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REVISED ADDENDUM TO HFC-134a LUBRICANT STUDY

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**SAE COOPERATIVE
RESEARCH PROGRAM**

**REVISED ADDENDUM TO
HFC-134a LUBRICANT STUDY**

**THIS REVISED ADDENDUM SUPERSEDES
THE ORIGINAL ADDENDUM DATED MAY 4, 1993
WHICH SHOULD BE DISCARDED**



INTRODUCTION

This report gives results of testing on three additional samples submitted to the Society of Automotive Engineers program entitled "SAE Cooperative Research Program - HFC-134a Lubricant Study." The initial study included eighteen other lubricants; test data on these may be found in Spauschus Associates, Inc. report titled as above dated November 6, 1992.

1.0 SAMPLES AND SAMPLE CHARACTERIZATION

Two liter samples of each lubricant were submitted to SAE, which were coded and forwarded for inclusion in the program. The lubricants are numbered LS-021, LS-022 and LS-023, in keeping with the numbering system given in the report referenced above.

Lubricants were stored in tightly capped containers during the course of the program and samples were withdrawn from these to carefully avoid contamination of the sample bottle. Some tests required withdrawing an amount slightly larger than actually consumed; any remaining lubricant was discarded and not returned to the sample bottle. The total amount of time that the samples were exposed to atmospheric conditions was kept to the bare minimum to avoid moisture absorption.

Basic property information requested from the suppliers included:

- Viscosity at 40°C
- Viscosity Index
- Moisture Content (as shipped)
- Water Solubility
- Moisture Absorption
- HFC-134a Miscibility
- Sealed Tubes Stability

This information is summarized in Tables 1, 2 and 3 which follow.

<u>Sample</u>	<u>Viscosity at 40C(104F)</u>	<u>Viscosity Index</u>	<u>Moisture¹ as shipped</u>	<u>Moisture at saturation</u>	<u>Water solubility</u>
LS-021	133.8	206	> 100	N/A ²	No
LS-022	133.8	206	> 100	N/A	No
LS-023	72.0	210	200	1.7%	N/A

¹Units are ppm.

²N/A = Not Available

**TABLE1: SUPPLIER BASIC PROPERTY DATA -
VISCOSITY, MOISTURE AND WATER SOLUBILITY**

<u>Sample</u>	<u>PHASE SEPARATION TEMPERATURES IN DEGREES CELSIUS</u>					
	<u>10% Lubricant</u>		<u>20% Lubricant</u>		<u>30% Lubricant</u>	
	<u>Lower</u>	<u>Upper</u>	<u>Lower</u>	<u>Upper</u>	<u>Lower</u>	<u>Upper</u>
LS-021	N/A ¹	N/A	N/A	N/A	N/A	N/A
LS-022	N/A	N/A	N/A	N/A	N/A	N/A
LS-023	N/A	N/A	N/A	N/A	N/A	N/A

¹N/A = Not Available

**TABLE 2: SUPPLIER BASIC PROPERTY DATA - MISCIBILITY
OF NEAT LUBRICANTS WITH HFC-134a**

<u>Sample</u>	<u>Color Change</u>		<u>Fluoride Ion Generated, ppm</u>	<u>TAN¹</u>		<u>Visual Inspection</u>
	<u>From</u>	<u>To</u>		<u>From</u>	<u>To</u>	
LS-021	L0.5 ²	L0.5	N/A ³	N/A	N/A	N/A
LS-022	L0.5	L0.5	N/A	N/A	N/A	N/A
LS-023	N/A	L0.5	N/A	N/A	N/A	No deposits, no corrosion, slight copper transfer on steel, none on Al.

¹Total Acid Number

²N/A = Not Available

³Color Scale Not Defined

TABLE 3: SUPPLIER BASIC PROPERTY DATA - STABILITY



2.0 HYGROSCOPICITY

Lubricant samples were placed in clean, dry 250 ml beakers, inside diameter 2.5 in., which were filled to 1.875 in. height, giving a surface area to depth ratio of 2.628 in. The samples were placed in a controlled environment at average conditions of 25°C, 50% relative humidity for a period of one week. Samples of each lubricant were taken periodically at a depth of 1 in. beneath the surface of the oil and analyzed by Karl Fischer Model 447 Coulomatic Titrimeter. Results are given in Figures 1 through 3, which show the data points along with a smooth curve found by least squares regression. The "as received" moisture content and the moisture content after 168 hours exposure is given in Table 4.

<u>Sample</u>	<u>As Received ppm</u>	<u>After 168 hrs ppm</u>
LS-021	25	5,330
LS-022	159	5,230
LS-023	171	11,930

TABLE 4: MOISTURE CONTENT OF LUBRICANT SAMPLES
"AS RECEIVED" AND AT 168 HOURS EXPOSURE

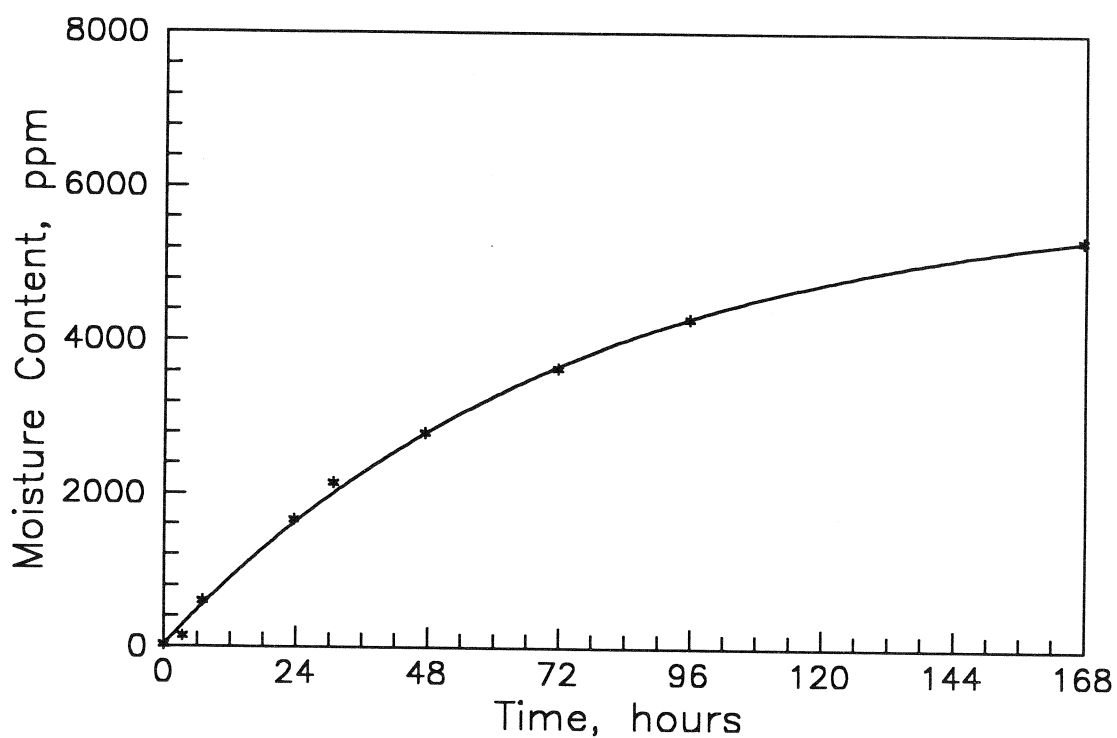
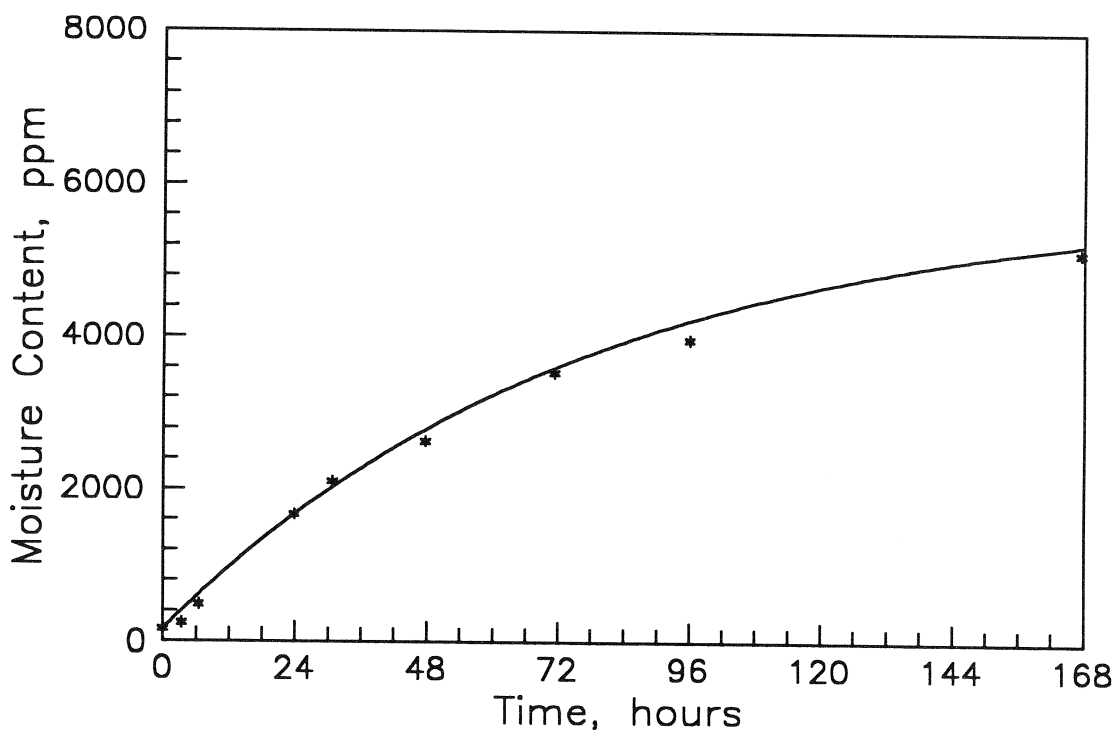
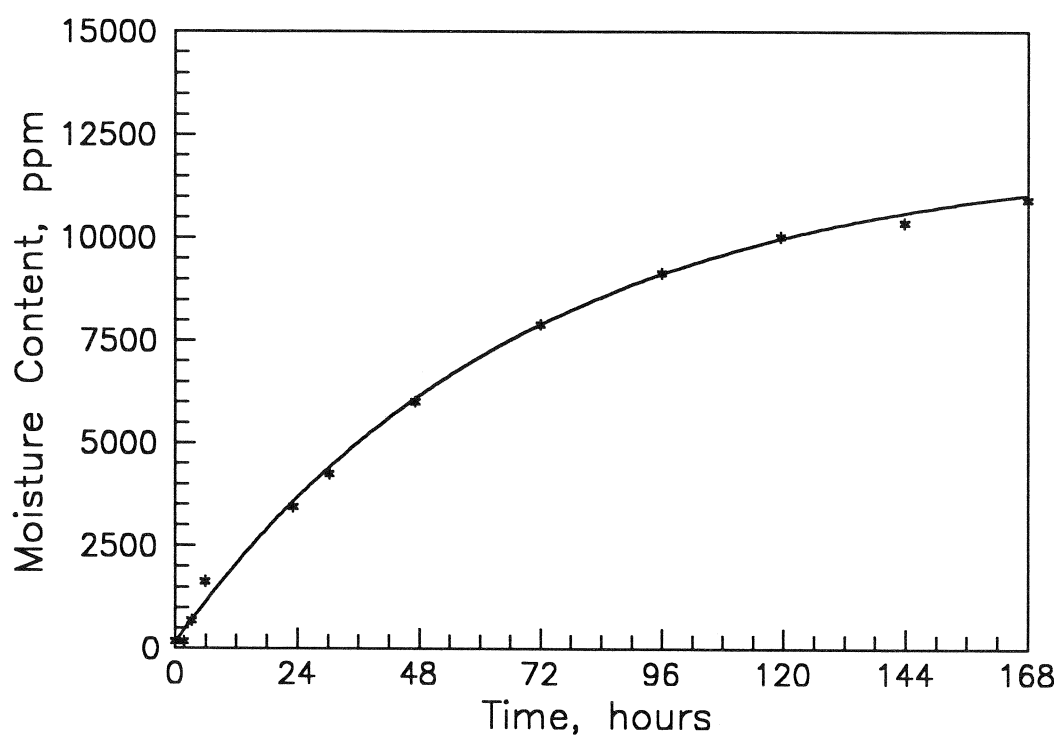
FIG 1: MOISTURE PICKUP OF SAMPLE LS-021**FIG 2: MOISTURE PICKUP OF SAMPLE LS-022**



FIG 3: MOISTURE PICKUP OF SAMPLE LS-023



3.0 MISCIBILITY

Sealed glass tubes were prepared containing 20% lubricant/80% HFC-134a, where 20% lubricant consists of 90% synthetic/10% mixed mineral oil. Mixed mineral oil is a 50/50% mixture of Idemitsu YN-9 and BVM-100N.

The tubes were sealed and allowed to warm to room temperature, where the phase conditions were noted; all were miscible (one phase) at 25°C. The samples were then cooled from room temperature to -40°C at a very slow rate; observations were made of the temperature at which a third phase appeared. A similar procedure was followed for the heating data, placing the tubes into an oven and gradually raising temperature while observing the contents for phase separation. The temperature at which this third phase appeared is given in Table 5.

<u>Sample</u>	<u>Cooling Phase Separation Temp, C</u>	<u>Heating Phase Separation Temp, C</u>
LS-021	< -40	31
LS-022	< -40	31
LS-023	< -40	41

TABLE 5: MISCIBILITY OF LUBRICANT SAMPLES WITH HFC-134a



4.0 STABILITY

Sealed glass tubes were prepared according to ANSI/ASHRAE 97-1989 containing equal volumes (1 cc) of oil and HFC-134a, where the 1cc of oil consisted of 90% synthetic/10% mixed mineral oil, and the HFC-134a was doped with 1% CFC-12. This series of tubes was prepared at two moisture levels, using the synthetic lubricants as received and samples at the moisture level given in Table 4 after 168 hours exposure. Each tube also contained metal catalysts of a copper/aluminum/steel couple linked by aluminum wire, arranged so that the copper is not in direct contact with the steel.

Five replicate tubes were prepared for each sample at the two moisture levels, for a total of 30 tubes. These tubes were then aged in large aluminum blocks for 14 days at 175°C; after aging, visual inspection was performed for corrosion, film formation, extracts, wall deposits and copper transfer. Color change of the liquid phase was determined by comparison to ASTM D 1500 standards. Two of the aged tubes were used for chloride and fluoride determination by specific ion electrode and two tubes were opened for measurement of total acid number according to ASTM D 664. Acid number of the as received lubricants was also measured; both titrations were carried out to a final pH of 11.0

Tables 6 and 7 which follow give the results of these tests for the as received and the high moisture tubes respectively. Percent refrigerant reacted has been calculated by assuming that each halide atom present was the result of the destruction of one refrigerant molecule. The quantity of chloride found in the tubes after aging is reported in micrograms.

<u>Sample</u>	<u>Color Change</u>		<u>Chloride ion, μg</u>	<u>%R-134a Reacted</u>	<u>TAN Change</u>		<u>Visual Inspection</u>
	<u>From</u>	<u>To</u>			<u>From</u>	<u>To</u>	
LS-021	2.0	2.5	65	0.022	<0.02	0.20	Al clean and bright.
			102	0.024	<0.02	0.17	Small amount of white flaky extracts. Fe slightly tarnished, Cu slightly pink.
LS-022	2.0	3.0	48	0.138	<0.02	0.41	Al clean and bright.
			0	0.111	<0.02	0.29	Heavy white extracts. Fe slightly tarnished, Cu slightly pink.
LS-023	2.0	4.5	12	0.002	<0.02	0.88	Al and Fe clean and
			12	0.001	<0.02	0.87	bright. Cu slightly pink. Small amount of extracts present.



<u>Sample</u>	<u>Color Change</u>		<u>Chloride ion, μg</u>	<u>%R-134a Reacted</u>	<u>TAN Change</u>		<u>Visual Inspection</u>
	<u>From</u>	<u>To</u>			<u>From</u>	<u>To</u>	
LS-021	2.0	2.5	199	0.007	<0.02	0.20	Al clean and bright.
			199	0.007	<0.02	0.18	Small smount of white flaky extracts. Fe slightly tarnished, Cu slightly pink.
LS-022	2.0	2.5	12	0.002	<0.02	<0.02	Al clean and bright.
			79	<0.001	<0.02	<0.02	Heavy white extracts. Fe slightly tarnished, Cu slightly pink.
LS-023	2.0	4.5	33	0.009	<0.02	2.62	Al and Fe clean and
			37	0.009	<0.02	2.33	bright. Cu slightly pink. Small amount of extracts present.

TABLE 7: STABILITY DATA - HIGH MOISTURE LUBRICANTS