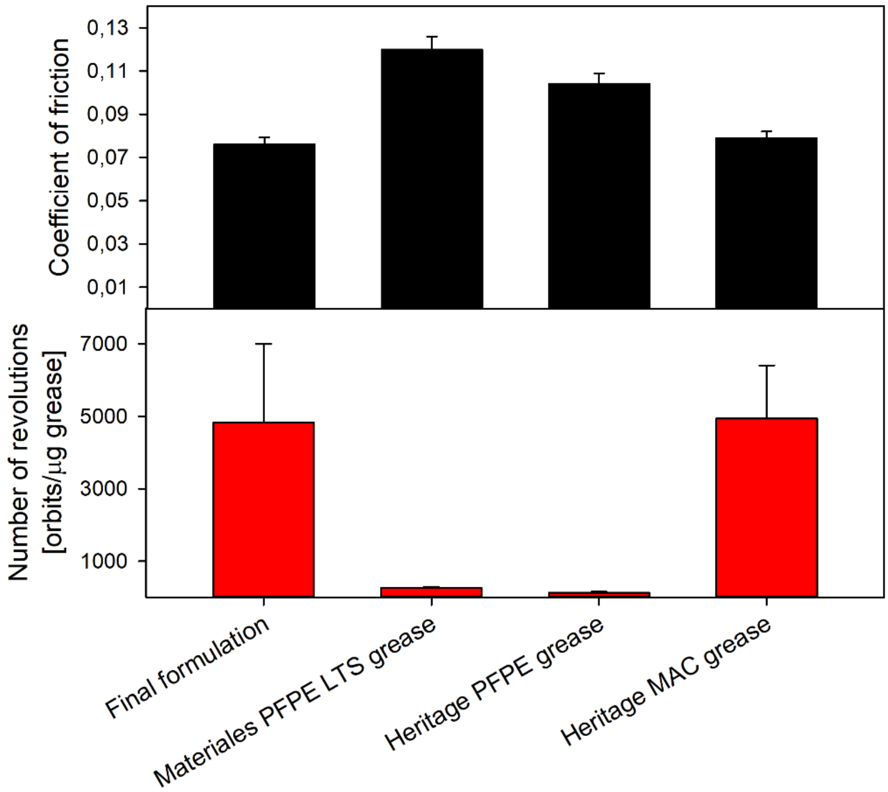

Figure 9: Depiction of SOT lifetime (in number of revolutions) and coefficient of friction for the final formulation, and comparison with Materiales LTS grease development (4000137211_21_NL_KML_rk) and heritage lubricants. Tests were conducted in high vacuum

5. Conclusions

Oil formulations based on ILs that are non-corrosive and that comply with important boundary condition parameters for space application could be developed successfully. A PFAS-free IL was identified as candidate, which performed best in the tribological evaluation. We regard the TRL of the base oil formulation to be 3-4. Until today, no completely PFAS-free greases for space application exist. It is therefore proposed to focus the further IL lubricant development towards a PFAS-free grease, which requires the development of an adequate thickener technology as the next step.