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VOLATILITY AND WEAR CHARACTERISTICS OF A VARIETY OF LIQUID LUBRICANTS FOR SPACE APPLICATIONS

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Abstract

The vapor pressures and wear characteristics are critical properties for liquid lubricants to assure long-term reliability and performance in space applications. Vapor pressures, obtained using a Knudsen cell technique, and wear properties, obtained using a vacuum four-ball apparatus, were measured for a series of unformulated liquid lubricants. These included: two multiply alkylated cyclopentanes (MACs) (X-1000 and X-2000), two linear perfluoropolyalkylethers (PFPAEs) (Z-25 and 815Z), and four silahydrocarbons (a tri-, a tetra- and two pentas). Vapor pressures were measured at three elevated temperatures (423, 448 and 498K) and extrapolated to room temperature 298K. The lowest 298K vapor pressure of 5.7×10^{-14} Pa, was obtained with the PFPAE fluid (815Z) and the highest value with the low molecular weight MAC (X-1000) at 3.6×10^{-7} Pa. In addition, vacuum wear rates were determined for some of the lubricants. The lowest wear rates (approximately 3×10^{-11} mm^3/mm) were observed for three of the silahydrocarbons while the highest wear rates (approximately 2×10^{-9} mm^3/mm) were observed with the two PFPAE fluids (Z-25 and 815Z). The MAC (X-2000) yielded a wear rate of about 10^{-10} mm^3/mm. The results indicated that the silahydrocarbon class of liquid lubricants offers the better potential for space applications.

Keywords

Fluorocarbons, Hydrocarbons, Space Vehicles, Synthetic Lubricants, Vapor Pressure, Wear Testing Device

Introduction

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Space applications require lubricants that have low vapor pressures, low creep rates, high viscosity indices (VI), good viscosity-pressure coefficients, low wear rates, and low tribochemical degradation rates (Jones, 1995), (Jones and Jansen, 2000), (Zaretsky). The lubricant class of choice for many space applications is the perfluoropolyalkylethers (PFPAEs), due to their low vapor pressures and high viscosity indices (Jones, 1995). However, PFPAEs have been shown to suffer degradation under boundary lubrication conditions that have been attributed to reactions with steel bearing surfaces (Jones, 1995; Carré, 1986). The steel surfaces are converted to FeF_3, which further catalyzes the degradation of the PFPAE. To avoid this degradation process, other lubricants that do not contain fluorine, such as silahydrocarbons are under investigation. The structures of silahydrocarbons vary depending on the number of silicon atoms, as specified in the names of the compounds. For example, the four silahydrocarbons studied in this paper varied in nomenclature from tri-, tetra-, to penta-silahydrocarbon. The differences in molecular weights should correlate to the viscosity of the lubricant, a critical parameter in selecting a lubricant (Sharma, et al). The availability of various viscosities (structures) facilitates the blending of these oils to arrive at an optimum viscosity for the selected application.

Two multiply alkylated cyclopentanes (MACs) (Venier and Casserly) were studied: the standard tri-substituted space lubricant Pennzane X-2000, as well as a lower molecular weight, di-substituted version X-1000. This less viscous fluid (X-1000) is primarily used for applications at low temperature. Consequently, this fluid is expected to have a much higher vapor pressure. The formulated version of the X-2000 fluid has performed very well in space as well as in land base life tests (Dietz et al.; Brown et al.).

Silahydrocarbons are unimolecular materials containing either three (Snyder et al.; Pettigrew; Paciorek et al., 1990), four (Paciorek et al., 1991) or five silicon atoms. However, most of the molecule is made up of carbon and hydrogen, so they solubilize conventional additives like ordinary hydrocarbons. They contain no oxygen and therefore are not silicone-based, which typically perform poorly in space applications. Due to their unimolecular nature, they should possess much better volatility characteristics than ordinary hydrocarbons (i.e. polyalpha olefin or mineral oils, which have a range of molecular weights). They should also exhibit constant viscosity even if some of the fluid is lost to evaporation.

As mission lifetimes increase, more burden will be placed on lubricant systems for moving mechanical assemblies. Since lubrication is necessary for sliding or rolling surfaces, lubricant loss due to evaporation can result in component failure. This study was conducted to investigate the vapor pressures of several unformulated liquid lubricants from various manufacturers including MACs, PFPAEs (Sianesi, et al.), and silahydrocarbons at temperatures of 423, 448, and 498K using a Knudsen cell apparatus. The measured vapor pressures were extrapolated to room temperature (298K) (Jones and Jansen, 2000) to make preliminary determinations of the volatility rates. When sufficient amounts of lubricant were available, room temperature wear rates were determined using a vacuum four-ball apparatus (Masuko et al.; Jones et al., 1999). The vacuum four-ball results will also aid in the preliminary determination of desirable space lubricants under boundary lubrication conditions by measuring wear rates.

Experimental Materials

The MACs, PFPAEs, and silahydrocarbons used in this study are listed in Table 1 along with some basic property data and the manufacturing source. All fluids are unformulated and the fluids were tested as received. The four silahydrocarbons were used in this study to allow for an examination of molecular weight effects and differing structures. The molecular weights were calculated from the molecular formulas with the exception of the two PFPAEs (Z-25 and 815Z), which was provided by the manufacturers. Table 2 provides the kinematic viscosities and viscosity indices for all test fluids.

Two MACs (X-1000 and X-2000) were studied: the standard tri-substituted space lubricant Pennzane X-2000, as well as a lower molecular weight, di-substituted version X-1000. Two linear PFPAEs (Z-25 and 815Z) were studied. These PFPAEs are commercial mixtures of perfluorinated polymeric chains and have an average molecular weights of about 9500 grams/mole. The 815Z fluid has undergone additional manufacturer processing and therefore may have a narrower distribution to remove low molecular weight impurities. Four silahydrocarbons (2-94-96, 6-88-134, MJD990405, and MJD991029) were chosen for this paper in order to study the behavior of its molecular weight range.

MACs are either unimolecular compounds or mixtures of two or three components. Gel permeation chromatography (GPC) was performed on X-2000 and the four silahydrocarbons. The X-2000 analysis indicated the primary peak to be the trisubstituted compound. GPC analysis of the two pentasilahydrocarbons yielded single peaks having polydispersities for the C_6 compound (MJD991029) of 1.02 while that of the C_{12} (MJD990405) was 1.16.

Infrared analysis of the two pentasilahydrocarbon compounds indicated the absence of both Si-H and Si-O-Si impurities. The tri and tetrasilahydrocarbons were synthesized under an Air Force Materials Laboratory program. The synthesis and characterization of these two compounds are reported in Paciorek et al., 1990 and

1991. No volatile impurities in the trisilahydrocarbon were detected by gas chromatographic (GC) analysis, and in support of this finding, no weight loss was observed below 473K under vacuum TGA.

Experimental Methods

Vapor Pressure

A number of techniques, that are all based on weight loss due to evaporation, can be used to determine the volatilities of space lubricants including: Knudsen cell (Daniels and Alberty); thermogravimetric analysis (TGA) (Scialdone); and non-equilibrium weight loss measurements under vacuum (Carré and Bertrand, 1999). The classical Knudsen cell technique was chosen for these experiments, in which one measures the weight loss of an oil sample, due to the effusion of its vapor through a small hole into a vacuum.

Vapor pressures were determined by the weight loss from a brass cell, shown in Figure 1. Approximately 100 to 400 mg of the liquid was placed in this cell. The exploded view in Figure 1 depicts the Knudsen cell (approximate weight is 20 grams), where the oil is placed in the cell cup; an orifice disk covers the cup in order to define the vapor escape route. A slip cover cell cap holds the cup and the orifice disk in place, thus allowing vapors to exit through only one opening. The orifice diameter of the disk was 0.28 cm, and the disk thickness was 0.06 cm, which corresponds to a Clausing factor of 0.82. The Clausing factor takes into account the thickness of the orifice where gases hit the edge of this opening at angles that allow the molecules to be deflected back into the sample cup. Oil samples in the Knudsen cell were loaded into the heating chamber, which had been preheated to 25K less than the desired test temperature. The system was evacuated and heat up to the test temperature was initiated. Approximately 8-12 minutes elapsed for pump evacuation, before the test temperature was established and test time was started. The measured pressure was less than 1×10^{-4} Pa for the duration of the test. Test times were from 30 minutes to 65 hours and the weight loss ranged from 0.1 - 80 mg. The entire Knudsen cell shown in Figure 1 was weighed. The sample weight loss was determined using a Mettler AT20 electronic balance to the nearest ± 2 μg. Each reported weight represents the average of five individual weighings. The weight loss data was used to calculate the vapor pressure by the Langmuir equation (Dushman, Equation 1.57):

$$P = \frac{(17.14)(g)(\sqrt{T})}{AtF\sqrt{M}} \tag{1}$$

where P is the calculated vapor pressure in torr; g is the weight loss in grams; T is temperature in Kelvin; A is the area of the orifice in cm^2; t is time in seconds; F is the Clausing factor; and M is the molecular weight in grams/mole.

Test temperatures of 423, 448, 498K were used. The test chamber's nitrogen purge was activated when it was not under vacuum to minimize moisture condensation from the air. After each test, the Knudsen cell was ultrasonically cleaned (10 min each) in a series of solvents. The cleaning procedure used after a test for hydrocarbon based oils was three washes in hexanes followed by one wash in methanol. After fluorinated oils were used, cleaning was done with three washes in 1,1,2-trichlorotrifluorethane followed by one wash in methanol.

Wear Rates

When a sufficient amount of lubricant was available for additional testing, a modified vacuum four-ball apparatus shown in Figure 2, was used to determine wear rates. AISI 440C stainless steel, grade 25, 0.953 cm diameter balls were used. The balls were ultrasonically cleaned in solvent baths of hexanes, acetone, and methanol, sequentially for 10 minutes each. As shown in Figure 3, the top ball is mounted on a rotating spindle and was connected to a rotary vacuum feed-through. The sample cup, containing three stationary balls, was mounted on a stage which moved upward with a pneumatic loading system to contact the top-rotating ball. Test conditions were a load of 200 Newtons, a pressure less than 6.7×10^{-4} Pa and a speed of 100 rpm. The sample cup, containing the three stationary balls, was filled with approximately 4 mL of oil and then placed in a separate glass jar connected to a mechanical vacuum pump to remove dissolved air. After degassing for one hour, the sample cup was removed and immediately placed in the vacuum four-ball apparatus. At one-hour intervals, wear diameters on the bottom three stationary balls were measured with an optical microscope modified to accommodate the sample cup without removing the balls. The expression for the wear volume (equation 2), assuming a flat circular wear surface can be derived from analytic geometry and is given as follows:

$$\frac{\pi}{3}\left[2R^3 - (2R^2 + r^2)\sqrt{R^2 - r^2}\right] \tag{2}$$

where R = radius of the ball and r = radius of the wear scar. The total test length was four hours, resulting in four measurements (one per hour). These measurements were used to calculate an average wear volume that was plotted as a function of sliding distance. Average wear rates (mm^3/mm) were calculated from the slope of the straight-line fits.

A computer system (Labview™) controlled start up and shut down when pre-established limits had been reached. When the chamber was not under vacuum, a nitrogen purge was activated to minimize the amount of moisture condensation from the air. For each lubricant change, the sample cup was cleaned in an ultrasonic bath for 10 minutes with hexanes for hydrocarbon-based lubricants or 1,1,2-trichlorotrifluorethane for fluorinated lubricants. Following this wash, a levigated alumina slurry was used to scrub all sample cup surfaces. The sample cup was rinsed with deionized water, followed by another 15 - 20 minutes ultrasonic bath in water. The assembly was dried with nitrogen and then sequentially cleaned in ultrasonic baths of hexanes, acetone and methanol for 10 minutes each.

Results and Discussion:

Vapor Pressure

Table 3 lists the results of the measured vapor pressures at 373, 423, 448, and 498K along with the vapor pressure extrapolated to room temperature (298K). Measured data at each temperature, for each lubricant, are shown in two charts for ease of readability: Figure 4 contains data for MACs and PFPAEs; Figure 5 contains data for silahydrocarbons. The slope of each line in the figures, represents an Arrhenius dependency, and was used to determine the vapor pressure predicted at room temperature. The four silahydrocarbons' measured vapor pressures range between MACs and PFPAEs. As shown in Figure 5, MJD990405 and MJD991029 both exhibit a higher temperature dependence, which results in lower vapor pressures when extrapolated to room temperature. An upper limit for the room temperature vapor pressure at the 80% confidence interval was determined and is included in Table 3 (Keller). One would expect the higher the molecular weight, the lower the volatility, however for silahydrocarbons, not all the vapor pressures correspond with the molecular weights trends. As mentioned earlier, GPC analysis of the two pentasilahydrocarbons indicate minimum purities for both compounds, but with the C_{12} compound being less pure than the C_6. The C_{12} compound is more likely to be a mixture due to steric hindrance. Because the C_6 is distillable, there is no catalyst present in the material. On the other hand, the C_{12} compound may contain traces of catalyst not removed in the final cleaning step of filtration through a fluorosil column. This indicates that the C_{12} compounds are less pure than the C_6 counterpart.

In Figure 6, extrapolated room temperature vapor pressures for all materials are shown. The 815Z (PFPAE) has the lowest extrapolated room temperature vapor pressure of 5.7 x 10^{-14} Pa, therefore indicating the best performance in volatility measurements for this study. The lowest vapor pressure reported for the silahydrocarbon is MJD991029 at 3.0x10^{-12} Pa. The second PFPAE, Z-25, at 1.2 x 10^{-11} Pa is comparable to the second lowest silahydrocarbon, MJD990405 of 1.2 x 10^{-11} Pa. As with PFPAEs and silahydrocarbons, there is some overlap in vapor pressure values between silahydrocarbons and MACs. Overall, the two pentasilahydrocarbons (MJD990405 and MJD991029) with the lowest vapor pressures are comparable to a PFPAE (Z-25) which is the lubricant class used in current space applications.

Vapor pressure measurements were obtained from the three manufacturers at two room temperatures, as indicated in Table 1. Comparing these values to the ones reported (Table 3) in this study, via the Knudsen cell method, the manufacturers' vapor pressure measurements are one to three orders of magnitude greater. These poor agreements may be due to uncertainties in the extrapolations to room temperatures. Readers must take this into account when evaluating these comparisons. Confidence intervals are also reported in Table 3 for the vapor pressure measurements found in this paper.

The results from this study can be compared to those found in the literature. Snyder reports results of volatility measurements for several lubricants that were obtained by TGA techniques. Using Snyder's TGA result for a silahydrocarbon plot with a molecular weight of 1704 g/mole, the rate of weight loss (g/s) can be estimated to range from 1.3 x 10^{-5} - 2.7 x 10^{-5} g/s at 660K, in a nitrogen containing environment at 56 Pa. Since no further TGA plots were shown, the other tested silahydrocarbons and the PFPAE volatility rates could not be estimated. For comparison, the lowest and highest rates of weight loss of the four silahydrocarbons studied in this paper were calculated using equation (1) and the vapor pressures from Figure 5 extrapolated to 660K. The silahydrocarbon with the lowest extrapolated weight loss at 660K is 2-94-96 (with a molecular weight of 1663 g/mole) with a rate of 1.4x10^{-4} g/s; the highest extrapolated weight loss demonstrated by MJD991029 (with a molecular weight of 1330g/mole) is 7.4x10^{-3}g/s. The weight loss rate range in this paper, using a Knudsen cell, is approximately an order of magnitude greater than the findings reported by Snyder via TGA technique. The difference may be attributed to: 1) Only one TGA plot was reported therefore, a range of weight loss for all tested samples could not be made. 2) Assumptions were made in order to estimate Snyder's weight loss rates such as the actual temperatures and time when the oil began to lose weight and when all the oil was gone. 3) Other discrepancies in the comparison may be linked to differing vacuum conditions and surface area of the sample holders as well as expected differences due to use of two independent volatility measurement techniques.

An evaporation method, which is not a Knudsen technique, was initially used by Snyder but later abandoned. However, some weight loss results were obtained using this technique, for a PFPAE and two silahydrocarbons at 423K, for times of 72 hours at 60 Pa. The reported percent weight loss for PFPAE was 0.1% as compared to 815Z at 0.04% and Z-25 at 0.09% from our study. The weight loss of two silahydrocarbons studied in Synder's paper ranged from 4.3 to 8.0%. Our weight loss range on the lowest vapor pressure lubricant MJD990405, at 423K is 0.11% and on MJD991029, the highest vapor pressure is 0.86%. Agreement between the two studies is reasonable considering the unknown surface area differences, unknown molecular weights of Snyder's samples, and the two different vacuum conditions.

Wear Rates

Results for a typical set of silahydrocarbon (MJD991029) wear tests are shown in Figure 7, where the average wear volume is plotted versus the sliding distance. Wear rates (mm^3/mm) are calculated by averaging the slopes of these straight lines. Averaging the slopes reduced uncertainties in reported wear rates due to normal variation in test set-up, which are reflected in the variable intercepts for each trial. Three to four tests were performed for each lubricant. Vacuum four-ball mean wear rates and standard deviations at room temperature are shown in Figure 8. The data demonstrate that all evaluated silahydrocarbon oils have lower wear rates compared to the MAC and PFPAEs. The average wear measurement for all silahydrocarbons is approximately 3×10^{-11} mm^3/mm, while the MAC (X-2000) is an order of magnitude greater, and the PFPAEs are greater by two orders of magnitude at 2×10^{-9} mm^3/mm. It should be noted that these wear rates are for unformulated base fluids. Since the silahydrocarbon and MAC lubricant classes solubilize conventional antiwear additives, their formulated versions are expected to exhibit lower wear rates than the corresponding base fluids.

Conclusion:

Vapor pressures and wear rates were determined for liquid lubricants including MACs, PFPAEs, and silahydrocarbons. PFPAEs have been historically the lubricant of choice for space applications; however, volatility and degradation are key factors in the life expectancies of these fluorinated lubricants. Silahydrocarbons compare favorably in vapor pressure results and yield much lower wear rates. These findings indicate that silahydrocarbons should be further investigated as possible candidates for space applications due to their excellent performance.

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Table 1. Designated Names and Type of Liquid Lubricants.

Manufacturer	**Designated Name**	**Type**	**Molecular Formula**	**Vapor Pressure (Pa) at 293K Vendor Data**	**Molecular Weight (g/mole)**
Pennzoil	X-1000	Hydrocarbon (MAC)	$C_{45}H_{90}$		630
Pennzoil	X-2000	Hydrocarbon (MAC)	$C_{65}H_{130}$	$^{+}4.7 \times 10^{-9}$	910
Ausimont	Z-25	PFPAE	$CF_3\text{-}(OCF_2CF_2)_n\text{-}(OCF_2)_n\text{-}OCF_3$	3.9×10^{-10}	9,500
Castrol	815Z	PFPAE	$CF_3\text{-}(OCF_2CF_2)_x\text{-}(OCF_2)_y\text{-}OCF_3$	5.3×10^{-11}	9,500
Technolube	2-94-96	Trisilahydrocarbon	$(C_{12}H_{25})_2Si[C_8H_{16}Si(C_{12}H_{25})_3]_2$		1,663
Technolube	6-88-134	Tetrasilahydrocarbon	$n\text{-}C_8H_{17}Si[C_3H_6Si(n\text{-}C_{12}H_{25})_3]_3$		1,876
Nye Lubricants	MJD990405	Pentasilahydrocarbon	$Si[(CH_2)_3Si(n\text{-}C_{12}H_{25})_3]_4$		2,340
Nye Lubricants	MJD991029	Pentasilahydrocarbon	$Si[(CH_2)_3Si(n\text{-}C_6C_{13})_3]_4$		1,330

$^{+}$Nye Lubricants Vapor Pressure Method at 298K

Table 2. Viscosity Properties of Test Fluids.

Designated Name	Kinematic Viscosity, cs @313K	Kinematic Viscosity, cs @373K	Viscosity Index	Polydispersity Data from Nye
X-1000	50	8.3	140	NM
X-2000	108	15	137	1.12
Z-25	155	47	355	NM
815Z	148^{+}	45^{++}	350	NM
2-94-96	133^{+}	20.4^{++}	177	1.14
6-88-134	105	17.5^{++}	183	1.15
MJD990405	206	31.2	184	1.16
MJD991029	76.7	12.6	164	1.02

$^{+}$ 311K

$^{++}$372K

NM = Not Measured

Table 3. Vapor Pressure Measurements (Pa) at 373, 423, 448, and 498K. Numbers in [ ] Indicate the Number of Tests.

Designated Name	**498K**	**448K**	**423K**	**373K**	***298K**	****298K Max**
X-1000	–	1.2[1]	6.0×10^{-1}[1]	3.3×10^{-3}[1]	3.6×10^{-7}	1.1×10^{-5}
X-2000	6.1×10^{-1}[2]	1.5×10^{-2}[3]	2.3×10^{-3}[3]	---	3.1×10^{-10}	5.1×10^{-10}
Z-25	2.0×10^{-3}[2]	2.2×10^{-4}[2]	1.1×10^{-5}[1]	---	1.2×10^{-11}	7.3×10^{-10}
815Z	2.0×10^{-3}[2]	6.3×10^{-5}[3]	3.5×10^{-6}[4]	---	5.7×10^{-14}	2.8×10^{-13}
2-94-96	6.0×10^{-4}[2]	1.7×10^{-4}[3]	3.1×10^{-5}[2]	---	1.1×10^{-8}	4.6×10^{-8}
6-88-134	1.6×10^{-3}[2]	3.8×10^{-4}[2]	8.1×10^{-5}[3]	---	1.9×10^{-8}	5.7×10^{-8}
MJD990405	6.0×10^{-3}[3]	1.2×10^{-4}[3]	1.9×10^{-5}[3]	---	1.2×10^{-11}	2.1×10^{-10}
MJD991029	8.8×10^{-2}[1]	1.8×10^{-3}[1]	1.5×10^{-4}[1]	---	3.0×10^{-12}	6.4×10^{-12}

*298K Represents Extrapolated Results.

**298K Max Represents the 80% Upper Confidence Predicted Limit of *298K (Keller).

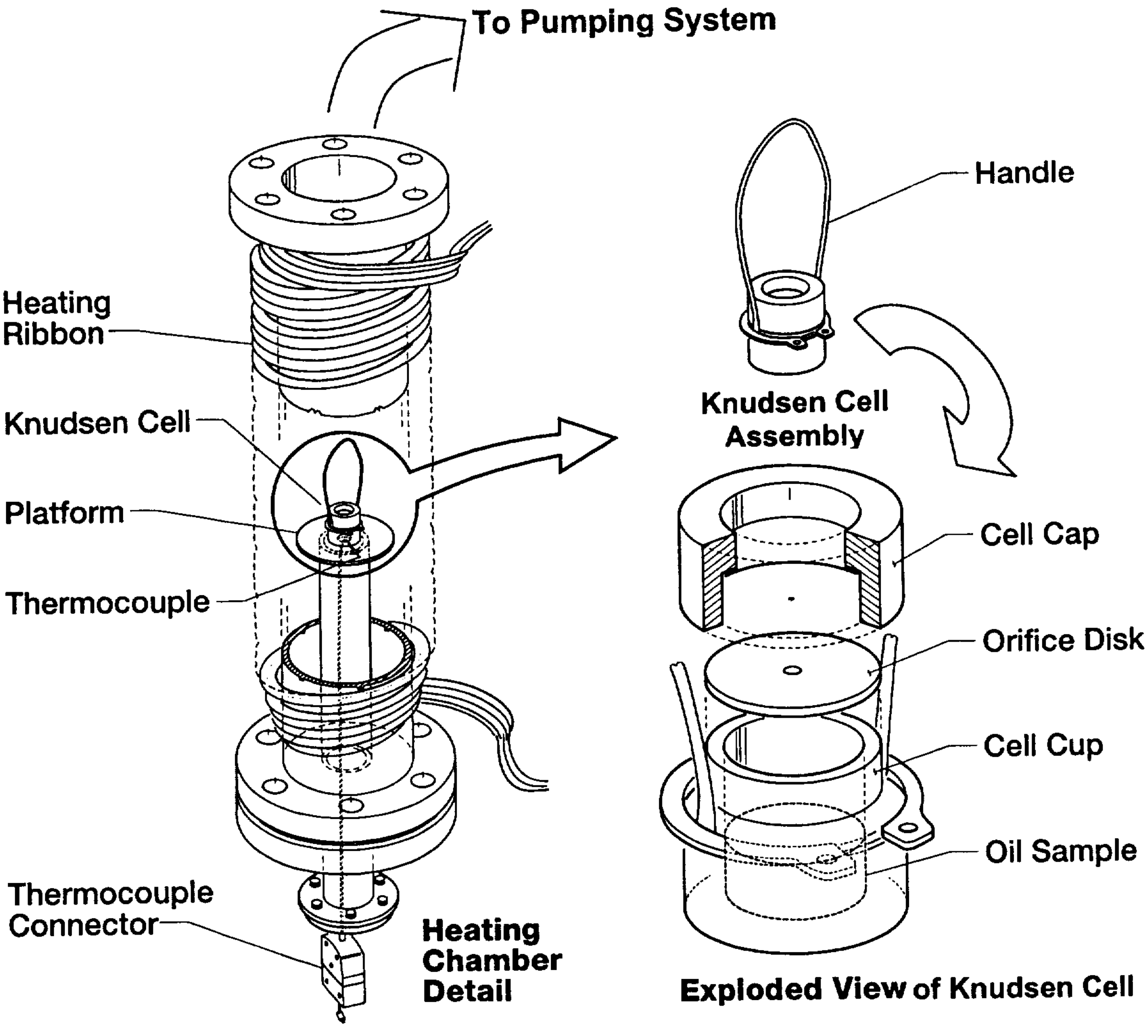

Figure 1. Knudsen Cell Schematic

Figure 1. Lead Author: QuynhGiao N. Nguyen

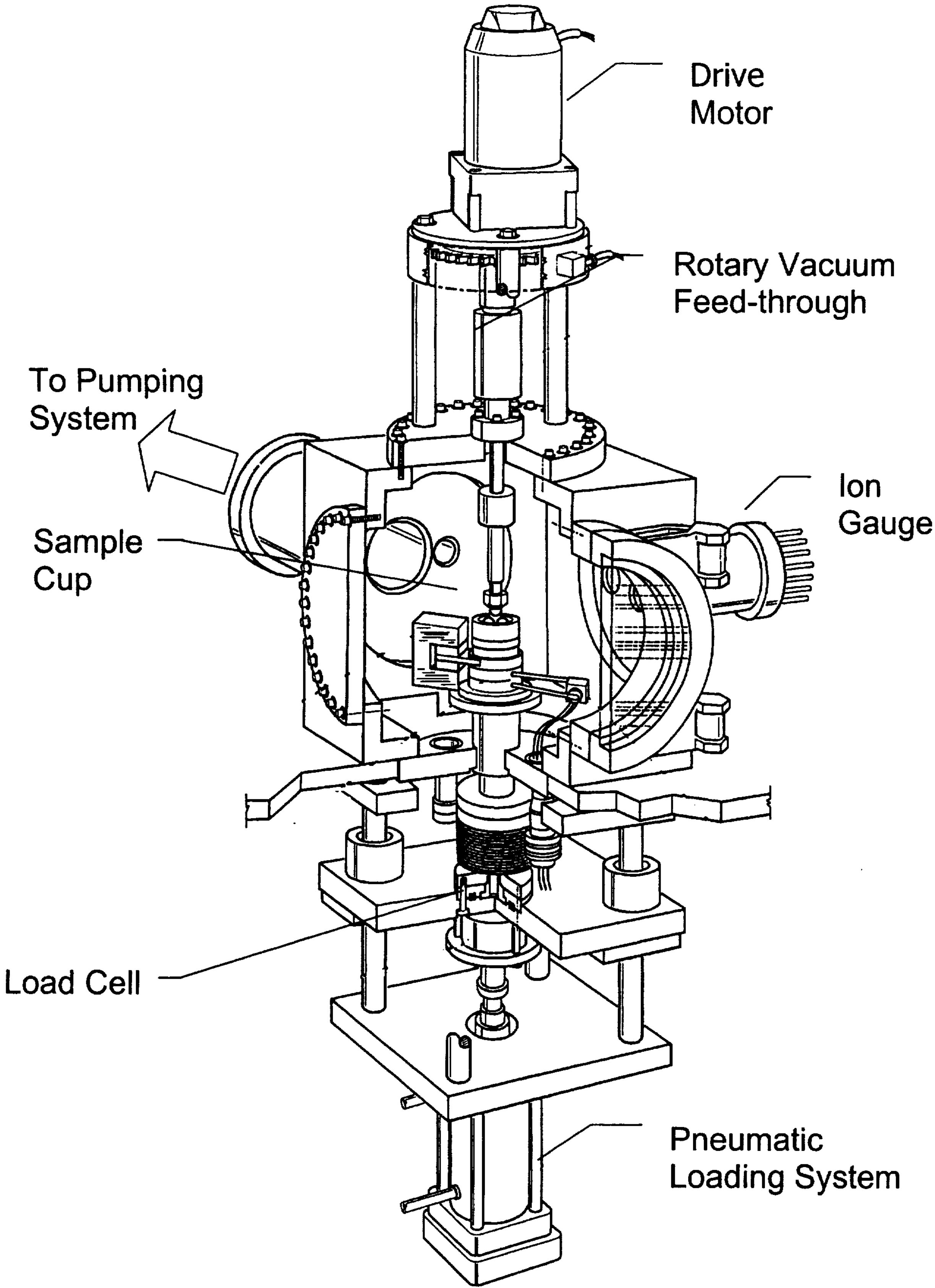

Figure 2. Vacuum Four-Ball Schematic

Figure 2. Lead Author: QuynhGiao N. Nguyen

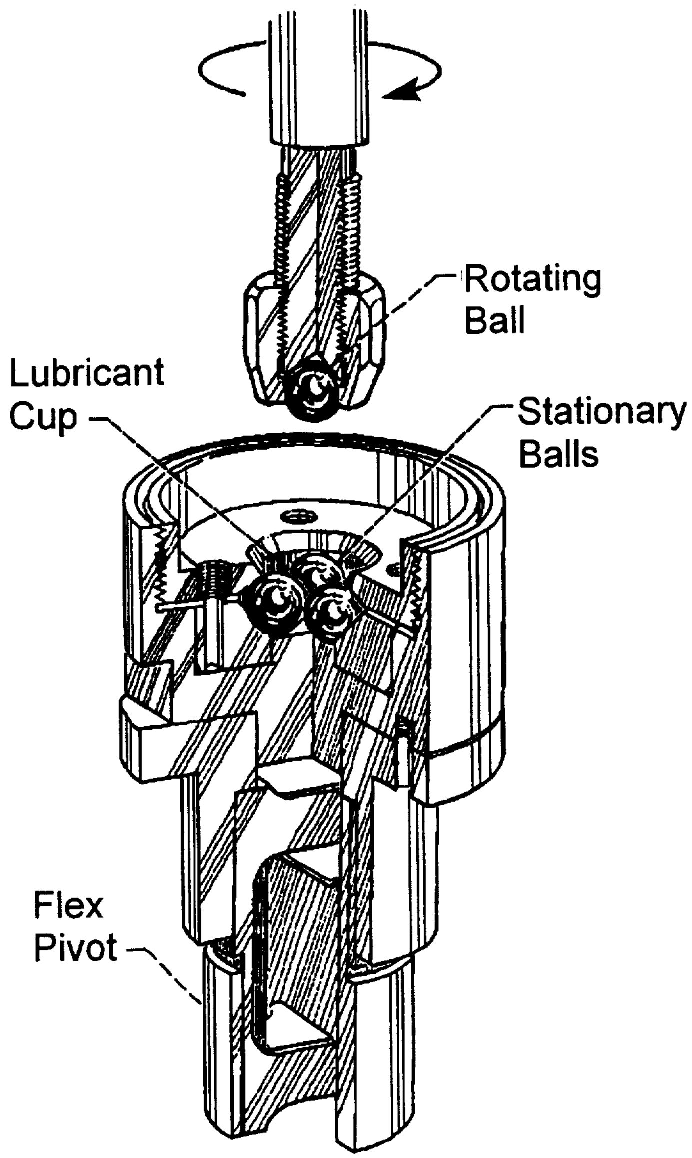

Figure 3. Detail of Four-Ball Sample Cup

Figure 3. Lead Author: QuynhGiao N. Nguyen

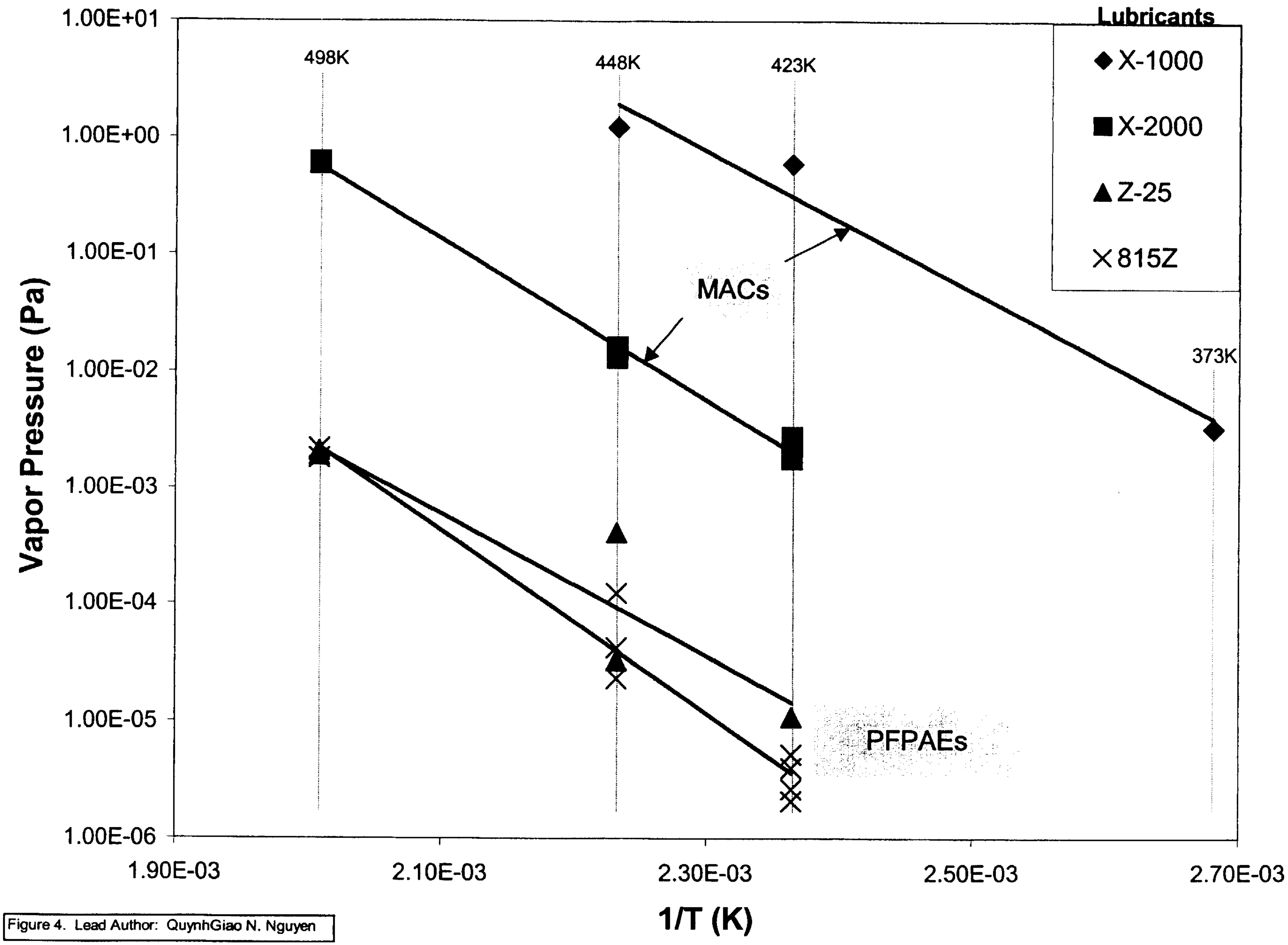

Figure 4. Lead Author: QuynhGiao N. Nguyen

Figure 4. Vapor Pressure Measurements of Multiply Alkylated Cyclopentanes (MACs) and Perfluoropolyalkylethers (PFPAEs).

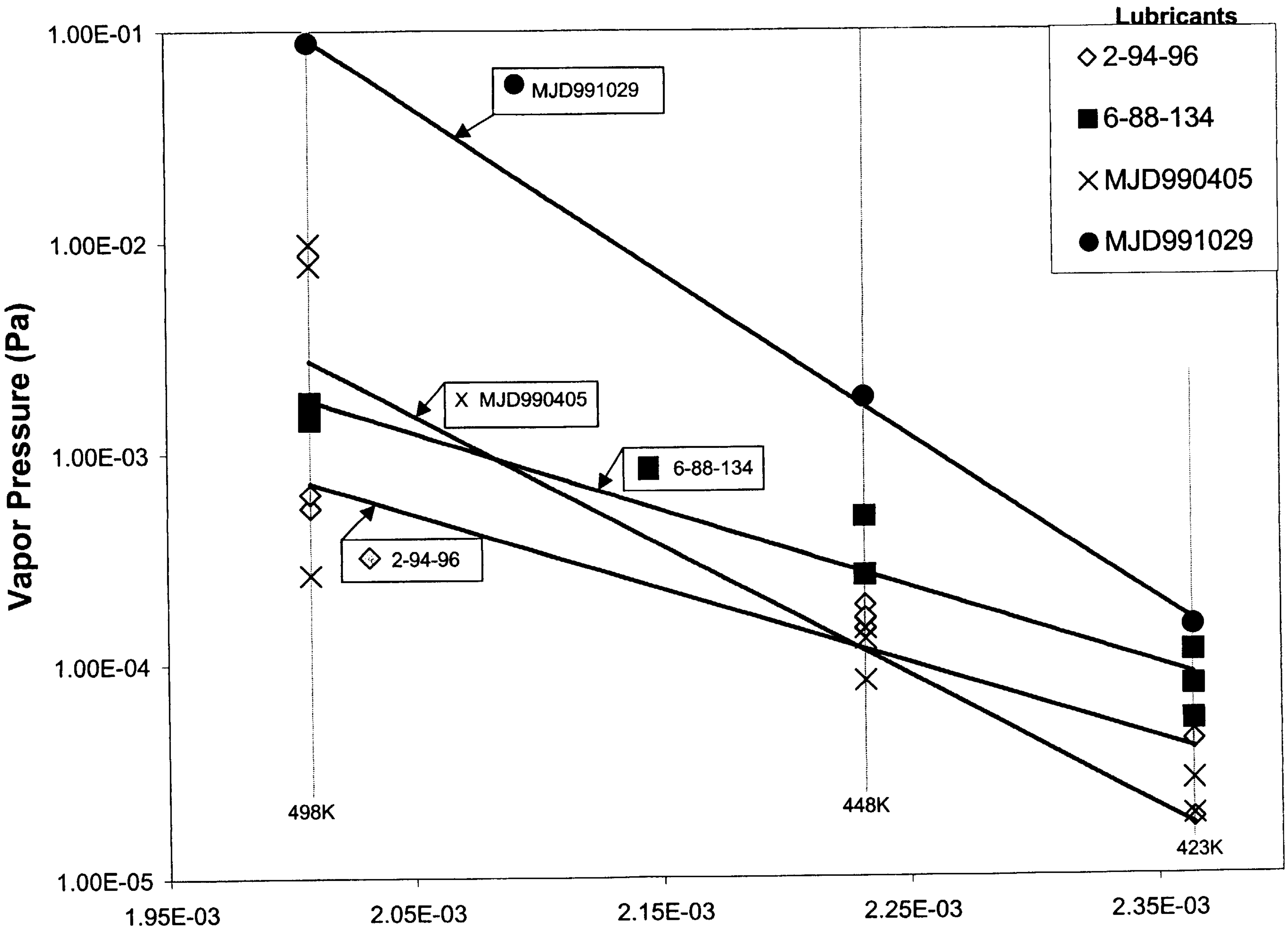

Figure 5. Lead Author: QuynhGiao N. Nguyen

Figure 5. Vapor Pressure Measurements of Silahydrocarbons.

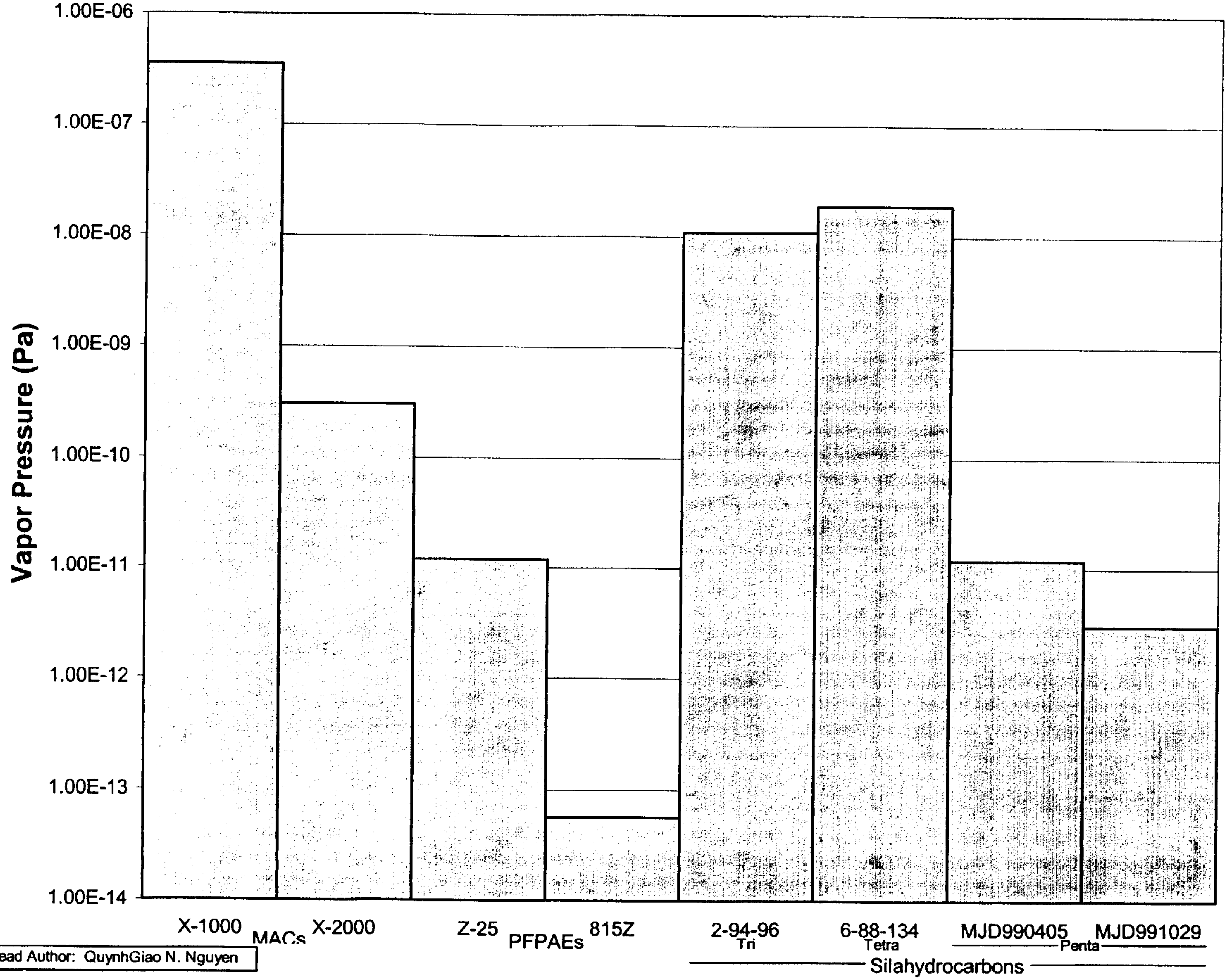

Figure 6. Lead Author: QuynhGiao N. Nguyen

Figure 6. Extrapolated Vapor Pressures at 298K.

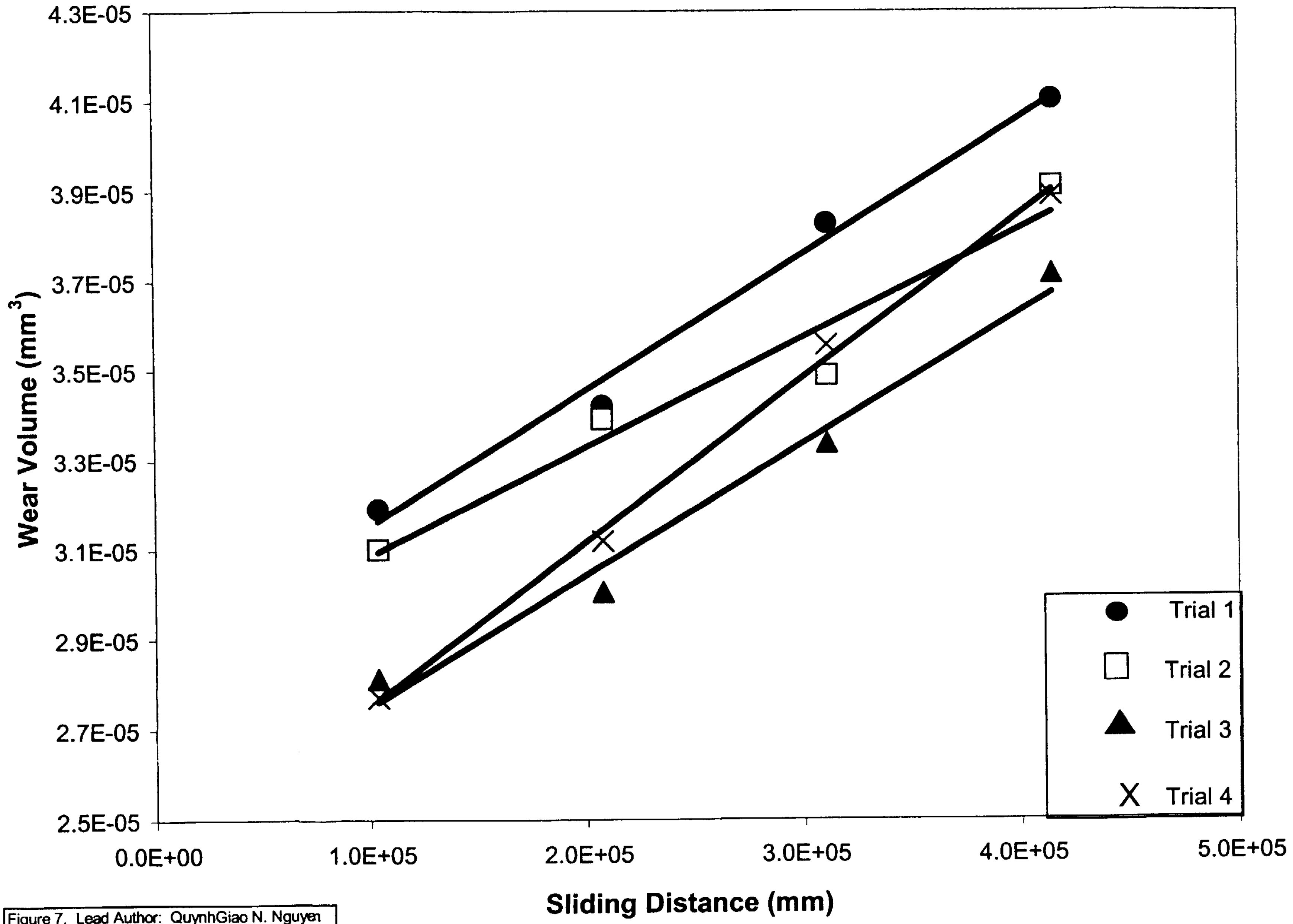

Figure 7. Lead Author: QuynhGiao N. Nguyen

Figure 7. Vacuum Four-Ball Results for Pentasilahydrocarbon (MJD991029).

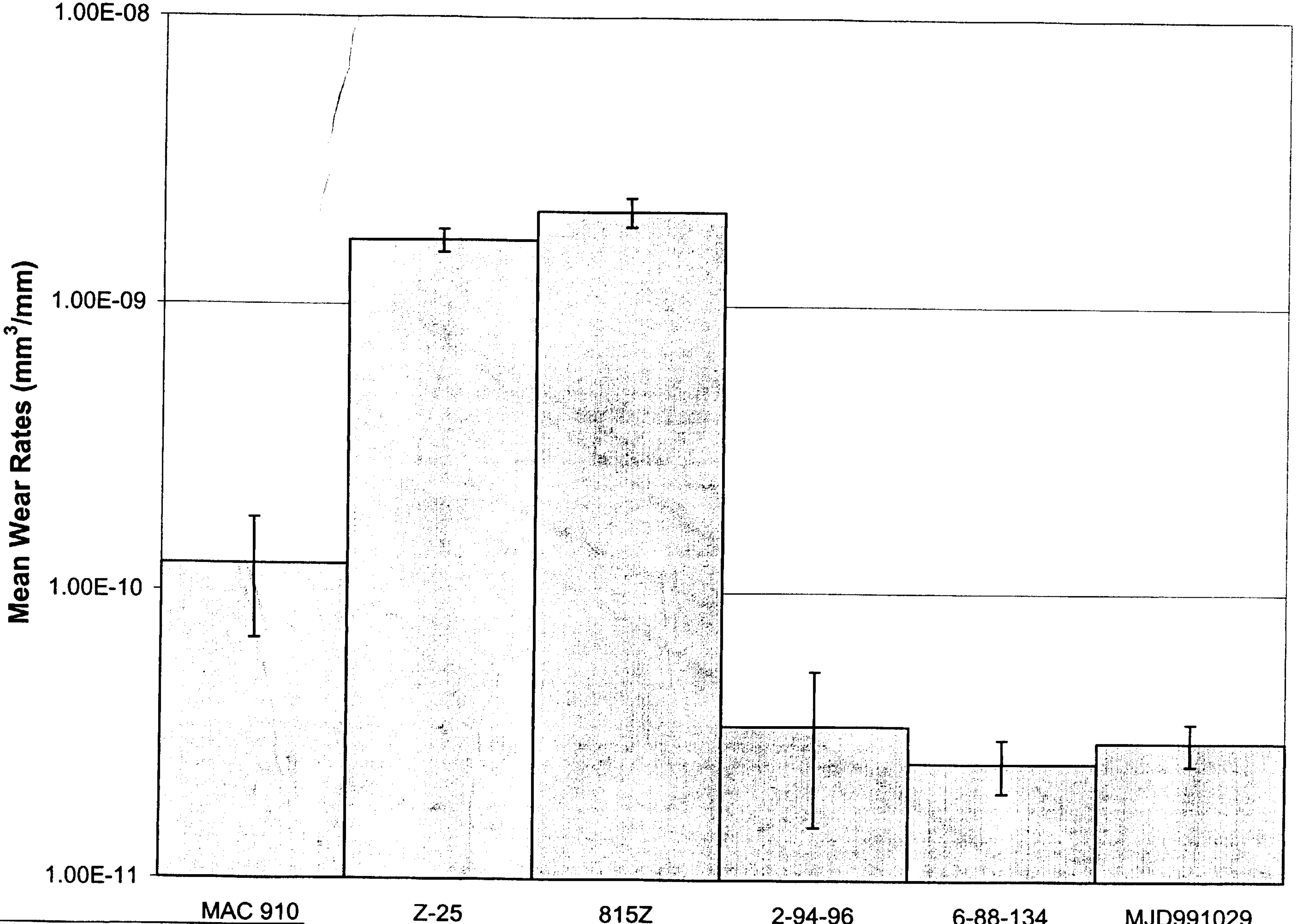

Figure 8. Lead Author: QuynhGiao N. Nguyen

Figure 8. Vacuum Four-Ball Results at Room Temperature. The Error Bars Represent One Standard Deviation of the Data.