



(86) Date de dépôt PCT/PCT Filing Date: 1998/08/25  
 (87) Date publication PCT/PCT Publication Date: 1999/03/04  
 (45) Date de délivrance/Issue Date: 2008/08/19  
 (85) Entrée phase nationale/National Entry: 2000/02/24  
 (86) N° demande PCT/PCT Application No.: US 1998/017536  
 (87) N° publication PCT/PCT Publication No.: 1999/010455  
 (30) Priorité/Priority: 1997/08/25 (US08/918,177)

(51) Cl.Int./Int.Cl. *C10M 105/38* (2006.01),  
*C09K 5/10* (2006.01), *C10M 169/04* (2006.01),  
*F04C 29/02* (2006.01)  
 (72) Inventeurs/Inventors:  
MCHENRY, MICHAEL A., US;  
CARR, DALE D., US;  
SCHAEFER, THOMAS G., US  
 (73) Propriétaire/Owner:  
HATCO CORPORATION, US  
 (74) Agent: BLAKE, CASSELS & GRAYDON LLP

(54) Titre : ESTER DE POLY(NEOPENTYL POLYOL) A BASE DE REFRIGERANTS ET CONDITIONNEMENT A BASE  
D'ADDITIFS PERFECTIONNE  
 (54) Title: POLY(NEOPENTYL POLYOL) ESTER BASED COOLANTS AND IMPROVED ADDITIVE PACKAGE

(57) **Abrégé/Abstract:**

A synthetic lubricant composition is a blend of a polyol ester blend admixed with effective amounts of an antioxidant, yellow metal pacifier and rust inhibitors. The polyol ester blend includes a major proportion of poly(neopentyl polyol) ester blend formed by reacting poly(pentaerythritol) partial esters with at least one C<sub>7</sub> to C<sub>12</sub> carboxylic acid mixed with an ester formed by reacting a polyol having at least two hydroxyl groups and at least one C<sub>8</sub>-C<sub>10</sub> carboxylic acid. Preferably, the acids are linear and avoid those which can cause odors during use. Effective additives include secondary arylamine antioxidants, triazole derivative yellow metal pacifier and an amino acid derivative and substituted primary and secondary amine and/or diamine rust inhibitor.

**PCT**WORLD INTELLECTUAL PROPERTY ORGANIZATION  
International Bureau

## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

<p>(51) International Patent Classification <sup>6</sup> : C10M 105/38, 169/04, 171/00 // (C10M 105/38, 105:38) (C10M 169/04, 105:38, 105:38, 129:10, 133:06, 133:08, 133:12, 133:16, 133:44), C10N 40:30</p>	A1	<p>(11) International Publication Number: <b>WO 99/10455</b></p> <p>(43) International Publication Date: 4 March 1999 (04.03.99)</p>
<p>(21) International Application Number: PCT/US98/17536</p> <p>(22) International Filing Date: 25 August 1998 (25.08.98)</p> <p>(30) Priority Data: 08/918,177 25 August 1997 (25.08.97) US</p> <p>(71) Applicant (for all designated States except US): HATCO CORPORATION [US/US]; 1020 King George Post Road, Fords, NJ 08863 (US).</p> <p>(72) Inventors; and (75) Inventors/Applicants (for US only): MCHENRY, Michael, A. [US/US]; 10 Evelyn Terrace, Sayerville, NJ 08879 (US). CARR, Dale, D. [US/US]; 54 Wetmore Avenue, Morristown, NJ 07960 (US). SCHAEFER, Thomas, G. [US/US]; 35 Driftwood Drive, Parlin, NJ 08859 (US).</p> <p>(74) Agent: WOLFSON, Michael, I.; Cowan, Liebowitz &amp; Latman, P.C., 1133 Avenue of the Americas, New York, NY 10036-6799 (US).</p>	<p>(81) Designated States: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, GH, GM, HR, HU, ID, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).</p> <p><b>Published</b> With international search report.</p>	
<p>(54) Title: POLY(NEOPENTYL POLYOL) ESTER BASED COOLANTS AND IMPROVED ADDITIVE PACKAGE</p>		
<p>(57) Abstract</p> <p>A synthetic lubricant composition is a blend of a polyol ester blend admixed with effective amounts of an antioxidant, yellow metal pacifier and rust inhibitors. The polyol ester blend includes a major proportion of poly(neopentyl polyol) ester blend formed by reacting poly(pentaerythritol) partial esters with at least one C<sub>7</sub> to C<sub>12</sub> carboxylic acid mixed with an ester formed by reacting a polyol having at least two hydroxyl groups and at least one C<sub>8</sub>-C<sub>10</sub> carboxylic acid. Preferably, the acids are linear and avoid those which can cause odors during use. Effective additives include secondary arylamine antioxidants, triazole derivative yellow metal pacifier and an amino acid derivative and substituted primary and secondary amine and/or diamine rust inhibitor.</p>		

**POLY (NEOPENTYL POLYOL) ESTER BASED****5 COOLANTS AND IMPROVED ADDITIVE PACKAGE**BACKGROUND OF THE INVENTION

10 This invention relates to synthetic ester compositions and in particular to compositions based on a blend of poly(neopentyl) polyol ester and an ester of a polyol containing at least two hydroxyl groups, which is inhibited with a unique combination of additives. The compositions are particularly well suited for use as a coolant/lubricant in a rotary screw air compressor. The coolant/lubricant compositions minimize use of esters formed from highly odoriferous acids, yet provide extended life, higher temperature operation, excellent demulsibility, 20 biodegradability, rust protection and hydrolytic stability -- a combination of properties not available in existing synthetic compressor coolant/lubricant compositions.

It is well known to use hydrocarbon lubricating oils in rotary screw compressors. The oil seals the rotors, lubricates 25 the bearings, cools the compressed gases and removes water condensed from the compressed gases. The high temperature and pressure and presence of water causes the hydrocarbon oils to break down and create sludge in a relatively short time.

Efforts to lengthen the useful life of coolants for air 30 compressors have led to the use of synthetic esters as basestocks. Typical operating temperatures are between about 80° to 104°C (170° to 220°F). While these temperatures are not high for a synthetic lubricant, the environment is highly oxidative so that mineral oils tend to break down after about 35 1,000 hours of use and must be changed. Several of the synthetic coolant/lubricants presently in use extend the intervals between changing lubricants to as much as 8,000 hours.

A synthetic ester coolant based on diesters of adipic acid and phthalic acid have been used for more than 25 years in a variety of compressors including rotary screw compressors. Other synthetic coolants in use are based on synthetic hydrocarbons or poly alpha olefins (PAO's), polyalkylene glycols (PAG's), silicones, and mixtures of synthetic hydrocarbons and dibasic acid esters. All of these products provide extended lubricant life when compared to petroleum based coolants used in the highly oxidative environment in a screw compressor. However, none of the available lubricants are able to provide all of the desired properties, in particular the combination of biodegradability with hydrolytic stability, rust protection, and demulsibility while avoiding offensive odors.

In addition to these performance properties, certain physical properties are required, such as providing efficient cooling, low temperature start-up, sealing of the rotors, and lubrication of the bearings. In particular, the desirable physical properties include a viscosity at 100°C (212°F) of at least 8 cSt, a viscosity index of at least 140, a flash point of at least 260°C (500°F), and a pour point of at most -50°C (-58°F).

A wide operating temperature range for the coolant is highly desirable. In order to permit low temperature starting, a lubricant having a pour point less than -50°C (-58°F) is required. Suitable high temperature viscosity properties and low volatility are needed to permit suitable lubrication at higher operating temperatures. Thus, the coolant composition should have a flash point above 260°C which is required by some manufacturers for safety reasons.

All screw compressor coolant/lubricants will come into contact with water condensed from the compressed air. Therefore, any lubricant must exhibit good hydrolytic stability and provide good rust and corrosion protection. It is an added advantage to provide a lubricant exhibiting good demulsibility.

This will permit separation of water from the spent lubricant and facilitate disposal and recycling of the spent lubricant.

Increased biodegradability is also highly desirable. In addition, the elimination of heavy metals, utilized in some commercial coolants, is also desirable from an environmental point of view.

It is also highly desirable to avoid objectionable odors. Thus, it becomes desirable to avoid or minimize the use of esters formed from carboxylic acids having less than seven carbon atoms. Finally, adequate regulatory listing is desirable.

Existing petroleum based and synthetic air compressor coolant/lubricants are not capable of providing all of the desired performance and physical properties for modern rotary screw compressors. Often, modification of one component of a synthetic lubricant will improve a certain desired property, but at the expense of another. For example, coolants based on PAO's have poor biodegradability; coolants based on adipate diesters cannot provide the viscosity, or flash point properties; coolants based on phthalate diester have poor biodegradability and viscosity indices; coolants based on silicones have poor biodegradability and rust protection; coolants based on polyalkylene glycol have poor demulsibility and inadequate rust protection; and coolants based on mixtures of PAO's and diester do not provide the desired biodegradability.

The following table summarizes the desired properties generally available from current types of rotary screw compressor coolant/lubricants. While several different types may be noted as having satisfactory performance of a particular property, there will be variations within acceptable ranges, some of which may be significant to the user. An "X" indicates that the referenced type of commercially available coolant/lubricant can provide the desired property, and an "O" indicates that it is not fully acceptable.

**TABLE 1**  
**SIGNIFICANT DESIRED PROPERTIES FOR**  
**ROTARY SCREW COMPRESSOR COOLANT/LUBRICANT COMPOSITIONS**

Desired Properties	Desired Composition	Mineral Oil	PAO (Polyalpha Olefin)	Diester	PAO/Ester	PAG (Polyalkylene Glycol)	Silicone	Phosphate	PAG/Ester
Pour Point <-50°C	X	0	X	0	X	X	X	0	X
Flash Point >260°C	X	0	0	X	0	X	X	X	X
Good Rust Prevention	X	X	X	X	X	0	0	0	0
Good Demulsibility	X	X	X	X	X	0	X	0	0
Good Hydrolytic Stability	X	X	X	0	X	X	X	0	X
Biodegradable	X	0	0	0	0	0	0	0	0
Viscosity Index >140	X	0	X	0	X	X	X	0	X

---

\* The poly(neopentyl polyol) ester based compositions prepared in accordance with the invention exhibit all of the desired properties.

One of the most widely used high performance rotary screw compressor coolants is a PAG/ester synthetic lubricant of the type described in U.S. Patent No. 4,302,343 to Carswell et al. The Carswell PAG/ester lubricant is an inhibited blend of a polyalkylene glycol with hindered alkanolic ester of aliphatic polyhydric alcohols having three to eight hydroxyl groups and five to ten carbon atoms. When properly blended and mixed with additives, the resulting synthetic coolant/lubricant has been found to satisfy the high temperature viscosity requirements and exhibit stability to the heat, air and water environment. However, ferrous metal corrosion resistance and demulsibility has been found less than satisfactory. The presence of the heavy metal barium also presents disposal concerns.

U.S. Patent No. 4,175,045 to Timony describes a compressor lubricant formed of a polyol ester of a carboxylic acid having from about four to thirteen carbon atoms. The polyols utilized are pentaerythritol, dipentaerythritol, trimethylolpropane, or combinations thereof. The lubricants are blends of the pentaerythritol ester and dipentaerythritol esters. While providing satisfactory useful life and temperature characteristics, the viscosity at 100°C (212°F) and pour point do not meet the desired values as set forth in Table I. In addition, the lubricants are based on substantial amounts of esters formed from valeric acid and thus in use present undesirable odor problems. Improvements in rust prevention and demulsibility are also needed.

U.S. Patent No. 3,694,382 to Kleiman, Malek and Lonsker discloses an ester lubricant which is a blend of 1.5 - 2.5 parts of a trimethylolpropane ester of aliphatic monocarboxylic acids containing from 4 - 12 carbon atoms with 0.75 - 2 parts of a dipentaerythritol ester of a mixture aliphatic monocarboxylic acids containing 4 - 10 carbon atoms. It is disclosed that the dipentaerythritol may include minor amounts of monopentaerythritol and higher condensation products of

pentaerythritol. The preferred blends include more than 50 weight percent dipentaerythritol ester. The products are suitable as synthetic lubricants in turbines and turbojet engines.

High temperature compressor oils are described in U.S. Patent No. 5,156,759 to Culpon Jr.. The compressor oils include a polyalphaolefin base lubricating oil together with well-known additive compositions including amine antioxidants and rust inhibitors/metal passivators which are a triazole and/or alkenylsuccinic acid rust inhibitor. Lubricant additives are also shown in U.S. Patent No. 2,830,019 to Fields and Brehm. These additives are reaction products of an amine and carboxylic acids for improving color stability and providing corrosion resistance. Additionally, aspartic acid derivatives as corrosion inhibitors of the type utilized herein are disclosed in Kugle and Blank, U.S. Patent No. 5,275,749. Such compositions are utilized to improve demulsibility as well as anti-corrosion and anti-wear properties of the lubricant.

European Patent Application No. 498 152 of CPI Engineering provides lubricant compositions miscible in non-chlorinated fluorinated hydrocarbon refrigerants. These lubricants are based on polyhydric alcohol esters of trimethylolpropane, mono- and/or dipentaerythritol esters made with branched carboxylic acids. Here, miscibility with the refrigerant composition is essential which requires the use of the branched esters.

As noted, the prior art describes a wide variety of synthetic lubricants based on various polyol esters and blends thereof admixed with various additives to improve performance. While these synthetic rotary screw air compressor coolant/lubricants are presently in wide use, it remains desirable to provide an improved coolant/lubricant composition

which can provide all the significant desired properties noted in Table I, particularly increased oxidative stability,

improved demulsibility, improved rust protection, increased biodegradability and low odor in use.

5

SUMMARY OF THE INVENTION

Generally speaking, in accordance with the invention, an improved synthetic coolant/lubricant composition for a rotary screw compressor providing superior performance based exclusively on a polyol ester base stock and suitable additives is provided. The ester portion of the composition is a blend of a major amount of poly(neopentyl polyol) ester and a minor amount of a polyol ester formed from a polyol having at least two hydroxyl groups. The composition includes antioxidants, yellow metal pacifiers, rust inhibitors, hydrolytic stability improver and may include an antifoam additive. This unique approach of using a poly (pentaerythritol) ester based preparation was found necessary to obtain all the desired performance properties, particularly the combination of extended life, low pour point, high flash point, good demulsibility and reduced odor. This approach differs from the esters described in the patent literature and synthetic coolant/lubricant compositions in use today.

The poly(neopentyl polyol) ester component is a poly(pentaerythritol) ester formed by esterifying partial esters of pentaerythritol, dipentaerythritol, tripentaerythritol, tetrapentaerythritol, etc. with at least one monocarboxylic acid or a mixture of acids having from about six to twelve carbon atoms. The poly(pentaerythritol) ester is formed by first reacting pentaerythritol with the selected carboxylic acid or a mixture of acids with an excess of hydroxyl groups to carboxyl groups to form the partial esters.

In the preferred embodiments, the carboxylic acid is a linear acid having from seven to twelve carbon atoms.

The polyol ester component is utilized to balance the properties of the poly (pentaerythritol) ester, especially to

lower the pour point and improve biodegradability without adversely affecting any other desired properties. It is prepared by reacting a hindered polyol having from five to eight carbon atoms and at least two hydroxyl groups with a monocarboxylic acid having from seven to twelve carbon atoms. In the preferred embodiments, the polyol is trimethylolpropane and the acid is a linear acid having from seven to twelve carbon atoms.

The coolant/lubricant composition is formed by mixing from 50 to 80 weight percent poly(neopentyl polyol) ester and 20 to 50 percent polyol ester, and adding effective amounts of additives, for example between 0.5 to 10 weight percent antioxidants, yellow metal pacifier, rust inhibitors and an antifoam agent.

Accordingly, it is the object of the invention to provide an improved rotary screw compressor coolant/lubricant.

Another object of the invention is to provide an improved rotary screw compressor coolant/lubricant of a blend of poly(neopentyl polyol) esters and traditional ester polyol.

A further object of the invention is to provide an improved rotary screw compressor coolant/lubricant having increased oxidative stability, improved demulsibility, improved rust protection, increased biodegradability, and reduced volatility compared to conventional synthetic compressor lubricants.

Yet another object of the invention is to provide an improved additive package designed to provide improved demulsibility, enhanced rust protection and improved hydrolytic stability for a synthetic ester rotary screw compressor coolant/lubricant.

Yet a further object of the invention is to provide a method of lubricating a rotary screw compressor with a coolant/lubricant composition based entirely on polyol esters.

Another object of the invention is to provide a synthetic ester rotary screw compressor coolant/lubricant meeting all the desired properties without including significant amounts of

AMENDED CLAIMS  
- 1999/02/24

esters based on acids having unpleasant odors.

Still another object of the invention is to provide a method of lubricating a rotary screw compressor with a coolant/lubricant composition based on a blend of poly(neopentyl polyol) esters and polyol esters.

Still a further object of the invention is to provide a new additive package for a synthetic ester lubricant to improve rust protection, hydrolytic stability and demulsibility.

Still other objects and advantages of the invention will in part be obvious and will in part be apparent from the specification.

The invention accordingly comprises a composition of matter possessing the characteristics, properties, and the relation of components which will be exemplified in the composition hereinafter described, and the scope of the invention will be indicated in the claims.

#### **DESCRIPTION OF THE PREFERRED EMBODIMENTS**

The polyol ester based compressor coolant/lubricant compositions prepared in accordance with the invention include a polyol ester blend and performance additives designed to provide improved demulsibility, biodegradability, provide excellent ferrous metal rust prevention and improved hydrolytic stability. There are no odoriferous heavy components or metals in the composition.

The synthetic ester portion of the lubricant is a blend of poly(neopentyl polyol) ester and polyol ester. The lubricant includes from 50-80 weight percent of the poly(neopentyl polyol) ester and from 20 to 50 weight percent polyol ester. Preferably, the poly(neopentyl polyol) ester is present between 55 to 75 weight percent, and in the most preferred embodiments, between 65-70 weight percent, based on the total weight of the composition with the balance the polyol ester and the additives.

Preferably, the poly(neopentyl polyol) ester, which is a

mixture of esters of pentaerythritol, dipentaerythritol, tripentaerythritol, tetrapentaerythritol, etc. formed by reacting pentaerythritol with at least one monocarboxylic acid having from about 6 to 12 carbon atoms in the presence of an  
5 excess of hydroxyl groups to carboxyl groups. Most preferably, the acids have from 7 to 10 carbon atoms and are linear. In the most preferred aspects of the invention, the acid component of the poly(neopentyl polyol) ester is a linear monocarboxylic acid, or a mixture of linear acids with about five weight  
10 percent or less branched acid.

Acids having less than six carbon atoms are not within the scope of the invention, because they may impart objectionable odors in use. Suitable acids include, but are not limited to oenanthic acid, caprylic acid, pelargonic acid  
15 and capric acid. Preferably, the straight chain acid is a mixture of heptanoic (C<sub>7</sub>) and caprylic-capric (C<sub>8</sub>-C<sub>10</sub>). The caprylic-capric acid is usually identified as being a mixture of 8 and 10 carbon acids, but actually includes C<sub>6</sub> to C<sub>12</sub> acids, including trace amounts of C<sub>6</sub> acid (less than about 5 weight %).  
20 Use of only linear acids to prepare the esters adds to the biodegradability and viscosity index of the coolant/lubricant composition.

The initial stage of the reaction to form the poly (neopentyl polyol) ester is conducted in the manner described  
25 by Leibfried in U.S. Patent No. 3,670,013. Here, a reaction mixture of pentaerythriol (272 w) and valeric acid (217 v) was placed into a reactor with extra valeric acid (38 v) in a condenser to assure a constant level of valeric acid in the reaction mixture. The mixture was heated to a temperature of  
30 171°C and concentrated sulfuric acid (1.0 w) diluted with water (2 v) was added. The reaction mixture was heated to 192°C and maintained until 50.5 v of water was removed after about 1.4 hours. His analysis of the product was pentaerythritol, dipentherythritol, tripentaerythritol and tetrapentaerythritol  
35 at weight ratios of 34:38:19:8.

The neopentyl polyol and selected acid or acid mixture are

mixed in the presence of a strong acid catalyst and heated. The reaction is continued until the desired viscosity of the reaction mixture is reached. At this point when the starting neopentyl polyol is pentaerythritol, the mixture includes  
5 partial esters of pentaerythritol, dipentaerythritol, tripentaerythritol, tetrapentaerythritol and the like. In order to complete the esterification of the partial esters, an excess of the acid is added to the reaction mixture which is then heated, water of reaction removed and acid returned to the  
10 reactor.

In the preferred embodiment the invention, the poly(neopentyl polyol) partial ester is poly(pentaerythritol) formed from pentaerythritol. The poly(pentaerythritol) is prepared by introducing a reaction mixture of pentaerythritol  
15 and a linear monocarboxylic acid having from 7 to 12 carbon atoms in an initial mole ratio of carboxyl groups to hydroxyl groups of 0.25:1 to 0.5:1 and an effective amount of an acid catalyst material into a reaction zone as described in Liebfried.

20 The acid catalyst is at least one acid esterification catalyst. Examples of acid esterification catalysts include mineral acids, preferably, sulfuric acid, hydrochloric acid, and the like, acid salts such as, for example, sodium bisulfate, sodium bisulfite, and the like, sulfonic acids such as, for  
25 example, benzene sulfonic acid, toluene sulfonic acid, polystyrene sulfonic acid, methyl sulfonic acid, ethyl sulfonic acid, and the like. The reaction mixture is heated to between about 150° and 200°C while withdrawing acid vapor and water vapor to yield the poly(pentaerythritol) partial ester product.

30 Prior to esterifying, the partial ester product will include a variety of condensation products of pentaerythritol. The mixture will include significantly more dipentaerythritol than the 10 to 15 weight percent generally present in commercially available dipentaerythritol. Depending on the  
35 initial ratio of carboxyl groups to hydroxyl groups and selection of reaction conditions, the partial ester product may include the following components in the weight ranges specified in the following table.

	<u>Pentaerythritol Component</u>	<u>Weight Percent</u>
	Pentaerythritol	30 to 45
	Dipentaerythritol	30 to 45
	Tri/tetrapentaerythritol	20 to 35
5	Others	3 to 15

The amount of the preferred heptanoic and caprylic-capric acid mixture for preparing the poly(neopentyl polyol) ester may vary widely. Initially, an excess of hydroxyl groups to carboxylic acid groups is present to form the partial esters of pentaerythritol, dipentaerythritol, tripentaerythritol, tetrapentaerythritol, etc. The excess of hydroxyl groups is necessary to promote the condensation of the polyol into partial esters during the reaction. The molar ratio of acid mixture to the polyol can be varied depending on the desired degree of condensation and the ultimate desired viscosity of the lubricant. After formation of the partial esters, generally, a 10 to 25 percent excess of the mixture of heptanoic acid and C<sub>8</sub>-C<sub>10</sub> acid is added to the reactor vessel and heated. Water of reaction is collected during the reaction while the acids are returned to the reactor. Presence of a vacuum will facilitate the reaction. When the hydroxyl value is reduced to a sufficiently low level, the bulk of the excess acid is removed by vacuum distillation. Any residual acidity is neutralized with an alkali. The resulting poly(neopentyl polyol) ester is dried and filtered.

The polyol ester preferably is an ester of a polyol having between 5 and 8 carbon atoms and at least two hydroxyl groups and a linear monocarboxylic acid having from 7 to 12 carbon atoms. Specific examples of polyol useful in the present invention include neopentyl glycol, pentaerythritol, dipentaerythritol, tripentaerythritol, trimethylolpropane, trimethylolethane etc. Mixtures of any of the above polyol can be utilized.

The acid component of the polyol ester can be the same or vary from that utilized to prepare the poly(neopentyl polyol)

ester. Accordingly, monocarboxylic acids having between 7 to 12 carbon atoms, such as heptanoic (C<sub>7</sub>) and caprylic-capric (C<sub>8</sub>-C<sub>10</sub>) with minimal C<sub>6</sub> and lower acids are preferred. In the most preferred embodiments, a linear acid, namely caprylic-capric acid is utilized to form the polyol ester. The amount of acid present in the reaction mixture can vary widely. Since the desire is to completely esterify the polyol, an excess generally in the amount of between 10 to 25 percent excess acid relative to the stoichiometric amount is added.

10 The polyol ester is formed by reacting the polyol with an excess of the carboxylic acid, removing the water of reaction and returning the unreacted acid to the heated reactor vessel. The reactor is equipped with a mechanical stirrer, thermocouple, thermoregulator, Dean Stark trap, condenser, nitrogen sparger and vacuum source. The esterification may or may not be carried out in the presence of an esterification catalyst, which are well known in the art, such as stannous oxalate. The polyol is preferably trimethylolpropane which is esterified with a C<sub>8</sub>-C<sub>10</sub> acid to form the desired triester. The trimethylolpropane-C<sub>8</sub>-C<sub>10</sub> triester is blended with the poly(pentaerythritol) esters to form the lubricant so that the triester is present between 20 to 50 weight percent of the lubricant, preferably between 25 to 40 weight percent. In the preferred embodiment 30 to 35 weight percent is used.

25 The polyol, and the monocarboxylic acid in excess of 10 to 15 percent, are charged to the reactor vessel. The vessel is heated and water of reaction is collected in the trap during the reaction. The acids are returned to the reactor. Vacuum is applied to maintain the reaction. When the hydroxyl value is reduced to a sufficiently low level, the bulk of the excess acid is removed by vacuum distillation. The residual acidity is neutralized with an alkali. Finally, the resulting polyol ester product is dried and filtered.

In order to improve the properties of the (polyneopentyl

AMENDED SHEET  
PREPARED

polyol) based ester compositions prepared in accordance with the invention as coolant/lubricants, effective amounts of various additives are added. For example, the oxidative stability of the ester based coolant/lubricant can be improved by adding an effective amount of at least one antioxidant. Examples of suitable antioxidants which can be used are secondary arylamines, and phenyl naphthylamines, i.e. both alpha and beta-naphthyl amines; diphenyl amine; iminodibenzyl; p, p'-dioctyl-diphenylamine; and related aromatic amines. Other suitable antioxidants are hindered phenolics, such as 2-t-butylphenol, 2,6-di-t-butylphenol and 4-methyl-2,6-di-t-butylphenol and the like.

Generally, between 0.5 to 10 weight percent antioxidant is included in the ester composition. Preferably, between 1 to 5 weight percent is included. The preferred antioxidant is a mixture of secondary arylamines, such as dioctyldiphenyl amine and phenylalphanaphthyl amine. When these two latter amines are utilized in combination, between 0.01 to 5 weight percent of each is included, and preferably between 0.1 to 0.5 weight percent of each is included.

A rust inhibitor is included to inhibit ferrous metal corrosion which is a serious problem due to the condensation of water from the compressed gases. Rust inhibitors have traditionally been compounds containing heavy metals which are desirable to avoid. However, a combination of amino acid derivatives with substituted primary and secondary amines have been found to provide a synergistic effect -- not only providing improved ferrous metal rust prevention and improved hydrolytic stability, but doing this without adversely affecting demulsibility. The amino acid derivative found suitable is available as KCORR-100E (50% active) from King

WO99/10455

13-A

PCT/US98/17536

Industries which is an N-acyl-N-alkoxyalkyl substituted amino acid ester and a mixture of substituted primary and secondary amines and diamines known as Duomeen TDO and Ethomeen T-12 from Akzo Nobel. The primary amine and/or diamine may be a substituted propyl diamine. The secondary amine may be a substituted ethoxylated amine. The primary or secondary amine may be substituted with tallow. Ethomeen™ TDO comprises N-tallow alkyl-2,2'-imino bisethanol. Duomeen™ TDO comprises N-tallow alkyl trimethylene diamine dioleates. These rust inhibitors are utilized in combination to maintain

good demulsibility, and provide excellent rust prevention and improved hydrolytic stability.

The amino acid derivative and substituted amines and diamines present as rust inhibitors are included in an amount  
5 between 0.05 to 10 weight percent of the lubricant. Preferably, between 0.1 to 6 weight percent is included. In the preferred embodiments, a total of 0.50 weight percent of the rust inhibitor, such as KCORR-100E, Ethomeen T-12 and Duomeen TDO, are included.

10 Cuprous metal deactivators, known as yellow metal pacifiers, may also be included. Examples are imidazole, benzimidazole, pyrazole, benzotriazole, tolyltriazole, 2-methyl benzimidazole, 3,5-dimethylpyrazole and methylene bis-benzotriazole. Preferably, an aryltriazole, such as  
15 tolyltriazole is utilized. Such a yellow metal pacifier is included in an effective amount, generally 0.001 to 0.5 weight percent of the lubricant. Preferably, between 0.01 to 0.2 weight percent is included. In the preferred embodiment, about 0.05 weight percent tolyltriazole is utilized as a yellow metal  
20 pacifier.

In addition to these antioxidants, yellow metal pacifiers and rust inhibitors, it may be desirable to include an effective amount of a defoamer which will prevent undesirable foaming of the lubricant as it is worked between the screws of the  
25 compressor. A silicone fluid present in a minor amount is effective. Generally, 0.001 - 10 parts per million of defoamer is sufficient. In the preferred embodiment, 1 ppm is utilized.

The lubricant is formed by placing the desired amounts of the poly(neopentyl polyol) ester and the polyol ester blend in a  
30 vessel equipped with a mechanical agitator, thermocouple, thermoregulator and nitrogen sparge. The mixture is heated to approximately 100°C (212°F). At this time, the antioxidants and yellow metal pacifier additives are added and agitated until dissolved. The mixture is cooled to less than 50°C (122°F) and  
35 the amino acid derivative, and mixture of substituted primary

and secondary amines are added. The mixture is agitated, filtered and then defoamer added.

The ester coolant/lubricants prepared in accordance with the invention are specifically designed to be utilized in a rotary screw air compressor. Accordingly, it is designed to have a viscosity in the range of 5 to 15 centistokes at 100°C (212°F) and preferably 7 to 10 centistokes at 100°C (212°F) and a pour point in the range of -29° to -54°C (-20° to -65°F).

The invention will be better understood with reference to the following examples. All percentages are set forth in percentages by weight, except where molar quantities are indicated. These examples are presented for purposes of illustration only, and not intended to be construed in a limiting sense.

15        Example 1

A poly(neopentyl) polyol ester based coolant/lubricant prepared in accordance with the invention was made as follows. Into a vessel equipped with a mechanical agitator, thermocouple, thermoregulator and nitrogen sparge was charged the poly(pentaerythritol) ester and trimethylolpropane ester in the quantity set forth in Table II. The ester mixture was heated to approximately 100°C. When the mixture reached 100°C, the secondary arylamines and a triazole derivative were added and agitated until dissolved. The mixture was then cooled to less than 50C and the amino acid derivative, and substituted primary and secondary amines were added to the mixture. The mixture was agitated thoroughly, filtered, and then defoamer added.

TABLE II

<u>COMPONENT</u>	<u>WEIGHT %</u>
Poly-pentaerythritol ester	66
Trimethylolpropane ester	32
Dioctyldiphenylamine (secondary arylamine)	0.9
Phenylalphanaphthylamine (secondary arylamine)	0.9
Tolyltriazole (triazole)	0.05
K-Corr 100E (amino acid derivative)	0.25
Ethomeen T-12 (substituted primary and secondary amines)	0.17
Duomeen TDO (substituted primary and secondary amines)	0.08
Silicone fluid (defoamer)	1ppm

Example 2

The poly(neopentyl) polyol ester based coolant/lubricant composition prepared in accordance with the invention as described in Example 1 was analyzed to determine its physical properties. Utilizing the same ASTM tests, a sample of a commercially available rotary screw compressor lubricant denominated SSR Ultra (the current industry standard) and believed prepared in accordance with the Carswell U.S. Patent No. 4,302,343 was also tested. The results of the comparison of physical and chemical properties and performance characteristics is as set forth in Table III.

**TABLE III**Physical and Performance Properties

<u>PROPERTY</u>	<u>EXAMPLE 1 PREPARATION</u>	<u>SSR ULTRA (Current Standard)</u>	<u>TEST METHOD</u>
Viscosity @ 100°C (212°F) (cSt) @ 40°C (104°F)	8.48 48.4	8.82 48.5	ASTM D 445 ASTM D 445
ISO Viscosity Grade	46	46	ASTM D 2422
Viscosity Index	153	163	ASTM D 2270
Pour Point, °C (°F)	-51 (-60)	-50 (-58)	ASTM D 97
Flash Point, °C (°F)	274 (525)	282 (540)	ASTM D 92
Fire Point, °C (°F)	307 (585)	299 (570)	ASTM D 92
Heavy Metal Content, %	None	1,200 ppm Barium	Atomic Emission
Fatty Acids < C <sub>7</sub> , Wt. %	None*	12	GC
Demulsibility @130°F, minutes	25	No separation (3 days)	ASTM D 1401
Biodegradability, %	85.4	66.8	CEC L-33-A-93
Ferrous Metal Corrosion Sea Water @ 24 hrs. Sea Water @ 48 hrs.	Pass (no rust) Pass (no rust)	Fail Fail	ASTM D 665 B
Rotary Bomb Oxidation Hours to 25 psi drop	15.0	10.1	ASTM D 2272
Modified Rotary Bomb Oxidation Pressure Drop @ 15 hrs, 10% H <sub>2</sub> O, psi Pressure Drop @ 15 hrs, 0% H <sub>2</sub> O, psi	20 3	44 20	
Hydrolytic Stability 48 hrs. @ 93.3°C (200°F) % Visc. change @ 40°C Oil acid change, mgKOH/g Water acid change, mgKOH/g	-0.2 +0.32 +0.11	+1.4 +0.04 +0.72	ASTM D 2619
Evaporation Rate, % loss 6.5 hrs @ 400°F	1.34	5.58	ASTM D 972
Volatility @ 400°F, % loss @ 6 hrs. @ 21 hrs. @ 55 hrs. @ 83 hrs.	0.1 0.3 0.9 1.2	0.4 0.9 3.0 3.9	Hatco Method

\* Includes trace amounts of C<sub>6</sub> from commercially available

5 C<sub>8</sub> - C<sub>10</sub> acid.

A comparison of the chemical and physical properties shows that the coolant/lubricant composition prepared in accordance with the invention as set forth in Example 1 meets and/or exceeds each of the chemical and physical properties relevant for use as a compressor coolant/lubricant. A review of the performance tests in Example 2, shows that the coolant/lubricant of Example 1 significantly exceeds each of the performance characteristics considered important for a lubricant to be used in a rotary screw air compressor.

10        Example 3

A sufficient quantity of coolant/lubricant prepared in accordance with Example I was placed in three different rotary screw air compressors of 5, 7.5 and 30 horsepower. The condition of the fluid was inspected at 500 hour intervals. After running the 30hp compressor for 3000 hours, the coolant/lubricant was inspected and tested and found to exceed the minimum specifications as called for in Table I.

These remarkable results obtained in accordance with the invention are due to the synergistic effect of the ester blend and additives, namely major amount of the poly(neopentyl) polyol ester combined with the polyol ester and the amino acid derivative and mixture of substituted amines rust inhibitors. The improvement in rust prevention and hydrolytic stability is achieved without any negative impact on biodegradability, demulsibility or oxidative stability. These improvements are attained without the use of any heavy metal containing compounds which greatly facilitates disposal of spent lubricant.

**WE CLAIM:**

1. **A synthetic coolant/lubricant composition, comprising an ester mixture of:  
50 to 80 weight percent of poly(neopentyl polyol) ester formed by reacting a poly(neopentyl polyol) partial ester and a first at least one linear monocarboxylic acid having from 6 to 12 carbon atoms;  
20 to 50 weight percent of a polyol ester formed by reacting a polyol having 5 to 8 carbon atoms and at least two hydroxyl groups with a second at least one linear monocarboxylic acid having from 6 to 12 carbon atoms,  
the weight percents based on the total weight of the composition, wherein the first and second linear monocarboxylic acids can be the same or different; and  
an effective amount of an additive package,  
wherein the composition has a viscosity in the range of 5 to 15 mm<sup>2</sup>/s at 100°C.**
2. **The composition of claim 1, wherein the first at least one linear monocarboxylic acid has from 7 to 12 carbon atoms.**
3. **The composition of claim 1 or 2, wherein the ester mixture includes between 55 to 75 weight percent poly(neopentyl polyol) ester and 25 to 40 weight percent polyol ester.**
4. **The composition of claim 1, wherein the poly(neopentyl polyol) is poly(pentaerythritol).**
5. **The composition of claim 1, wherein the first and second at least one linear monocarboxylic acids are selected from the group consisting of C<sub>7</sub> and C<sub>8</sub>-C<sub>10</sub> acids and mixtures thereof.**
6. **The composition of claim 5, wherein the first and second at least one linear monocarboxylic acid includes less than 5 weight percent branched acid.**

7. The composition of claim 1, wherein the poly (neopentyl polyol) ester includes between 65 to 85 mole percent C<sub>7</sub> acid and the balance C<sub>8</sub>-C<sub>10</sub> acid.
8. The composition of claim 1, wherein the polyol ester comprises a polyol having 5 to 8 carbon atoms and three hydroxyl groups.
9. The composition of claim 1, wherein the polyol ester is a triester.
10. The composition of claim 9, wherein the polyol is trimethylolpropane.
11. The composition of claim 1, wherein the second at least one linear monocarboxylic acid used to form the polyol ester is a C<sub>8</sub>-C<sub>10</sub> linear acid.
12. The composition of claim 11, wherein the C<sub>8</sub>-C<sub>10</sub> linear acid includes less than 5 weight percent branched acids.
13. The lubricant composition of claim 1, wherein the polyol ester is the reaction product formed by reacting trimethylolpropane and a C<sub>8</sub>-C<sub>10</sub> linear acid.
14. The composition of claim 1, wherein the poly (neopentyl polyol) ester is the reaction product formed by reacting a poly(pentaerythritol)partial ester and a mixture of C<sub>7</sub> and C<sub>8</sub>-C<sub>10</sub> linear acids.
15. The composition of claim 1, further including effective amounts of at least one of each of an antioxidant, a rust inhibitor and a yellow metal pacifier.
16. The composition of claim 15, wherein the antioxidant is a secondary arylamine, the yellow metal pacifier is a triazole derivative and the rust inhibitor is a mixture of an amino acid derivative and substituted primary and secondary amines and/or diamines.

17. The composition of claim 15, wherein the rust inhibitor is a combination of amino acid derivative and substituted primary and secondary amine and/or diamines.
18. The lubricant composition of claim 16, wherein the primary amine and/or diamine is a substituted propyl diamine.
19. The lubricant composition of claim 16, wherein the secondary amine is a substituted ethoxylated amine.
20. The lubricant composition of claim 16, wherein the primary amine is substituted with tallow.
21. The lubricant composition of claim 16, wherein the secondary amine is substituted with tallow.
22. The lubricant composition of claim 1 admixed with an effective amount of a rust inhibitor including a mixture of an amino acid derivative and substituted primary and secondary amines.
23. The lubricant composition of claim 1, comprising an additive package for improving corrosion resistance of the lubricant, comprising, an effective amount of an N-acyl-N-alkoxyalkyl aspartate ester and at least one substituted primary and secondary amine and/or diamines.
24. A method of cooling and lubricating a compressor which comprises contacting moving components of the compressor to be cooled and lubricated with an effective amount of the synthetic lubricant of any one of claims 1 to 23.
25. The method of cooling and lubricating a compressor of claim 24, wherein the compressor is a rotary screw compressor.