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Evaluation of Ikon®-12 Refrigerant for Motor Vehicle Air Conditioning

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ABSTRACT

A proprietary refrigerant, called Ikon®-12, was evaluated as an alternative to HFC (hydrofluorocarbon)-134a for automotive air conditioning. The evaluation was motivated by concern over the relatively high global warming potential of HFC-134a. In preliminary tests, Ikon®-12 was found to be compatible with a polyolester lubricant and engineering materials. Refrigeration capacity and efficiency for Ikon®-12 compared favorably to those for HFC-134a. In a preliminary durability test, Ikon®-12 refrigerant showed no significant chemical breakdown after extended operation with an elevated compressor discharge temperature. Further testing would be required to determine if stability and materials compatibility are acceptable for long-term use.

INTRODUCTION

HFC-134a is currently used as the refrigerant in new and retrofitted motor vehicle air conditioners, replacing the stratospheric ozone depleting refrigerant, CFC (chlorofluorocarbon)-12. Although HFC-134a has no ozone depletion potential, it has a global warming potential approximately 3300 times that of CO_2 (carbon dioxide) over a 20 year time horizon, 1300 times that of CO_2 over a 100 year time horizon, or 420 times that of CO_2 over a 500 year time horizon.[1] By the year 2005, approximately 260 million vehicles with HFC-134a air conditioners will be in service in developed countries and will require an estimated 74,800 metric tons of refrigerant annually including 33,400 metric tons for new vehicles and 41,400 metric tons for service usage.[2] Refrigerant emissions due to service usage represent an annual, CO_2-equivalent of 53.8 million metric tons, based on a 100 year time horizon. In 1994, the estimated total U.S. greenhouse gas emissions were 6109 million metric tons CO_2-equivalent.[3] Increased concern over global climate change could eventually lead to restrictions on HFC-134a consumption. For this reason, the U.S. EPA (Environmental Protection Agency) has evaluated alternatives for HFC-134a in motor vehicle air conditioning.[4]

A proprietary, near-azeotropic blend of an FIC (fluoroiodocarbon) and an HFC, called Ikon®-12, has been proposed as a refrigerant for automotive air conditioning and other applications.[5] The global warming potential for Ikon®-12 has been reported to be 4 percent of that for HFC-134a.[5] Ikon®-12 has been reported to be nonflammable according to testing based on ASTM (American Society for Testing and Materials) methods E681-85 and E918-83.[6-7] Extensive toxicity testing has been reported for both components of the refrigerant blend. Reported results have been generally favorable, although toxicity may be somewhat higher than for HFC-134a. Under EPA's SNAP (Significant New Alternatives Policy) Program, Ikon®-12 has been approved for use in motor vehicles subject to use conditions including the use of unique fittings, the use of descriptive labels, a prohibition against topping off one refrigerant with another, and appropriate recovery or recycling equipment.[8]

LUBRICANT MISCIBILITY

Miscibility was tested for the Ikon®-12 refrigerant with a PAG (polyalkylene glycol) lubricant and a POE (polyolester) lubricant. The PAG lubricant was supplied by an OEM (original equipment manufacturer). Its viscosity was reported to be 135 cSt at 40°C and 25 cSt at 100°C. It was reported to contain proprietary additives. The POE lubricant was supplied by a chemical manufacturer. Its viscosity was reported to be 134 cSt at 40°C and 15 cSt at 100°C. It also contained proprietary additives. Sealed glass tubes were prepared with the Ikon®-12 refrigerant and lubricant at lubricant concentrations of 2, 5, 10, 20, and 30 weight percent.

Complete miscibility with the PAG lubricant was found at low temperatures with all sample concentrations remaining miscible down to -45°C. However, separation of the refrigerant and PAG lubricant occurred at approximately 95°C for the 2 and 5 weight percent samples. The 10, 20, and 30 weight percent samples remained miscible up to 125°C, at which point the test was terminated.

Miscibility with the POE lubricant was tested over the temperature range of -45 to +115°C. No phase separation was observed for all concentrations, indicating complete miscibility of the Ikon®-12 refrigerant and the POE lubricant at the conditions tested.

STABILITY AND MATERIALS COMPATIBILITY

As a preliminary evaluation of the thermal and hydrolytic stability of Ikon®-12 and its compatibility with common materials of construction used in automotive air conditioners, sets of sealed glass tube samples were prepared. Materials were selected in preparation for testing the refrigerant in an automotive air-conditioning system designed for HFC-134a. Materials compatibility and stability were tested in accordance with the methods described in ANSI/ASHRAE Standard 97-1989.[9] Stability tests were conducted by sustained heating of the Ikon®-12 refrigerant by itself and with copper, aluminum, and steel in sealed glass tubes at 175°C for 14 days. Compatibility tests were conducted by sustained heating of elastomers, polymers, desiccant, cast iron, and brass with the refrigerant at 125°C for 14 days. One set of tubes was prepared without any lubricant, one set was prepared with PAG lubricant, and one set was prepared with POE lubricant. Duplicate samples were prepared and tested for each combination.

Sealed tubes containing Ikon®-12 refrigerant by itself were visually inspected after sustained heating at 175°C for 14 days. The fluid changed from colorless to purple color, indicating the presence of iodine. Volatiles released upon breaking the tubes under vacuum were analyzed by gas chromatography, mass spectrometry, and infrared spectrometry. The resultant chromatograms and spectra were compared to those obtained for pure refrigerant. A small amount of breakdown of the FIC was found as evidenced by the formation of a minute quantity of CF_3H (trifluoromethane). The addition of copper, aluminum, and steel did not affect the results significantly. The addition of a small amount of water also did not affect the results.

Sealed tubes containing Ikon®-12 refrigerant in combination individually with buna-n, neoprene, 6/6 nylon, Teflon, cast iron, and brass were maintained at a temperature of 125°C for 14 days. Chemical analysis of the volatiles released from those tubes indicated the formation of a minute amount of CF_3H.

Visual inspection of sealed tubes containing Ikon®-12 and PAG lubricant showed substantial decomposition as evidenced by the presence of carbonized solids after the 14-day aging period. Vapor-phase contents of one tube containing only the Ikon®-12 and PAG lubricant were analyzed by gas chromatography and mass spectrometry. A host of degradation products were evident from the analyses indicating that the incompatibility of Ikon®-12 and PAG lubricant was independent of the presence of other materials. Vapor-phase components identified from the analyses suggest that the PAG lubricant and both components of the Ikon®-12 blend decomposed. Analysis of the contents of other tubes containing materials with the PAG lubricant was not continued because of the obvious instability. The PAG lubricant was considered unacceptable for use with the Ikon®-12 refrigerant.

Sealed tubes containing Ikon®-12 and the POE lubricant appeared from visual inspection to have better chemical stability than the tubes containing the PAG lubricant. Stability of the refrigerant and lubricant combination was tested by sustained heating at 175°C for 14 days. Volatiles released upon breaking the tubes were analyzed. The addition of the POE lubricant resulted in a greater decomposition of the FIC than without the lubricant, but only a relatively small amount of decomposition was found. Further work would be required to quantify the decomposition. For comparison, HFC-134a with PAG lubricant typically has no discernible breakdown at 175°C. However, mineral oil lubricant used with CFC-12 refrigerant typically has substantial breakdown at 175°C.

When copper, aluminum, and steel were added to the mixture of refrigerant and lubricant, greater decomposition of the FIC was found as evidenced by the formation of larger concentrations of CF_3H and the deposition of salts on the metal surfaces. The combination of Ikon®-12, POE lubricant, and metals also yielded trace amounts of CO (carbon monoxide) and monofluoroethene. The presence of the monofluoroethene indicated a probable degradation of the HFC component of the refrigerant blend.

Sealed tubes containing Ikon®-12 and the POE lubricant in combination individually with buna-n, neoprene, 6/6 nylon, Teflon, XH-7 desiccant, cast iron, and brass were maintained at a temperature of 125°C for 14 days. Chemical analysis of the volatiles released from those tubes indicated a small amount of decomposition of the refrigerant. In the presence of the desiccant, there was a very slight formation of monofluoroethene, again indicating a probable degradation of the HFC component of the refrigerant blend.

Data for materials tested with Ikon®-12 in sealed tubes with and without POE lubricant are summarized in Table 1. Average values for each pair of tubes are reported. Hardness values were not obtained for nylon

Table 1. Materials Compatibility Test Results

Material	Weight Change (Percent)	Volume Change (Percent)	Linear Swell (Percent)	Dia. Change (Percent)	Hard. Change (Percent)
Buna-N	+ 37.2	+ 8.4	+ 9.6	+ 4.1	+ 7.3
Buna-N (with POE lube)	+ 20.1	+ 10.0	+ 5.7	+ 4.9	+ 12.9
Neoprene	+12.5	+7.5	+2.8	+2.3	+13.7
Neoprene (with POE lube)	+7.3	+10.2	+2.7	+4.1	+2.1
Teflon	+ 4.8	- 8.9	+ 5.2	- 4.6	not tested
Teflon (with POE lube)	+ 2.7	- 6.4	+ 4.7	- 3.3	not tested
Nylon 6/6	+ 12.9	+ 5.8	+1.1	not tested	not tested
Nylon 6/6 (with POE lube)	- 9.4	- 10.1	- 4.4	not tested	not tested

and Teflon since these harder materials were not amenable to the test method. With the POE lubricant, both Teflon and nylon shrank in volume while buna-n swelled in volume. For Teflon and nylon, volume change was excessive compared to generally accepted criteria for volume change from -5 to +25 percent.

It was concluded that the stability and compatibility of the Ikon®-12 refrigerant and POE lubricant combination were adequate to proceed with performance testing at temperatures not exceeding 125°C. Further laboratory and field testing would be required to determine if stability and materials compatibility are acceptable for long-term use.

Refrigeration Capacity and Energy Efficiency

Refrigeration capacity and COP (coefficient of performance) were experimentally determined for Ikon®-12 and for HFC-134a with an instrumented, automotive air-conditioning system. Instrumentation was similar to that described in a previous paper.[10] The automotive air-conditioning system was an OEM unit designed for HFC-134a. Refrigeration capacity was measured with an uncertainty of approximately ±100W and a repeatability of approximately ±30W. COP was determined from the measured power input to the compressor with an uncertainty of approximately ±75W and a repeatability of ±50W.

The OEM air-conditioning system had a suction line accumulator and orifice tube expansion device. Refrigerant was added to the system until liquid was present in the accumulator as indicated by minimal superheat at the compressor inlet. The refrigerant charge size was 920 g for HFC-134a and 1520 g for Ikon®-12. The same orifice tube was used for both refrigerants. Approximately the same amount of subcooling was measured at the orifice tube inlet for both refrigerants at each test condition. Test results are shown in Figure 1. Refrigeration capacity and COP for Ikon®-12 and HFC-134a were measured at three compressor rotational speeds, three evaporating temperatures, and three condensing temperatures. Over the range of test conditions, refrigeration capacity for Ikon®-12 averaged 5 percent higher than for HFC-134a. COP for Ikon®-12 averaged 11 percent higher than for HFC-134a. Compressor discharge temperatures with Ikon®-12 averaged 2°C higher than with HFC-134a. Over the range of test conditions, the compressor discharge temperature with Ikon®-12 varied from 0.8°C lower to 6°C higher than with HFC-134a.

Durability

Following the performance tests, the test system was run for an extended period of time with an elevated compressor discharge temperature to determine if refrigerant decomposition would occur. Test conditions were adjusted to obtain a compressor discharge temperature of 110°C. Compressor suction pressure was 338 kPa and discharge pressure was 2614 kPa. Compressor rotational speed was 2050 rpm, and compressor ambient temperature was 70°C. After 192 hours of continuous operation at those conditions, mechanical failure of a compressor suction reed valve occurred. The Ikon®-12 refrigerant was recovered from the system, and the compressor was disassembled and inspected. The broken reed valve damaged the cylinder surface, but there was no evidence of excessive wear or any other problem. Failure of the reed valve was apparently caused by metal fatigue due to the severe operating conditions. The lubricant remained clear, and no deposits were found on the internal surfaces of the compressor.

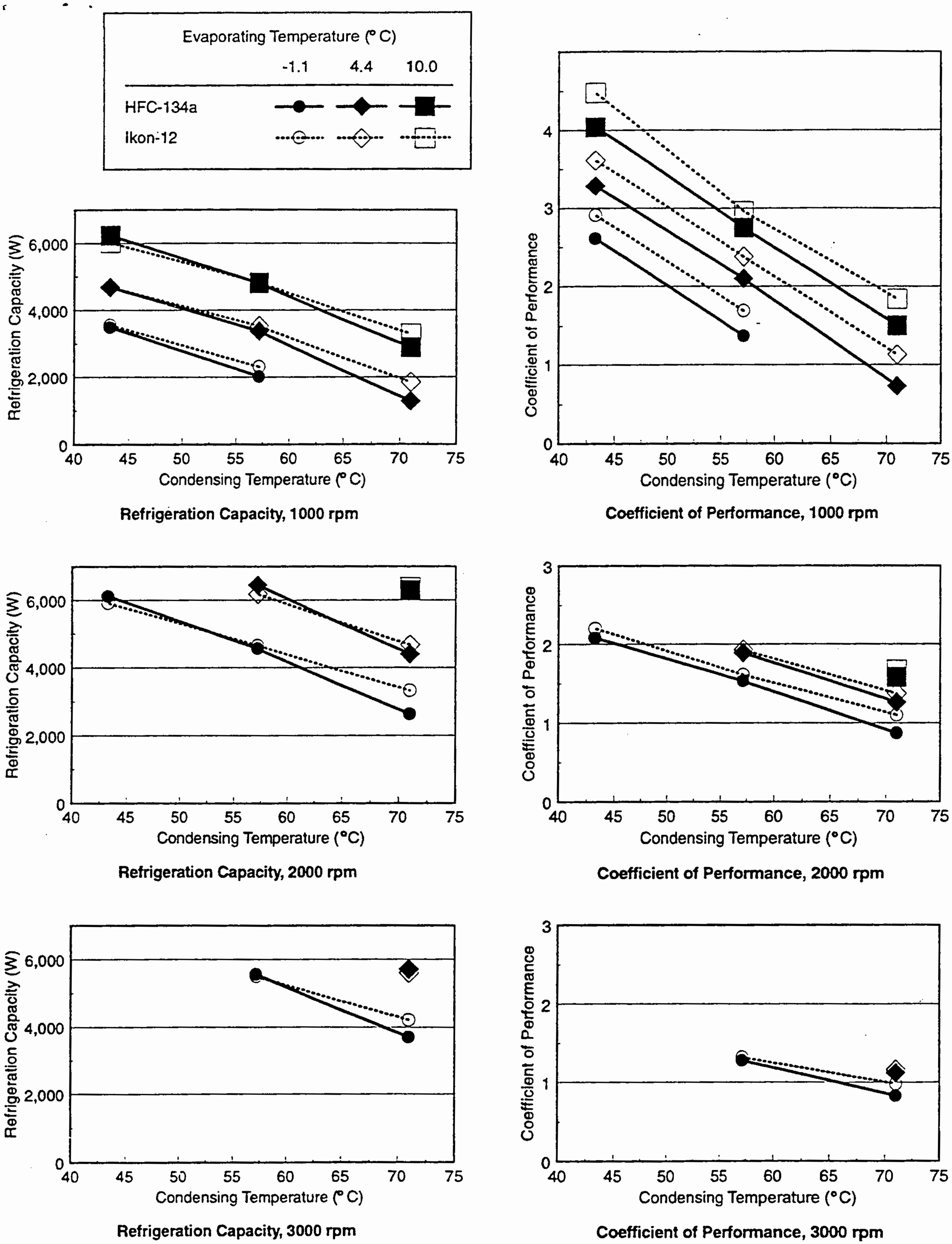

Figure 1. Refrigeration Capacity and Coefficient of Performance of Ikon-12 Compared to HFC-134a

Following the compressor failure, samples of refrigerant and lubricant were analyzed. Vapor- and liquid-phase materials were analyzed with gas chromatography, mass spectrometry, and infrared spectrometry. The resultant chromatograms and spectra were compared to those obtained for pure refrigerant and lubricant. There was no evidence of refrigerant breakdown in the vapor or liquid. Purity of the Ikon®-12 was greater than 99.5 percent.

The failed compressor was replaced with a similar, new compressor. Before installation, PAG lubricant was flushed from the new compressor with POE lubricant, and approximately 40 ml of the new POE lubricant was added to the new compressor to replace the amount removed with the failed compressor. The total amount of lubricant in the air-conditioning system was approximately 240 ml. The same Ikon®-12 refrigerant used for the previous testing was recharged into the system. Test conditions were adjusted to maintain the 110°C compressor discharge temperature, but the compressor rotational speed was reduced to prevent premature failure of the valves. Compressor rotational speed was 1000 rpm, and compressor ambient temperature was 90°C. Compressor suction pressure was 479 kPa and discharge pressure was 2867 kPa.

Refrigerant and lubricant samples were analyzed after 467 hours and again after 632 hours of operation, including the 192 hours of operation on the first compressor. Gas chromatography/mass spectrometry analyses of the refrigerant vapor and gas chromatography/Fourier transform infrared analyses of the lubricant showed no significant changes over the 632 hour period.

After 732 hours of operation, a leak in a refrigerant line resulted in the loss of the refrigerant charge. A lubricant sample was analyzed with gas chromatography/Fourier transform infrared, and no significant change was found. During inspection of the test system, it was found that the compressor shaft could not be rotated by hand. When torque was applied to the compressor shaft with a wrench, the moving parts loosened, and the shaft could then be turned by hand. The compressor was disassembled and inspected. A dark, viscous fluid was found in small quantities on some bearing surfaces and in some cavities inside the compressor. No excessive wear was found on any surfaces, and no indication of mechanical failure was observed. Lubricant found inside the compressor remained clear. Since no similar dark substance was found inside the first compressor after 192 hours of operation, and since Ikon®-12 refrigerant was known to be incompatible with PAG lubricant, it was suspected that the foreign substance resulted from a small amount of PAG lubricant remaining in the second compressor after flushing. An infrared spectral analysis of the dark, viscous fluid exhibited general features resembling both PAG and POE lubricants. However, the fluid could not be unambiguously traced to residual PAG lubricant left in the compressor. Further durability testing and chemical analyses were beyond the scope of the project. Further testing would be required to determine if stability and durability are acceptable for long-term use.

Summary

1. A proprietary, near-azeotropic, low-GWP, refrigerant blend of an FIC and an HFC, called Ikon®-12, was evaluated as an alternative to HFC-134a for automotive air conditioning. The evaluation was motivated by concern over the relatively high global warming potential of HFC-134a.

2. Ikon®-12 was not compatible with a PAG lubricant, but was sufficiently compatible and stable with a POE lubricant and engineering materials to proceed with testing in an automotive air-conditioning system. Over the temperature range of -35 to +100°C, the Ikon®-12 refrigerant was completely miscible with the POE lubricant.

3. Refrigeration capacity and COP were determined experimentally with an instrumented, OEM, automotive air-conditioning system. Refrigeration capacity for Ikon®-12 averaged 5 percent higher than for HFC-134a over the range of test conditions. COP for Ikon®-12 averaged 11 percent higher than for HFC-134a.

4. Durability of the refrigerant and lubricant was tested by operating the test system with an elevated compressor discharge temperature. During the course of the testing, a compressor suction reed valve was broken. Failure of the reed was apparently caused by metal fatigue due to the severe operating conditions. After 632 hours of operation with a discharge temperature of 110°C, no significant breakdown of the refrigerant or lubricant was detected from chemical analyses by gas chromatography, mass spectroscopy, and infrared spectroscopy.

5. Following the durability test, the compressor was disassembled and inspected. A small quantity of dark, viscous fluid was found inside the compressor and may have been caused by residual PAG lubricant. However, the fluid could not be positively identified by chemical analyses.

6. Further laboratory and field testing would be required to determine if stability and materials compatibility are acceptable for long-term use.

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16. ABSTRACT The paper gives results of an evaluation of a proprietary refrigerant, Ikon-12, as an alternative to hydrofluorocarbon (HFC)-134a for automotive air conditioning. The evaluation was motivated by concern over the relatively high global warming potential of HFC-134a. In preliminary tests, Ikon-12 was found to be compatible with a polyolester lubricant and engineering materials. Refrigeration capacity and efficiency for Ikon-12 compared favorably to those for HFC-134a. In a preliminary durability test, Ikon-12 refrigerant showed no significant chemical breakdown after extended operation with an elevated compressor discharge temperature. Further testing would be required to determine if stability and materials compatibility are acceptable for long-term use.

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Pollution	Pollution Prevention	13B
Refrigerants	Mobile Sources	13A
Motor Vehicles	Ikon-12	13F
Air Conditioning	Hydrofluorocarbons	
Halohydrocarbons	Global Warming	07C
Greenhouse Effect		04A
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