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ABSTRACT

Miscibility data is being obtained for a variety of non-CFC refrigerants and their potential lubricants under a program initiated and supported by the Air-Conditioning and Refrigeration Technology Institute. At least ten (10) different refrigerants and seven (7) different lubricants are being investigated. Experiments are being performed in two phases: Phase I focuses on performing screening tests and Phase II consists of developing miscibility plots. The miscibility tests are being performed in a test facility consisting of a series of miniature test cells submerged in a constant temperature bath. The bath temperature can be precisely controlled over a temperature range of -50°C to 100°C. The test cells are constructed to allow for complete visibility of lubricant-refrigerant mixtures under all test conditions. Tables and plots of critical solubility temperatures versus lubricant and refrigerant concentrations will be developed.

INTRODUCTION

Miscibility data is needed for lubricant-refrigerant mixtures consisting of the new refrigerants (non-CFCs) and a variety of lubricants. This data will allow refrigeration system designers to select lubricants that are miscible with refrigerants so that evaporator heat transfer, lubricant return, and compressor operation are not adversely affected. The Air-Conditioning and Refrigeration Technology Institute (ARTI) is sponsoring a research project to measure the miscibility of a variety of refrigerant-lubricant mixtures consisting of alternative refrigerants and their potential lubricants.

The objectives of this ARTI project are:

- To provide information on the miscibility of both current and new lubricants with potential substitutes for CFC refrigerants.
- To obtain necessary data on lubricant-refrigerant miscibility to accelerate development of equipment using the substitute refrigerants.

PROJECT SCOPE

Phase I - Preliminary Screening

Miscibility tests will be performed for refrigerant concentrations of 95%, 50% and 10% by weight. These tests will be performed by keeping the liquid phase visible at all times, by controlling temperatures to ±1°C, and by providing agitation of the test cells.

The following refrigerants will be tested for miscibility in 10°C (18°F) increments over the temperature ranges indicated below:

- R-22: -50 – +60°C (-58 – +140°F)
- R-32: -50 – +60°C (-58 – +140°F)
- R-123: -50 – +60°C (-58 – +140°F)
- R-124: -50 – +90°C (-58 – +194°F)
- R-125: -50 – +60°C (-58 – +140°F)
- R-134a: -50 – +90°C (-58 – +194°F)
- R-142b: -50 – +90°C (-58 – +194°F)
- R-143a: -50 – +60°C (-58 – +140°F)
- R-152a: -50 – +90°C (-58 – +194°F)
- E-134: -50 – +60°C (-58 – +140°F)

Each of the above refrigerants will be tested for miscibility with the lubricants listed below. In most cases, the lubricant viscosity will be 32 cSt. The purity requirements stated will also be met during all miscibility tests.

- mineral oils (<30 ppm moisture, acid number <0.01)
  - naphthenic mineral oil
- alkylbenzenes (<30 ppm moisture, acid number <0.01)
- polyglycols(<50 ppm moisture, acid number <0.05)
  - polypropylene glycol butyl monoether
  - polypropylene glycol diol
  - modified polyglycol
- polyolesters(<50 ppm moisture, acid number <0.05)
  - penta erythritol ester mixed-acid
  - penta erythritol ester branched-acid

During the above miscibility tests, the contents of each test cell will be observed for signs of cloudiness, floc or precipitate formation, and liquid layer formation. These observation will be made at each required temperature after equilibrium (i.e., steady states) conditions have been reached. Applicable miscibility data will consist of refrigerant/lubricant concentrations, the temperature at equilibrium, and observations of the test cell contents for evidence of immiscibility.

Phase II - Derivations Of Miscibility Plots

Phase II will consist of additional tests made up of an expanded concentration range. In addition to Phase I lubricant concentrations (i.e., 5, 50, 90%), lubricant concentrations by weight of 10, 20, 35, 65 and 80% will be tested for miscibility. Miscibility plots consisting of critical solution temperatures will be developed.

The basis for selecting lubricant-refrigerant combinations to be tested in Phase II will be that the combination must have demonstrated single-phase miscibility in at least one of the three test points obtained in Phase I. Assuming that these criteria are met, each of the refrigerants will be tested for miscibility with the lubricants listed below. The lubricant viscosities to be used in the tests are also noted.

- mineral oils (30 ppm moisture, acid number <0.01)
  - naphthenic mineral oil, 32 and 68 cSt
- alkylbenzenes (<30 ppm moisture, acid number <0.01), 32 and 65 cSt
- polyglycols (<50 ppm moisture, acid number <0.05)
  - polypropylene glycol butyl monoether, 32 and 68 cSt
  - polypropylene glycol diol, 32 and 100 cSt
  - modified polyglycol, 32 and 68 cSt
- polyolesters (<50 ppm moisture, acid number <0.05)
  - penta erythritol ester mixed-acid, 22 cSt and 46 cSt
  - penta erythritol ester branched-acid, 32 and 100 cSt

TEST FACILITY

The test facility to be used in this project was designed for the purpose of measuring the miscibility and solubility characteristics of refrigerant-lubricant mixtures by using a series of miniature test cells submerged in a constant temperature bath. The test cells are constructed to allow for complete visibility of the oil/refrigerant mixture at all test conditions. The temperature of the cells is fixed by placing them in a constant temperature bath. A temperature sensor can be inserted in each cell or in adjoining reference cells exposed to the same heating or cooling conditions. During charging, each cell can be filled so that the vapor space volume is less than 5% of the total volume. In addition, the temperature and pressure data allows for correcting the liquid concentration due to vapor space refrigerant, if necessary. A detailed description of the test facility along with data analysis and correlation techniques is presented below.

Each test cell consists of a double-port seal-cap type liquid indicator, which is essentially a 1.25 inch pipe cross with sight windows screwed into opposing ports. The sight windows allow for complete visibility of the oil/refrigerant mixture. Valves for charging the oil and refrigerant into the cell are screwed into the other two ports. In line with one of the valves is a pressure transducer (optional). The pressure transducer is included so that solubility data can be obtained simultaneously, if needed. The overall volume of each test cell varies slightly; however all were measured to have volumes around 65 ml. Figure 1 is a photograph of a test cell assembly.

The test cells (up to a total of 12 for each bath) are submersed in a 20 x 12 x 12 inch glass bath. The glass allows for visibility of the test cells and, therefore, the oil/refrigerant mixtures throughout the test. Figure 2 is a photograph showing an array of four test cells in the bath. The bath fluid is a 65% pure ethylene glycol and water solution.

Pure ethylene glycol is used so that the bath fluid will be transparent. The bath is cooled with the use of an R-502 refrigeration system. A temperature controller and a heater are installed to regulate the temperature of the bath. The bath is insulated on all sides to ensure a uniform bath temperature. Part of the insulation can be removed to allow for evaluation of the miscibility characteristics of each cell. A fluorescent light, mounted behind the bath, is used to help increase visibility.

Type T thermocouples are installed at several locations in the bath to measure an average bath temperature which is used by the temperature controller. The precise temperature of the bath fluid is measured by two internal resistance temperature detectors (RTD). These primary temperature probes consist of a platinum RTD connected to a signal conditioner/current transmitter that provides a linear response over the temperature range -60°F (-51°C) to 300°F (149°C). A microcomputer and data acquisition hardware under the direction of a data acquisition program monitors and records signals from all instrumentation. Table 1 gives a summary of the range and precision of the sensors.

Table 1. Summary of instruments, ranges, and precision

Instrument	Range	Precision
Platinum RTD and Signal Conditioner	-50°C + 150°C	±0.1°C
Critical Solubility Temperature	-50°C + 150°C	±1.0°C
Concentration Measurements	0% to 100%	±1%

One cell is assembled with an internal thermocouple to determine equilibrium (i.e., steady state) conditions. The temperature difference between this thermocouple and another mounted on the outside of the cell indicates when thermal equilibrium between the cell and the bath has been achieved. The cell is charged with pure oil to provide a "worst case" heat transfer. Steady-state conditions are typically achieved about one to two hours after a (e.g., 5° − 10 C) change in the circulating bath temperature.

EXPERIMENTAL PROCEDURE

Experimental procedures have been developed for measuring refrigerant-lubricant miscibility by using the test facility described previously. The desired amounts of refrigerant and lubricant are conveniently injected into each test cell from a charging station which was built with the specific purpose of simplifying the oil and refrigerant injection process. The injection process uses a piston cylinder, which acts similar to a syringe which is evacuated and filled with oil or refrigerant. Nitrogen pressure is then used to drive the fluid into the cell being charged. The distance in which the piston rod travels is correlated to the volume of fluid being injected. Since the density of the fluid being charged is known, the mass that is charged can be calculated.

Prior to the injection of any fluid for testing, the test cells are rinsed and cleaned to remove traces of any oil that had been previously tested. The seals around the windows may be replaced or fittings tightened if a failure to hold a vacuum or set pressure indicates that this is necessary, Next, the cells are evacuated, and the connections from the charging station are attached to a valve on the test cell.

Measured amounts of refrigerant and oil are injected to provide the desired volume and concentration of liquid. In addition, the cells are weighed on a scale before and after the injection of the oil and the refrigerant. The scale has an uncertainty of ±0.01 gram. The concentration of the liquid in each cell is calculated from the masses of refrigerant and oil injected along with corrections for the refrigerant vapor present in each cell. It is important to note that since the refrigerant vapor density changes as the temperature and pressure change, then the refrigerant vapor mass also varies. As a result, the liquid concentration varies slightly as the temperature and pressure change. This variation is discussed in more detail in the next section. For the experimental approach presented in this proposal, the vapor volumes are kept small; less than 5% of the total space is vapor. Therefore, the overall variation in refrigerant concentration as temperature and pressure are changed during any particular test are small.

The cells are then placed in the bath and cooled to desired temperatures whereupon pressures and temperatures are read after checking to ensure that equilibrium conditions exist in each cell. At each test temperature, the bath insulation is removed to facilitate visual inspection of each cell. The characteristics of the fluid in each cell are then noted. The temperature at which the liquid mixture separates into two phases is noted and called the critical solubility temperature for that concentration.

DATA ANALYSIS AND CORRELATION

For each lubricant and refrigerant pair, the data set consists of the temperature, composition, and miscibility characteristics for a series of test points. The miscibility characteristics consist of critical solubility temperature, the temperature at which a given concentration of a liquid refrigerant-lubricant mixture separates into two phases. These temperatures will be plotted on a graph of temperature versus refrigerant concentration. In addition, a nonlinear regression analysis will be performed to obtain equations for critical solubility temperatures as functions of refrigerant and lubricant concentrations. A sample plot of critical solubility temperatures versus concentrations for R-22 and a 3GS naphthenic oil is shown in Figure 4.

It is important to note that the refrigerant concentration data can be affected by pressure changes in the test cell as the temperature changes. At low temperatures such as -50°C, the density of the refrigerant vapor is negligible so that all of the refrigerant charged into a test cell (or sealed glass tube for that matter) can be considered to be in the liquid phase. Therefore, if the concentration of refrigerant in the liquid is 10%, then at temperatures higher than -50°C, the vapor density increases so that the liquid phase refrigerant concentration decreases. The vapor density can be found from the temperature and pressure (solubility data) for the mixture. With the vapor density and the vapor volume known, the mass of refrigerant in the vapor can be calculated. By subtracting the mass of refrigerant in the vapor, the mass of refrigerant in the liquid can be found, and therefore the concentration can be calculated. Table 2 shows a summary of how the liquid concentration varies with temperatures for a sample case of R-22 and 3GS naphthenic oil. The vapor volume in test cells are maintained less than 5% of the total volume, and, as shown in Table 2 the change in liquid concentration is negligible.

Table 2. Change in liquid refrigerant concentration due to pressure changes in vapor space (sample case is for R-22 and 3GS Naphthenic oil)

Volume of Vapor Space (% of total volume)	Refrigerant Concentration in Liquid			
	-50°C	0°C	20°C	60°C
50%	10%	9.3%	8.3%	7.5%
30%	10%	9.7%	9.2%	8.9%
5%	10%	~10%	9.9%	9.9%

ACKNOWLEDGMENTS

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2. VanGaalen, N.A., S.C. Zoz, and M.B. Pate, "A Test Facility for Measurement of Solubility, Viscosity, and Miscibility of Oil/Refrigerant Solutions at Conditions Relevant to Refrigeration Cycles," presented at the XVIIIth International Congress of Refrigeration, August 1991, Montreal, Canada.

FIGURES

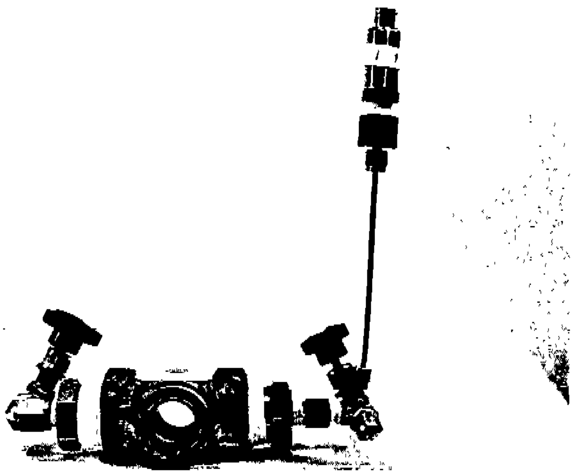

Figure 1 Miscibility and solubility test cell.

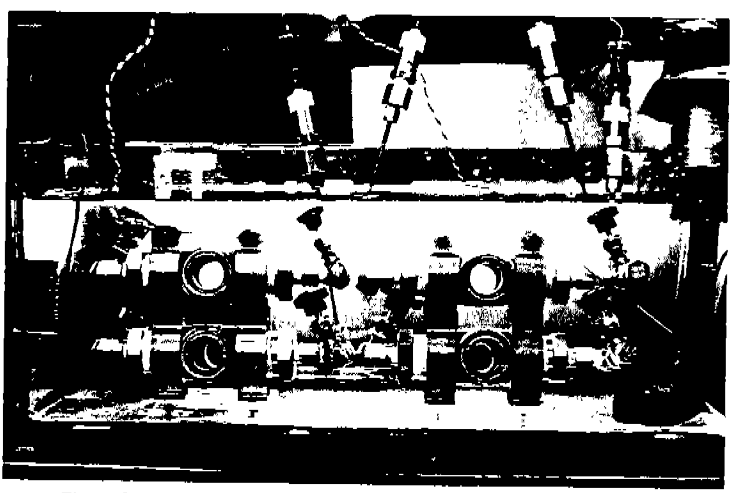

Figure 2 Temperature bath for the miscibility and solubility test facility.

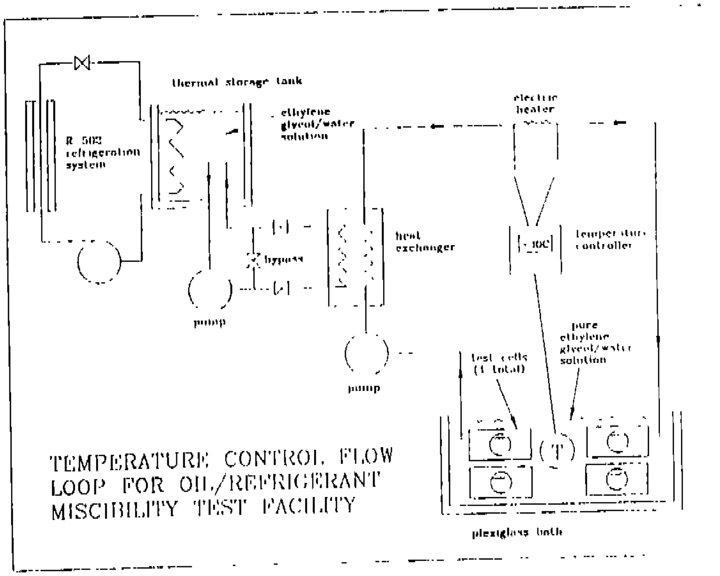

Figure 3 Temperature control flow loop for the miscibility and solubility test facility.

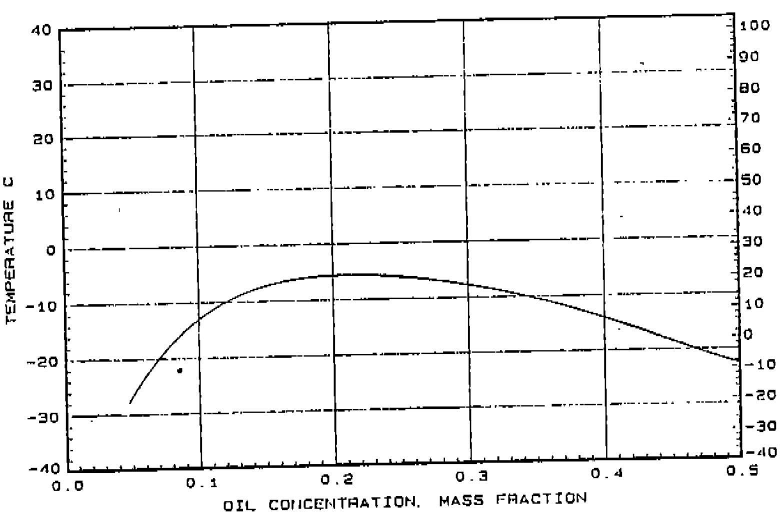

Figure 4 Critical solubility temperature for 3GS naphthenic oil and refrigerant 22 mixtures.