THE TRIBOLOGICAL PROPERTIES OF SEVERAL SILAHYDROCARBONS FOR USE IN SPACE MECHANISMS

W. R. Jones, Jr.¹, M. J. Jansen, L. J. Gschwender, C. E. Snyder, Jr., S. K. Sharma, R. E. Predmore, M. J. Dube

¹NASA John H. Glenn Research Center at Lewis Field, 21000 Brookpark Road, Cleveland, Ohio, 44135, (216) 433-6051, Fax: (216) 433-5170, email: William.R.Jones@grc.nasa.gov

ABSTRACT

Silahydrocarbons are members of a relatively new class of liquid lubricants with great potential for use in space mechanisms. They are unimolecular species consisting of silicon, carbon and hydrogen. They possess unique wear, viscosity and volatility properties while retaining the ability to solubilize conventional additives. The tribological properties of several members of this class, including tri-, tetra- and penta-compounds, are presented. These properties include: viscosity-temperature, viscosity-pressure, vapor pressure, lubricant lifetimes, traction, reciprocating and four ball wear rates. Lubricant lifetimes were determined using a vacuum ball bearing simulator, the spiral orbit tribometer (SOT). Wear was measured using a Cameron Plint reciprocating tribometer and wear rates with a vacuum four ball tribometer. Conventional viscometry was used for viscosity-temperature measurements and a Knudsen cell for vapor pressure. Thermogravimetric Analysis (TGA) was also used for volatility measurements. Pressure viscosity coefficients (α values) were estimated from EHL film thickness measurements. These properties are compared to existing state-of-the-art space lubricants.

INTRODUCTION

Many mechanical assemblies used in space rely on liquids to provide lubrication throughout their mission lifetimes.

The majority of current applications use mineral oils, polyalphaolefins (PAO), perfluoropolyalkylethers (PFPAE), or synthetic hydrocarbons. For a more detailed description of each of these classes of lubricants, see Reference 1. Recently, a relatively new class of lubricants, the silahydrocarbons, containing only silicon, carbon, and hydrogen have been developed (2 to 5).

Silahydrocarbons are unimolecular, have exceptionally low volatility, high viscosity indices and are available in a wide range of viscosities. There are three types: tri, tetra, and penta, based on the number of silicon atoms present in the molecule. Additionally, silahydrocarbons can accept conventional lubricant additives.

EXPERIMENTAL

MATERIALS

Six different silahydrocarbon materials were tested. These included a tri, three tetras and two pentas. Structures and designations are shown in Table 1. The three tetrasilas (SiHC-1 to 3) were synthesized at the Air Force Research Laboratory, Wright-Patterson Air Force Base, OH.. The two pentas (MJD990405 and 991029) were synthesized at Nye Lubricants, Inc., New Bedford, MA. Finally, the tri (2-94-96) and tetra (6-88-134) were prepared at Technolube Products, Los Angeles, CA.

Table 1 – Structure of Silahydrocarbons

Designation	Type	Formula
2-94-96	Tri	$(n-C_{12}H_{25})_2Si[C_8H_{16}Si(n-C_{12}H_{25})_3]_2$
MJD990405	Penta	$Si[C_3H_6Si(n-C_{12}H_{25})_3]_4$
MJD991029	Penta	$Si[C_3H_6Si(n-C_6H_{13})_3]_4$
SiHC-3	Tetra	$CH_3Si[CH_2CH_2Si-(C_6H_{13})_3]_3$
SiHC-2	Tetra	$CH_3Si[CH_2CH_2Si-(C_8H_{17})_3]_3$
SiHC-1	Tetra	$CH_3Si[CH_2CH_2Si-(C_{10}H_{21})_3]_3$
6-88-134	Tetra	$n-C_8H_{17}Si[C_3H_6Si(n-C_{12}H_{25})_3]_3$

PHYSICAL PROPERTY MEASUREMENTS

Viscosity-Temperature

Conventional viscometry was used to measure viscosity at two temperatures, 38°C and 99°C. ASTM viscosity versus temperature plots (1) for a series of silahydrocarbons appear in Figure 1. A state-of-the-art space lubricant (Pennzane P2001A) appears for comparison. Viscosity and viscosity index (VI) appear in Table 2. Extrapolated viscosity data to -20°C illustrates the advantage that silahydrocarbons possess at low temperature compared to conventional space lubricants, such as Pennzane 2001A.

Figure 1 – Kinematic viscosity as a function of temperature for a series of silahydrocarbons (1)

Table 2 – Physical properties of test fluids

Designation	Kinematic Vis. (cS)			Viscosity Index
	-20°C*	40°C**	100°C***	
2-94-96	6,000	133*	20**	177
6-88-134	4,000	105	18	183
SiHC-1	4,000	94	15	170
SiHC-2	3,000	71	12	169
SiHC-3	2,500	57	10	165
MJD991029	3,500	77	13	164
MJD990405	10,000	206	31	184
Pen. 2001A	8,500	106	14	139

* Extrapolated ** at 38°C *** at 99°C

Viscosity-Pressure

Film thickness (6) as a function of speed at 21°C for a tri and a pentasilahydrocarbon appears in Figure 2. Effective pressure-viscosity coefficients for these two fluids were estimated to be 16 ± 0.3 and 17 ± 0.3 Gpa⁻¹ at 21°C and 11 ± 1 and 13.5 ± 1 at 40°C, respectively. For comparison, Pennzane 2001A has pressure-viscosity coefficients of 11 at 21°C and 8.5 at 40°C by the same technique.

Figure 2 – Elastohydrodynamic properties for a tri- and pentasilahydrocarbon at 21°C (6)

Vapor Pressure and Volatility

Vapor pressure was measured with a Knudsen cell (7) at three temperatures, 150, 175, and 225°C. The log of the vapor pressure as a function of the reciprocal absolute temperature appear in Figure 3. Extrapolated values to room temperature appears in Table 3. Values for Pennzane P2001A and PFPAE 815Z are shown for comparison. Vacuum Thermogravimetric Analysis ($T_{1/2}$) at 33 Pa for silahydrocarbon (SiHC-1) (5) appears in Figure 4 compared to a series of conventional space lubricants.

Figure 3 – Vapor pressure (Pa) of test fluids

Table 3 – Vapor pressures (Pa) of test fluids at three temperatures and extrapolated values at 25°C (6)

Designation	225°C	175°C	150°C	25°C*
P2001A	6.1e-1	1.5e-2	2.3e-3	3.1e-10
815Z	2.0e-3	6.3e-5	3.5e-6	5.7e-14
2-94-96	6.0e-4	1.7e-4	3.1e-5	1.1e-8
6-88-134	1.6e-3	3.8e-4	8.1e-5	1.9e-8
MJD990405	6.0e-3	1.2e-4	1.9e-5	1.2e-11
MJD991029	8.8e-2	1.8e-3	1.5e-4	3.0e-12

* Extrapolated results at 25°C

Figure 4 – Volatility of candidate space lubricant base oils (5)

Traction

Traction characteristics (8) as a function of slide/roll ratio for two hydrocarbon based lubricants and a silahydrocarbon (SiHC-1) appear in Figure 5. As can be seen, the silahydrocarbon yields a lower traction coefficient than a conventional polyalphaolefin or paraffinic mineral oil.

Figure 5 – Traction data for base oils (5)

SPIRAL ORBIT TRIBOMETER

The NASA spiral orbit tribometer (SOT), was used to perform accelerated life testing on several silahydrocarbons. The SOT simulates an angular contact bearing under boundary lubrication conditions, with similar stress levels, rolling and pivoting motions at room temperature.

The SOT is shown in detail in Figure 6. It consists of two flat plates, which simulate bearing raceways, with a single 12.7 mm (1/2 in) ball sandwiched between them. The lower plate is fixed and the top plate is rotated at ~100 RPM. During rotation, the ball moves in a spiral orbit. This motion would eventually cause the ball to fall from between the raceways. A third plate, called the guide plate, is used to return the ball to its original orbit diameter every revolution. The force the ball exerts on the guide plate is measured and can be related to friction coefficient.

Figure 6 – The spiral orbit tribometer

Accelerated testing is achieved by limiting the amount of lubricant available during the test. Prior to the test, only the ball is lubricated with $\sim 50 \mu\text{g}$ of oil. During the test, all of the oil is consumed, eventually leading to an increased friction coefficient. Failure is defined as the point when the friction coefficient exceeds a predetermined value (0.28 in this case). Lifetime is defined as the number of ball orbits to failure divided by the amount of lubricant on the ball. A complete description of the tribometer can be found in Reference 9 and many other studies it was used for are described in Reference 10.

For these tests, all of the parts were made from hardened ANSI 440C stainless steel. The plates were polished to a surface roughness of less than $5 \mu\text{m}$. All parts were sequentially cleaned in an ultrasonic bath for five minutes using the following solvents: hexane, methanol, and distilled water. They were then rinsed a final time in methanol and treated for 15 minutes in a UV-ozone box [11]. The parts were removed, the ball lubricated, and then installed in the tribometer. Tests were conducted using a mean Hertzian stress of 1.5 GPa, room temperature ($\sim 23^\circ\text{C}$), a vacuum $< 1.3 \times 10^{-6}$ Pa, and a rotational speed of 200 RPM. Relative lifetimes for a series of space lubricants appear in Figure 7.

Figure 7 – Relative lifetimes at a mean Hertzian stress of 1.5 GPa of several space lubricants using the SOT

RECIPROCATING TRIBOMETER

Wear tests were conducted in a modified reciprocating tribometer fully described in Reference 12. Conditions are as follows: 100°C , a 9 mm stroke, 6 Hz, 2 hours, 2 ml test fluid, and a dry nitrogen purge. The test specimen was a 6 mm diameter x 6 mm long roller bearing pin with blended crown ends made from ANSI 52100 steel. The pin contacted a ANSI 52100 steel disk, with a 250 N load, yielding a maximum Hertzian stress of 0.7 GPa. The disks were polished to a surface roughness of 0.12 to 0.15 microns. The wear scars were oval and the area was calculated using the following:

$$area = \frac{\pi L W}{4}$$

Where L=the track length and W=the track width. The average of replicated data was 0.71 mm^2 compared to 1.02 mm^2 for Pennzane.

VACUUM FOUR-BALL TRIBOMETER

Wear tests were conducted in a vacuum four-ball tribometer. The tribometer uses a standard four-ball configuration, but uses 9.5 mm (3/8") diameter, AISI 440C stainless steel balls. The test conditions were a vacuum $< 5 \times 10^{-6}$ Torr, 200 N load, 100 RPM, and room temperature. Under these conditions, the test operates in the boundary lubrication mode. A complete description of this device appears in Reference 13.

Wear rates for several space lubricants appear in Figure 8.

Figure 8 – Vacuum 4-Ball results of several space lubricants at room temperature

VAPOR PRESSURE APPARATUS

Vapor pressure was determined using a Knudsen cell device. Vapor pressure is calculated by measuring the rate at which molecules in a system at equilibrium effuse through an opening to a vacuum (15) at various temperatures. For these tests, the following conditions were used: three temperatures (150, 175 and 225°C), a vacuum level of less than 10^{-4} Pa, and an orifice diameter of 0.28 cm. Vapor pressure is calculated using the Langmuir equation (16).

VACUUM BEARING TEST FACILITY

The bearing test facility used for these experiments is shown in Figure 9 (14). The facility uses a single angular contact bearing, which can be rotated from 0-1000 RPM. The bearing can be heated to 100°C. To determine what lubrication regime the bearing is operating in, cross-bearing resistance is also monitored. The rig also monitors bearing torque and axial load.

For these tests, a standard MPB 1219 bearing was used with 20 mg of free oil. It has ANSI 440C stainless steel balls and raceways. The bearing was operated at 600 RPM, 75°C, and with a 20 lb axial load for approximately 1000 hours at a vacuum level of 10^{-5} Pa. Under these conditions, the bearing should operate in the elastohydrodynamic regime.

Figure 9 – Bearing test facility

These long-term, elevated temperature tests were performed with a silahydrocarbon, Pennzane P2001A, and several Krytox formulations. All of the Krytox formulations were 'dry' and showed significant amounts of lubricant degradation upon completion of the test. Both the silahydrocarbon and Pennzane lubricated bearing showed no signs of degradation and still had ample free oil remaining at test conclusion. Post-test bearing condition is illustrated in Figure 10 for the silahydrocarbon base fluid.

Figure 10 – Bearing upon completion of 1,000 hour test in the bearing test facility (Pentasilahydrocarbon, 75°C, 600 RPM, 10^{-5} Pa)

SUMMARY OF RESULTS

1. Silahydrocarbon fluids are now commercially available in a variety of molecular structures and molecular weights resulting in lower and higher viscosities compared to Pennzane P2001A. Also, silahydrocarbons offer improved low temperature fluidity as compared to Pennzane.
2. Typically, silahydrocarbons yield pressure-viscosity coefficients (α) substantially greater than Pennzane, which will result in thicker EHL films.
3. Many of the silahydrocarbon structures have vapor pressures comparable to Pennzane but all are at least an order of magnitude greater than the gold standard for space lubrication (815Z).
4. In general, silahydrocarbons provide lower traction characteristics compared to paraffinic mineral oils or PAOs.
5. Relative lifetimes in vacuum measured in a ball bearing simulator (SOT) were about 1/3 the life of Pennzane, but an order of magnitude greater than PFPAEs (815Z and Z25).
6. From vacuum four ball wear measurements, a silahydrocarbon yielded a wear rate comparable to Pennzane and two orders of magnitude less than PFPAEs.
7. Reciprocating wear tests in dry nitrogen showed improved results with a silahydrocarbon as compared to Pennzane.
8. 1000 hour vacuum bearing tests at 75C yielded excellent results compared to a PFPAE (Krytox 143 AB) and comparable results to Pennzane.

DISCUSSION

The results of the above tests indicate that the silahydrocarbon class of liquid lubricants compare favorably in most areas to current space lubricants, such as Pennzane P2001A and Castrol 815Z and in some areas surpass these state-of-the-art materials.

Silahydrocarbon based lubricants can also be formulated with antiwear additives and work with commercial additives is ongoing. It is expected these additives will improve silahydrocarbon wear characteristics further.

Currently, a vacuum life test is underway on instrument scanner ball bearings at Lockheed Martin Missiles and Space using a formulated tetrasilhydrocarbon (SiHC-3). Life test details appear in Reference 17.

CONCLUSION

The silahydrocarbon class of lubricants are now commercially available and should be considered as a viable alternative for future lubrication requirements for space mechanisms, particularly for low temperature applications.

REFERENCES

1. Jones, W.R., Jr., Jansen, M.J., "Space Tribology", NASA TM 2000-209924, March, (2000).
2. Snyder, C.E., Jr., Gschwender, L.J., Randolph, B.B., Paciorek, K.J.L., Shih, J.G., Chen, G.J., "Research and Development of Low-Volatility Long Life Silahydrocarbon-Based Liquid Lubricants for Space", *Lub. Engr.*, **48**, 4, pp 325-328, (1992).
3. Paciorek, K.J.L., Shih, J.G., Kratzer, R.H., Randolph, B.B. and Snyder, C.E., Jr., "Polysilahydrocarbon Synthetic Fluids. 1. Synthesis and Characterization of Trisilahydrocarbons", *I & EC Research*, **29**, pp 1855-1858, (1990).
4. Paciorek, K.J.L., Shih, J.G., Kratzer, R.H., Randolph, B.B. and Snyder, C.E., Jr., "Polysilahydrocarbon Synthetic Fluids. 2. Synthesis and Characterization of Tetrasilhydrocarbons", *I & EC Research*, **30**, pp 2191-2194, (1991).
5. Gschwender, L.J., Snyder, C.E., Jr., Massey, M. and Peterangelo, S., "Improved Liquid/Grease Lubricants for Space Mechanisms", *Lubr. Engr.*, **56**, 12, pp 25-31, (2000).
6. Spikes, H.A., "Estimation of the Pressure-Viscosity Coefficients of Two Silahydrocarbon Fluids at Two Temperatures", Imperial College of Science, Technology & Medicine, Tribology Section, London, UK, Report TS024b/96, (1996).
7. Nguyen, Q.N., Jones, W.R., Jr., "Volatility and Wear Characteristics of a Variety of Liquid Lubricants for Space Applications", to be published, *Trib. Trans.*
8. Sharma, S.K., Snyder, Jr., C.E. and Gschwender, L.J., "Tribological Behavior of Some Candidate Advanced Space Lubricants", *Trib. Trans.*, **36**, pp 321-325, (1993).
9. Pepper, S.V., Kingsbury, E.P., Ebihara, B.T., "A Rolling Element Tribometer for the Study of Liquid Lubricants in Vacuum", NASA TP 3629, October, (1996).
10. Jones, W.R., Jr., Pepper, S.V., Jansen, M.J., Nguyen, Q.N., Kingsbury, E.P., Loewenthal, S.,

- Predmore, R.E., "A New Apparatus to Evaluate Lubricants for Space Applications – The Spiral Orbit Tribometer (SOT)", International Spring Fuels & Lubricants Meeting & Exposition", SAE Tech. Paper 2000-01-1828, Paris, France, June 19-22, (2000).
11. Vig, J.R., "UV-Ozone Cleaning of Surfaces", *J. Vac. Sci. Tech. A*, 3, pp 1027-1034, May/June 1985.
 12. Cavdar, B., Sharma, S.K., and Gschwender, L.J., "Wear-Reducing Surface Films Formed by a Fluorinated Sulfonamide Additive in a Chlorotrifluoroethylene-based Fluid", *Lube. Eng.*, 50, 11, pp 895-902, (1994).
 13. Masuko, M., Jones, W.R., Jr., Jansen, R.H., Ebihara, B.E., Pepper, S.V., Helmick, L., "A Vacuum Four-Ball Tribometer to Evaluate Liquid Lubricants for Space Applications", *Lub. Engr.*, 5, 11, pp 871-875, (1994).
 14. Jansen, M.J., Jones, W.R., Jr., Predmore, R.E., "Evaluation of Temperature and Material Combinations on Several Lubricants for Use in the Geostationary Operational Environmental Satellite (GOES) Mission Filter Wheel Bearings", NASA TM in process, (2001).
 15. Margrave, J.L., *The Characterization of High Temperature Vapors*, John Wiley & Sons, New York (1967).
 16. Dushman, S., *Scientific Foundations of Vacuum Technique*, Second Edition, John Wiley & Sons, New York (1962).
 17. Loewenthal, S., Jones, W.R., Jr., Predmore, R., "Life of Pennzane and 815Z-Lubricated Instrument Bearings Cleaned with Non-CFC Solvents", 8th European Space Mechanisms and Tribology Symp., SP-438, pp 225-231, (1999).