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**Sealed Tube Comparisons
of the Compatibility of Desiccants
with Refrigerants and Lubricants**

Quarterly Technical Progress Report

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I. Abstract

Continuing environmental concerns mandate replacement of CFC's with alternate refrigeration fluids. At this time relatively little testing has been reported in the literature for compatibility of desiccants in these new working fluids. Using bench scale test methods generally accepted throughout the industry today, this work will provide data necessary to assess the compatibility of virtually all of the currently used desiccant types - both bead and molded core, with thirteen refrigerant/lubricant combinations. The desiccants are tested by exposure to refrigerant and lubricant in sealed, glass tubes in accordance with ASHRAE/ANSI Standard 97-1989. After aging, the liquid phase is evaluated for acid anion formation, change in color, and presence of halide ions, the gas phase is analyzed for refrigerant decomposition by gas chromatography, and the desiccants are evaluated for changes in crush strength and for retention of acids and halide ions. Metal catalysts, also present in the sealed tubes, are visually examined for corrosion, copper plating, and appearance changes.

II. Scope

This project determines the compatibility of sixteen desiccants in thirteen refrigerant/lubricant combinations using bench-scale sealed tube tests. There are eight desiccant categories, as shown in Table II.1. In each category samples have been obtained from two suppliers to yield a total of sixteen desiccants. There are ten refrigerants and four lubricants combined to give the thirteen combinations shown in Table II.2. The tests are conducted with two levels of moisture - less than fifty parts per million moisture and at one thousand parts per million moisture.

Table II.1. Desiccants Under Study

1. 4A Molecular Sieve	7. Core Type without Carbon
2. 3A Molecular Sieve	15 to 30% molecular sieve type 3A
3. Alumina	Alumina
4. Silica Gel	10 to 20% phosphate binder
5. Core type with Carbon	8. Core Type without Carbon
10 to 25% molecular sieve type 3A	15 to 30% molecular sieve type 3A
Alumina	Alumina
5 to 15% carbon	10 to 20% phosphate binder
10 to 20% phosphate binder	
6. Core type with Carbon	
10 to 25% molecular sieve type 4A	
Alumina	
5 to 15% carbon	
10 to 20% phosphate binder	

Table II.2. Refrigerant/Lubricant Combinations Under Study

1. R-11 with naphthenic mineral oil
2. R-12 with naphthenic mineral oil
3. R-22 with naphthenic mineral oil
4. R-123 with naphthenic mineral oil
5. R-134a with penta erythritol mixed acid polyolester lubricant
6. R-134a with penta erythritol branched acid polyolester lubricant
7. R-152a with alkylbenzene lubricant (or with penta erythritol mixed acid polyolester lubricant)
8. R-32 with penta erythritol mixed acid polyolester lubricant
9. R-32 with penta erythritol branched acid polyolester lubricant
10. R-124 with alkylbenzene lubricant
11. R-125 with penta erythritol mixed acid polyolester lubricant
12. R125 with penta erythritol branched acid polyolester lubricant
13. R143a with penta erythritol branched acid polyolester lubricant

III. Methodology

A. Sample Preparation

The test tubes are cleaned by rinsing first with deionized water, followed by two rinses with methanol and one rinse with toluene. The tubes are then dried at 175°C. Cleaned tubes are kept dry in desiccators prior to use.

Lubricants are tested for moisture and dried by evacuation to below 50ppm prior to use. Refrigerants are tested for moisture content prior to use. Those high in moisture are rejected and replaced. No attempts are made to dry the refrigerants on-site.

The metal catalyst coupons are prepared by punching 3.3x19.3mm coupons from thin sheet. The coupons are held together by aluminum wire such that the steel and copper are separated by the aluminum. These prepared coupons are thoroughly cleaned and kept dry prior use.

The desiccant samples are activated at 260°C (500°F) for four hours prior to sealing in the tubes. Desiccant samples in bead form are weighed to within one bead of 0.5 gram. Core type desiccants are cut into small pellets prior to drying. Three pellets are chosen and weighed to yield the specified 0.5 gram.

B. Tube Preparation and Aging

First, the desiccant and metal catalysts coupon are placed in the tube. A small wad of glass wool is inserted on top of the desiccant to keep the lubricant and desiccant from violently boiling from the tube when later opened for analysis. The tube is then necked down to a size through which a standard cannula can fit. Next the 1.0 cc of oil is added accurately using a

syringe and cannula. The tube is then evacuated to 30 microns followed by accurate charging with refrigerant, and moisture if required, using a calibrated gas handling system. Finally, the tube neck is sealed and annealed. This procedure minimizes oil contamination of the seal area, and most importantly, produces a minimally stressed glass seal which can better withstand the high pressure encountered at the aging temperature. Three tubes are made for each moisture condition, desiccant, and refrigerant/lubricant combination.

Each test tube is placed in a drilled hole in a large aluminum block which is heated in an air circulating oven at 149°C for 28 days. The aluminum block serves two purposes. The first is safety. Should any tube break at elevated temperature, the aluminum block contains the pieces. Secondly, the large mass of the block provides a stable temperature environment for the tubes during the aging period.

C. Analysis

1. Visual Inspection

Visual inspections are made on each tube after fourteen days and after 28 days of aging. Each tube is compared to standard liquid color references, which give a numerical value for the amount of color change from water white to jet black. Similarly, changes in presence of solid particulates, and formation of copper plating will be noted and scaled numerically. Also noted and recorded are the appearance of the metal catalysts and the desiccant.

2. Refrigerant Decomposition

The quantitative degree of refrigerant decomposition is determined by gas chromatographic (GC) analysis on the vapor phase of one tube from each group of three. The vapor phase is captured in an evacuated gas handling system and introduced to the GC helium effluent stream by a gas sampling valve. The GC is equipped with a Porapak Q column, and thermal conductivity detector. The method utilized is that reported in the earlier publish MCLR Project Number 650-50200: Chemical and Thermal Stability of Refrigerant-Lubricant Mixtures with Metals. The final report for this project is Report Number DOE/CE/23810-5.

3. Lubricant Total Acid Number

The total acid number is determined for one tube from each group of three. The procedure used follows that for ASTM 664. The method is modified to accommodate the small 1cc sample size from the sealed tubes by reducing the alcoholic KOH titrant concentration from 0.1 Normal to 0.01 Normal. This yields sufficient sensitivity to determine acid numbers down to 0.1 with the 1cc sample size.

4. Halide Ions and Acid Anions

Anion concentrations are determined for the tube liquid phase and desiccant by ion chromatography (IC). After aging, the liquid and vapor phase of one tube is separated from the desiccant. The desiccant is washed with toluene. This toluene wash is added to the liquid phase from the tube, which is then extracted in deionized water for 24 hours. The washed desiccant is also extracted in deionized water. The aqueous phases from these two extractions are then analyzed by IC for fluoride ion, chloride ion, organic anions (such as formate, acetate, butyrate) and inorganic anions (such as nitrate, sulfate).

In the development of this method, acid and basic aqueous solutions were studied as extractants. Both caused degradation of some unaged lubricants producing the very same acid anions that are produced by aging. Also, methanol was tried as an extractant, but this produced severe interference in the IC chromatogram. Methanol eludes from the IC column in a very broad peak covering most of the time period that the anions of interest elude.

The only reason for considering alternate extractants is for cases where water does not effectively extract the anions of interest. To determine if water is an effective extractant. A sample of lubricant was doped with hexanoic acid. The doped lubricant was extracted with deionized water and the aqueous phase from the extraction was analyzed for hexanoate ion. The method yielded a concentration representing 85% of the total hexanoic acid that was originally added to the lubricant. Of the anions of interest in this study, hexanoic acid is one of the least water-soluble and least sensitive anions to the IC detector. Based on these results, deionized water appears to be satisfactory for extraction of these samples.

5. Desiccant Crush Strength

Desiccant crush strength is determined on freshly activated material and then on material that has been aged. The change in crush strength is of most importance. For bead type materials, the crush strength is determined for at least ten individual beads, with the average and 95% confidence (2 σ) limits reported. For core type desiccants it is not possible to include enough sample in the sealed tubes to perform a statistical analysis. Therefore, only the average crush strength can be obtained from the few available specimens. The method utilizes a digital force gauge which displays the peak force achieved while the specimen is slowly pressed between two parallel plates. It is recognized that this technique is limited. It will not distinguish between two close contenders for instance. It will, however, point out any gross incompatibilities that cause disintegration of the desiccant - rendering it a system contaminant.

D. Distribution of Tests Between Sealed Tubes

Table III.1 below shows which of the analyses is conducted on each of the three tubes in a group. The reasoning behind this distribution stems from the nature of the tests and the condition of the specimens after the tests. For example, the test for Total Acid Number, shown for tube #3 in the table below, completely consumes the tube contents. Thus, there are no other analyses that can be performed with the contents of this tube.

Table III.1. Distribution of Tests Between Sealed Tubes.

<u>Test</u>	<u>Tube #1</u>	<u>Tube #2</u>	<u>Tube #3</u>
Visual Inspection	✓	✓	✓
Desiccant Crush Strength	✓		
GC Refrigerant Decomposition	✓		
Lubricant Total Acid Number			✓
Liquid Phase Halide Ion/Acid Anion		✓	
Desiccant Halide Ion/Acid Anion		✓	

IV. Data For As-Received Materials

A. Desiccants

As mentioned above, there are 8 categories of desiccants studied in this work. In each category one sample from two suppliers has been obtained. Table IV.1 lists the desiccants obtained along with the manufacturer's published contents, and the desiccant code letter that is used throughout this report to identify the various desiccants.

After completion of some of the preliminary tests to develop methods for this work, it was discovered that many of the desiccants contained appreciable levels of fluoride and chloride ion. Consequently, each desiccant was tested for anion concentration in the "as-received" state. Table IV.1 includes the anion concentrations found for each desiccant studied at this time. This information is repeated in the "Summary Test Results" tables in Section V below. Also the individual ion chromatograms for the "as-received" desiccants are included in the appropriate appendices.

Table IV.1 Desiccant Specifications

Code	Desiccant Type	Published Contents	Anions Found	Crush Strength
A	4A Molecular Sieve	4Å zeolite 75-85% MgAl(SiO ₂) 23-15% Quartz 2-0%	F 190 ppm Cl 15 ppm Heptanoate 1737 ppm SO ₄ 163 ppm	17.8 lbs
E	4A Molecular Sieve	Al ₂ O ₃ < 30% Na ₂ O < 30% SiO ₂ < 50% MgO < 5%	F 10 ppm Cl 11 ppm Heptanoate 430 ppm SO ₄ 104 ppm	31.5 lbs
F	3A Molecular Sieve	3Å zeolite 75-85% MgAl(SiO ₂) 23-15% Quartz 2-0%	F 159 ppm Cl 23 ppm Heptanoate 1262 ppm SO ₄ 414 ppm	19.7 lbs
H	3A Molecular Sieve	Al ₂ O ₃ < 40% K ₂ O < 15% Na ₂ O < 30% SiO ₂ < 50% MgO < 5%	F 18 ppm Cl 4 ppm Heptanoate 184 ppm SO ₄ 260 ppm	33.6 lbs
I	Alumina	Al ₂ O ₃ 93.6% Fe ₂ O ₃ 0.02% Na ₂ O 0.35% SiO ₂ 0.02%	F 10 ppm Cl 4626 ppm Formate 891 ppm Acetate 655 ppm	11.4 lbs

Code	Desiccant Type	Published Contents	Anions Found	Crush Strength
J	Alumina	Al_2O_3 90+ % Fe_2O_3 <0.1 % Na_2O <0.6 % SiO_2 <0.2 %	F 7 ppm Cl 56 ppm Formate 64 ppm Acetate 91 ppm Heptanoate 242 ppm SO_4 17 ppm	
K	Silica Gel	$SiO_2 \cdot xH_2O$ >95 %	F 3 ppm Cl 14 ppm SO_4 47 ppm	76.1 lbs
L	Silica Gel	$SiO_2 \cdot xH_2O$ >95 %	F 14 ppm Cl 22 ppm SO_4 860 ppm	
M	Core type with Carbon and 3A molecular sieve	3Å zeolite 22 % Carbon 4 % Phosphate 17 % Binder		
N	Core type with Carbon and 3A molecular sieve	3Å zeolite 9 % Carbon 19 % Phosphate 27 % Binder		
T	Core type with Carbon and 4A molecular sieve	4Å zeolite 19 % Carbon 28 % Phosphate 19 % Binder		
V	Core type with Carbon and 4A molecular sieve	4Å zeolite 8 % Carbon 26 % Phosphate 15 % Binder		
W	Core Type No Carbon and 3A molecular sieve	3Å zeolite 10 % Phosphate 18 % Binder		
X	Core Type No Carbon and 3A molecular sieve	3Å zeolite 20 % Phosphate 15 % Binder		

Code	Desiccant Type	Published Contents	Anions Found	Crush Strength
Y	Core Type No Carbon and 4A molecular sieve	4Å zeolite 15% Phosphate 13% Binder		
Z	Core Type No Carbon and 4A molecular sieve	4Å zeolite 17% Phosphate 13% Binder		

B. Refrigerants

As-received refrigerants are tested for moisture content by Karl-Fisher titration and for impurities by Gas Chromatography. Table IV.2 lists the refrigerants with their moisture content and the GC detected impurity peak-area percents and retention times, if any. The "Air" peak contains all non-condensibles. Most of this is probably a result of the sampling technique.

Table IV.2 As-Received Refrigerant Properties

Refrigerant	Moisture Content	GC Peaks		
		Peaks Found		Peak Area Percents
R-11	5 ppm			
R-12	< 1 ppm	0.32 min Air 1.98 min R-12		0.5% 99.5%
R-22	37 ppm	0.33 Air 1.52 R-22		0.2% 99.8%
R-32				
R-123				
R-124	16 ppm			
R-125				
R-134a	8 ppm	0.33 Air 1.28 R134a 3.08 Unkn		0.2% 99.6% 0.2%
R-143a	46 ppm			
R-152				

C. Lubricants

Lubricants are tested for acid number, ion concentration, and moisture content. These properties are listed in table IV.3 below for the four lubricants used in this work. All lubricants are dried to less than 30ppm moisture before using. The moisture values listed below are for the lubricants "as-received". Moisture content is determined by Karl-Fisher titration.

Table IV.3 Lubricant Properties

Lubricant	Moisture Content	Total Acid Number	Ions Found
Naphthenic Mineral Oil	18 ppm	<0.1	Tin 9 ppm Phosphorus 14 ppm Zinc 2 ppm
Alkylbenzene	51 ppm	<0.1	Aluminum 1 ppm Lead 1 ppm
penta erythritol mixed acid polyolester	366 ppm	<0.1	Tin 9 ppm Phosphorus 17 ppm Antimony 13 ppm Pentanoate 4 ppm
penta erythritol branched acid polyolester	162 ppm	0.1	Acetate 0.07 ppm Formate 0.005 ppm Pentanoate 0.6 ppm

V. Test Results

At this time tests are completed for six of the bead type desiccants in R-12/mineral oil. Please read the following "Code Key For Test Results" for information necessary to decipher the data in the "Summary Test Results" tables.

Supporting test results are presented for each desiccant in the appendices at the end of this report. Each appendix is labelled by the code letter given the desiccant and by the descriptive type of desiccant. In each appendix there is a table of crush strength test results with statistical treatment, gas chromatograms, and ion chromatograms.

4Å Molecular Sieve (See Tables A and E, and Appendices A and E)

Both desiccants performed the same in ability to contain the various anions. Desiccant A, however, started out with significantly higher concentrations of fluoride ion and organic anions. The crush strength for sample E was almost twice that of sample A. Neither desiccant showed evidence of refrigerant decomposition as determined by gas chromatography.

3Å Molecular Sieve (See Tables F and H, and Appendices F and H)

These two samples differed widely in just about all of the tests performed on the new and aged desiccants, with Sample H performing better than sample F. Also, sample F was the only bead

desiccant to cause refrigerant decomposition as determined by gas chromatography. Crush strength for sample H was double that for sample F. It should be noted that the as-received sample F contained much higher concentrations of anions than did the as-received sample H. This could be the cause for sample F's poorer performance.

Alumina (See Table I, and Appendix I)

Data for the dry condition for only one sample of alumina (sample I) was available at the time of this writing. The surprisingly low crush strength is due to the bead size for the sample. Whereas, the other bead desiccants are nominal 1/8" in diameter, this material has only 1/16" diameter. The only other noteworthy observation for the limited data for this material is the high organic anion (acetate) concentration on the new desiccant.

Silica Gel (See Table K, and Appendix K)

As was the case for alumina, data for the dry condition for only one sample of silica gel (sample K) was available at the time of this writing. This material had the lowest overall concentration of anions on the new desiccant of all the bead desiccants tested at this time. Also, the crush strength for this material, at 76 pounds, was the highest of all those tested so far.

Code Key
For
Test Results Tables

Liquid Color

Colors follow ASTM Standard D1500. However, 8 mm internal diameter is much less than that specified. Therefore, colors "0" through "2" appear the same. The first number listed is the color before aging and the second number is the color after aging.

- 2.0 Water clear
- 2.5 Very Faint Yellow
- 3.0 Pale Yellow
- 3.5 Light Yellow
- 4.0 Yellow
- 4.5 Yellow-Orange
- 5.0 Light Orange
- 5.5 Orange
- 6.0 Orange-Brown
- 6.5 Brown
- 7.0 Dark Brown
- 7.5 Brown-Black
- 8.0 Black

Copper Plating

- 0 none
- 1 spots on edges
- 2 edges covered
- 3 spots on surface
- 4 Partially coated surface
- 5 Fully coated surface

Solids Formation

- 0 None
- 1 small amount
- 2 medium amount
- 3 heavy amount

Moisture

- Dry < 10 ppm
- Wet 1000 ppm

Crush Strength

The value entered is the average Crush Strength in pounds.

GC % Refrigerant Reacted

Based on peak area ratios for largest decomposition product detected.

F ion In Liquid

The ppm by weight for the concentration of F ion in the liquid phase from the aged tube.

Cl ion In Liquid

The ppm by weight for the concentration of Cl ion in the liquid phase from the aged tube.

Total Acid Number

mg of KOH per gram of oil.

F ion on Desiccant

ppm based on weight of desiccant.

Cl ion on Desiccant

ppm based on weight of desiccant.

Organic Acid on Desiccant

Sum of the ppm results for all organic anions found. Based on the desiccant weight.

Desiccant; A - 4Å Molecular Sieve

Summary Test Results

Code	System Fluids	Liquid Color (2-8)	Copper Plating (0-5)	Solids Formation (0-3)	Crush Strength (lbs)	GC % Ref Reacted (wt %)	F ion in Liquid (ppm)	Cl ion in Liquid (ppm)	Total Acid Number (mg KOH)	F ion on Desiccant (ppm)	Cl ion on Desiccant (ppm)	Org Acid on Desic (ppm)
A-New	None	-	-	-	17.3	-	-	-	-	190	15	1700
A-12	50 ppm Moisture											
	R11/mineral oil											
	R12/mineral oil	2.5/4.5	0	0	13.3	0%	4	28	<0.1	670	1560	2600
	R22/mineral oil											
	R123/mineral oil											
	R134a/ mixed ester											
	R134a/branched ester											
	R152a/alkylbenzene											
	R32/mixed ester											
	R32/branched ester											
	R124/alkylbenzene											
	R125/mixed ester											
A-42	1000 ppm Moisture											
	R11/mineral oil											
	R12/mineral oil	2.5/4.5	0	0	13.1	0%	2	21	0.1	1240	1390	2970
	R22/mineral oil											
	R123/mineral oil											
	R134a/ mixed ester											
	R134a/branched ester											
	R152a/alkylbenzene											
	R32/mixed ester											
	R32/branched ester											
	R124/alkylbenzene											
	R125/mixed ester											
	R125/branched ester											
	R143a/branched ester											

Desiccant; E - 4Å Molecular Sieve

Summary Test Results

Code	System Fluids	Liquid Color (2-8)	Copper Plating (0-5)	Solids Formation (0-3)	Crush Strength (lbs)	GC % Ref Reacted (wt %)	F ion in Liquid (ppm)	Cl ion in Liquid (ppm)	Total Acid Number (mg KOH)	F ion on Desiccant (ppm)	Cl ion on Desiccant (ppm)	Org Acid on Desic (ppm)
E-New	None	-	-	-	30.9	-	-	-	-	10	11	430
	50 ppm Moisture											
	R11/mineral oil											
E-12	R12/mineral oil	2.5/3.5	0	0	27.0	0%	1	18	<0.1	440	660	610
	R22/mineral oil											
	R123/mineral oil											
	R134a/ mixed ester											
	R134a/branched ester											
	R152a/alkylbenzene											
	R32/mixed ester											
	R32/branched ester											
	R124/alkylbenzene											
	R125/mixed ester											
	R125/branched ester											
	R143a/branched ester											
	1000 ppm Moisture											
	R11/mineral oil											
E-42	R12/mineral oil	2.5/3.0	0	0	23.6	0%	1	18	<0.1	570	720	719
	R22/mineral oil											
	R123/mineral oil											
	R134a/ mixed ester											
	R134a/branched ester											
	R152a/alkylbenzene											
	R32/mixed ester											
	R32/branched ester											
	R124/alkylbenzene											
	R125/mixed ester											
	R125/branched ester											
	R143a/branched ester											

Desiccant; F - 3Å Molecular Sieve

Summary Test Results

Code	System Fluids	Liquid Color (2-8)	Copper Plating (0-5)	Solids Formation (0-3)	Crush Strength (lbs)	GC % Ref Reacted (wt %)	F ion in Liquid (ppm)	Cl ion in Liquid (ppm)	Total Acid Number (mg KOH)	F ion on Desiccant (ppm)	Cl ion on Desiccant (ppm)	Org Acid on Desic (ppm)
F-New	None	-	-	-	20.0	-	-	-	-	159	23	1300
	50 ppm Moisture											
	R11/mineral oil											
F-12	R12/mineral oil	2.5/4.5	0	0	18.0	0.14%	41	130	1.3	1600	6500	1900
	R22/mineral oil											
	R123/mineral oil											
	R134a/ mixed ester											
	R134a/branched ester											
	R152a/alkylbenzene											
	R32/mixed ester											
	R32/branched ester											
	R124/alkylbenzene											
	R125/mixed ester											
	R125/branched ester											
	R143a/branched ester											
	1000 ppm Moisture											
	R11/mineral oil											
F-42	R12/mineral oil	2.5/4.5	0	0	18.6	0.14%	52	76	<0.1	1400	5800	2100
	R22/mineral oil											
	R123/mineral oil											
	R134a/ mixed ester											
	R134a/branched ester											
	R152a/alkylbenzene											
	R32/mixed ester											
	R32/branched ester											
	R124/alkylbenzene											
	R125/mixed ester											
	R125/branched ester											
	R143a/branched ester											

Desiccant; H - 3Å Molecular Sieve

Summary Test Results

Code	System Fluids	Liquid Color (2-8)	Copper Plating (0-5)	Solids Formation (0-3)	Crush Strength (lbs)	GC % Ref Reacted (wt %)	F ion in Liquid (ppm)	Cl ion in Liquid (ppm)	Total Acid Number (mg KOH)	FI ion on Desiccant (ppm)	Cl ion on Desiccant (ppm)	Org Acid on Desic (ppm)
H-New	None	-	-	-	34.8	-	-	-	-	18	4	184
	50 ppm Moisture											
H-12	R11/mineral oil											
	R12/mineral oil	2.5/3.5	0	0	24.4	0%	4	24	<0.1	1000	1600	810
	R22/mineral oil											
	R123/mineral oil											
	R134a/ mixed ester											
	R134a/branched ester											
	R152a/alkylbenzene											
	R32/mixed ester											
	R32/branched ester											
	R124/alkylbenzene											
	R125/mixed ester											
	R125/branched ester											
	R143a/branched ester											
	1000 ppm Moisture											
H-42	R11/mineral oil											
	R12/mineral oil	2.5/4.0	0	0	24.5	0%	2	21	<0.1	432	754	745
	R22/mineral oil											
	R123/mineral oil											
	R134a/ mixed ester											
	R134a/branched ester											
	R152a/alkylbenzene											
	R32/mixed ester											
	R32/branched ester											
	R124/alkylbenzene											
	R125/mixed ester											
	R125/branched ester											
	R143a/branched ester											

Desiccant; I - Alumina

Summary Test Results

Code	System Fluids	I-New									
Org Acid											
on Desicc											
(ppm)											
Cl ion on											
Desiccant											
(ppm)											
F ion on											
Desiccant											
(ppm)											
Total Acid											
Number											
(mg KOH)											
Cl ion in											
Liquid											
(ppm)											
F ion in											
Liquid											
(ppm)											
GC % Ref											
Reacted											
(wt %)											
Crush											
Strength											
(lbs)											
Solids											
Formation											
(0-3)											
Copper											
Plating											
(0-5)											
Liquid											
Color											
(2-8)											
50 ppm Moisture											
R1 1/mineral oil											
R1 2/mineral oil											
R2 2/mineral oil											
R1 23/mineral oil											
R1 34a/ mixed ester											
R1 34a/branched ester											
R1 52a/alkylbenzene											
R3 2/mixed ester											
R3 2/branched ester											
R1 24/alkylbenzene											
R1 25/mixed ester											
R1 25/branched ester											
R1 43a/branched ester											
1000 ppm Moisture											
R1 1/mineral oil											
R1 2/mineral oil											
R2 2/mineral oil											
R1 23/mineral oil											
R1 34a/ mixed ester											
R1 34a/branched ester											
R1 52a/alkylbenzene											
R3 2/mixed ester											
R3 2/branched ester											
R1 24/alkylbenzene											
R1 25/mixed ester											
R1 25/branched ester											
R1 43a/branched ester											
I-12											
R1 1/mineral oil											
R1 2/mineral oil											
R2 2/mineral oil											
R1 23/mineral oil											
R1 34a/ mixed ester											
R1 34a/branched ester											
R1 52a/alkylbenzene											
R3 2/mixed ester											
R3 2/branched ester											
R1 24/alkylbenzene											
R1 25/mixed ester											
R1 25/branched ester											
R1 43a/branched ester											
50 ppm Moisture											
R1 1/mineral oil											
R1 2/mineral oil											
R2 2/mineral oil											
R1 23/mineral oil											
R1 34a/ mixed ester											
R1 34a/branched ester											
R1 52a/alkylbenzene											
R3 2/mixed ester											
R3 2/branched ester											
R1 24/alkylbenzene											
R1 25/mixed ester											
R1 25/branched ester											
R1 43a/branched ester											
1000 ppm Moisture											
R1 1/mineral oil											
R1 2/mineral oil											
R2 2/mineral oil											
R1 23/mineral oil											
R1 34a/ mixed ester											
R1 34a/branched ester											
R1 52a/alkylbenzene											
R3 2/mixed ester											
R3 2/branched ester											
R1 24/alkylbenzene											
R1 25/mixed ester											
R1 25/branched ester											
R1 43a/branched ester											
1000 ppm Moisture											
R1 1/mineral oil											
R1 2/mineral oil											
R2 2/mineral oil											
R1 23/mineral oil											
R1 34a/ mixed ester											
R1 34a/branched ester											
R1 52a/alkylbenzene											
R3 2/mixed ester											
R3 2/branched ester											
R1 24/alkylbenzene											
R1 25/mixed ester											
R1 25/branched ester											
R1 43a/branched ester											
1000 ppm Moisture											
R1 1/mineral oil											
R1 2/mineral oil											
R2 2/mineral oil											
R1 23/mineral oil											
R1 34a/ mixed ester											
R1 34a/branched ester											
R1 52a/alkylbenzene											
R3 2/mixed ester											
R3 2/branched ester											
R1 24/alkylbenzene											
R1 25/mixed ester											
R1 25/branched ester											
R1 43a/branched ester											
1000 ppm Moisture											
R1 1/mineral oil											
R1 2/mineral oil											
R2 2/mineral oil											
R1 23/mineral oil											
R1 34a/ mixed ester											
R1 34a/branched ester											
R1 52a/alkylbenzene											
R3 2/mixed ester											
R3 2/branched ester											
R1 24/alkylbenzene											
R1 25/mixed ester											
R1 25/branched ester											
R1 43a/branched ester											
1000 ppm Moisture											
R1 1/mineral oil											
R1 2/mineral oil											
R2 2/mineral oil											
R1 23/mineral oil											
R1 34a/ mixed ester											
R1 34a/branched ester											
R1 52a/alkylbenzene											
R3 2/mixed ester											
R3 2/branched ester											
R1 24/alkylbenzene											
R1 25/mixed ester											
R1 25/branched ester											
R1 43a/branched ester											
1000 ppm Moisture											
R1 1/mineral oil											
R1 2/mineral oil											
R2 2/mineral oil											
R1 23/mineral oil											
R1 34a/ mixed ester											
R1 34a/branched ester											
R1 52a/alkylbenzene											
R3 2/mixed ester											
R3 2/branched ester											
R1 24/alkylbenzene											
R1 25/mixed ester											
R1 25/branched ester											
R1 43a/branched ester											
1000 ppm Moisture											
R1 1/mineral oil											
R1 2/mineral oil											
R2 2/mineral oil											
R1 23/mineral oil											
R1 34a/ mixed ester											
R1 34a/branched ester											
R1 52a/alkylbenzene											
R3 2/mixed ester											
R3 2/branched ester											
R1 24/alkylbenzene											
R1 25/mixed ester											
R1 25/branched ester											
R1 43a/branched ester											
1000 ppm Moisture											
R1 1/mineral oil											
R1 2/mineral oil											
R2 2/mineral oil											
R1 23/mineral oil											
R1 34a/ mixed ester											
R1 34a/branched ester											
R1 52a/alkylbenzene											
R3 2/mixed ester											
R3 2/branched ester											
R1 24/alkylbenzene											

Desiccant; K - Silica Gel

Summary Test Results

Code	System Fluids	Liquid Color (2-8)	Copper Plating (0-5)	Solids Formation (0-3)	Crush Strength (lbs)	GC % Ref Reacted (wt %)	F ion in Liquid (ppm)	Cl ion in Liquid (ppm)	Total Acid Number (mg KOH)	F ion on Desiccant (ppm)	Cl ion on Desiccant (ppm)	Org Acid on Desic (ppm)
K-New	None	-	-	-	76.4	-	-	-	-	3	14	0
	50 ppm Moisture											
	R11/mineral oil											
K-12	R12/mineral oil	2.5/2.5	0	0	50.8	0%	8	605	<0.1	335	322	10
	R22/mineral oil											
	R123/mineral oil											
	R134a/ mixed ester											
	R134a/branched ester											
	R152a/alkylbenzene											
	R32/mixed ester											
	R32/branched ester											
	R124/alkylbenzene											
	R125/mixed ester											
	R125/branched ester											
	R143a/branched ester											
	1000 ppm Moisture											
	R11/mineral oil											
	.12/mineral oil											
	R22/mineral oil											
	R123/mineral oil											
	R134a/ mixed ester											
	R134a/branched ester											
	R152a/alkylbenzene											
	R32/mixed ester											
	R32/branched ester											
	R124/alkylbenzene											
	R125/mixed ester											
	R125/branched ester											
	R143a/branched ester											

VI. Conclusions

Although the results are very limited, some trends in the data that are developing for the desiccants in R-12/mineral oil.

1. The addition of 1000 ppm moisture to the sealed tubes produced no significant difference in any of the test results with R-12/mineral oil.
2. When the anion analyses of the liquid phase and the desiccant, solid phase are compared for each sample, the desiccant, solid phase is found to contain most of the fluoride ion, chloride ion, inorganic anions, and organic anions. In general there is one hundred times the anion concentration on the desiccant as in the liquid phase. The one exception to this is the Silica Gel (Sample K) for which there was twice as much chloride in the liquid phase as on the desiccant.
3. Only one of the desiccants (Molecular Sieve 3Å, Sample F) caused a significant increase in acid number after aging. And, this was only true for the low (< 50 ppm) moisture condition. This same desiccant was also the only one to demonstrate refrigerant decomposition in the R-12/mineral oil system, as determined by gas chromatography.
4. All of these desiccants contained chloride and fluoride ions when received. Sulfate was present on most of the desiccants as received. Other anions detected on new desiccants were acetate, formate, and what appears to be heptanoate.
5. On the average, crush strengths were reduced by about 20% after aging. In no case was the crush strength reduced to a level that could cause the desiccant to disintegrate and become a system contaminant. Also the addition of 1000 ppm moisture to the tube caused no greater change in crush strength than observed for the desiccants aged in the dry condition.

APPENDIX A

Desiccant: A - 4Å Molecular Sieve

Crush Strength Test Results

Code	System Fluids	Test #										Total # of Tests	Crush Strength (lbs)			
		1	2	3	4	5	6	7	8	9	10		Avg	Std Dev	Upper 95%	Lower 95%
A-New	None	13.0	17.3	25.9	14.4	16.4	13.6	17.7	21.0	15.5	18.6	10.0	17.3	3.9	25.1	9.6
		50 ppm Moisture														
A-11	R11/mineral oil															
A-12	R12/mineral oil	14.5	15.9	12.1	10.8	12.9	11.4	12.6	16.4	10.8	15.1	10.0	13.3	2.1	17.4	9.1
A-13	R22/mineral oil															
A-14	R123/mineral oil															
A-15	R134a/ mixed ester															
A-16	R134a/branched ester															
A-17	R152a/alkylbenzene															
A-18	R32/mixed ester															
A-19	R32/branched ester															
A-20	R124/alkylbenzene															
A-21	R125/mixed ester															
A-22	R125/branched ester															
A-23	R143a/branched ester															
		1000 ppm Moisture														
A-41	R11/mineral oil															
A-42	R12/mineral oil	13.5	13.3	9.6	16.2	10.1	9.1	10.8	14.6	25.4	8.1	10.0	13.1	5.1	23.2	2.9
A-43	R22/mineral oil															
A-44	R123/mineral oil															
A-45	R134a/ mixed ester															
A-46	R134a/branched ester															
A-47	R152a/alkylbenzene															
A-48	R32/mixed ester															
A-49	R32/branched ester															
A-50	R124/alkylbenzene															
A-51	R125/mixed ester															
A-52	R125/branched ester															
A-53	R143a/branched ester															

Gas Chromatography Data

File: Sample: A12

File: Sample: A42

Data Reprocessed On 12/14/1993 09:48:29

```
=====
Sample Name: A "AS RECEIVED"          Date: 12/14/1993 09:33:52
Data File  : C:\DX\DATA\DATA1071.D03
Method     : C:\DX\METHOD\50MMOLAR.MET
ACI Address: 1 System: 1 Inject#: 3    Detector: COND
Analyst    :                          Column:
=====
```

Calibration	Volume	Dilution	Points	Rate	Start	Stop	Area	Reject
External	1	60	13500	5Hz	0.00	45.00		1000

***** Component Report: Components Found *****

Pk. Num	Ret Time	Component Name	Concentration ppm	Height	Area	Bl. Code	%Delta
1	3.47	Fluoride	190.506	88442	1087827	1	1.36
2	11.80	Chloride	14.843	1951	56912	1	0.51
3	17.00	Heptanoate	1737.436	2984	284388	1	0.00
4	35.10	Sulfate	163.208	5573	457323	1	-2.50
Totals			2105.993	98951	1886449		

***** Peak Report: Unknown Peaks *****

Pk. Num	Ret Time	Component Name	Concentration ppm	Height	Area	Bl. Code	%Delta
Totals			0.000	0	0		

File: DATA1071.D03 Sample: A "AS RECEIVED"

Data Reprocessed On 12/20/1993 10:52:15

```
=====
Sample Name: A-12 LIQUID                      Date: 12/17/1993 09:43:51
Data File  : C:\DX\DATA\DATA1111.D04
Method     : C:\DX\METHOD\50MMOLAR.MET
ACI Address: 1 System: 1 Inject#: 4           Detector: COND
Analyst    :                               Column:
=====
```

Calibration	Volume	Dilution	Points	Rate	Start	Stop	Area	Reject
External	1	30	13500	5Hz	0.00	45.00		1000

***** Component Report: Components Found *****

Pk. Num	Ret Time	Component Name	Concentration ppm	Height	Area	Bl. Code	%Delta
1	3.56	Fluoride	3.828	4943	43722	1	0.56
2	4.78	Formate	4.208	891	15296	1	0.42
3	12.00	Chloride	28.126	6199	215683	1	1.69
4	16.80	Heptanoate	59.127	470	19356	1	0.00
Totals			95.289	12502	294057		

***** Peak Report: Unknown Peaks *****

Pk. Num	Ret Time	Component Name	Concentration ppm	Height	Area	Bl. Code	%Delta
Totals			0.000	0	0		

File: DATA1111.D04 Sample: A-12 LIQUID

Data Reprocessed On 12/20/1993 10:50:02

```
=====
Sample Name: A-12 DESICCANT                      Date: 12/17/1993 12:53:15
Data File  : C:\DX\DATA\DATA1111.D08
Method     : C:\DX\METHOD\50MMOLAR.MET
ACI Address: 1 System: 1 Inject#: 8              Detector: COND
Analyst    :                               Column:
=====
```

Calibration	Volume	Dilution	Points	Rate	Start	Stop	Area	Reject
External	1	60	13500	5Hz	0.00	45.00		1000

***** Component Report: Components Found *****

Pk. Num	Ret Time	Component Name	Concentration ppm	Height	Area	Bl. Code	%Delta
1	3.58	Fluoride	673.250	307215	3844400	1	1.13
2	4.82	Formate	575.333	65951	1045773	1	1.26
3	11.64	Chloride	1560.748	164820	5984259	2	-1.36
4	16.40	Heptanoate	2028.111	4078	331968	2	0.00
5	34.80	Sulfate	145.280	4183	407087	2	-3.33
Totals			4982.723	546247	11613486		

***** Peak Report: Unknown Peaks *****

Pk. Num	Ret Time	Component Name	Concentration ppm	Height	Area	Bl. Code	%Delta
Totals			0.000	0	0		

File: DATA1111.D08 Sample: A-12 DESICCANT

Data Reprocessed On 12/16/1993 15:11:13

```
=====
| Sample Name: A-42 LIQUID                               Date: 12/16/1993 14:48:34
| Data File   : C:\DX\DATA\DATA1101.D02
| Method      : C:\DX\METHOD\50MMOLAR.MET
| ACI Address: 1 System: 1 Inject#: 2                     Detector: COND
| Analyst     :                                           Column:
=====
```

Calibration	Volume	Dilution	Points	Rate	Start	Stop	Area	Reject
External	1	30	13500	5Hz	0.00	45.00		1000

***** Component Report: Components Found *****

Pk. Num	Ret Time	Component Name	Concentration ppm	Height	Area	Bl. Code	%Delta
1	3.58	Fluoride	2.034	2818	23230	1	1.13
2	3.94	Acetate	5.905	524	6249	1	0.51
3	4.80	Formate	4.103	1125	14916	1	0.84
4	11.80	Chloride	20.514	6086	157311	1	0.00
5	17.16	Heptanoate	85.075	539	27851	1	-1.94
Totals			117.631	11092	229556		

***** Peak Report: Unknown Peaks *****

Pk. Num	Ret Time	Component Name	Concentration ppm	Height	Area	Bl. Code	%Delta
Totals			0.000	0	0		

File: DATA1101.D02 Sample: A-42 LIQUID

Data Reprocessed On 12/20/1993 13:40:33

```
=====
| Sample Name: A-42 DESICCANT                      Date: 12/20/1993 11:52:37
| Data File  : C:\DX\DATA\DATA1141.D03
| Method     : C:\DX\METHOD\50MMOLAR.MET
| ACI Address: 1 System: 1 Inject#: 3              Detector: COND
| Analyst    :                      Column:
=====
```

Calibration	Volume	Dilution	Points	Rate	Start	Stop	Area	Reject
External	1	60	13500	5Hz	0.00	45.00		1000

***** Component Report: Components Found *****

Pk. Num	Ret Time	Component Name	Concentration ppm	Height	Area	Bl. Code	%Delta
1	3.64	Fluoride	1240.208	493424	7081844	1	-0.55
2	4.90	Formate	821.404	86442	1493053	1	-0.41
3	11.92	Chloride	1386.766	142166	5317173	2	-2.30
4	16.44	Heptanoate	2153.643	4437	352513	2	0.00
5	35.04	Sulfate	139.429	4218	390691	1	-2.67
Totals			5741.450	730688	14635274		

***** Peak Report: Unknown Peaks *****

Pk. Num	Ret Time	Component Name	Concentration ppm	Height	Area	Bl. Code	%Delta
Totals			0.000	0	0		

File: DATA1141.D03 Sample: A-42 DESICCANT

APPENDIX E

Desiccant: E - 4Å Molecular Sieve

Crush Strength Test Results

Crush Strength Test Results													Total # of Tests	Crush Strength (lbs)				
Code	System Fluids	Test #										Avg		Std Dev	Upper 95%	Lower 95%		
		1	2	3	4	5	6	7	8	9	10							
E-New	None	25.3	20.5	26.8	23.2	26.4	45.6	46.2	35.5	28.3	30.9	10.0	30.9	8.9	48.7	13.1		
		50 ppm Moisture																
E-12	R11/mineral oil																	
	R12/mineral oil	38.7	27.2	20.5	10.6	28.6	34.4	31.3	16.8	38.4	23.1	10.0	27.0	9.3	45.5	8.4		
	R22/mineral oil																	
	R123/mineral oil																	
	R134a/ mixed ester																	
	R134a/branched ester																	
	R152a/alkylbenzene																	
	R32/mixed ester																	
	R32/branched ester																	
	R124/alkylbenzene																	
	R125/mixed ester																	
	R125/branched ester																	
	R143a/branched ester																	
		1000 ppm Moisture																
E-42	R11/mineral oil																	
	R12/mineral oil	17.4	27.7	19.8	27.5	34.9	12.2	22.6	24.3	33.1	16.0	10.0	23.6	7.4	38.3	8.8		
	R22/mineral oil																	
	R123/mineral oil																	
	R134a/ mixed ester																	
	R134a/branched ester																	
	R152a/alkylbenzene																	
	R32/mixed ester																	
	R32/branched ester																	
	R124/alkylbenzene																	
	R125/mixed ester																	
	R125/branched ester																	
	R143a/branched ester																	

Gas Chromatography Data

File: Sample: E12

File: Sample: E42

```

=====
Sample Name: E "AS RECEIVED"           Date: 12/13/1993 16:38:04
Data File  : C:\DX\DATA\DATA1061.D05
Method     : C:\DX\METHOD\50MMOLAR.MET
ACI Address: 1 System: 1 Inject#: 5      Detector: COND
Analyst    :                          Column:
=====

```

```

-----
Calibration Volume Dilution Points Rate Start Stop Area Reject
-----
External           1           60 13500 5Hz  0.00 45.00      1000
-----

```

***** Component Report: Components Found *****

Pk. Num	Ret Time	Component Name	Concentration ppm	Height	Area	Bl. Code	%Delta
1	3.47	Fluoride	10.339	4169	59039	1	1.36
2	11.53	Chloride	10.994	917	42154	2	-1.76
3	17.93	Heptanoate	430.858	884	70524	2	2.48
4	35.80	Sulfate	103.716	2469	290620	2	-0.56
Totals			555.907	8439	462337		

***** Peak Report: Unknown Peaks *****

Pk. Num	Ret Time	Component Name	Concentration ppm	Height	Area	Bl. Code	%Delta
Totals			0.000	0	0		

File: DATA1061.D05 Sample: E "AS RECEIVED"

Data Reprocessed On 12/20/1993 10:56:06

```
=====
Sample Name: E-12 LIQUID                      Date: 12/17/1993 10:32:12
Data File  : C:\DX\DATA\DATA1111.D05
Method     : C:\DX\METHOD\50MMOLAR.MET
ACI Address: 1 System: 1 Inject#: 5           Detector: COND
Analyst    :                               Column:
=====
```

```
-----
Calibration Volume Dilution Points Rate Start Stop Area Reject
-----
External          1          30 13500 5Hz  0.00 45.00          1000
-----
```

***** Component Report: Components Found *****

Pk. Num	Ret Time	Component Name	Concentration ppm	Height	Area	Bl. Code	%Delta
1	3.56	Fluoride	0.809	930	9239	1	0.56
2	4.80	Formate	3.036	776	11036	1	0.84
3	11.96	Chloride	18.071	4321	138577	1	1.36
4	16.84	Heptanoate	69.546	447	22767	1	0.00
Totals			91.462	6475	181620		

***** Peak Report: Unknown Peaks *****

Pk. Num	Ret Time	Component Name	Concentration ppm	Height	Area	Bl. Code	%Delta
Totals			0.000	0	0		

File: DATA1111.D05 Sample: E-12 LIQUID

```

=====
Sample Name: E-12 DESICCANT                      Date: 12/20/1993 15:08:09
Data File  : C:\DX\DATA\DATA1151.D02
Method     : C:\DX\METHOD\50MMOLAR.MET
ACI Address: 1 System: 1 Inject#: 2              Detector: COND
Analyst    :                               Column:
=====

```

```

-----
Calibration Volume Dilution Points Rate Start Stop Area Reject
-----
External          1          60 13500 5Hz  0.00 45.00      1000
-----

```

***** Component Report: Components Found *****

Pk. Num	Ret Time	Component Name	Concentration ppm	Height	Area	Bl. Code	%Delta
1	3.66	Fluoride	436.912	188610	2494857	1	0.00
2	4.94	Formate	272.112	30213	494612	1	0.41
3	12.08	Chloride	659.521	69343	2528753	2	-0.98
4	17.12	Heptanoate	337.073	883	55173	2	-2.17
5	35.40	Sulfate	133.467	4240	373986	1	-1.67
Totals			1839.085	293289	5947382		

***** Peak Report: Unknown Peaks *****

Pk. Num	Ret Time	Component Name	Concentration ppm	Height	Area	Bl. Code	%Delta
Totals			0.000	0	0		

File: DATA1151.D02 Sample: E-12 DESICCANT

```

=====
Sample Name: E-42 LIQUID                      Date: 12/16/1993 11:21:58
Data File  : C:\DX\DATA\DATA1091.D01
Method     : C:\DX\METHOD\50MMOLAR.MET
ACI Address: 1 System: 1 Inject#: 1           Detector: COND
Analyst    :                               Column:
=====

```

```

-----
Calibration Volume Dilution Points Rate Start Stop Area Reject
-----
External          1          30 13500 5Hz  0.00 45.75      1000
-----

```

***** Component Report: Components Found *****

Pk. Num	Ret Time	Component Name	Concentration ppm	Height	Area	Bl. Code	%Delta
1	3.58	Fluoride	1.008	1179	11484	1	1.13
2	3.94	Acetate	14.022	1014	14841	1	0.51
3	4.82	Formate	5.110	1232	18577	1	1.26
4	11.84	Chloride	18.409	5210	141165	1	0.34
5	17.12	Heptanoate	101.638	704	33273	1	-2.17
Totals			140.184	9339	219340		

***** Peak Report: Unknown Peaks *****

Pk. Num	Ret Time	Component Name	Concentration ppm	Height	Area	Bl. Code	%Delta
Totals			0.000	0	0		

File: DATA1091.D01 Sample: E-42 LIQUID

```

=====
Sample Name: E-42 DESICCANT                      Date: 12/20/1993 14:19:49
Data File  : C:\DX\DATA\DATA1141.D01
Method     : C:\DX\METHOD\50MMOLAR.MET
ACI Address: 1 System: 1 Inject#: 1              Detector: COND
Analyst    :                               Column:
=====

```

```

-----
Calibration Volume Dilution Points Rate Start Stop Area Reject
-----
External          1          60 13500 5Hz  0.00 45.00      1000

```

***** Component Report: Components Found *****

Pk. Num	Ret Time	Component Name	Concentration ppm	Height	Area	Bl. Code	%Delta
1	3.68	Fluoride	566.647	241644	3235675	1	0.00
2	4.92	Formate	218.740	23570	397600	1	0.00
3	12.04	Chloride	716.452	73270	2747038	2	-1.31
4	17.12	Heptanoate	499.444	1187	81750	2	-2.17
5	35.24	Sulfate	125.107	3789	350561	1	-2.11
Totals			2126.391	343460	6812623		

***** Peak Report: Unknown Peaks *****

Pk. Num	Ret Time	Component Name	Concentration ppm	Height	Area	Bl. Code	%Delta
Totals			0.000	0	0		

File: DATA1141.D01 Sample: E-42 DESICCANT

APPENDIX F

Desiccant: F - 3Å Molecular Sieve

Crush Strength Test Results

Crush Strength Test Results																		
Code	System Fluids	Test #										Total # of Tests	Crush Strength (lbs)					
		1	2	3	4	5	6	7	8	9	10		Avg	Std Dev	Upper 95%	Lower 95%		
F-New	None	22.8	23.2	17.7	23.8	22.5	13.0	19.0	25.3	15.1	18.0	10.0	20.0	4.1	28.2	11.9		
		50 ppm Moisture																
F-12	R11/mineral oil R12/mineral oil R22/mineral oil R123/mineral oil R134a/ mixed ester R134a/branched ester R152a/alkylbenzene R32/mixed ester R32/branched ester R124/alkylbenzene R125/mixed ester R125/branched ester R143a/branched ester	16.4	16.0	16.2	21.6	15.4	18.6	18.5	22.9	14.5	19.9	10.0	18.0	2.8	23.6	12.4		
		1000 ppm Moisture																
F-42	R11/mineral oil R12/mineral oil R22/mineral oil R123/mineral oil R134a/ mixed ester R134a/branched ester R152a/alkylbenzene R32/mixed ester R32/branched ester R124/alkylbenzene R125/mixed ester R125/branched ester R143a/branched ester	14.8	21.1	14.7	19.8	16.6	23.2	25.4	14.6	16.6	19.4	10.0	18.6	3.8	26.2	11.0		

Gas Chromatography Data

File: Sample: F12

File: Sample: F42

Data Reprocessed On 12/13/1993 15:59:59

```
=====
Sample Name: F "AS RECEIVED"          Date: 12/13/1993 15:49:55
Data File  : C:\DX\DATA\DATA1061.D04
Method     : C:\DX\METHOD\50MMOLAR.MET
ACI Address: 1 System: 1 Inject#: 4    Detector: COND
Analyst    :                          Column:
=====
```

Calibration	Volume	Dilution	Points	Rate	Start	Stop	Area	Reject
External	1	60	13500	5Hz	0.00	45.00		1000

***** Component Report: Components Found *****

Pk. Num	Ret Time	Component Name	Concentration ppm	Height	Area	Bl. Code	%Delta
1	3.43	Fluoride	159.240	66005	909295	1	0.39
2	11.57	Chloride	22.897	2531	87792	1	-1.48
3	17.60	Heptanoate	1261.821	2320	206538	1	0.57
4	35.67	Sulfate	414.752	10499	1162167	1	-0.93
Totals			1858.710	81355	2365792		

***** Peak Report: Unknown Peaks *****

Pk. Num	Ret Time	Component Name	Concentration ppm	Height	Area	Bl. Code	%Delta

Totals			0.000	0	0		

File: DATA1061.D04 Sample: F "AS RECEIVED"

Data Reprocessed On 12/20/1993 10:54:37

```
=====
Sample Name: F-12 LIQUID                      Date: 12/17/1993 11:22:15
Data File  : C:\DX\DATA\DATA1111.D06
Method     : C:\DX\METHOD\50MMOLAR.MET
ACI Address: 1 System: 1 Inject#: 6           Detector: COND
Analyst    :                               Column:
=====
```

Calibration	Volume	Dilution	Points	Rate	Start	Stop	Area	Reject
External	1	30	13500	5Hz	0.00	45.00		1000

***** Component Report: Components Found *****

Pk. Num	Ret Time	Component Name	Concentration ppm	Height	Area	Bl. Code	%Delta
1	3.56	Fluoride	41.131	37694	469728	1	0.56
2	4.80	Formate	17.385	4403	63201	1	0.84
3	11.88	Chloride	129.729	29889	994821	2	0.68
4	16.84	Heptanoate	121.816	683	39878	2	0.00
5	34.52	Sulfate	8.445	470	47329	2	-4.11
Totals			318.506	73139	1614957		

***** Peak Report: Unknown Peaks *****

Pk. Num	Ret Time	Component Name	Concentration ppm	Height	Area	Bl. Code	%Delta
Totals			0.000	0	0		

File: DATA1111.D06 Sample: F-12 LIQUID

```

=====
Sample Name: F-12 DESICCANT                      Date: 12/20/1993 15:52:11
Data File  : C:\DX\DATA\DATA1151.D03
Method     : C:\DX\METHOD\50MMOLAR.MET
ACI Address: 1 System: 1 Inject#: 3              Detector: COND
Analyst    :                               Column:
=====

```

```

-----
Calibration Volume Dilution Points Rate Start Stop Area Reject
-----
External          1          60 12655 5Hz  0.00 42.18      1000
-----

```

***** Component Report: Components Found *****

Pk. Num	Ret Time	Component Name	Concentration ppm	Height	Area	Bl. Code	%Delta
1	3.68	Fluoride	1584.039	630788	9045191	1	0.55
2	4.94	Formate	817.497	86983	1485951	1	0.41
3	11.68	Chloride	6547.612	535186	25105013	2	0.00
4	16.92	Heptanoate	1083.800	2301	177399	2	0.00
5	35.28	Sulfate	413.367	11409	1158287	1	-2.00
Totals			10446.314	1266668	36971841		

***** Peak Report: Unknown Peaks *****

Pk. Num	Ret Time	Component Name	Concentration ppm	Height	Area	Bl. Code	%Delta
Totals			0.000	0	0		

File: DATA1151.D03 Sample: F-12 DESICCANT

Data Reprocessed On 12/16/1993 13:36:04

```
=====
Sample Name: F-42 LIQUID                      Date: 12/16/1993 12:08:52
Data File  : C:\DX\DATA\DATA1091.D02
Method     : C:\DX\METHOD\50MMOLAR.MET
ACI Address: 1 System: 1 Inject#: 2           Detector: COND
Analyst    :                               Column:
=====
```

```
-----
Calibration Volume Dilution Points Rate Start Stop Area Reject
-----
External          1          30 13500 5Hz  0.00 45.00      1000
-----
```

***** Component Report: Components Found *****

Pk. Num	Ret Time	Component Name	Concentration ppm	Height	Area	Bl. Code	%Delta
1	3.58	Fluoride	52.093	53277	594920	1	1.13
2	4.82	Formate	26.123	6476	94967	1	1.26
3	11.80	Chloride	76.154	18643	583986	2	0.00
4	17.12	Heptanoate	66.901	519	21901	2	-2.17
Totals			221.272	78915	1295774		

***** Peak Report: Unknown Peaks *****

Pk. Num	Ret Time	Component Name	Concentration ppm	Height	Area	Bl. Code	%Delta
Totals			0.000	0	0		

File: DATA1091.D02 Sample: F-42 LIQUID

Data Reprocessed On 12/20/1993 13:38:25

```
=====
: Sample Name: F-42 DESICCANT                      Date: 12/20/1993 11:03:02
: Data File  : C:\DX\DATA\DATA1141.D02
: Method     : C:\DX\METHOD\50MMOLAR.MET
: ACI Address: 1 System: 1 Inject#: 2              Detector: COND
: Analyst    :                      Column:
=====
```

Calibration	Volume	Dilution	Points	Rate	Start	Stop	Area	Reject
External	1	60	13500	5Hz	0.00	45.00		1000

***** Component Report: Components Found *****

Pk. Num	Ret Time	Component Name	Concentration ppm	Height	Area	Bl. Code	%Delta
1	3.64	Fluoride	1389.518	559294	7934440	1	-0.55
2	4.88	Formate	816.513	85612	1484161	1	-0.81
3	11.60	Chloride	5765.006	485570	22104327	2	0.00
4	16.68	Heptanoate	1306.809	2710	213902	2	0.00
5	34.80	Sulfate	418.664	11642	1173129	1	-3.33
Totals			9696.509	1144828	32909958		

***** Peak Report: Unknown Peaks *****

Pk. Num	Ret Time	Component Name	Concentration ppm	Height	Area	Bl. Code	%Delta
Totals			0.000	0	0		

File: DATA1141.D02 Sample: F-42 DESICCANT

APPENDIX H

Desiccant: H - 3Å Molecular Sieve

Crush Strength Test Results

[illegible]

Gas Chromatography Data

File: Sample: H12

File: Sample: H42

Data Reprocessed On 12/13/1993 15:12:14

Sample Name: H "AS RECEIVED" Date: 12/13/1993 15:01:44
Data File : C:\DX\DATA\DATA1061.D03
Method : C:\DX\METHOD\50MMOLAR.MET
ACI Address: 1 System: 1 Inject#: 3 Detector: COND
Analyst : Column:

Calibration	Volume	Dilution	Points	Rate	Start	Stop	Area	Reject
External	1	60	13500	5Hz	0.00	45.00		1000

***** Component Report: Components Found *****

Pk. Num	Ret Time	Component Name	Concentration ppm	Height	Area	Bl. Code	%Delta
1	3.43	Fluoride	18.361	7396	104845	1	0.39
2	11.60	Chloride	3.822	564	14654	1	-1.19
3	17.93	Heptanoate	183.717	482	30071	2	2.48
4	35.67	Sulfate	259.528	6865	727217	2	-0.93
Totals			465.427	15308	876787		

***** Peak Report: Unknown Peaks *****

Pk. Num	Ret Time	Component Name	Concentration ppm	Height	Area	Bl. Code	%Delta
Totals			0.000	0	0		

File: DATA1061.D03 Sample: H "AS RECEIVED"

```

=====
Sample Name: H-12 LIQUID                      Date: 12/16/1993 17:14:25
Data File  : C:\DX\DATA\DATA1101.D05
Method     : C:\DX\METHOD\50MMOLAR.MET
ACI Address: 1 System: 1 Inject#: 5           Detector: COND
Analyst    :                               Column:
=====

```

```

-----
Calibration Volume Dilution Points Rate Start Stop Area Reject
-----
External          1          30 13500 5Hz  0.00 45.00      1000
-----

```

***** Component Report: Components Found *****

Pk. Num	Ret Time	Component Name	Concentration ppm	Height	Area	Bl. Code	%Delta
1	3.56	Fluoride	3.584	4328	40927	1	0.56
2	4.82	Formate	4.616	1092	16781	1	1.26
3	11.84	Chloride	23.659	6068	181431	2	0.34
4	17.12	Heptanoate	106.215	700	34771	2	-2.17
Totals			138.074	12188	273910		

***** Peak Report: Unknown Peaks *****

Pk. Num	Ret Time	Component Name	Concentration ppm	Height	Area	Bl. Code	%Delta
Totals			0.000	0	0		

File: DATA1101.D05 Sample: H-12 LIQUID

Data Reprocessed On 12/20/1993 13:42:05

```
=====
Sample Name: H-12 DESICCANT                      Date: 12/20/1993 12:39:54
Data File  : C:\DX\DATA\DATA1141.D04
Method     : C:\DX\METHOD\50MMOLAR.MET
ACI Address: 1 System: 1 Inject#: 4              Detector: COND
Analyst    :                                  Column:
=====
```

Calibration	Volume	Dilution	Points	Rate	Start	Stop	Area	Reject
External	1	60	13500	5Hz	0.00	45.00		1000

***** Component Report: Components Found *****

Pk. Num	Ret Time	Component Name	Concentration ppm	Height	Area	Bl. Code	%Delta
1	3.66	Fluoride	1015.780	418267	5800318	1	0.00
2	4.90	Formate	532.077	55971	967148	1	-0.41
3	11.92	Chloride	1600.632	158508	6137184	2	-2.30
4	17.20	Heptanoate	284.636	846	46590	2	-1.71
5	35.08	Sulfate	249.102	6680	698003	1	-2.56
Totals			3682.228	640272	13649243		

***** Peak Report: Unknown Peaks *****

Pk. Num	Ret Time	Component Name	Concentration ppm	Height	Area	Bl. Code	%Delta
Totals			0.000	0	0		

File: DATA1141.D04 Sample: H-12 DESICCANT

Data Reprocessed On 12/16/1993 11:18:07

```
=====
Sample Name: H-42 LIQUID                      Date: 12/16/1993 10:31:00
Data File  : C:\DX\DATA\DATA1081.D04
Method     : C:\DX\METHOD\50MMOLAR.MET
ACI Address: 1 System: 1 Inject#: 4           Detector: COND
Analyst    :                               Column:
=====
```

Calibration	Volume	Dilution	Points	Rate	Start	Stop	Area	Reject
External	1	30	13500	5Hz	0.00	45.00		1000

***** Component Report: Components Found *****

Pk. Num	Ret Time	Component Name	Concentration ppm	Height	Area	Bl. Code	%Delta
1	3.58	Fluoride	1.768	2369	20191	1	1.13
2	3.94	Acetate	7.903	752	8365	1	0.51
3	4.82	Formate	4.514	1240	16410	1	1.26
4	11.88	Chloride	20.525	5755	157396	2	0.68
5	17.08	Heptanoate	87.375	596	28603	2	-2.40
Totals			122.085	10711	230966		

***** Peak Report: Unknown Peaks *****

Pk. Num	Ret Time	Component Name	Concentration ppm	Height	Area	Bl. Code	%Delta
Totals			0.000	0	0		

File: DATA1081.D04 Sample: H-42 LIQUID

```

=====
Sample Name: H-42 DESICCANT                      Date: 12/17/1993 13:49:18
Data File  : C:\DX\DATA\DATA1111.D09
Method     : C:\DX\METHOD\50MMOLAR.MET
ACI Address: 1 System: 1 Inject#: 9              Detector: COND
Analyst    :                               Column:
=====

```

```

-----
Calibration Volume Dilution Points Rate Start Stop Area Reject
-----
External          1          60 13500 5Hz  0.00 45.00      1000
-----

```

***** Component Report: Components Found *****

Pk. Num	Ret Time	Component Name	Concentration ppm	Height	Area	Bl. Code	%Delta
1	3.58	Fluoride	431.937	195416	2466449	1	1.13
2	4.82	Formate	495.873	54724	901340	1	1.26
3	11.76	Chloride	753.921	82235	2890701	2	-0.34
4	17.04	Heptanoate	249.489	841	40837	2	-2.63
5	35.00	Sulfate	216.691	6334	607186	1	-2.78
Totals			2147.911	339551	6906513		

***** Peak Report: Unknown Peaks *****

Pk. Num	Ret Time	Component Name	Concentration ppm	Height	Area	Bl. Code	%Delta
Totals			0.000	0	0		

File: DATA1111.D09 Sample: H-42 DESICCANT

APPENDIX I

Desiccant: 1 - Alumina

Crush Strength Test Results

[illegible]

Gas Chromatography Data

File: Sample: 112

```

=====
| Sample Name: I "AS RECEIVED"                      Date: 12/13/1993 14:12:43
| Data File   : C:\DX\DATA\DATA1061.D02
| Method      : C:\DX\METHOD\50MMOLAR.MET
| ACI Address: 1 System: 1 Inject#: 2                Detector: COND
| Analyst     :                      Column:
=====

```

```

-----
Calibration Volume Dilution Points Rate Start Stop Area Reject
-----
External          1          60 13500 5Hz  0.00 45.00          1000
-----

```

***** Component Report: Components Found *****

Pk. Num	Ret Time	Component Name	Concentration ppm	Height	Area	Bl. Code	%Delta
1	3.43	Fluoride	2.006	1437	11452	2	0.39
2	3.83	Acetate	3759.051	91989	1989250	3	0.88
3	4.63	Formate	112.486	9585	204464	4	-0.14
4	11.60	Chloride	58.791	6843	225419	1	-1.19
Totals			3932.334	109854	2430586		

***** Peak Report: Unknown Peaks *****

Pk. Num	Ret Time	Component Name	Concentration ppm	Height	Area	Bl. Code	%Delta
Totals							
			0.000	0	0		

File: DATA1061.D02 Sample: I "AS RECEIVED"

```

=====
Sample Name: I-12 LIQUID                      Date: 12/16/1993 16:25:06
Data File  : C:\DX\DATA\DATA1101.D04
Method     : C:\DX\METHOD\50MMOLAR.MET
ACI Address: 1 System: 1 Inject#: 4           Detector: COND
Analyst    :                               Column:
=====

```

```

-----
Calibration Volume Dilution Points Rate Start Stop Area Reject
-----
External          1          30 13500 5Hz  0.00 45.00       1000
-----

```

***** Component Report: Components Found *****

Pk. Num	Ret Time	Component Name	Concentration ppm	Height	Area	Bl. Code	%Delta
1	3.56	Fluoride	3.304	4154	37735	1	0.56
2	4.80	Formate	3.876	1156	14091	1	0.84
3	11.80	Chloride	21.458	6174	164547	1	0.00
4	17.08	Heptanoate	85.855	601	28106	1	-2.40
Totals			114.493	12085	244478		

***** Peak Report: Unknown Peaks *****

Pk. Num	Ret Time	Component Name	Concentration ppm	Height	Area	Bl. Code	%Delta
Totals			0.000	0	0		

File: DATA1101.D04 Sample: I-12 LIQUID

```

=====
Sample Name: I-12 DESICCANT                      Date: 12/20/1993 16:38:32
Data File  : C:\DX\DATA\DATA1151.D04
Method     : C:\DX\METHOD\50MMOLAR.MET
ACI Address: 1 System: 1 Inject#: 4              Detector: COND
Analyst    :                               Column:
=====

```

```

-----
Calibration Volume Dilution Points Rate Start Stop Area Reject
-----
External          1          60 13500 5Hz  0.00 45.00      1000

```

***** Component Report: Components Found *****

Pk. Num	Ret Time	Component Name	Concentration ppm	Height	Area	Bl. Code	%Delta
1	3.68	Fluoride	9.746	5936	55652	1	0.55
2	4.08	Acetate	655.176	21963	346712	1	0.49
3	4.96	Formate	890.572	95307	1618777	1	0.81
4	11.80	Chloride	4626.078	403551	17737422	1	0.00
Totals			6181.572	526757	19758564		

***** Peak Report: Unknown Peaks *****

Pk. Num	Ret Time	Component Name	Concentration ppm	Height	Area	Bl. Code	%Delta
Totals			0.000	0	0		

File: DATA1151.D04 Sample: I-12 DESICCANT

APPENDIX K

Desiccant: K - Silica Gel

Crush Strength Test Results

[illegible]

Gas Chromatography Data

File: Sample: K12

Data Reprocessed On 12/13/1993 13:36:46

```
=====
Sample Name: K "AS RECEIVED"          Date: 12/13/1993 13:26:29
Data File  : C:\DX\DATA\DATA1061.D01
Method     : C:\DX\METHOD\50MMOLAR.MET
ACI Address: 1 System: 1 Inject#: 1    Detector: COND
Analyst    :                          Column:
=====
```

Calibration	Volume	Dilution	Points	Rate	Start	Stop	Area	Reject
External	1	60	13500	5Hz	0.00	45.00		1000

***** Component Report: Components Found *****

Pk. Num	Ret Time	Component Name	Concentration ppm	Height	Area	Bl. Code	%Delta
1	3.47	Fluoride	3.064	1447	17496	1	1.36
2	11.63	Chloride	13.884	1896	53234	1	-0.91
3	35.57	Sulfate	47.247	1874	132390	1	-1.20
Totals			64.195	5217	203120		

***** Peak Report: Unknown Peaks *****

Pk. Num	Ret Time	Component Name	Concentration ppm	Height	Area	Bl. Code	%Delta
Totals			0.000	0	0		

File: DATA1061.D01 Sample: K "AS RECEIVED"

```

=====
Sample Name: K-12 LIQUID                      Date: 12/16/1993 15:38:55
Data File  : C:\DX\DATA\DATA1101.D03
Method     : C:\DX\METHOD\50MMOLAR.MET
ACI Address: 1 System: 1 Inject#: 3           Detector: COND
Analyst    :                               Column:
=====

```

```

-----
Calibration Volume Dilution Points Rate Start Stop Area Reject
-----
External          1          30 13500 5Hz  0.00 45.00          1000
-----

```

***** Component Report: Components Found *****

Pk. Num	Ret Time	Component Name	Concentration ppm	Height	Area	Bl. Code	%Delta
1	3.58	Fluoride	8.262	9833	94359	1	0.58
2	4.80	Formate	14.952	3830	54354	1	0.04
3	11.64	Chloride	604.589	142285	4636257	1	-1.36
4	17.16	Heptanoate	130.432	829	42699	1	-1.94
Totals			758.234	156777	4827670		

***** Peak Report: Unknown Peaks *****

Pk. Num	Ret Time	Component Name	Concentration ppm	Height	Area	Bl. Code	%Delta
Totals			0.000	0	0		

File: DATA1101.D03 Sample: K-12 LIQUID

```

=====
Sample Name: K-12 DESICCANT                      Date: 12/20/1993 10:13:49
Data File  : C:\DX\DATA\DATA1131.D01
Method     : C:\DX\METHOD\50MMOLAR.MET
ACI Address: 1 System: 1 Inject#: 1              Detector: COND
Analyst    :                                  Column:
=====

```

```

-----
Calibration Volume Dilution Points Rate Start Stop Area Reject
-----
External          1          60 13500 5Hz  0.00 45.00      1000
-----

```

***** Component Report: Components Found *****

Pk. Num	Ret Time	Component Name	Concentration ppm	Height	Area	Bl. Code	%Delta
1	3.62	Fluoride	334.519	143091	1910172	1	2.26
2	4.86	Formate	9.846	972	17897	1	2.10
3	12.08	Chloride	321.878	33030	1234146	1	-0.98
4	34.72	Sulfate	48.238	1480	135166	1	-3.56
Totals			714.479	178583	3297382		

***** Peak Report: Unknown Peaks *****

Pk. Num	Ret Time	Component Name	Concentration ppm	Height	Area	Bl. Code	%Delta
Totals			0.000	0	0		

File: DATA1131.D01 Sample: K-12 DESICCANT

DATE

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5 / 16 / 94

END