

COMPATIBILITY OF MANUFACTURING PROCESS FLUIDS WITH HFC REFRIGERANTS AND ESTER LUBRICANTS

First Draft of Final Report of Part One and
Quarterly Report of Part

Two

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ABSTRACT

Included in this report is a compiled partial list of presently used processing materials in the air-conditioning and refrigeration industry and the manufacturers, intended uses, and applications of each. Also listed are the processing materials that have received final acceptance for this contracted study. An analytical methodology discussion is presented, including the final decision and the limitations of said methodology, as well as how to establish a level of confidence in observed immiscible material components in two 32 ISO VG polyolesters: 1) Mobil EAL Arctic 32; and 2) ICI Emkarate RL32H; both with HFC 134a refrigerant solutions.

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1 SCOPE

Part one of this study consists of compiling a list of actively used processing materials within the compressor and systems component industry. The objective of this list is to provide producers and suppliers of HVAC compressors and equipment with guidelines for selecting a class of processing materials compatible with currently used polyolesters (POE) and HFC alternative refrigerants. The results of this study can provide processing material manufacturers with additional guidelines for designing chemical formulations most likely to be successful assembly and production facilities.

1.1 Statement and Chemical Properties of Lubricants and Processing Fluids

This project uses samples of processing materials provided by the manufacturers. To the best of our knowledge, the materials are representative of the published compositions as reproduced in the appendices.

This program also uses two commercial proprietary lubricants which are pentaerythritol polyolesters that may or may not be additized. Due to proprietary concerns, only the product type of the polyolester will be reported. Information concerning specific structural properties of the polyolester remains with the manufacturer. The most effective method of reporting lubricant type is to determine alcohol type, and stoichiometry of carboxylic acids used in synthesis. Miscibility and solubility of process materials may be affected by polyolesters chemical compositions.

2 SIGNIFICANT RESULTS

One hundred ten process materials gathered from suppliers have reported little to no compatibility information with HFCs. However, according to the names and intended uses of each product, a decision can be made in regards to the selection and potential compatibility or incompatibility of each with HFC-134a and polyolester lubricant solutions.

A list of processing materials has been compiled that is representative of presently used compounds in HVAC component manufacturing facilities. The importance of this study is to quantify the acceptable materials in terms of observed immiscibility in HFC polyolester solutions. Immiscibility may signal that a material could have questionable use.

Capillary tubing expansion devices are subject to blockage as a result of processing chemicals remaining in the heat exchanger tubing of refrigerators, heat pumps, and other HVAC equipment. Polyolesters are able to solubilize the trace fats and waxes from process chemicals. Use of POEs with system components contaminated by process chemicals combine with discharged refrigerant gas and develop on the condenser as microscopic particulate. The ensuing solid particles are suspended in a high dilution ratio of HFC to POE liquid. As insoluble material, the contaminants can build up on the walls of tubing, resulting in blockage.

2.1 Brazing Fluxes

Appendix A, Table A.1, contains a list of brazing fluxes considered. Tube brazing procedures are one of the last operations in the compressor, heat exchanger, and tubing connections in HVAC systems. The composition of most brazing fluxes is sodium and potassium salts of fluorine, tetra and penta borates, or a potassium tetra fluoro aluminate. Although these brazing materials are likely to be insoluble in the refrigerant and POE, there could be some unexpected reactions with the POE at elevated temperatures. No Rez 65™ uses hydrobromic acid and ethylene diamine as the volatile complex. If not removed from the brazing surfaces, it could react with the POE and then is not suitable. However, with NoRez 65™ after proper use and washing, the remaining brazed metal surface will be clean and suitable for use in HVAC systems.

Table A.1 Brazing Fluxes

Product	Manufacturer	Published Composition	Working Concentration
No Rez 65	Novamax	Water, > 60% Hydrobromic acid < 25%; Glycol ether, > 5% Amino alcohol, > 5% Ethylene diamine, < 5%	Flux: water 1:1 to 1:3 v/v
Stay Silv Black	J. W. Harris	Boric acid, 30-50% Potassium bifluoride 25-40% Water, 15-20% Potassium tetraborate, undisclosed amount Potassium pentaborate, undisclosed amount	Use as packaged; may be thinned with water.
Stay Silv White	J. W. Harris	Boric acid, 30-50% Potassium bifluoride 25-40% Water, 15-20% Potassium tetraborate, undisclosed amount Potassium pentaborate, undisclosed amount	Use as packaged; may be thinned with water
Mocolok 100	Soluay	Potassium Tetra Aluminate, (KAlF ₄) undisclosed amount	Use as packaged
DAG - 137	Acheson Colloids	Graphite (?) Colloid undisclosed amount	Use as packaged
Flux 604 PD	Superior	Potassium bifluoride 30- 35 % Potassium pentaborate 30 - 30 %	As packaged; may be thinned with water
Flux DB	Lucas-Milhaupt, Inc.	Potassium; salt of Boron and Fluoride Potassium Boron Oxide	Use as packaged
Handy Flux D	Lucas-Milhaupt, Inc.	Potassium bifluoride 30- 35 % Potassium pentaborate 30 - 30 %	As packaged; may be thinned with water
Handy Flux D-2	Lucas-Milhaupt, Inc.	Potassium bifluoride 30- 35 % Potassium pentaborate 30 - 30 %	As packaged; may be thinned with water

2.2 Cleaners, Degreasers and Detergents

Appendix B, Table B.1. contains a list of cleaners, degreasers, and detergents. All are salts of silicates and organic emulsifiers. Used as the final wash before assembly of compressor parts, the cleaners, degreasers, and detergents submitted, will remain in contact with the working fluid system. Alkaline surfaces will react with: 1) POEs in the presence of water retained from assembly; and 2) in POE charged into the system. It is likely that POE on bearing surfaces will result in detracting from the surface wetting action, however, most inorganic, thin film residues will not be transported by the POE HFC combination.

Table B.1 Cleaners, Degreasers, and Detergents

Product	Manufacturer	Published Composition	Working Concentration
Careclean MP	Dubois	company refused to participate	
Cerfakleen 5380	E.F. Houghton	proprietary; alkaline	2-3% v/v with water
Cerfakleen 5382	E.F. Houghton	proprietary; alkali salts and surfactants	2% v/v with water
Cerfakleen 5384	E. F. Houghton	proprietary, alkali salts and surfactants	1-3% v/v with water
Cerfakleen 5391	E.F. Houghton	proprietary.	2-4% v/v with water
Citrus Cleaner (aerosol)	3M	d-limonene, 70-80% propane (propellant) 10-20% proprietary surfactants; 7-13%	as packaged
Dustryp	Dubois	company refused to participate	
FAC 106	Dubois	company refused to participate	
Liftaway	Dubois	company refused to participate	
Oakite LSD	Oakite	sodium hydroxide, 10-20%, sodium xylenesulfonate, < 5% sodium silicate, < 5%, nonylphenoxy polyethoxy ethanol, 5% undisclosed nonhazardous ingredients	
Bruln 815 GD	Bruln Corporation	proprietary; includes detergents, alkaline builders, and inhibitors	1:10-1:20 (v/v) product:water depending on use
Bruln 1990 GD	Bruln Corporation	proprietary; includes detergents, phosphates, silicates, and inhibitor	1:20 (v/v) product:water
Bruln 63-G	Bruln Corporation	proprietary; includes detergents, phosphates, silicates, and inhibitor	1:20 (v/v) product:water

Table B.1 (continued) Cleaners, Degreasers, and Detergents

Brulin 224	Brulin Corporation	proprietary; includes water, alkaline builders, and surfactants	1:64-1:128 (v/v) product:water depending on use
Brulin 815 QR (degreaser)	Brulin Corporation	proprietary; includes detergents, phosphates, silicates, and amines	1:10-1:20 (v/v) product:water for most uses
Oakite 77 (degreaser)	Oakite	sodium hydroxide, 40-50 % sodium dodecylbenzene sulfonate, < 10 % sodium metasilicate, < 10 % sodium carbonate, < 10 % dipentene, < 5 % undisclosed nonhazardous ingredients	unknown
Gillite 0650	Man-Gill Chemical	none	unknown
Dubois 422	Dubois	company refused to participate	
Parco 142	Parker/Amchem	proprietary; includes surfactants	5-10% w/w with other cleaners
Parco 328	Parker/Amchem	proprietary; include potassium hydroxide (10-30%).	1.5 - 4% v/v with water
Simple Green	Sunshine Makers	proprietary, includes nonionic surfactants	depends on use; 1:10 (v/v) product:water for most industrial applications
D-Limonene	Florida Chemical Co.		
Okemclean	Oakite		
Inproclean 1300	Oakite		
Rustripper	Oakite		
Castrol Kleen 3625	Castrol Industrial		
Solvox Special #474 - R - I Hard Surface	Solvox Manufacturing Co.	alcohols, ethoxylated or propoxylated; sodium metasilicate; 2 - butoxyethanol; sodium nitrite	unknown

2.3 Coolants

Appendix C, Table C.1 contains a list of coolants. The coolants listed are synthetic materials, some of unknown compositions, and may consist of lubricity additives, emulsifiers, trifunctional amine complexes, and carboxylic acid salt complexes. All are diluted with water and none are reported to contain chlorine, phosphate or sulfur heteroatom additives. Generally, coolant materials are washed away by an appropriate aqueous cleaner during final assembly.

Table C.1 Coolants

Product	Manufacturer	Published Composition	Working Concentration
Chemtool 415	Chemtool	synthetic fluid	not given
Chemtool 500	Chemtool	synthetic fluid	not given
Chemtool 643	Chemtool	synthetic fluid	not given
Cimstar 3700T	Cincinnati Milacron	proprietary; alkaline	1:20 (v/v) product:water
Cimstar 40	Cincinnati Milacron	proprietary <i>w/mo</i> <i>semi-synthetic</i>	1:20 (v/v) product:water
Cimtech 3900	Cincinnati Milacron	proprietary; alkaline	1:20 (v/v) product: water
Tech Cool	Chemical Technologies, Inc.	proprietary	1:10-1:40 (v/v) product:water, depending on use
Solene 1000	Solene Industrial Lubricants	alkanolamine proprietary additives	1:9 to 1:30 dilution with water
Solene 4000	Solene Industrial Lubricants	alkanolamine proprietary additives	1:9 to 1:30 dilution with water
Controlant 127S	Oakite		
Safety Cool 800	Castrol Industrial Central, Inc.	petroleum oil sodium tetraborate	(?%)

2.4 Iron Phosphatizers

Appendix D, Table D.1 contains a list of iron phosphatizers. Iron phosphatizers have been used in the treatment of cast iron and steel shaft surface materials. Uses of this processing material in compressors or systems construction were not included and are unknown.

Table D.1 Iron Phosphatizers

Product	Manufacturer	Published Composition	Working Concentration
IPI 25	Dubois	company refused to participate	
IS 1	Mc Gean-Rohco	detergent, accelerator, phosphoric acid	0.5 - 2% v/v with water, depending on use
Crysoat 747	Oakite	phosphates, activator agents, surfactants, and fluorides	3-5% v/v with water
Crysoat 2147	Oakite		
Crysoat 1127	Oakite		
Crysoat Ultra Seal	Oakite		

2.5 Processing Lubricants

Processing lubricants submitted are listed in Appendix E, Table E.1. The recorded usages are with the following: assembly, ball (tube) expanding, drawing, forming, honing, tube bending, tube fitting, die lubes, and extruding oils. These processing lubricants are used in the production of compressor and system parts and will likely remain in the HVAC system. Some materials intentionally remain in the machines and some remain due to inadequate washing or vendor quality. Drawing oils generally contain extreme pressure lubricants such as calcium, barium, and/or sodium sulfonate contents. These soaps serve to lubricate, as well as assist and ease removal of caustic degreasers. In low temperature systems, remaining hydrocarbon oils are generally unacceptable contaminants in HFC applications. Particularly in capillary systems, paraffin hydrocarbon is unacceptable. A specific objectionable lubricant includes Suniso 160 assembly oil. Paraffin oil is stabilized toward oxidation with Zinc dithioaryl or alkyl phosphates (ZDTPs), however, it may be useful for improved oxidation stability of POEs.

Table E.1 Lubricants

Product	Manufacturer	Published Composition	Working Concentration
Oakite SPO2 (assembly)	Oakite		
Suniso 3GS	Witco	refined mineral oil	insoluble in water
Suniso 4GS (assembly)	Witco	refined mineral oil	insoluble in water
Oakite 59 (ball expanding)	Oakite		
Oak 50-5 (drawing)	Oak International		
Quaker Ferrocote 5684 (drawing)	Quaker Chemical		
Oak 14B-1 (drawing)	Oak International		HFC - compatible (miscible)
Oak 14B-2 (expansion)	Oak International		HFC - compatible (miscible)
Oak 60-1 (forming)	Oak International		not in contact with internals
Oak Oil HC (forming)	Oak International		not in contact with internal
Tower A-1830 (forming)	Tower Chemical		
Honilo 480 (honing)	Castrol Industrial		
Care Tech (honing)	Castrol Industrial		
F4422 Oil (honing)	Schrader Bellows	severely refined heavy paraffinic petroleum oil (90 - 100 %)	as packaged
Maaco HEG-18 (honing)	Witco		
Oak Oil 11C (tube bending)	Oak International		not compatible with HFC's?
Master Draw 1969-A-1 (tube fitting)	Etna Products	synthetic wetting agents, fats, and emulsifiers. water soluble	~ 10% v/v with water, depending on use.
Baker and Grubbins D1337 (tube fitting)			

Table E.1 (continued) Lubricants

XE - 6368	Solene	90-92 % aliphatic petroleum distillates	Use as packaged
XE - 6512	Solene	polyalkalene glycol + additives	Use as packaged
XE - 6338 Drawing Lube Cutting Lube	Solene	petroleum based with chlorinated paraffin	Use as packaged.
Rex Draw 176	Benz Oil, Inc.	mineral oil 40% chlorinated paraffin 40% proprietary emulsifier 3% Ba & Na sulfonate 10%	
Safety Kleen 140	Safety Kleen Corporation	petroleum naphtha 100%	
Lab Oil #720	Heatbath Corporation	sorbitan monooleate 2.4% naphthenic oil 64.9% tetraethylene glycol 2.4% polyglycol 400 1.5% tripropylene glycol 1.0% Alox 575 27.8%	
Piedmont 562 Drawing & Extruding	Piedmont Chemical Co.	distillates (petroleum) hydro-treated, heavy naphthenic > 5% solvent refined paraffinic distillates > 5% sulfonic acid, petroleum, calcium salts, overbased (calcium sulfonate) > 5%	
Draw Clean V	Oakite	aliphatic solvent 100%	
ZEROL 150	Shrieve Chemical Products Co.	alkylbenzene	
Oakite Formula #59	Oakite		
Etna 566	Etna Products		
Suniso 160	Witco		
CutMax-570			

2.6 Rust Inhibitors

Appendix F, table F.1 contains a list of rust inhibitors. Compositions range from paraffin hydrocarbons to complex tertiary amine carboxylic acid salts. Some inhibitor systems are inorganic and use borax, sodium nitrite, and/or a carboxylic acid. These systems should be considered intermediate inhibitors, applied only for storage purposes and not for final assembly. Rust inhibitor compatibility with HFCs and POE should consider surface adhesion and substance solubility. As well, the thermal stability of the lubricant system should be thoroughly investigated.

Table F.1 Rust Inhibitors

Product	Manufacturer	Published Composition	Working Concentration
Resco 27-ABE-4	Research Metal Fluids	amine carboxylate salts	
Maaco 1961	Witco	none	
Maaco 102LK-6	Witco	none	
Rustarest 1073	Man-Gill Chemical	non-hazardous material	dilute with water
Novamax R44	Novamax	alkanolamine borates ethanolamine 3-6%	
Rustproof 4200 Alkaline Corrosion Inhibitor	Ardrox	monoethanolamine 15%	dilute with water
Protech 1300	Chemical Technologies, Inc.	petroleum naphtha and non-hazardous additives	
Gilcote 4127	Man-Gill Chemical	Borax (10 mole) dodecanoic acid sodium nitrite	dilute with water
Meca Lube	Puma Technologies	ethyleneglycol monobutyl ether <58% aliphatic hydrocarbon solvent <2.0% mineral oil >40%	Use as packaged
Tectyl 900	Ashland Chemical, Inc.	barium petroleum sulfonate neutral 1-5% lubricating oil base 55-70% petroleum hydrocarbon 10-25%	use as packaged
Eaken A/R - 10	R. W. Eaken Inc.	aliphatic hydrocarbon proprietary additive	
Resco Oxykoate Syn .	Research Metal Fluids		

Table F.1 (continued) Rust Inhibitors

W&B Rust Inhibitor 3340	White & Babley of Michigan, Inc.	triethanolamine water boric acid caprylic acid oxygenated hydrocarbon partially neutralized with triethanolamine fatty amide concentrate	
Diversey E-314 Emulsion Cleaner / RP	Diversey Corp.	unknown but contains hydrocarbon mixture	dilute in water
Rustproof Oil 00564	Texaco, Inc.	petroleum oil 35-50% additive package containig barium, sulfur 1-5% additive package containing phosphorous < 1%	
Inhibitor W	Madison Chemical Co., Inc.	sodium nitrite < 60%	dilute in water
Rust Prevent 100	Dubois Chemicals, Inc.	non-hazardous	water reducible
Rust-Ban 343	Exxon Company, U.S.A.	distillate petroleum < 90% proprietary additives < 10%	use as packaged
Rust Vito 4214	E. F. Houghton & Co.	stoddard solvent > 60% mineral oil 1-10%	unknown
Dri-Touch IRP1	Birchwood Casey	mineral spirits > 84% heavy petroleum oxygenates, barium neutralized ,12% propylene glycol monomethyl ether < 4%	unknown
Met-Chem 211	Met-Chem, Inc.	mixture of triethanolamine (85%) plus Aqualox 232 of amine salts of organic acids	dilute in water
Anti-Rust Oil #2-V	Amoco Oil Co.	stoddard solvent solvent refined paraffinic petroleum oil	Use as packaged
Ferrocote 368	Quaker Chemical Corporation	diethylene glycol monobutyl ether 1-5% barium sop of oxygenated hydrocarbon 5-10%	unknown
Resco Ultra Koate XP	Reserch Metal Fluids		
Dubois 200 NT	Dubois Chemicals, Inc.	none given	Unknown

2.7 Rust Preventatives

Appendix G, Table G.1 contains a list of rust preventatives. Generally, rust preventatives are used for protection during storage of aluminum bloom and steel surfaces. In terms of final assembly usage, the submitted list is undesirable. However, the Diversey E-314 emulsion cleaner in Table F.1 is a hydrocarbon solvent mixture and constitutes as a rust preventative, yet is listed by the manufacturer as a rust inhibitor. This material may be useful for final assembly.

Table G.1 Rust Preventatives

Product	Manufacturer	Published Composition	Working Concentration
Almco 2420	Almco	proprietary; mildly alkaline	0.5 - 2% v/v with water
Almco H10	Almco	proprietary	not stated
Rustilo 924	Castrol Industrial		
Rustilo DWX 30	Castrol Industrial		
Chemtool 625 ND	Chemtool	proprietary: "synthetic metalworking fluid"	not stated
Chemtool 1256	Chemtool	proprietary: "synthetic metalworking fluid"	not stated
Inpro-tect	Oakite	amines, phosphates, surfactants	3-7% v/v with water
Prevon 501	Parker	proprietary.	3-10% v/v with water for spray, 5-20% for dip
Rust Preventative #308	Mullen Circle Brand, Inc.	mineral spirits < 1% glycol ether > 1%	

2.8 Sealants and Other Compounds

Appendix G, Table G.1 contains a list of burnishing compounds, anaerobic sealants, biocides, and thread retaining compounds. Certainty of material usage is found only in the sealants and retaining compounds. Cross-linked anaerobic sealants are insoluble in most lubricants. Some retaining compounds, in the presence of POEs, and under sealed tube conditions and higher temperatures, show a failure mode of softening in some selected POE solutions. When HFCs are added to these POE solutions, the solubilized polymers show reduced solution and precipitation of a solid upon cooling.

Table H.1 Sealants and Other Compounds

Product	Manufacturer	Published Composition	Working Concentration
Roto-Brite (R) L-543 Burninshing Compound	Roto-Finish	proprietary	1:12
Loctite 640 Sealant	Loctite	polyurethane methyl acrylate resin 55-60% polyglycol dimethyl acrylate 25-30%	use as packaged
Loctite 515 Gasket Eliminator Sealant	Loctite	polyurethane methyl acrylate resin 70-75% polyglycol dimethyl acrylate 25-30%	use as packaged
Renovator	Oakite	phosphates, surfactants, fluorides, sequestrants	
Oakite 80 Biocide	Oakite	hexahydro -1,3,5-tris (2- hydroxyethyl) - s -triazine 78.5% proprietary ingredients 21.5%	

Table H.1 (continued) Sealants and Other Compounds

<p>Loctite RC 620 Retaining Compound</p>	<p>Loctite</p>	<p>aromatic dimethacrylate ester 75-80% maleimide resin 10-15% cumene hydroperoxide 1-3 hydroxyalkyl methacrylate 1-3 methyl alcohol 1-3 silica, amorphous treated 1-3 1-acetyl-2-phenylhydrazine 0.1-1 N,N-dialkyltoluidines 0.1-1 maleic Acid 0.1-1 saccharin 0.1-1</p>	<p>Use as packaged</p>
<p>Loctite Liter Retaining Compound 620</p>	<p>Loctite</p>	<p>aromatic dimethacrylate ester 75-80% maleimide resin 10-15% cumene hydroperoxide 1-3 methyl alcohol 1-3 silica, amorphous treated 1-3 1-acetyl-2-phenylhydrazine 0.1-1 N,N-dialkyltoluidines 0.1-1 maleic Acid 0.1-1 saccharin 0.1-1</p>	<p>Use as packaged</p>

2.9 Published Information

Two papers (DeKleva, T. W., et al, 1992) and (Greig, B. D., et al, 1992) have been published and have identified organic and inorganic materials that could potentially cause expansion device restrictions and plugging. It is generally accepted that chlorinated contaminants and materials such as CFC-11 or CFC-12 in HFC-134a systems have a negative effect on system chemical stability. Without water present, many chlorinated contaminants are acceptable. According to these published papers, many unidentified process materials were examined and consisted of the same type of chemicals tested in this program with the exception of paraffin winding lubricant wax.

Chemical compounds suggested in these two published papers include: abietic acid, bisphenol A, carbon residue epoxides, immiscible ester lubricant, 1-isocyanato-2-methyl benzene, paraffinic hydrocarbons, metal carboxylated oxidized/polymerized mineral oil, paraffin wax, polyethylene glycol terephthalate, HMW polyalphaolefins, polyamides and silicone.

The inorganic compounds reported were aluminum, antimony, copper, sodium, iron salts, sulfur compounds, metal chlorides, oxides, phosphides, and sulfides, metal silicates. The assignment of these organic and inorganic materials is a result of a combination of analytical methods that use FTIR peak assignments, mass spectroscopy, and EDAX.

2.10 Replacements for Some Selected Materials

From the initial list of 52 selected manufacturer process materials, at least 7 materials were not submitted or were no longer being sold. Therefore, substitutions have been made as follows:

Figure 1 - Replacements for Some Selected Materials

Original	Replacement
Master Draw 1969	Master Draw 176
W & B Rust Inhibitor	Novamax Protech 1300
Chemtool 625 ND	Chemtool CT625
Rustilo DWX22 and DWX30	Rustilo DW924
MAACO 196	Resco Ultra Koate XP
Oakite SP02	Oakite Formula 59
OAK 14B-1 (not supplied)	Resco Oxy Kleen 4926A**
OAK 14B-2 (not supplied)	Crysocoat 1127**

**Resco Oxy Kleen 4926A and Crysocoat 1127 can serve as the last coating applied prior to assembly of the system components.

3 ANALYTICAL PROCEDURES

One of the experimental objectives was to evaluate the solubility temperatures of process materials dissolved in two polyolesters: 1) Mobil EAL Arctic 32; and 2) ICI RL32H. Concentrations of 0.01%, 0.1%, and 0.5% w/w will be evaluated followed by the production of sealed tubes of these mixtures with HFC-134a at 90: 10 lubricant: refrigerant and refrigerant: lubricant ratios.

As a result, a protocol has been designed wherein the processing materials are reduced to a residue of constant weight in a glass dish. This is accomplished by first dehydrated in an open hood for five days. These materials are then dehydrated further at 45°C in a circulating air bath oven for at least 3-8 days until constant weight. To make the three concentrations of 0.01%, 0.1% and 0.5% by weight, the process material is weighed into test tubes containing the polyolesters. Tables 1 and 2 show the process materials tested thus far. As seen, most of the materials are not soluble in the POEs. Conditions of 100°C for five days continue to indicate incomplete solubility.

A small amount of the residue applied to a steel coupon and then exposed to HFC-134a RL32H or Mobil EAL 32 at a 1:9 ratio (in a 2 gram system) after 5 days at 175°C also failed to give any clear indication of solubility. The tables indicate four arbitrarily selected residues that were found insoluble in the ICI Emkarate RL32H. We then evaluated these residues with the Mobil EAL Arctic 32 and refrigerant system.

Because of the uncertainty of the amount in solution, it is difficult to determine complete miscibility in a test tube of lubricant. As well, it is equally difficult to estimate how much has gone into solution with a refrigerant and oil mixture in a sealed tube. It is important to report a reasonably accurate level of solubility.

It is necessary to know how much of a particular processing material residue goes into solution with a particular polyolester. Our results indicate only incomplete solubility of the processing material residues, however, selective solubility of specific kind of component could cause problems in a system in the future. An additional problem with this study of processing materials is that most water based materials may leave a partially soluble layer on machine parts. Therefore, surface effects must be taken into account and steel and aluminum chips are coated with a representative amount of processing materials. As previously stated, testing only a small amount of residue in a tube is an analytical failure mode. To compensate for this effect, we have an alternate, instrumental approach that will help evaluate processing materials

with partial solubility. The alternate procedure(s) being considered and evaluated uses either or both sizes of exclusion chromatography (SEC) and/or UV spectroscopy using standard analytical methodologies.

The first part of the SEC approach was to find a common solvent for all processing material residues (see Table 3). The results of this search include several solvent systems that solubilize the processing materials and are also compatible with the chosen SEC solvent system. The common solvent must also solubilize the polyolester. Aqueous alcohol (methanol, ethanol, or isopropyl) water dilutions were first attempted. More exotic materials, such as aqueous NMP, DMF or DMSO, then followed. These all failed due to viscosity limitations within the SEC column. We now believe the correct solvent system is an aqueous tetra hydro furan (THF) mobile phase using the UV detector on an absorbency knee of the THF solvent. The solutions listed in Tables 4 and 5 make up the standards of the processing materials in the aqueous THF system. It is then possible to filter out the polyolester from the processing material using SEC. This procedure is as follows: The molecular weight of a polyolester is much larger than that of a processing material. The polyolester has an estimated molecular weight of 600 to 650 whereas, processing materials that are oils and water reducible are about 175 to 580 molecular weight. A typical SEC column acts like a filter for large molecular weight materials. The heavier (larger) components elute first, in this case the polyolester, followed by the lighter (smaller) components, in this case the processing materials. An example of this procedure with Oakite Crysoat 747TM is seen in Figure 2. Figure 3 indicates the Mobil Arctic EAL 32 response. The response and sensitivity is present and visible. At this time, all of the peaks in the processing materials are being grouped as total area counts. However, from each processing material residue that appears to be essentially insoluble, it is also possible to see which components are soluble. Therefore, processing materials that are partially soluble in POEs at 100°C can be identified if they fail the CST of HFC-134a and lubricant dilutions.

Figure 2 - Processing Materials Procedure with Oakite Cryscoat 747™

Figure 3 - Processing Materials Procedure with Mobil Arctic EAL 32

Following this analytical method, standards of the processing materials will be produced in the correct solvent combination at 0.01% and 0.5% w/w concentration. The total peak area counts will become the total amount dissolved. In a 250ml bottle at the 0.5% concentration, the polyolester will be subjected to the processing material residue coated on 10-20g of cast iron turnings. The bottle and its contents will then be tumbled for 5 days at 100 °C. Cast iron fillings provide a large surface area for any enhanced solubility. After 5 days, a hot aliquot of the polyolester will be taken and subjected to HFC-134a miscibility determinations. The error in a visual analysis of CST (partial or cloud point) formation is that the low levels of processing materials that can cause a problem in an operating system may not be visible in the 0.01% w/w range. Therefore, the SEC analytical procedure is useful. For the final analysis, the flow chart in Figure 3 is necessary. The tubes that fail the CST test are then checked by SEC. The area counts of which peaks are responsible for immiscibility will be calculated for its concentration in POE. Because all of the processing materials put onto the metal surface were lubricant insoluble, only the component that was partially soluble will be determined. Once the semi-quant data is known, a more realistic value can be made on the compatibility of the processing material remaining in the compressor or HVAC system.

The above mentioned analytical routines for lubricant soluble compounds is now in progress and follows the flow chart in Figure 3. Miscibility and immiscibility is seen as white and black blocks in Tables 4 and 5.

All of the sealed miscibility tubes are constructed at a ratio of 95: 5 refrigerant to lubricant mixture. The sealed tubes are constructed with iron, aluminum, and copper coupons for aging at 14 days and 175°C. Miscibility will be determined and listed as additional columns in Tables 4 and 5. One of the primary objectives of this research is to identify if any chemistry is taking place with the soluble components, and also to identify if any immiscibility can occur.

Figure 4 - Processing Materials Flow Diagram

4 CONTRACT AGREEMENT

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

5 PRINCIPAL INVESTIGATOR EFFORT

The principal investigator and support personnel have devoted 1980 hours toward the completion of this contract. Additional activities include: reporting early refrigerant miscibility information to the Technical Oversight Group and the continuous establishment of experimental methodologies.

6 REFERENCES

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