**MEASUREMENT OF VISCOSITY, DENSITY, AND GAS
SOLUBILITY OF REFRIGERANT BLENDS IN SELECTED
SYNTHETIC LUBRICANTS**

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ABSTRACT

Liquid/liquid miscibilities of four different 32 ISO VG polyolesters and one alkylbenzene at three concentrations has been determined with five refrigerant blends, one blend comprising HC-290. A vapor lubricant equilibrium (VLE) viscosity reduction of 32 ISO VG mineral oil with HCFC-22 has been completed. Composite Viscosity reduction information by the fractionate components from R-502 in 32 ISO VG mineral oil has been obtained from -10°C (14°F) to 125°C (257°F) isotherms.

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1 SCOPE

The purpose of this study is to measure the viscosity, density and solubility of refrigerant/lubricant mixtures using various blended refrigerants. The two lubricants chosen for this study comprise a fully and a partially miscible 32 ISO VG branched acid, and a mixed branched acid 32 ISO VG polyolester, respectively. The refrigerant mixtures to be tested are labeled blends A, B, C, D, E and F. As shown in Table 1, these blends comprise refrigerant proportions expressed in percentages by weight. The refrigerant gas equilibrium solubility information is the fractionation of the individual gases in the lubricants. We will conduct similar measurements on the individual gases that comprise the refrigerant mixtures, i.e. HFC-32, HFC-125, HFC-134a, HFC-143a.

Table 1. Blends with Refrigerant Proportions in Percentages by Weight

Blend	HFC-32 %	HFC-125 %	HFC-134a %	HFC-143a %	HFC-290 %
A	60	40			
B	30		70		
C	30	10	60		
D		44	2	54	
E	30	55	20		5
F		45		55	

Refrigerant mixtures A, B, C, D, E and F will be used in refrigerant/lubricant combinations to determine the miscibilities in the five different lubricants (Table 2.) The miscibility information facilitates the selection of the two lubricants. We will use only mixtures A, C, and D for viscosity determinations. A base-line evaluation of HCFC-22 and R-502 in a 32 ISO VG mineral oil is included to show a comparison of viscosity, gas solubility and density properties.

Table 2. Lubricants

Fluid	Name	Manufacturer	Type	Registered Trademark, Trademark, or None
A	Icematic SW32	Castrol	Branched Acid Polyolester	Registered Trademark
B	Emery 2927a	Henkel, Emery Group	Branched Acid Polyolester	Registered Trademark
C	RL-32S	ICI Chemicals and Polymers, Ltd	Mixed Acid Polyolester	Registered Trademark
D	Artic EAL 224R	Mobil	Mixed Acid Polyolester	Registered Trademark
E	Shrieve Zerol 150	Shrieve Chemical Company	Alkylbenzene	Registered Trademark
F	Suniso 3GS	Witco Corporation	Naphthenic Mineral Oil	Registered Trademark

1.1 Statement and Chemical Properties of Lubricants

Our program uses proprietary lubricants, and pentaerythritol polyolesters. The most effective method to report lubricant kind is to determine alcohol type, and stoichiometry of carboxylic acids used in synthesis. It is necessary to examine the amount of acids used to provide the viscosity and liquid miscibility of the product with the various refrigerants. Unfortunately, due to proprietary concerns, only the miscibility and viscosity differences between the polyolesters are reported. Information about specific structural properties of the polyolester remain with the manufacturer.

2 METHODS

2.1 Viscosity Determinations

In this study, we use a method similar to the gas equilibrium concept used by Albright (1956-59), Little (1956) and Parmelee (1964.) These authors use the gas equilibrium concept, the saturation of the liquid with vapor, to study the viscosity reduction of hydrocarbon refrigeration oils in refrigerant gases. In conjunction with the gas equilibrium approach, we use temperature and pressure limitations to determine refrigerant gas concentrations.

To determine accurately viscosity and density, we use a fourth generation viscometer (Figure 1), which was taken from a report by Nissen. This viscometer system comprises an oscillating body device enclosed in a low volume pressure tube. The active part of this system is a solid, highly polished, stainless steel oscillating cylinder connected to a precisely thermostatted spring inside a stainless steel pipe to measure accurately

density and viscosity. This cylindrical bob is actuated by an external electromagnet, which causes oscillatory motion. We are able to determine the decrement of any solution in the viscometer by measuring the rate of sinusoidal decay. A quick decay represents a high viscosity, while a slow decay represents a low viscosity. After having measured the decrement and density, the viscosity of the solution can then be determined.

One benefit of the fourth generation viscometer is the extremely wide range (0.10 to 1800cP) of viscosity determinations, using a single oscillating bob. In this study, we use polyolesters (Blends B and C) and 32 ISO VG mineral oil, viscosity of water, and viscosity and density of test fluids that have been established as certified standards by an independent laboratory (NIST traceable.) These standards are calibrated from -25°C to 125°C and are reported in both cP and cSt values. The readability of the viscometer is 0.06 cP. The density of the oils is also reported. The wide temperature range of various calibration oils results in a significant overlap that serves as an internal standard and self check. A comparison of one calibration standard to another standard is $\pm 0.1\%$ for low viscosity solutions. Consequently, this increases to $\pm 1.5\%$ for high viscosity solutions.

The densitometer portion of the viscometer uses the same polished stainless steel bob as for viscosity determinations. We determine the density of the surrounding fluid by observing the position changes of the bob. The density sensitivity is within a density reading of 0.000X g/ml. Consistent density at constant temperature and constant pressure is a very accurate measure method of fluid consistency. Accurate density measurements are very important because it allows the investigator to calculate centistokes (cSt) from centipoise (cP). The density range of the instrument is calibrated with known, readily available pure fluids that are dried over a molecular sieve prior to use. Subsequently, we are able to establish a straight calibration line for density at specified temperatures. The precision of this determination is $\pm 0.3\%$

The fluid refrigerant mixture is pumped from the bottom of the viscometer and is sprayed into the refrigerant vapor space at specific pressures and temperatures. Consequently, we are able to monitor density for equilibrium conditions of the fluid and to measure the decrement and calculate viscosity. This fluid is visually examined several times to determine the true solution conditions. This procedure is repeated several times before sampling the fluid. The fluid sample is then drawn through a very low volume capillary line (380 μ l) into a deeply evacuated, light weight glass sampling bulb where a total charge is retained. We are then able to determine the percentage by weight of gas to liquid oil. The concentration measurement is reproducible within $\pm 0.5\%$ by weight at a given isothermal pressure test point. Sampling the refrigerant lubricant mixture at each isothermal pressure test point is an essential feature of our measurement technique.

Figure 1 Oscillating Body Viscometer

Oscillating Body Viscometer

The physical, chemical solution properties of refrigerant lubricant combinations are such that inconsistent ranges of solubility may exist. For example, this viscometer does not allow the conditions of immiscibility to occur. However, a pressure cell filled with a liquid refrigerant and a partially miscible lubricant could become immiscible in the liquid refrigerant at lower temperature measurements. The gas equilibrium concept does not produce immiscible layers unless forced liquefied gas saturation occurs. It is possible to maintain oil saturated with gas at a specific pressure and a specific temperature by accurately monitoring the gas pressure, which is always below or at the saturation pressure. However, liquid refrigerant can be added to the viscometer under pressure, and fluid properties can be measured for viscosity, density, pressure and refrigerant concentration with oils that are almost infinitely miscible. At this point, the refrigerant gas pressure is saturated, the viscosity is refrigerant, and the oil dilutions have reached saturation pressure. We observe that unless there is a zero void space in a pressurized viscometer cell, the refrigerant-oil pair combinations will change. For example, a closed system (viscometer) containing a fluid with 6.6% refrigerant by weight at 100°C (212°F) is cooled to 10°C(50°F), and the refrigerant concentration increases by a factor of 3 (22.0%), which is effected directly by the available gas space. When refrigerant blends are used, the vapor space of the viscometer is maintained to equal the gas composition of the pure refrigerant blend. At this point, we determine the fractionation of the mixed gases at every temperature and pressure. Samples of the fluid/refrigerant mixture are collected to determine the percent refrigerant by weight in the fluid. The ratio of gases is determined by gas chromatography, stated in: Section 2.2.

The fluid measurement portion of the viscometer is equipped with two high temperature and pressure sight glasses, upper and lower. The sight glasses are placed directly adjacent to the suspended stainless steel solid cylinder. We continually observe the solution quality for the formation of any immiscible particulates or haze of the fluid and refrigerant, which could occur during the study. In addition, to measure accurately the density/viscosity properties of the solution, it is necessary for the cylindrical bob to be completely immersed. We are able to verify, through the upper sight glass, if the viscometer is sufficiently charged with fluid. The lower sight glasses are adjacent to the gas introduction port and to the oil sampling port. The configuration of the mixed lubricant sample and the gas introduction port that is directly in line with the sight glasses ensures the mixed fluid is always a pure solution. Finally, we install a third sight glass at the top of the vapor space directly adjacent to the pump exit where we are able to observe the foaming qualities of the lubricant and to guarantee that the mixed system is always at the stated measurement temperature.

For the best reproducible method, we measure pressure with an accuracy of ± 0.2 psia with a temperature compensated 660 degree rotation Bourdon tube gauge (calibrated with both gas and liquid). This gauge is traceable to NIST standards. We measure the equilibrium vapor pressure, and the soluble gases are responsible for the fluid properties at those steady state conditions. The amount of gas content in the Bourdon tube is unnecessary because we always measure the gas content in the fluid.

Density, viscosity and vapor pressure are always measured under isothermal conditions. The viscometer temperature is controlled by a circulating constant temperature glycol bath using a Platinum RTD ($\pm 0.1^\circ\text{C}$) microprocessor controller. The RTD probe is mounted at the surface of the viscometer tube inside the liquid bath. The other temperature zones are controlled by electric heaters, using a microprocessor controller ($\pm 0.1^\circ\text{C}$) with type "J" thermocouples.

We pump the oil in the viscometer by a magnetically coupled impeller located in the pump body and spray the oil into a soluble gas vapor space at the top of the viscometer. We are able to simulate the lubricant fluid refrigerant pair conditions that exist in the operating compressor systems, according to the pressure or the temperature of the measurement. Test fluids are degassed at 60°C (140°F) to 20 millitorr for 24 hours and are generally dried to 50 ppm. Similarly, the viscometer is evacuated to 20 millitorr for several hours, purged with the refrigerant gas several times, and evacuated. The test oil is then drawn through the oil charging valve and re-evacuated to 20 millitorr. The oil is purged with refrigerant gas and evacuated again. We determine the non-condensable gas content that is not allowed to be higher than 10 ppm or equivalent to the refrigerant used.

2.2 Refrigerant Blend Sampling

After obtaining refrigerant lubricant samples from the viscometer, the glass container is weighed with the appropriate rubber stopper. The glass bulb is placed on the apparatus (Figure 2) sample input port with the lower stop cock closed. The upper stop cock is opened and attached to the vacuum pump. The upper stop cock remains open until a sufficient vacuum (30-50 millitorr) has been pulled on the gas chamber. When the upper stop cock is closed, the lower stop cock is opened. The lower pressure in the gas chamber pulls the refrigerant out of the sample in the glass bulb. To separate the refrigerant from the lubricant, the glass bulb is heated carefully with a propane torch. The lower stop cock is closed within 30 seconds of heating. One minute after closing the lower stop cock, equilibrium is established. We then take a sample to determine the ratio of the gases, which are drawn off the top oil. A second gas sample is obtained to determine if the ratio has changed and if equilibrium has been established. We use gas chromatography to

analyze the refrigerant sample. Separate measurement studies indicate that more than 80% of the refrigerant contained in the liquid sampling bulb has been removed.

3 RESULTS

3.1 Miscibility Determinations

We have examined the miscibility of four different polyolesters and one alkylbenzene at three different refrigerant/lubricant concentrations with six refrigerant blends (Appendix A.) We use alkylbenzene to examine the aromatic properties of miscibility. Surprisingly, we observe a partial miscibility for blend D as a result of lower temperature. This suggests a possibility for good oil return and inverse miscibility at higher temperatures, which indicates undiluted hydrocarbon lubrication.

The fluids show that levels of immiscibility may significantly effect the fractionation of individual gases at different temperatures and pressures. For example, Lubricant B is a 32 ISO VG branched acid polyolester, which is believed to be the most miscible, and represents a polyolester of very high liquid/liquid miscibility suggesting very little influence on the fractionation of the various gases that comprise refrigerant blends.

Although Lubricant C has similar viscosity to Lubricant B, it is determined to be the least miscible. Consequently, we use Lubricant C in our study to verify the impact of partial liquid miscibility to the gas solubility of various gases in refrigerant blends. We could also use Lubricants A and D, which are in the intermediate range of miscibility.

3.2 Viscosity of 32 ISO VG Mineral Oil and HCFC-22

Appendix B (Raw Data Table B-1) illustrates the isothermal viscosity, density and solubility of HCFC-22 in 32 ISO VG mineral oil. One of the more useful engineering features of isothermal determination is a snapshot view of refrigerant lubricant solubility knees, which are unique properties of refrigerant lubricant combinations. Solubility knees become apparent as the concentration of refrigerants approach the critical phase of refrigerant/refrigerant gas solutions. The viscosity determinations cease at -20°C , the lowest temperature. We attempted to measure the viscosity at a -40°C , but without success because the fluid became immiscible at the refrigerant concentration greater than 9%. Figure 3 presents viscosity as a function of temperature, and includes isobaric pressure lines. Figure 4 presents a modified "Daniel Plot" that shows viscosity and pressure at constant concentration as a function of temperature. Figure 5 shows density as a function of temperature at constant concentration. Appendices B and C Raw Data Tables (B-1 and C-1) illustrate density values.

Figure 2 Refrigerant Blend Sampling Apparatus

Refrigerant Blend Sampling Apparatus

Viscosity vs Temperature HCFC-22 in 32 ISO VG Mineral Oil

Figure 3 Viscosity vs. Temperature HCFC-22 in 32 ISO VG Mineral Oil

Viscosity and Pressure at Constant Concentrations HCFC-22 in 32 ISO VG Mineral Oil

Figure 4 Viscosity and Pressure at Constant Concentrations HCFC-22 in 32 ISO VG Mineral Oil

Density vs Temperature HCFC-22 in 32 ISO VG Mineral Oil

Figure 5 Density vs. Temperature HCFC-22 in 32 ISO VG Mineral Oil

3.3 Comparison of Published Data on HCFC-22 and R-502 Viscosity, Solubility and Density of 32 ISO VG Mineral Oil

We have reviewed the data written by Michael Pate, Iowa State University (ISU), who has published the most recent work on R-22 and R-502 with 32 ISO VG Mineral Oil, and alkylbenzene as part of ASHRAE RP-580. In addition, we have reviewed the most recent work by Spauschus Associates, Inc.(SAI) who has also reported on the R-22 data with 32 ISO VG Mineral Oil in their work for ARTI on measuring the "Solubility, Viscosity and Density of Refrigerant/Lubricant Mixtures." We will only address 32 ISO VG Mineral Oil, although there are many other fluids in the SAI report to be reviewed.

We obtain measurements at isothermal conditions with varying refrigerant gas pressures at equilibrium conditions. Total refrigerant gas concentration is double checked following each viscosity and density measurement. Experimental evidence shows that minute changes in refrigerant concentration have a dramatic effect on the measured viscosity and the density of the mixture. Internal fluid consistency is measured before each viscosity measurement, which is indicated by no change in density. Prior to each series of measurements, the certified NIST traceable test fluid compares the viscometer at several temperatures, which forms the basis of calibration for the viscometer.

The use of an oscillating body viscometer/densitometer instrumental technique is new to the refrigeration industry and constitutes a unique analytical method for accurately determining four valuable working fluid properties. The four properties comprise viscosity, density, equilibrium refrigerant gas solubility, and pressure over a broad temperature (-40°C to 125°C) range. This fundamental approach will provide research quality data upon which meaningful empirical equations of state can be formulated.

A reliable test for the correlation of data is designed to match the pressures, viscosity and concentration measurements of SAI and ISU laboratories to those measurements obtained from our laboratory. If our pressure matches the other pressures from other laboratories, that data should be identical to our reported viscosity measurements (Tables 3,4.) From this simple analysis, the most dramatic difference is the refrigerant solubility or concentration measurements. Both SAI and ISU laboratories show dramatically lower refrigerant concentration, and therefore, obtain higher viscosity at lower pressures. Our measurements provide a good representation of the refrigerant in oil concentration because we gravimetrically measure the weight of the refrigerant in oil at each pressure point. Furthermore, our study shows the dramatic changes in viscosity and in pressure with very small changes in refrigerant concentrations.

Table 3. SAI = Spauschus & Associates, Inc. ISU = Iowa State University.
IRI = Imagination Resources, Inc. TEMPERATURE = 40°C

Pressure psia	Viscosity cSt			% Refrigerant		
	SAI	ISU	IRI	SAI	ISU	IRI
75	13		12.8	10.0		6.4
80		13	12.0		10.0	6.9
107			8.41			10.0
130	6		5.94	20.0		13.2
140		5	5.05		20.0	14.8
167			3.09			20.0
175	3		2.62	30		21.9
180		2.8	2.25		30.0	23.2
201			1.43			30.0

Table 4 TEMPERATURE = 100°C

Pressure psia	Viscosity cSt			% Refrigerant		
	SAI	ISU	IRI	SAI	ISU	IRI
170	2.9		2.3	10.0		6.45
225		2.0	1.99		10.0	8.36
271.6			1.76			10.0
315	2.0		1.58	20.0		11.57
390		1.2	1.31		20.0	14.41
447	1.4		1.14	30.0		16.75
519			0.96			20.0
687			0.65			30.0

3.4 Viscosity of Mineral Oil and R-502

Appendix C shows the isothermal viscosity, density and solubility of R-502 in 32 ISO VG mineral oil. Each isothermal plot shows the fractionation of R-502 as percent R-22 of the total gas in solution. The lowest temperature of the viscosity determination is -10°C, the higher limit of 125°C and 500 psia. Mixed refrigerant gas is purged through the lubricant to achieve an equilibrium of the fractionated components at specified temperatures and pressures. The equilibrium is maintained by making the refrigerant gas above the lubricant equal to the proportion of the mixed lubricant refrigerant. The percent concentration of the total refrigerant represents the total of both gases that are soluble in the fluid at that pressure and temperature. Figure 6 presents viscosity as a function of temperature and includes isobaric pressure lines. Figure 7 presents a modified "Daniel Plot" that shows viscosity and pressure at constant concentration as a function of temperature. Figure 8 shows density as a function of temperature at constant concentration.

Viscosity vs Temperature of R-502 in 32 ISO VG Mineral Oil

Figure 6 Viscosity vs. Temperature R-502 in 32 ISO VG Mineral Oil

Viscosity and Pressure at Constant Concentrations R-502 in 32 ISO VG Mineral Oil

Figure 7 Viscosity and Pressure at Constant Concentrations R-502 in 32 ISO VG Mineral Oil

Density vs Temperature R-502 in 32 ISO VG Mineral Oil

Figure 8 Density vs. Temperature R-502 in 32 ISO VG Mineral Oil

3.5 Comparison of the Published Data on R-502 Viscosity, Solubility and Density of 32 ISO VG Mineral Oil

The R-502 data obtained from IRI and ISU show conflicting results. ISU investigators report that they have found zones of immiscibility in the measured fluids at the higher concentrations. The ISU case records higher pressures, which may attribute to the increased content of R-115 separation in the gas phase. In our study, the first addition of R-502 has the head space that is very rich in R-115 until it is purged with additional R-502.

On the contrary, our experimental evidence indicates R-502 fractionation in 32 ISO VG Mineral Oil (Appendix C) and the ratio changes with pressure and temperature. The principle component of solution is R-22, which is found to be between 62%-68% over the measured pressure and temperature range.

4 ARTI Recommendations

4.1 Refrigerant and Gas Solubility Studies of 32 ISO VG Mineral Oil by Oscillating Body Viscometer and Canon Fenske Viscometer

Due to different data results between SAI and IRI, ARTI requested that we include a supplemental study to reaffirm some of our original data results. Therefore, we are providing data using larger sample sizes and step by step details of our study.

A) Please provide complete step-by-step details as to how concentration determinations are made, identify the variables involved, and provide error band analysis for each of these variables.

Following the addition (or removal) of refrigerant the mixture is stirred then calculated to ensure that the solution is homogenous. A determination of the vapor equilibrium status can be made by monitoring the pressure of the mixture. For a given refrigerant-lubricant mixture, concentration determinations are established after equilibrium conditions are established. Concentration determinations are made using the following seven step process:

- 1) Two empty 200 ml glass bulbs each with an associated rubber stopper, (Fisherbrand® micro solid red rubber stopper, Cat No. 21-381) are weighed by an OHAUS® GA200 digital scale. The GA200 has a resolution of 0.0001 gram.

2) Prior to sampling the mixture at a given temperature-pressure, the capillary tube connecting the body of the viscometer to the sample port is purged to ensure that the sample represents the conditions of the solution in the viscometer.

Purging is accomplished by placing a 200 ml glass bulb on the sample port, evacuating the bulb and opening the sample port valve. The amount of mixture purged is approximately that of a typical sample (1g), which represents a volume well in excess of that of the capillary tube (approximately 380 μ l).

3) For a given temperature-pressure point, the mixture is purged prior to viscosity determination. The first of the two pre-weighed 200 ml bulbs is placed on the sample port and then evacuated to 50 millitorr. The sample is then drawn into the bulb until the pressure inside the bulb is just under one atmosphere. The bulb is then removed from the sample port and capped with the appropriate rubber stopper, and weighed on the GA200 scale. After each test of the sample an appropriate amount of refrigerant is added to the viscometer to equilibrate a set pressure. This ensures that identical operating conditions are maintained from the first through the fourth sample.

4) To remove refrigerant from the samples, the bulbs are placed on a vacuum manifold, and warmed with a propane torch. When the vacuum inside the bulbs maintains a constant 30 millitorr, all refrigerant is assumed to have been removed. This procedure lasts between two to four hours.

5) After the refrigerant is removed, the sample bulbs are capped with the appropriate stoppers and re-weighed.

6) The percent refrigerant concentration by weight is calculated by comparing the weight of the oil in the sample to the weight of the total sample. The weight of the oil is obtained by the difference between the sample, with the refrigerant removed, and the weight of the empty flask. The weight of the refrigerant-oil sample is the difference in the weight between the bulb with the refrigerant-lubricant sample and the empty bulb.

7) If the points do not agree by weight refrigerant (within $\pm 1\%$), both bulbs are placed on the vacuum manifold and re-weighed. Most refrigerant concentration comparisons vary by $\pm 0.3\%$ by weight. If the refrigerant concentration determinations still do not agree by weight, then the viscosity, density and solubility are measured again. The data given in Tables 5 and 6 represent an average of two separate samples at a given point.

Table 5: % Refrigerant comparison between large and small sample sizes
Temperature = 40°C

Pressure (psia)	Sample Wt. (g) 200 ml	% Refrigerant conc.	Sample Wt. (g) 1000 ml	% Refrigerant conc.	Difference (%)
52.0	1.288	4.631	8.789	4.533	.098
78.5	1.468	7.192	4.943	6.882	.310
104.0	1.511	9.777	9.543	9.543	.234
152.0	.916	16.491	9.597	16.904	-.413
199.0	.765	34.805	6.284	34.491	.314
212.5	.652	62.138	4.083	62.454	-.316

Table 6: % Refrigerant comparison between large and small sample sizes
Temperature = 100°C

Pressure (psia)	Sample Wt. (g) 200 ml	% Refrigerant conc.	Sample Wt. (g) 1000 ml	% Refrigerant conc.	Difference (%)
50.0	2.523	1.904	8.844	1.887	.017
100.0	1.710	3.137	11.002	2.948	.189
150.0	1.932	4.863	10.217	4.695	.168
200.0	1.254	7.175	9.752	6.974	.210
319.0	1.429	12.124	8.558	11.153	.971
445.0	1.260	17.492	8.073	18.466	-.974

In addition to the above standard operating procedure, we use heated head space GC and the FTIR methods to verify that all the refrigerant had been removed from the samples. The IRI oscillating body viscometer uses a temperature compensated Heise™ NIST traceable pressure gauge with a resolution of 0.2 psia and a maximum pressure reading of 500 psia. We use Omega CN9000 Series controllers to maintain the constant temperature bath with a resolution of 0.1°C. As previously stated, the sensitivity of the OHAUS® GA200 is 0.0001 gram.

B) Please re-verify the purity of the Suniso 3GS that was used in the tests.

The viscosity of neat 3GS is determined at 1 atmosphere, and 20°C to 100°C. We then compare the results with a Cannon Fenske viscometer. In addition, the sample material is tested by gas chromatography. In both cases, the results confirm the purity of the sample.

C) Please Provide viscosity Measurements of the pure 32 ISO VG Mineral Oil lubricant (e.g., without refrigerant) -- over a range of 0°C to 100°C -- that were obtained utilizing your individual procedures for determining viscosity's of lubricant-refrigerant mixtures.

Table 7 compares the viscosity values of 32 ISO VG Mineral Oil in centistokes using the IRI oscillating bob and the Cannon Fenske viscometer. The viscosity of 32 ISO VG Mineral Oil at 0°C is too high to obtain an accurate value.

Table 7: Viscosity of 32 ISO VG Mineral Oil

Temperature (°C)	IRI Oscillating body Viscosity (cSt)	Cannon Fenske Viscosity (cSt)
20.0	95.08	96.20
40.0	28.44	28.90
60.0	12.16	-
80.0	6.05	6.09
100.0	3.68	-

D) Most importantly, concern was expressed that weight measurement of 1 gram samples could impact the accuracy of the measurement; especially if any lubricant had previously "hung up" in the capillary tube. Please determine concentration values for a R-22/Mineral oil mixture at 40 °C and 100 °C utilizing a sample size much greater than your current 1 gram specimen. Additionally, once you have determined that the refrigerant has been completely driven from the lubricant, it is requested that you perform an analysis on the lubricant to confirm that the lubricant is absent of refrigerant.

The amount of sample obtained from a given refrigerant-lubricant blend depends on several factors, such as the current operating pressure and the volume of the sample flask. When we measure the mixtures at high pressures, a small sample is sufficient to increase the bulb pressure to ambient conditions. We are able to obtain larger samples at a given pressure-temperature point when we increase the volume of the vessels.

Currently, sample vessels have a volume of 200 ml. We use specially designed bulbs with a volume of 1000 ml, as recommended in article D. On the average, the new bulbs weigh 260 grams, opposed to the 110 grams for the smaller 200 ml bulbs. This increased bulb weight requires the use of a larger OHAUS® Model GT410 scale. The GT410 scale has 1/10 the resolution (.001g vs. .0001g) of the GA200. However, the increase in sample size negates this loss in sensitivity.

We measured the refrigerant concentration and the viscosity at the recommended pressures and temperatures. We took four samples, two with the 200 ml and two with the 1000 ml bulb and determined the refrigerant concentration for each sample. Error analysis determined no significant differences between the large and small samples. We compared the samples in the 200 ml vs. the 1000 ml bulbs, at 40°C and 100°C, which are shown in

Tables 5 and 6. Tables 5 and 6 compare the average weights and percent refrigerant concentrations of samples obtained using a 200 ml and 1000 ml glass bulb, respectively. The last column in Tables 5 and 6 compares the differences in the percent refrigerant concentration by weight with the two different bulb sizes.

The average increase in sample size, using the 1000 ml vs. the 200 ml volume bulbs, were 6.6 and 5.5 times for 40 and 100°C respectively. Table 7 indicates a larger sample size may increase the accuracy, but there is no consistent trend between the sample size and the refrigerant concentration observed.

Concern was expressed over lubricant being "hung up" in the capillary tube connecting the viscometer body with the sampling port. This would result in consistent pattern between the refrigerant concentration determinations obtained from the first and second sample, however, no such pattern was observed.

5 Contract Agreement

Imagination Resources, Inc. is in compliance with the contract agreement.

6 Principal Investigator Effort

The principal investigators have devoted 640 hours toward the completion of this contract. Activities include reporting early refrigerant miscibility information to the Technical Oversight Group. Experimental facilities are being established.

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APPENDIX A

Miscibility of Refrigerant Blends A-F

Miscibility of Refrigerant Blend A

HFC-32(60%) and HFC-125(40%)

Miscibility of Refrigerant Blend B

HFC-32(30%) and HFC-134a(70%)

Miscibility of Refrigerant Blend C

HFC-32(30%), HFC-125(10%), and HFC-134a(60%)

Miscibility of Refrigerant Blend D

HFC-125(44%), HFC-143a(52%) and HFC-134a(4%)

Miscibility of Refrigerant Blend E

HFC-32(30%), HFC-125(55%), HFC-134a(20%), and HC-290(5%)

Miscibility of Refrigerant Blend F

HFC-125(45%) and HFC-143a(55%)

APPENDIX B

Viscosity and Solubility of 32 ISO VG Mineral Oil at Various Temperatures with HCFC-22

Viscosity and Solubility of 32 ISO VG Mineral Oil **Determined at 125 °C with HCFC-22**

Viscosity via Gas Solubility Equilibrium
 Oil degassed to 20 Millitorr
 Figure B-1

Viscosity and Solubility of 32 ISO VG Mineral Oil **Determined at 100 °C with HCFC-22**

Viscosity via Gas Solubility Equilibrium
 Oil degassed to 20 Millitorr
 Figure B-2

Viscosity and Solubility of 32 ISO VG Mineral Oil Determined at 80 °C with HCFC-22

Viscosity via Gas Solubility Equilibrium
Oil degassed to 20 Millitorr
Figure B-3

Viscosity and Solubility of 32 ISO VG Mineral Oil Determined at 60 °C with HCFC-22

Viscosity via Gas Solubility Equilibrium
Oil degassed to 20 Millitorr
Figure B-4

*Viscosity and Solubility of 32 ISO VG Mineral Oil
Determined at 40 °C with HCFC-22*

Viscosity via Gas Solubility Equilibrium
Oil degassed to 20 Millitorr
Figure B-5

*Viscosity and Solubility of 32 ISO VG Mineral Oil
Determined at 20 °C with HCFC-22*

Viscosity via Gas Solubility Equilibrium
Oil degassed to 20 Millitorr
Figure B-6

Viscosity and Solubility of 32 ISO VG Mineral Oil Determined at 0°C with HCFC-22

Viscosity via Gas Solubility Equilibrium
Oil degassed to 20 Millitorr
Figure B-7

Viscosity and Solubility of 32 ISO VG Mineral Oil Determined at -20°C with HCFC-22

Viscosity via Gas Solubility Equilibrium
Oil degassed to 20 Millitorr
Figure B-8

Viscosity, Density, and Solubility of HCFC-22 in ISO VG Mineral Oil Raw Data Table B-1

Temperature: 125 C
P_{max} = 500 psia

Density g/mL	Pres. psia	Conc % by wt.	Visc. Cp	Visc. Cst
0.8600	450.00	11.232	1.216	1.414
0.8585	450.00	11.036	1.235	1.438
0.8480	334.00	8.368	1.358	1.602
0.8480	334.00	8.369	1.332	1.571
0.8376	214.00	4.956	1.499	1.789
0.8361	213.00	4.857	1.501	1.796
0.8271	114.00	2.521	1.716	2.075
0.8271	116.00	2.817	1.731	2.093
0.8136	19.00	0.287	1.904	2.340
0.8122	18.00	0.255	1.897	2.336

Temperature: 40 C
P_{sat} = 222.4 PSIA

Density g/mL	Pres. psia	Conc % by wt.	Visc. Cp	Visc. Cst
0.8824	12.00	1.530	22.010	24.942
0.8794	12.00	1.699	21.963	24.974
0.9064	69.00	5.724	10.598	11.694
0.9019	69.00	4.418	10.661	11.821
0.9318	128.50	11.579	5.188	5.568
0.9288	128.00	12.680	5.183	5.580
0.9572	168.00	19.199	2.660	2.779
0.9557	168.00	18.754	2.720	2.846
1.0260	217.50	37.893	0.937	0.913
1.0275	218.00	38.110	0.949	0.924

Temperature: 100 C
P_{max} = 500 psia

Density g/mL	Pres. psia	Conc % by wt.	Visc. Cp	Visc. Cst
0.9183	489.00	19.232	1.135	1.236
0.9153	489.00	19.913	1.146	1.252
0.8989	375.00	13.422	1.396	1.553
0.9004	375.00	13.411	1.381	1.534
0.8779	260.00	9.538	1.809	2.060
0.8779	256.00	8.984	1.721	1.960
0.8525	144.00	5.969	2.315	2.715
0.8525	144.00	5.966	2.287	2.683
0.8256	10.00	1.312	3.234	3.917
0.8271	10.00	2.152	3.228	3.902

Temperature: 20 C
P_{sat} = 132.0 PSIA

Density g/mL	Pres. psia	Conc % by wt.	Visc. Cp	Visc. Cst
0.9034	9.00	2.460	72.050	79.758
0.8974	9.00	2.231	73.090	81.448
0.9198	44.00	5.840	27.539	29.940
0.9243	44.00	5.398	29.367	31.772
0.9452	78.50	11.600	11.363	12.022
0.9437	78.50	11.548	11.626	12.319
0.9811	114.00	19.898	3.804	3.877
0.9811	113.00	22.316	3.873	3.948
1.1067	134.00	55.410	0.601	0.543
1.1052	134.00	56.658	0.615	0.557

Temperature: 80 C
P_{max} = 500 psia

Density g/mL	Pres. psia	Conc % by wt.	Visc. Cp	Visc. Cst
0.8361	20.00	1.353	5.033	6.019
0.8361	20.00	1.827	4.932	5.899
0.8600	122.50	5.494	3.517	4.090
0.8615	122.50	5.178	3.289	3.818
0.9004	248.00	12.444	1.955	2.171
0.8959	248.00	12.030	1.960	2.188
0.9318	405.00	22.534	1.142	1.225
0.9333	405.00	23.117	1.124	1.205

Temperature: 0 C
P_{sat} = 72.2 PSIA

Density g/mL	Pres. psia	Conc % by wt.	Visc. Cp	Visc. Cst
1.1516	72.50	59.767	0.896	0.778
1.1546	72.50	63.300	0.884	0.766
1.0095	63.00	22.691	8.592	8.511
1.0035	63.00	23.521	8.678	8.646
0.9572	45.00	13.005	37.550	39.230
0.9587	45.00	11.140	37.463	39.078
0.9392	28.50	8.589	108.350	115.358
0.9378	28.50	7.268	107.432	114.564
0.9228	12.50	1.800	413.270	447.844
0.9228	12.50	1.982	414.480	449.155

Temperature: 60 C
P_{sat} = 352.0 PSIA

Density g/mL	Pres. psia	Conc % by wt.	Visc. Cp	Visc. Cst
0.9871	332.50	36.030	0.858	0.870
0.9886	332.50	34.971	0.841	0.850
0.9437	260.00	21.078	1.692	1.793
0.9437	260.00	22.778	1.687	1.788
0.9153	172.00	10.788	3.317	3.624
0.9138	170.00	11.337	3.297	3.608
0.8944	97.00	6.043	5.497	6.146
0.8914	98.00	6.736	5.531	6.205
0.8495	19.00	0.826	9.232	10.867
0.8510	18.50	1.983	9.083	10.673

Temperature: -20 C
P_{sat} = 40.3 PSIA

Density g/mL	Pres. psia	Conc % by wt.	Visc. Cp	Visc. Cst
0.9811	25.50	9.280	209.777	213.815
0.9811	25.50	9.803	209.777	213.815
0.9901	32.50	15.002	52.942	53.472
0.9916	32.50	15.850	53.060	53.510
1.0170	34.50	35.474	23.410	23.019
1.0185	34.50	21.255	23.561	23.134
1.1665	35.50	59.910	3.474	2.978
1.1665	35.50	56.968	3.231	2.770

APPENDIX C

Viscosity, Solubility and Gas Fractionation at Various Temperatures with R-502

Viscosity, Solubility, and Fractionation of 32 ISO VG Mineral Oil Determined at 125°C with R-502

Viscosity via Gas Solubility Equilibrium
Oil degassed to 20 Millitorr
Figure C-1

Viscosity, Solubility, and Fractionation of 32 ISO VG Mineral Oil Determined at 100°C with R-502

Viscosity via Gas Solubility Equilibrium
Oil degassed to 20 Millitorr
Figure C-2

Viscosity, Solubility, and Fractionation of 32 ISO VG Mineral Oil Determined at 70°C with R-502

Viscosity via Gas Solubility Equilibrium
Oil degassed to 20 Millitorr
Figure C-3

Viscosity, Solubility, and Fractionation of 32 ISO VG Mineral Oil Determined at 20°C with R-502

Viscosity via Gas Solubility Equilibrium
Oil degassed to 20 Millitorr
Figure C-4

Viscosity, Solubility, and Fractionation of 32 ISO VG Mineral Oil Determined at 0°C with R-502

Viscosity via Gas Solubility Equilibrium
Oil degassed to 20 Millitorr
Figure C-5

Viscosity, Solubility, and Fractionation of 32 ISO VG Mineral Oil Determined at -10°C with R-502

Viscosity via Gas Solubility Equilibrium
Oil degassed to 20 Millitorr
Figure C-6

Viscosity, Density, Solubility, and Gas Fractionation of R-502 in 32 ISO VG Mineral Oil Raw Data **Table C-1**

Temperature: 125 C
P_{sat} = 500.0

Density g/mL	Pres. psia	Conc % by wt.	Visc. Cp	Visc. Cst	% R-22
0.8073	34.00	2.308	1.963	2.432	66.200
0.8073	33.50	2.041	1.912	2.368	66.000
0.8281	116.00	4.934	1.880	2.271	63.500
0.8264	116.00	4.926	1.873	2.267	64.300
0.8503	274.50	8.731	1.805	1.867	66.400
0.8473	274.50	9.121	1.580	1.865	66.400
0.8799	466.50	12.437	1.481	1.663	66.200
0.8771	467.00	12.666	1.502	1.712	65.600

Temperature: 20 C
P_{sat} = 147.9

Density g/mL	Pres. psia	Conc % by wt.	Visc. Cp	Visc. Cst	% R-22
0.8902	36.00	4.070	48.818	54.836	67.0
0.8876	39.00	4.095	47.774	53.626	66.0
0.9124	74.25	8.271	20.619	22.617	64.5
0.9124	74.50	8.655	20.364	22.341	64.0
0.9820	140.00	24.739	4.709	4.796	62.0
0.9835	140.00	22.537	4.702	4.781	62.0
1.0293	147.00	34.072	2.357	2.289	62.4
1.0279	147.00	35.249	2.360	2.296	61.5

Temperature: 100 C
P_{sat} = 500.0

Density g/mL	Pres. psia	Conc % by wt.	Visc. Cp	Visc. Cst	% R-22
0.8266	25.00	6.224	3.597	4.352	66.200
0.8251	25.00	6.366	3.443	4.173	66.000
0.8364	110.00	6.602	2.921	3.464	63.500
0.8414	110.00	5.776	2.890	3.435	64.300
0.8917	375.00	10.169	1.993	2.235	66.400
0.8932	376.00	11.320	1.990	2.226	66.400
0.9163	451.00	16.205	1.691	1.841	66.200
0.9169	451.00	15.432	1.729	1.865	65.600

Temperature: 0 C
P_{sat} = 83.1

Density g/mL	Pres. psia	Conc % by wt.	Visc. Cp	Visc. Cst	% R-22
0.8976	33.00	6.864	66.435	96.293	62.000
0.8947	33.00	7.262	96.257	107.590	61.000
0.9169	45.75	10.829	61.846	89.266	65.800
0.9169	46.00	10.496	73.366	80.051	65.300
0.9272	59.50	12.427	51.374	55.406	64.700
0.9243	60.00	13.275	50.979	55.156	65.500
0.9539	70.25	15.747	23.530	24.666	62.000
0.9539	70.25	15.763	23.922	25.079	61.900
0.9728	80.50	23.402	12.496	12.847	59.600
0.9731	80.50	22.980	12.615	12.963	59.000

Temperature: 70 C
P_{sat} = 467.67

Density g/mL	Pres. psia	Conc % by wt.	Visc. Cp	Visc. Cst	% R-22
0.8414	37.00	3.022	7.044	8.372	57.9
0.8414	37.00	2.746	7.107	8.446	57.2
0.8651	153.00	9.333	4.601	5.319	66.4
0.8665	153.00	8.442	4.458	5.144	66.2
0.9080	271.00	16.703	2.679	3.171	65.9
0.9095	271.00	16.200	2.974	3.270	65.7
0.9391	367.50	22.826	1.946	2.072	63.8
0.9391	367.50	21.836	1.972	2.100	64.6
0.9635	455.00	32.450	1.284	1.305	60.8
0.9635	455.00	32.410	1.284	1.305	60.9

Temperature: -10 C
P_{sat} = 60.1

Density g/mL	Pres. psia	Conc % by wt.	Visc. Cp	Visc. Cst	% R-22
0.9701	55.00	19.272	26.540	27.356	72.400
0.9672	55.00	19.334	25.811	26.666	72.200
0.9716	57.50	22.308	20.556	21.157	70.900
0.9701	57.50	25.672	20.016	20.632	69.500
0.9509	49.00	15.507	48.092	50.574	73.800
0.9479	49.00	16.705	49.249	51.953	73.900
0.9257	32.50	11.137	65.690	92.563	65.000
0.9243	32.50	10.278	67.351	94.506	64.300
0.9272	40.75	10.573	122.164	131.774	63.200
0.9257	40.75	10.514	124.060	134.032	62.500
0.9376	44.25	14.452	63.635	69.202	69.000
0.9391	44.25	13.191	63.557	66.979	69.600

Temperature: 40 C
P_{sat} = 243.2

Density g/mL	Pres. psia	Conc % by wt.	Visc. Cp	Visc. Cst	% R-22
0.8739	70.50	5.267	11.645	13.325	59.5
0.8754	70.50	7.597	11.552	13.196	59.6
0.9183	155.00	13.119	6.317	6.876	56.4
0.9183	155.00	16.656	6.379	6.947	56.6
0.9479	208.00	23.976	3.656	3.656	60.7
0.9494	208.00	22.737	3.643	3.637	60.9
1.0027	243.00	36.026	1.613	1.609	59.3
1.0027	243.00	36.082	1.613	1.609	56.6

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