

[72] Inventors **Arthur W. Sawyer**
Hamden;
David A. Csejka, Orange, both of Conn.
 [21] Appl. No. **717,996**
 [22] Filed **Apr. 1, 1968**
 [45] Patented **Dec. 7, 1971**
 [73] Assignee **Olin Mathieson Chemical Corporation**
Continuation-in-part of application Ser. No.
653,338, July 14, 1967. This application
Apr. 1, 1968, Ser. No. 717,996

[54] **WATER-INSENSITIVE HYDRAULIC FLUIDS**
CONTAINING BORATE ESTERS
24 Claims, No Drawings

[52] U.S. Cl. **252/75,**
252/49.6, 252/74, 252/77, 252/78
 [51] Int. Cl. **C09k 3/00,**
C10m 3/48
 [50] Field of Search..... **252/74, 75,**
77, 78, 49.6; 260/462

[56]

References Cited

UNITED STATES PATENTS

3,080,412	3/1963	Young.....	260/462
3,316,287	4/1967	Nunn et al.	260/462
3,377,288	4/1968	Sawyer.....	252/75
3,414,519	12/1968	Beynon	252/75

Primary Examiner—Leon D. Rosdol

Assistant Examiner—D. Silverstein

Attorneys—Walter D. Hunter, Donald F. Clements, Thomas P.
 O'Day, Gordon D. Byrkit, Ellen P. Trevors, Richard S.
 Strickler and George J. Koeser

ABSTRACT: A water-insensitive hydraulic fluid composition comprising from about 54.5 to about 92 percent by weight of at least one borate ester, from 0 to about 20 percent by weight of a polyoxyalkylene glycol and from about 3 to about 43 percent by weight of a glycol monoether or diether diluent together with minor amounts of an alkaline buffer and an antioxidant, if desired. Such water-insensitive hydraulic fluids are high boiling compositions suitable for use as brake fluids.

WATER-INSENSITIVE HYDRAULIC FLUIDS CONTAINING BORATE ESTERS

This is a continuation-in-part of application, Ser. No. 653,338, filed July 14, 1967 and now abandoned.

This invention relates to new and improved, water-insensitive hydraulic pressure transmission fluids for use in fluid pressure operating devices such as hydraulic brake systems, hydraulic steering mechanisms, hydraulic transmissions, etc. More particularly, this invention relates to water-insensitive hydraulic fluids which employ as the main lubricant one or more borate esters of glycol monoethers.

One of the basic objects of this invention is to provide hydraulic pressure transmission fluids for use in hydraulic systems which are extremely high boiling compositions and which maintain high boiling points even when water is added to the initial fluid composition. Another object of this invention is to provide a hydraulic pressure transmission fluid having a boiling point of at least about 450° F. A further object of this invention is to provide a hydraulic pressure transmission fluid having a high degree of lubricity while maintaining desired viscosities within a predetermined range under wide variation of temperature conditions.

A great number of hydraulic fluid compositions have been suggested in the art. Commonly, the hydraulic pressure transmission fluids, such as brake fluids are made up of three principal units. The first is a base or lubricant for the system which may include heavy bodied fluids such as polyglycols, castor oil, mixtures of these materials, etc. Diluents, which are employed for the purpose of controlling the viscosity of the fluid as represented by glycol ethers, glycols, alcohols, etc., form the second basic unit. Finally, the third basic unit is represented by an inhibitor system comprising small quantities of inhibitors which are added to reduce oxidation, to improve wetting and flow and to maintain the pH of the hydraulic system above 7 in order to minimize corrosion. Although the hydraulic fluids of the prior art possesses one or more of the desired characteristics of viscosity temperature relationship, volatility, or pour-point, they all suffer from one or more disadvantage and their use is handicapped by the fact that a wide range of suitable properties cannot be obtained. Fluids known in the art are not water insensitive; they suffer from lack of lubricity; some are not stable against oxidation or deterioration; with others it is found that over long periods of use insoluble materials are formed which greatly reduce their efficiency; and in some instances exposure to oxidizing conditions also results in the formation of insoluble compositions. Frequently, it is found that these fluids are also corrosive and that they do not possess the required rubber swelling properties.

It has been discovered that the hydraulic fluids of this invention which contain as the main base or lubricant at least one borate ester of a glycol monoether are of low cost; they possess a high boiling point and are essentially odorless and colorless; they possess a high degree of compatibility with other fluids; they exhibit a very low rate of corrosivity; and, especially desirable, they have a high degree of water tolerance.

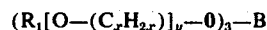
Another feature of this invention is the highly satisfactory rubber compatibility of the novel fluids as shown in tests carried out according to SAE Standard J70c. Fluids previously employed have utilized expensive materials such as 2-ethylhexanol, heptanols, butyl ethers of glycols, or diethers of glycols in an amount from about 10 to about 30 percent by weight of the total composition to achieve the desired rubber swelling characteristics. The rubber swelling properties of the fluids of this invention are derived from the lubricant or base portion. The importance of the rubber swelling properties of the fluid cannot be overlooked since too little swelling will result in leakage of the fluid past the rubber cup sealing means and past the piston in hydraulic cylinders with corresponding loss of power. On the other hand fluids which cause too much rubber swelling are not desirable in that they destroy the structural properties of the rubber sealing cups in hydraulic cylinders, in turn, results in malfunction or inoperativeness of the unit.

Lubricant or Base Compositions

The lubricant or base compositions employed in the novel hydraulic fluids of this invention comprise at least one borate ester of a glycol monoether as the main base or lubricant and, optionally, may contain a minor amount of a polyoxyalkylene glycol having a molecular weight of not less than 150. From about 54.5 to about 92 percent by weight of the final fluid will consist of the borate ester and from 0 to about 20 percent of a polyoxyalkylene glycol will be present in the final fluid, the percent by weight being based on the total fluid weight. Preferably, from about 60 percent to about 90 percent by weight of the final fluid composition will be the borate ester compound or compounds while 0 to about 10 percent by weight of the final fluid composition will be the polyoxyalkylene glycol or mixture of polyoxyalkylene glycols. The polyethylene glycols represent a preferred group of polyoxyalkylene glycols.

A wide range of polyoxyalkylene glycols can be employed in the lubricant or base compositions of this invention. Useful polyoxyalkylene glycols include those having molecular weights ranging from not less than about 150 to about 400, and preferably from about 200 to about 350. Suitable polyoxyalkylene glycols include polyethylene, polypropylene and polybutylene glycols.

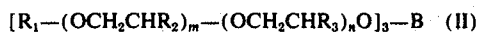
Although a wide variety of borate esters can be employed as lubricants in the novel hydraulic fluids of this invention, an especially useful class of borate esters are the so-called triborate esters of glycol monoethers having the general formula:



wherein R_1 is a lower alkyl radical containing from one to four carbon atoms, x is an integer from 2 to 4 inclusive and y is an integer from 2 to 4 inclusive. Borates of the above-mentioned type include, for example, $(CH_3(OCH_2CH_2)_2O)_3-B$, $(C_2H_5(OCH_2CH_2)_2O)_3-B$, $(C_3H_7(OCH_2CH_2)_2O)_3-B$, $(CH_3(OCH_2CH(CH_3)_2O)_3-B$, $(CH_3(OCH_2CH(CH_3)_2O)_3-B$, $(C_2H_5(OCH_2CH(CH_3)_2O)_3-B$, $(C_4H_9(OCH_2CH(CH_3)_2O)_3-B$, $(C_3H_7(OCH_2CH(CH_3)_2O)_3-B$, and $(CH_3(OCH_2CH(CH_3)_2O)_3-B$.

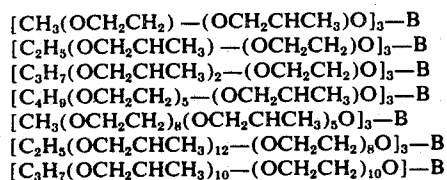
Borates of the above-mentioned type can be conveniently prepared by reacting orthoboric acid and the glycol monoether while in the presence of a water-azeotrope forming solvent. Water formed in the esterification reaction is continuously removed as the azeotrope. At first, the temperature of the reaction mixture is maintained between about 0° C. and about 190° C. and desirably at the distillation temperature of the water-solvent azeotrope. After essentially complete removal of the water formed during the esterification the excess solvent is conveniently removed from the reaction mixture by distillation. The borate ester product, which is left in a residue, may then be recovered by distilling under reduced pressure or by extraction with a suitable solvent followed by evaporation of the solvent. For example, the compound $(C_2H_5(OCH_2CH_2)_2O)_3-B$ can be prepared by reacting 2 moles of $C_2H_5(OCH_2CH_2)_2OH$, 0.67 mole of orthoboric acid and 700 ml. of ethylbenzene with heating and mixing to yield 198 grams of the ester, a water-white liquid boiling at 222°-223° C. (5 mm. Hg). The preparation of the esters of this type is more completely described in U.S. Pat. No. 3,080,412.

A second highly useful class of borate esters includes compounds of the general formula:

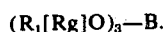


wherein R_2 and R_3 are independently selected from the group consisting of hydrogen and methyl, m and n are positive integers whose sum is from 2 to 20 and R_1 is alkyl of from one to four carbon atoms and with proviso that one of R_2 and R_3 is methyl and one of R_2 and R_3 is hydrogen. Borate esters of type II can be prepared in the general way as those esters previously described (type I) above, utilizing the so-called block type glycol monoethers. The preparation of esters of type II is described in detail in U.S. Pat. No. 3,316,287.

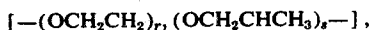
Type II borate esters useful in preparing the novel fluids of this invention include, for example:



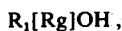
Another class of borate esters useful in the fluid compositions of this invention include esters having heteric oxyalkylene chains, that is, oxyalkylene chains in which oxyethylene and oxypropylene units are distributed randomly throughout the chain. These type III esters have the general formula:



Rg represents a heteric oxyalkylene chain having the formula:

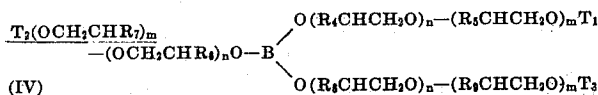


where the sum of r and s is not more than 20 and wherein the weight percent of oxyethylene units in the said chain is not less than 20 based on the total weight of all the oxyalkylene units in the chain and R_1 is alkyl of from one to four carbon atoms. The preparation of type III esters can be accomplished in the same general manner as the preparation of types I and II described above by reacting orthoboric acid in the presence of toluene with a heteric glycol monoether of the formula:



Where R_1 and Rg have the same meaning as previously set forth. Glycol monoethers of this class can be conveniently prepared by methods well known in the art such as the process described in U.S. Pat. No. 2,425,845.

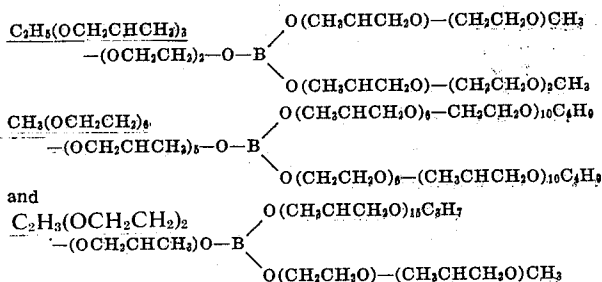
A fourth type of borate esters suitable for use in the fluid compositions of this invention have the general formula:



wherein T_1 , T_2 and T_3 are each an independently selected alkyl group having from one to four carbon atoms, R_4 , R_5 , R_6 , R_7 , R_8 , and R_9 are independently selected from the group consisting of hydrogen and methyl, n and m are positive integers independently selected in each chain and whose sum in each chain is from 2 to 20, and with the proviso that in no more than two of the chains is the sum of n and m the same.

Borate esters of this type can be prepared in the same way as the process described for type I esters previously mentioned.

Type IV borate esters suitable for use in the fluids of this invention include, for example:



Diluents

The diluent portion of the novel fluid composition of this invention can constitute from about 3 to about 43 percent by weight based on the total weight of the fluid composition of one or more diluents which are glycol monoethers, or diethers of the formula:



wherein R is alkyl from one to four carbon atoms, R' is hydrogen or alkyl of from one to four carbon atoms, x is an

integer from 2 to 4, and y is an integer from 2 to 4. Preferably, the hydraulic fluid composition will contain from about 6.5 to about 35 percent by weight, based on the total weight of the fluid composition, of the glycol monoether or diether. Useful glycol monoethers and diethers, many of which are commercially available include, for example, diethylene glycol monomethyl ether, diethylene glycol monoethyl ether, diethylene glycol monoisobutyl ether, triethylene glycol monomethyl ether, triethylene glycol monoethyl ether, triethylene glycol mono- n -butyl ether, tetraethylene glycol monomethyl ether, tetraethylene glycol monoethyl ether, dipropylene glycol monomethyl ether, dipropylene glycol monoethyl ether, tripropylene glycol monomethyl ether, tripropylene glycol monoethyl ether, tripropylene glycol mono- n -butyl ether, tetrapropylene glycol monomethyl ether, dibutylene glycol monomethyl ether, tributylene glycol mono- n -propyl ether, tetrabutylene glycol mono- n -butyl ether and the corresponding diethers thereof.

Additives

When desired, inhibitors for pH and corrosion control, such as alkaline inhibitors as exemplified by the alkali metal borates, can be employed in an amount sufficient to maintain alkaline conditions in the fluid compositions, e.g., a pH value of from about 7.0 to about 11.5. These inhibitors are generally added in an amount of from about 0.5 to about 5.0 percent by weight based on the total weight of the fluid composition and preferably from about 0.8 to about 5.0 percent by weight on the same basis. Useful inhibitors include alkali metal borates, such as sodium borate, potassium tetraborate, etc.; sodium meta arsenite; alkali metal salts of fatty acids, such as potassium oleate, the potassium soap of rosin or tall oil; alkylene glycol condensates with alkali metal borates, such as the ethylene glycol condensate of potassium tetraborate; amines, for example, ethanalamine, methyl diethanolamine, diethanolamine, di(2-ethylhexyl) amine, di- N -butyl amine, monoamyl amine, diamylamine, dioctylamine, salicylal monoethanolamine, di- β -naphthyl- p -phenylene diamine, N,N' -disalicylidene-1,2-propanediamine, N,N' -disalicylal ethylene diamine, dicyclohexylamine, and amine salts such as mono- or dibutyl ammonium borate; phosphites, such as triphenyl phosphite, tri(tert amylphenyl) phosphite, diisopropyl phosphite, etc.; mercaptobenzotriazole; morpholine compounds including alkyl morpholines having from one to four carbon atoms in the alkyl group such as N -ethyl morpholine, N -isopropyl morpholine, N -butyl morpholine; N -phenyl morpholine, N -(2-aminoethyl) morpholine, N -(2-hydroxyethyl) morpholine, etc., phosphates, including the alkali metal phosphates, dibutyl amine phosphates, the dialkyl acid o -phosphates and amine salts thereof; triazoles including benzotriazole, 1,2-naphthotriazole, 4-nitrobenzotriazole, aminobenzotriazoles such as 5-acylamino benzotriazole, and alkyl triazoles having one to 10 carbon atoms in the alkyl group as exemplified by methyl triazole, ethyl triazole, n -propyl triazole, tertiary butyl triazole, hexyl triazole, isodecyl triazole, etc. Other useful corrosion inhibitors include adenine, 4-methylimidazole, 3,5-dimethyl pyrazole, 6-nitroimidazole, imidazole, benzimidazole, guanine, indazole, ammonium dinonylnaphthalene sulfonate, dioleil thiodipropionate, ethylbenzoate, ethyl- p -aminobenzoate, cyclohexyl ammonium nitrite, diisopropyl ammonium nitrite, butynediol, 1,3,5-trimethyl-2,4,6-tris (3,5-di-tert. butyl-4-hydroxybenzoyl), 4,4'-methylene bis(2,6-di tert. butylphenol), 4-hydroxymethyl-2,6-di-tert. butylphenol, 4,4'-methylene bis(4-methyl-6-tert. butylphenol), salicylal- o -aminophenol, 2,6-di-tert. butyl-2-dimethylamino- p -cresol, 4,4'-thio bis(6-tert. butyl- o -cresol). Mixtures of the above-mentioned inhibitors can be employed if desired.

Preferably, the hydraulic fluid compositions of this invention contain from about 0.001 to about 1.0 percent by weight of an antioxidant based on the total weight of the fluid composition, to protect the diluents. Typical antioxidants include phenolic compounds, such as 2,2-di-(4-hydroxyphenyl) propane, phenothiazine, phenothiazine carboxylic acid esters, N -alkyl or N -arylphenothiazines, such as N -ethyl

phenothiazine, N-phenyl phenothiazine, etc.; polymerized trimethyldihydroquinoline; amines, such as phenyl-alphanaphthylamine, phenyl-betanaphthylamine, N,N'-diocetyl diphenylamine, N,N-di- β -naphthyl-p-phenylene diamine, p-isopropoxy diphenylamine, N,N-dibutyl-p-phenylene diamine, diphenyl-p-phenylene diamine, N,N'-bis(1,4-dimethylpentyl)-p-phenylene diamine, N,N'-diisopropyl-p-phenylene diamine, p-hydroxydiphenylamine, etc.; hindered phenols such as dibutyl cresol, 2,6-dimethyl-p-cresol, butylated 2,2-di-(4-hydroxyphenyl) propane, n-butylated aminophenol, butylated hydroxyanisoles, such as 2,6-dibutyl-p-hydroxyanisole; anthraquinone, dihydroxyanthraquinone, hydroquinone, 2,5-di-tertiarybutylhydroquinone, 2-tertiary butylhydroquinone, quinoline, p-hydroxydiphenylamine, phenyl benzoate, 2,6-dimethyl p-cresol, p-hydroxyanisole, nordihydroguaiaretic acid, pyrocatechol, styrenated phenol, polyalkyl polyphenols, sodium nitrite, etc. Mixtures of the above-mentioned antioxidants can be employed, if desired. It should be emphasized that with a variety of the fluids of this invention, which are suitable for a wide range of industrial application, a separate antioxidant is not required.

Formulation of the novel fluid of this invention is accomplished by blending the components to a homogeneous stage in a mixing vessel. The preferable blending temperature is from about 50°–125° F. It is preferable to warm the solution during preparation to facilitate dissolution. The blending of the compounds is conveniently conducted at atmospheric pressure in the absence of moisture.

In general, any suitable method can be used in preparing the liquid compositions of this invention. The components can be added together or one at a time, in any desired sequence. It is preferable, however, to add the antioxidant and alkaline inhibitor as a solution in the glycol ether component. All components are mixed until a single phase composition is obtained.

The following examples which illustrate various embodiments of this invention are to be considered not limitative:

EXAMPLE I

	Percent by Weight
[CH ₂ (OCH ₂ CH ₂) ₃ O] ₃ -B	67.39
Triethylene glycol monomethyl ether	23.20
Diethanolamine	1.78
Polyethylene glycol (Mol. wt. 300)	7.62
Sodium Nitrite	0.01

EXAMPLE II

	Percent by Weight
[CH ₂ (OCH ₂ CH ₂) ₃ O] ₃ -B	89.48
Triethylene glycol monomethyl ether	6.68
Polyethylene glycol (Mol. wt. 300)	1.83
Methyldiethanolamine	1.99
Sodium Nitrite	0.02

The reflux boiling point₁ of the above fluid was measured and found to be 552° F. at atmospheric pressure. To test the water insensitivity of the fluid composition of example II a composition consisting of 100 parts by volume of the fluid plus 3.5 parts by volume of water was prepared and it was found to have a reflux boiling point at atmospheric pressure of 402° F. indicating that the fluid is exceptionally water insensitive. For example, when a conventional brake fluid which boils above 500° F. is tested in the same manner, the reflux boiling point drop is about 250° F. or more.

1 - All reflux boiling point measurements in this example and in the other examples of this specification were conducted in accordance with the procedure of SAE Standard J70c.

EXAMPLE III

	Percent by Weight
[CH ₂ (OCH ₂ CH ₂) ₃ O] ₃ -B	66.0
Triethylene glycol monomethyl ether	21.9
N-ethyl morpholine	4.4
Polyethylene glycol (Mol. wt. 300)	7.7

The reflux boiling point of this field was measured and found to be 440° F. at atmospheric pressure. On addition of 3.5 parts by volume of water to 100 parts by volume of the fluid there was obtained a fluid having a reflux boiling point at atmospheric pressure of 353° F. When tested according to the procedure of SAE Standard J70c, this fluid passed the evaporation requirement for SAE heavy duty type 70R3brake fluid.

EXAMPLE IV

	Percent by Weight
[CH ₂ (OCH ₂ CH ₂) ₃ O] ₃ -B	67.04
Triethylene glycol monomethyl ether	21.72
N-phenyl morpholine	1.89
Diethanolamine	0.85
Polyethylene glycol (Mol. wt. 300)	8.50
Properties	
Reflux boiling point at atmospheric pressure	498° F.
Reflux boiling point at atmospheric pressure of 3.5 parts by volume of water and 100 parts by volume of fluid of example IV	367° F.

EXAMPLE V

	Percent by Weight
[C ₂ H ₅ (OCH ₂ CH ₂) ₃ O] ₃ -B	44.68
[CH ₂ (OCH ₂ CH ₂) ₃ O] ₃ -B	31.63
Diethylene glycol monobutyl ether	3.81
Triethylene glycol monomethyl ether	11.05
Diethanolamine	1.83
Polyethylene glycol (Mol. wt. 300)	6.98
Sodium nitrite	0.02
Properties	
Reflux boiling point at atmospheric pressure	510° F.
Reflux boiling point at atmospheric pressure of 3.5 parts by volume of water and 100 part by volume of fluid of example V	372° F.
-40° F. = 1,620 cs.	Viscosity
212° F. = 2.74 cs.	
Water Tolerance Test	
Passed water tolerance test at -40° F. and 140° F. according to procedure of SAE J70c.	

7

EXAMPLE VI

	Percent by Weight
$[\text{C}_2\text{H}_5(\text{OCH}_2\text{CH}_2)_2\text{O}]_3\text{-B}$	82.78
Triethylene glycol monoethyl ether	10.00
Methyldiethanolamine	2.20
Polyethylene glycol (Mol. wt. 300)	5.00
Sodium nitrite	0.02
Properties	
Reflux boiling point at atmospheric pressure	554° F.
Reflux boiling point at atmospheric pressure of 3.5 parts by volume of water and 100 parts by volume of fluid of example VI	383° F.
Viscosity	
-40° F. = 1,910 cs.	
212° F. = 2.82 cs.	
Water Tolerance Test	
Passed -40° F. and 140° F. water tolerance test according to procedure of SAE J70c.	

EXAMPLE VII

	Percent by Weight
$[\text{CH}_3(\text{OCH}_2\text{CH}_2)_2\text{O}]_3\text{-B}$	70.13
Triethylene glycol monomethyl ether	20.00
Diethanolamine	1.86
Triethylene glycol (Mol. wt. 150)	8.00
Sodium nitrite	0.01
Properties	
Reflux boiling point at atmospheric pressure	500° F.
Reflux boiling point at atmospheric pressure of 3.5 parts by volume of water and 100 parts by volume of fluid of example VII	381° F.
Viscosity	
-40° F. = 2,050 cs.	
212° F. = 2.80 cs.	
Corrosion Test	
Passed corrosion test according to procedure of SAE J70c.	
Water Tolerance Test	
Passed water tolerance test at -40° F. and 140° F. according to procedure of SAE J70c.	

EXAMPLE VIII

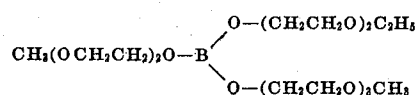
	Percent by Weight
$[\text{C}_2\text{H}_5(\text{OCH}_2\text{CH}_2)_2\text{O}]_3\text{-B}$	28.29
$[\text{CH}_3(\text{OCH}_2\text{CH}_2)_2\text{O}]_3\text{-B}$	55.58
Triethylene glycol monomethyl ether	8.05
Polyethylene glycol (Mol. wt. 300)	6.11
Methyldiethanolamine	1.94
Sodium nitrite	0.03
Properties	
Reflux boiling point at atmospheric pressure	562° F.
Reflux boiling point at atmospheric pressure of 3.5 parts by volume of water and 100 parts by volume of fluid of example VIII	400° F.
Viscosity	
-40° F. = 2,540 cs.	
212° F. = 3.27 cs.	
Water Tolerance Test	
Passed water tolerance test at -40° F. and 140° F. according to procedure of SAE J70c.	

8

EXAMPLE IX

	Percent by Weight
Borate Ester D (Described below)	80.46
Triethylene glycol monobutyl ether	11.40
Diethanolamine	2.04
Polyethylene glycol (Mol. wt. 300)	6.08
Sodium nitrite	0.02
Properties	
Reflux boiling point at atmospheric pressure	500° F.
Reflux boiling point at atmospheric pressure of 3.5 parts by volume of water and 100 parts by volume of fluid of example IX	385° F.
Viscosity	
-40° F. = 1,470 cs.	
212° F. = 2.64 cs.	
Water Tolerance Test	
Passed water tolerance test at -40° F. and 140° F. according to procedure of SAE J70c.	

Borate Ester D employed in the above fluid, which is an example of a type IV ester, was prepared in the following manner: One mole (61.8 grams) of orthoboric acid was added to a solution of one mole (164 grams) of triethylene glycol monomethyl ether and 50 ml. of toluene. The mixture was heated to the boiling point and one mole (18 ml.) of water was removed as the azeotrope with toluene. The solution was cooled slightly below the boiling point and one mole (134 grams) of diethylene glycol monoethyl ether was added. Heating was resumed until another mole (18 ml.) of water was removed from the reaction mixture. Again the solution was cooled below the boiling point and one mole (120 grams) of diethylene glycol monomethyl ether was added. The solution was heated to boiling and a third mole (18 ml.) of water was removed following which the toluene remaining in the reaction mixture was removed to yield a tri borate ester of the formula:



The boiling point of the ester was 610° F. at atmospheric pressure and it exhibited a viscosity of 775 cs. at -40° F.

Analysis	Percent B
Calculated	2.58
Found	2.83

EXAMPLE X

	Percent by Weight
Borate Ester E (Described below)	29.18
$[\text{CH}_3(\text{OCH}_2\text{CH}_2)_2\text{O}]_3\text{-B}$	55.48
Triethylene glycol monomethyl ether	11.90
Diethanolamine	0.91
Polyethylene glycol (Mol. wt. 300)	2.52
Sodium nitrite	0.01
Properties	

9

Reflux boiling point at atmospheric pressure	555° F.
Reflux boiling point at atmospheric pressure of 3.5 parts by volume of water and 100 parts by volume of fluid of example X	383° F.
Viscosity	
-40° F. = 2,380 cs.	
212° F. = 3.38 cs.	
Water Tolerance Test	
Passed water tolerance test at -40° F. and 140° F. according to procedure of SAE J 70c.	

Borate Ester E employed in the above fluid composition, which is a type III borate ester, was prepared as follows: One mole (354 grams) of a random addition product of ethylene oxide and propylene oxide with butyl alcohol, prepared by reacting a mixture containing 50 weight percent ethylene oxide and 50 weight percent propylene oxide with the alcohol, and 60 ml. of toluene were mixed with one-third mole (20 grams) of orthoboric acid. The mixture was heated to its boiling point and one mole (18 ml.) of water was removed as the azeotrope with toluene after which the toluene remaining in the reaction mixture was removed under vacuum. The resulting tris borate ester (Borate Ester E) was clear, water-white liquid having a boiling point of approximately 625°-30° F.

EXAMPLE XI

	Percent by Weight
$[\text{C}_2\text{H}_5(\text{OCH}_2\text{CH}_2)_2\text{O}]_3\text{-B}$	80.00
Triethylene glycol monoethyl ether	12.78
Methyldiethanolamine	2.20
Polyethylene glycol (Mol. wt. 300)	5.00
Diisopropyl ammonium nitrite	0.02

EXAMPLE XII

	Percent by Weight
$[\text{CH}_3(\text{OCH}_2\text{CH}_2)_2\text{O}]_3\text{-B}$	70.30
Triethylene glycol monomethyl ether	19.95
Diethanolamine	1.85
Polyethylene glycol (Mol. wt. 300)	7.85
Di(2-ethylhexyl) ammonium nitrite	0.05

EXAMPLE XIII

	Percent by Weight
$[\text{CH}_3(\text{OCH}_2\text{CH}_2)_2\text{O}]_3\text{-B}$	66.00
Triethylene glycol monomethyl ether	25.90
Diethanolamine	1.85
Polyethylene glycol (Mol. wt. 300)	6.00
Benzotriazole	0.25

EXAMPLE XIV

	Percent by Weight
$[\text{C}_2\text{H}_5(\text{OCH}_2\text{CH}_2)_2\text{O}]_3\text{-B}$	80.00
Triethylene glycol monoethyl ether	15.0
Polyethylene glycol (Mol. wt. 300)	5.0

10

EXAMPLE XV

	Percent by Weight
$[\text{CH}_3(\text{OCH}_2\text{CH}_2)_2\text{O}]_3\text{-B}$	67.40
Triethylene glycol monomethyl ether	25.81
Diethanolamine	1.78
Polyethylene glycol (Mol. wt. 300)	5.00
Sodium nitrite	0.01

EXAMPLE XVI

	Percent by Weight
$[\text{CH}_3(\text{OCH}_2\text{CH}_2)_2\text{O}]_3\text{-B}$	75.00
Triethylene glycol monomethyl ether	6.20
Tetraethylene glycol monoethyl ether	17.00
Diethanolamine	1.68
Sodium nitrite	0.02
Toltriazole	0.10

The fluid composition of Example XVI was tested according to the procedures set forth in Motor Vehicle Brake Fluid - SAE Standard J1703. Pertinent data which illustrates the outstanding properties of this fluid is shown below:

PROPERTIES OF WATER INSENSITIVE BRAKE FLUID OF EXAMPLE XVI

Test	SAE J 1703	Test Results
Boiling Point min.	374°	532° F.
Flash Point	179.6° F. min.	320° F.
Viscosity at 212° F.	1.5 cs. min.	2.5 cs.
Viscosity at -40° F.	1,800 cs. max.	1,530 cs.
pH	7.0 to 11.5	7.5
Evaporation Loss	80 percent max.	62 percent

EXAMPLE XVII

	Percent by Weight
$[\text{CH}_3(\text{OCH}_2\text{CH}_2)_2\text{O}]_3\text{-B}$	70.00
Tetraethylene glycol monoethyl ether	21.00
Triethylene glycol monomethyl ether	7.20
Diethanolamine	1.68
Toltriazole	0.10
Sodium nitrite	0.20
Reflux boiling point at atmospheric pressure	528° F.
Viscosity at -40° F.	1,447 cs.
pH 7.3	

EXAMPLE XVIII

	Percent by Weight
$[\text{CH}_3(\text{OCH}_2\text{CH}_2)_2\text{O}]_3\text{-B}$	70.00
Tetraethylene glycol monoethyl ether	25.00
Triethylene glycol monomethyl ether	3.20
Diethanolamine	1.68
Toltriazole	0.10
Sodium nitrite	0.02
Reflux boiling point at atmospheric	

pressure
Viscosity at -40° F. 1,552 cs.
pH 7.35

528° F.

EXAMPLE XIX

	Percent by Weight
$[\text{CH}_2(\text{OCH}_2\text{CH}_2)_3\text{O}]_3\text{-B}$	66.00
Tetraethylene glycol monoethyl ether	29.00
Triethylene glycol monomethyl ether	3.20
Diethanolamine	1.68
Toltriazole	0.10
Sodium nitrite	0.02
Reflux boiling point at atmospheric pressure	526° F.
Viscosity at -40° F. 1,501 cs.	
pH 7.5	

EXAMPLE XX

	Percent by Weight
$[\text{CH}_2(\text{OCH}_2\text{CH}_2)_3\text{O}]_3\text{-B}$	75.00
Tetraethylene glycol monoethyl ether	23.20
Diethanolamine	1.68
Toltriazole	0.10
Sodium nitrite	0.02
Reflux boiling point at atmospheric pressure	535° F.
Viscosity at -40° F. 1,663	

EXAMPLE XXI

	Percent by Weight
$[\text{CH}_2(\text{OCH}_2\text{CH}_2)_3\text{O}]_3\text{-B}$	75.00
Triethylene glycol monomethyl ether	11.25
Tetraethylene glycol monomethyl ether	12.00
Diethyl amino ethoxyethanol	1.75

EXAMPLE XXII

	Percent by Weight
$[\text{CH}_2(\text{OCH}_2\text{CH}_2)_3\text{O}]_3\text{-B}$	70.00
Triethylene glycol monomethyl ether	13.25
Tetraethylene glycol monomethyl ether	15.00
Methyl diethanolamine	1.75

EXAMPLE XXIII

	Percent by Weight
$[\text{CH}_2(\text{OCH}_2\text{CH}_2)_3\text{O}]_3\text{-B}$	72.00
Triethylene glycol monoethyl ether	16.25
Tetraethylene glycol monoethyl ether	10.00
Hydroxyethyl hydrazine	1.75

EXAMPLE XXIV

	Percent
--	---------

by Weight

$[\text{CH}_2(\text{OCH}_2\text{CH}_2)_3\text{O}]_3\text{-B}$	66.00
Triethylene glycol monoethyl ether	5.00
Triethylene glycol monomethyl ether	17.00
Tetraethylene glycol monomethyl ether	10.00
Diethyl amino ethanol	2.00

EXAMPLE XXV

	Percent by Weight
$[\text{CH}_2(\text{OCH}_2\text{CH}_2)_3\text{O}]_3\text{-B}$	73.00
Diethylene glycol monoethyl ether	5.00
Triethylene glycol monomethyl ether	10.00
Tetraethylene glycol monomethyl ether	10.00
Dimethyl amino ethoxyethanol	2.00

EXAMPLE XXVI

	Percent by Weight
$[\text{CH}_2(\text{OCH}_2\text{CH}_2)_3\text{O}]_3\text{-B}$	73.00
Triethylene glycol monomethyl ether	10.00
Tetraethylene glycol monomethyl ether	15.00
Diethanolamine	1.78
Sodium nitrite	0.02

EXAMPLE XXVII

	Percent by Weight
$[\text{CH}_2(\text{OCH}_2\text{CH}_2)_3\text{O}]_3\text{-B}$	7.500
Triethylene glycol monomethyl ether	6.00
Triethylene glycol monoethyl ether	7.00
Tetraethylene glycol monoethyl ether	10.00
Hydroxyethyl hydrazine	1.90
Toltriazole	0.10

EXAMPLE XXVIII

	Percent by Weight
$[\text{CH}_2(\text{OCH}_2\text{CH}_2)_3\text{O}]_3\text{-B}$	71.00
Triethylene glycol monomethyl ether	18.00
Tetraethylene glycol monomethyl ether	9.00
Diethanolamine	1.93
Sodium nitrite	0.02
Benzotriazole	0.05

EXAMPLE XXIX

	Percent by Weight
$[\text{C}_2\text{H}_4(\text{OCH}_2\text{CH}_2)_3\text{O}]_3\text{-B}$	75.00
Triethylene glycol monomethyl ether	6.00
Triethylene glycol monoethyl ether	7.00
Tetraethylene glycol monoethyl	

ether	10.00
Hydroxyethyl hydrazine	1.90
Tolutriazole	0.10

EXAMPLE XXX	
	Percent by Weight
$[\text{C}_2\text{H}_4(\text{OCH}_2\text{CH}_2)_3\text{O}]_3\text{-B}$	75.00
Triethylene glycol monomethyl ether	6.00
Triethylene glycol monoethyl ether	7.00
Tetraethylene glycol monoethyl ether	10.00
Hydroxyethyl hydrazine	1.90
Tolutriazole	0.10

EXAMPLE XXXI

	Percent by Weight
$[\text{CH}_3(\text{OCH}_2\text{CH}_2)_3\text{O}]_3\text{-B}$	65.0
Tripropylene glycol dimethyl ether	22.9
N-ethyl morpholine	4.4
Polyethylene glycol (Mol. wt. 250)	7.7

The fluid composition of example I was tested according to the procedures set forth in Hydraulic Brake Fluid - SAE Standard J70c for heavy duty types 70R1 and 70R3 hydraulic brake fluid. Pertinent data relating to these tests, which illustrates the outstanding properties of these novel fluids, is shown in table 1. The fluid of example I was found to satisfy completely the requirements for SAE heavy duty type 70R3 hydraulic brake fluid. The fluid of example II, which was tested according to the procedures of J70c for water tolerance and evaporation, was found to pass both of these tests.

TABLE 1.—PROPERTIES OF WATER INSENSITIVE BRAKE FLUID OF EXAMPLE I

[Tests conducted according to procedures set forth in Society of Automotive Engineers Standard J70c]

Test	SAE 70R3 specification	Test results water in- sensitive fluid of Example I
Boiling point (min.) °F.....	374	497
Flash point (min.) °F.....	179.6	330
Viscosity:		
—40° F. (max.) cs.....	1,800	1,790
122° F. (min.) cs.....	4.2	7.34
212° F. (min.) cs.....	1.5	2.71
pH.....	7–11.5	7.70
Stability at high temperature:		
Boiling point (min.) °F.....	374	495
Boiling point change (max.) °F.....	±9.0	—2
Corrosion test (wt. change mg./sq. cm.):		
Copper.....	0.4	—0.18
Brass.....	0.4	—0.14
Cast iron.....	0.2	+0.01
Aluminum alloy.....	0.1	0.00
Steel.....	0.2	0.00
Tinned iron.....	0.2	0.00
Sedimentation, percent by vol. (max.)..	0.10	<0.01
Jelling fluid/water mixture.....	None	None
Crystalline deposit jar walls or strips...	None	None
pH fluid/water mixture.....	7–11.5	7.50
Rubber cups:		
Swelling (max.) in.....	0.055	0.007
Softening (max.) deg.....	—15	—3

Test	SAE 70R3 specification	Test results water in- sensitive fluid of Example I
5 Fluidity and appearance: (Cold test) —40° F., six days:		
Black contrast lines, HP chart.....	(1)	(2)
Stratification-sedimentation.....	None	None
Air bubble travel time (max.) sec.....	10	2
—58° F., six hours:		
Black contrast lines, IIP chart.....	(1)	(2)
Stratification-sedimentation.....	None	None
Air bubble travel time (max.) sec.....	35	5
10 Evaporation test:		
Percent loss (max.) percent.....	80	69
Gritty or abrasive residue.....	None	None
Pour point (max.) °F.....	23	23
Water tolerance:		
15 —40° F., 24 hours:		
Black contrast lines, HP chart.....	(1)	(2)
Stratification-sedimentation.....	None	None
Air bubble travel time (max.) sec.....	10	3
140° F., 24 hours:		
Stratification.....	None	None
Sedimentation, percent by vol. (max.).....	0.05	0.01
20 Compatibility:		
—40° F., 24 hours:		
Black contrast lines, HP chart.....	(1)	(2)
Stratification-sedimentation.....	None	None
140° F., 24 hours:		
Stratification.....	None	None
Sedimentation, percent by vol. (max.).....	0.05	0.01
25 Resistance to oxidation (Peroxide test):		
Pitting of metal strips:		
Aluminum.....	None	None
Cast iron.....	None	None
Weight loss in mg./sq. cm.:		
Aluminum alloy (max.).....	0.05	0.00
Cast iron (max.).....	0.30	+0.01
30 Effect on rubber—158° F.:		
Hardness decrease, deg.:		
Natural (max.).....	10	2
SBR (max.).....	10	3
Base diameter increase, in.:		
Natural.....	0.006–0.055	0.013
SBR.....	0.006–0.055	0.019
35 Disintegration rubber cups.....	None	None
Effect on SBR rubber—248° F.:		
Hardness decrease (max.) deg.....	15	4
Base diameter increase, in.....	0.006–0.055	0.033
Disintegration rubber cups.....	None	None
Specific gravity 60/60° F.....		1.085
Pounds per gallon 60° F.....		(3)
40 Color.....		

¹ Clearly discernible.

² Clear.

³ Straw.

TABLE 1.—EFFECT OF ADDED WATER ON BOILING POINT

Water, percent by vol.	Reflux boiling point, ° F.	
	Conventional Fluid A	Water insensi- tive fluid of Example I
0.0.....	484	497
1.0.....	380	460
3.38 ¹	294	373
¹ 3.5 ml. H ₂ O/100 ml. brake fluid.		
NOTE.—The composition of conventional Fluid A was as follows:		
	Weight Percent	
A 50–50 weight percent ethylene oxide-propylene oxide random addition reaction product with butyl alcohol (ave. mol wt. 1,000).....	20.0	
Triethylene glycol monomethyl ether.....	19.0	
Triethylene glycol monoethyl ether.....	40.2	
Triethylene glycol monobutyl ether.....	19.0	
Ethylene glycol.....	1.5	
Diphenylol propane.....	0.1	
Condensate of 7 moles of ethylene glycol with 1 mole of potassium tetraborate.....	0.2	
Total.....	100.0	

TABLE 1.—PROPERTIES OF WATER INSENSITIVE BRAKE FLUID OF EXAMPLE I

Simulated service performance procedure No. 2 (85,000 strokes; 248° F/1,000 p.s.i.)

Test	SAE 70R3 specification	Test results water insensitive fluid of Example I
Corrosion of metal parts as evidenced by discernible pitting	None	None
Change in initial diameter of any cylinder or piston (max.) in	0.005	None
Lip diameter interference set rubber cups, percent:		
Wheel cylinder cups (max.)	65	7.2
Master cylinder cups:		
Primary (max.)	65	7.2
Secondary (max.)	65	0.0
Hardness decrease of rubber cups, deg.:		
Wheel cylinder cups (max.)	15	4
Master cylinder cups:		
Primary (max.)	15	6
Secondary (max.)	15	5
Operating condition of rubber cups as evidenced by excessive tackiness, scoring, scuffing, blistering, cracking, chipping, or change in shape		
Fluid loss during any 24,000 stroke period (max.) ml.	36	7
Freezing or malfunction of cylinder pistons	None	None
Gum deposited on metal parts of cylinder walls that are abrasive or cannot be removed with ethanol	2	None
Deposit formed or adhering to cylinder walls that are abrasive or cannot be removed with ethanol	None	None
Fluid loss during 100 strokes at end of test (max.) ml.	36	5
Condition of fluid and brake cylinders by evidence of sludging, gelling or grittiness likely to cause improper brake action	None	None
Percent sediment in fluid drained from wheel cylinders, 72 hours equilibrium (max.)	1.5	0.5
Percent sediment in fluid drained from cylinder, 72 hours equilibrium (max.)	1.5	0.05
Decrease in base diameter of rubber cups, in.		
Wheel cylinder cups (max.)	0.035	0.020
Master cylinder cups:		
Primary (max.)	0.035	0.025
Secondary (max.)	0.035	0.035

1 Satisfactory.

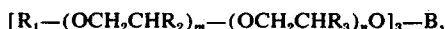
2 Trace.

What is claimed is:

1. A hydraulic fluid composition consisting essentially of (A) from about 54.5 to about 92 percent by weight, based on the total weight of the fluid composition, of at least one base or lubricant selected from the group consisting of (a) a borate ester of the formula:



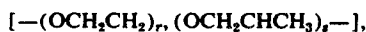
wherein R_1 is alkyl of from one to four carbon atoms, x is an integer of from 2 to 4 and y is an integer of from 2 to 4; (b) a borate ester of the formula:



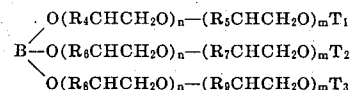
wherein R_1 is alkyl of from one to four carbon atoms, R_2 and R_3 are independently selected from the group consisting of hydrogen and methyl, m and n are positive integers whose sum is from 2 to 20, and with the proviso that one of R_2 and R_3 is methyl and one of R_2 and R_3 is hydrogen; (c) a borate ester of the formula:



wherein R_1 is alkyl of from one to four carbon atoms, Rg is a heteric oxyalkylene chain of the formula:



wherein the sum of r and s is not more than 20 and wherein the weight percent of the oxyethylene units is not less than 20 based on the total weight of all the oxyalkylene units; and (d) a borate ester of the formula:

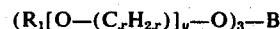


wherein T_1 , T_2 and T_3 are each an independently selected alkyl group having from one to four carbon atoms; R_4 , R_5 , R_6 , R_7 , R_8 and R_9 are independently selected from the group consisting of hydrogen and methyl, n and m are positive integers independently selected in each chain and whose sum in each chain is from 2 to 20, and with the proviso that in no more than two of the chains is the sum of n and m the same; (B) from 0 to about 20 percent by weight based on the total weight of the fluid composition of a polyoxyalkylene glycol having a molecular weight of not less than 150 and up to about 400, and (C) from about 3 to about 43 percent by weight based on the total weight of the fluid of at least one diluent having the formula:

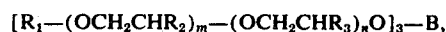


wherein R is alkyl of from one to four inclusive carbon atoms, R' is selected from the group consisting of hydrogen and alkyl of from one to four inclusive carbon atoms, x is an integer of from 2 to 4 inclusive and y is an integer of from 2 to 4 inclusive.

2. A hydraulic fluid composition consisting essentially of (A) from about 54.5 to about 92 percent by weight, based on the total weight of the fluid composition of at least one base or lubricant selected from the group consisting of (a) borate ester of the formula:



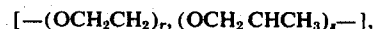
wherein R_1 is alkyl of from one to four carbon atoms, x is an integer of from 2 to 4 and y is an integer of from 2 to 4; (b) a borate ester of the formula:



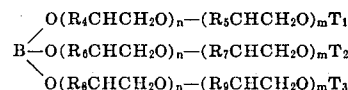
wherein R_1 is alkyl of from one to four carbon atoms, R_2 and R_3 are independently selected from the group consisting of hydrogen and methyl, m and n are positive integers whose sum is from 2 to 20, and with the proviso that one of R_2 and R_3 is methyl and one of R_2 and R_3 is hydrogen; (c) a borate ester of the formula:



wherein R_1 is alkyl of from one to four carbon atoms, Rg is a heteric oxyalkylene chain of the formula:



wherein the sum of r and s is not more than 20 and wherein the weight percent of the oxyethylene units is not less than 20 based on the total weight of all the oxyalkylene units; and (d) a borate ester of the formula:



wherein T_1 , T_2 and T_3 are each an independently selected alkyl group having from one to four carbon atoms; R_4 , R_5 , R_6 , R_7 , R_8 and R_9 are independently selected from the group consisting of hydrogen and methyl, n and m are positive integers independently selected in each chain and whose sum in each chain is from 2 to 20 and with the proviso that in no more than two of the chains is the sum of n and m the same; (B) from 0 to about 20 percent by weight, based on the total weight of the fluid composition, of a polyoxyalkylene glycol having a molecular weight of not less than 150 and up to about 400, (C) from about 3 to about 43 percent by weight, based on the total weight of the fluid, of at least one diluent having the formula:



wherein R is alkyl of one to four inclusive carbon atoms, R' is selected from the group consisting of hydrogen and alkyl of from one to four inclusive carbon atoms, x is an integer of from 2 to 4 inclusive, and y is an integer of from 2 to 4 inclusive and (D) from about 0.5 to about 6.0 percent by weight, based on the total weight of the fluid of an inhibitor for pH and corrosion control.

3. The hydraulic fluid composition of claim 2 wherein the said base or lubricant is:



4. The hydraulic fluid composition of claim 2 wherein the said polyoxyalkylene compound is polyethylene glycol having a molecular weight of 300.

5. The hydraulic fluid composition of claim 2 wherein the said diluent is a mixture of triethylene glycol monomethyl ether and tetraethylene glycol monoethyl ether.

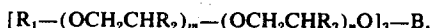
6. The hydraulic fluid of claim 2 wherein the said inhibitor is N-ethyl morpholine.

7. The hydraulic fluid composition of claim 2 wherein the said inhibitor is a mixture of diethanolamine and tolutriazole.

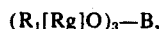
8. A hydraulic fluid composition consisting essentially of (A) from about 54.5 to about 92 percent by weight, based on the total weight of the fluid composition of at least one base or lubricant selected from the group consisting of (a) borate ester of the formula:



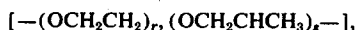
wherein R₁ is alkyl of from one to four carbon atoms, x is an integer of from 2 to 4 and y is an integer of from 2 to 4; (b) a borate ester of the formula:



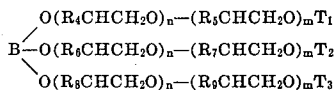
wherein R₁ is alkyl of from one to four carbon atoms, R₂ and R₃ are independently selected from the group consisting of hydrogen and methyl, m and n are positive integers whose sum is from 2 to 20, and with the proviso that one of R₂ and R₃ is methyl and one of R₂ and R₃ is hydrogen; (c) a borate ester of the formula:



wherein R₁ is alkyl of from one to four carbon atoms, R_g is a heteric oxyalkylene chain of the formula:



wherein the sum of r and s is not more than 20 and wherein the weight percent of the oxyethylene units is not less than 20 based on the total weight of all the oxyalkylene units; and (d) a borate ester of the formula:



wherein T₁, T₂, and T₃ are each an independently selected alkyl group having from one to four carbon atoms; R₄, R₅, R₆, R₇, R₈ and R₉ are independently selected from the group consisting of hydrogen and methyl, n and m are positive integers independently selected in each chain and whose sum in each chain is from 2 to 20, and with the proviso that in no more than two of the chains is the sum of n and m the same; (B) from 0 to about 20 percent by weight based on the total weight of the fluid composition of an polyoxyalkylene glycol having a molecular weight of not less than 150 and up to about 400, (C) from about 3 to about 43 percent by weight based on the total weight of the fluid of at least one diluent having the formula:



wherein R is alkyl of one to four inclusive carbon atoms, R' is selected from the group consisting of hydrogen and alkyl of from one to four inclusive carbon atoms, x is an integer of from 2 to 4 inclusive and y is an integer of from 2 to 4 inclusive, (D) from about 0.05 to about 6.0 percent by weight, based on the weight of the total fluid, of an inhibitor for pH and corrosion control and (E) from about 0.001 to about 1.0 percent by weight of an antioxidant.

9. The hydraulic fluid composition of claim 8 wherein the said base or lubricant is:



10. The hydraulic fluid composition of claim 8 wherein the said polyoxyalkylene glycol is polyethylene glycol having a molecular weight of 300.

11. The hydraulic fluid composition of claim 8 wherein the said diluent is a mixture of triethylene glycol monomethyl ether and tetraethylene glycol monoethyl ether.

12. The hydraulic fluid composition of claim 8 wherein the said antioxidant is sodium nitrite.

13. The hydraulic fluid composition of claim 8 wherein the said inhibitor is a mixture of diethanolamine and tolutriazole.

14. The hydraulic fluid composition of claim 8 consisting essentially of the following ingredients in the approximate percentage by weight indicated:

	Percent by Weight
$[CH_3(OCH_2CH_2)_3O]_3-B$	67.39
Triethylene glycol monomethyl ether	23.20
Diethanolamine	1.78
Polyethylene glycol (Mol. wt. 300)	7.62
Sodium nitrite	0.01

15. The hydraulic fluid composition of claim 8 consisting essentially of the following ingredients in the approximate percentage by weight indicated:

	Percent by Weight
$[CH_3(OCH_2CH_2)_3O]_3-B$	75.00
Triethylene glycol monomethyl ether	6.20
Tetraethylene glycol monoethyl ether	17.00
Diethanolamine	1.68
Tolutriazole	0.10
Sodium nitrite	0.02

16. The hydraulic fluid composition of claim 3 consisting essentially of the following ingredients in the approximate percentage by weight indicated:

	Percent by Weight
$[CH_3(OCH_2CH_2)_3O]_3-B$	65.0
Tripropylene glycol dimethyl ether	22.9
N-ethyl morpholine	4.4
Polyethylene glycol (Mol. wt. 250)	7.7

17. The hydraulic fluid composition of claim 3 consisting essentially of the following ingredients in the approximate percentage by weight indicated:

	Percent by Weight
$[CH_3(OCH_2CH_2)_3O]_3-B$	67.04
Triethylene glycol monomethyl ether	21.72
N-phenyl morpholine	1.89
Diethanolamine	0.85
Polyethylene glycol	

8.50

5

Triethylene glycol monobutyl ether	11.40
Diethanolamine	2.04
Polyethylene glycol (Mol. wt. 300)	6.08
Sodium nitrite	0.02

10

23. The hydraulic fluid composition of claim 8 consisting essentially of the following ingredients in the approximate percentage by weight indicated:

15

	Percent by Weight
Borate Ester [E] of the formula (c) prepared as described below:	
(CH ₃ (OCH ₂ CH ₂) ₃ O) ₃ B	29.18
Triethylene glycol monomethyl ether	55.48
Diethanolamine	11.9
Polyethylene glycol (Mol. wt. 300)	0.91
Sodium nitrite	2.52
	0.01

20

The borate ester of the formula (c) noted above is prepared by adding a random addition product of ethylene oxide, propylene oxide, and butyl alcohol to a mixture of toluene and ortho boric acid and heating the combined mixture to its boiling point.

25

24. In the operation of a fluid pressure operating device which uses hydraulic pressure transmission fluid, the improvement comprising using as said hydraulic pressure transmission fluid a composition consisting essentially of from about 54.5 to about 92 percent by weight, based on the total weight of the fluid composition, of at least one borate ester selected from the group consisting of (a) a borate ester of the formula:

25

$$(R_1-(OCH_2CHR_2)_m(OCH_2CHR_3)_nO)_3-B,$$

10

wherein R₁ is alkyl of from one to four carbon atoms, R₂ and R₃ are independently selected from the group consisting of hydrogen and methyl, *m* and *n* are positive integers whose sum is from 2 to 20, and with the proviso that one of R₂ and R₃ is methyl and one of R₂ and R₃ is hydrogen; (c) a borate ester of the formula:

$$(R_1(R_g)O)_3-B,$$

wherein R_1 is alkyl of from one to four carbon atoms, R_g is a heteric oxyalkylene chain of the formula:

50

$$(-(\text{OCH}_2\text{CH}_2)_r, (\text{OCH}_2\text{CHCH}_3)_s-),$$

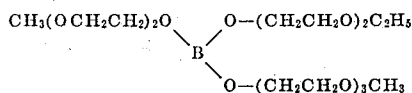
wherein the sum of r and s is not more than 20 and wherein the weight percent of the oxyethylene units is not less than 20 based on the total weight of all the oxyalkylene units; and (d) a borate ester of the formula:

$$\begin{array}{c} \text{O(R}_4\text{CHCH}_2\text{O)}_n\text{---(R}_5\text{CHCH}_2\text{O)}_m\text{T}_1 \\ \diagup \\ \text{B---O(R}_6\text{CHCH}_2\text{O)}_n\text{---(R}_7\text{CHCH}_2\text{O)}_m\text{T}_2 \\ \diagdown \\ \text{O(R}_8\text{CHCH}_2\text{O)}_n\text{---(R}_9\text{CHCH}_2\text{O)}_m\text{T}_3 \end{array}$$

50

wherein T_1 , T_2 and T_3 are each an independently selected alkyl group having from one to four carbon atoms; R_4 , R_5 , R_6 , R_7 , R_8 and R_9 are independently selected from the group consisting of hydrogen and methyl, n and m are positive integers independently selected in each chain and whose sum in each chain is from 2 to 20, and with the proviso that in no more than two of the chains is the sum of n and m the same.

9



UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,625,899 Dated December 7, 1971

Inventor(s) Arthur W. Sawyer and David A. Csejka

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

Col.2, line 36, " $(CH_3(OCH_2HCH_3)_2O)_3-B$ " should read
-- $(CH_3(OCH_2CHCH_3)_2O)_3-B$ ---.

Col.2, line 37, " $(C_2H_5(OCH_2CHCH_3)_4O)_3-B$ " should read
-- $(C_2H_5(OCH_2CHCH_3)_4O)_3-B$ ---.

Col.3, line 5, " $\sqrt[7]{CH_3(OCH_2CH_2)_8(OCH_2CHCH_3)_5O}_3-B$ " should read
-- $\sqrt[7]{CH_3(OCH_2CH_2)_8-(OCH_2CHCH_3)_5O}_3-B$ ---.

Col.3, line 7, " $\sqrt[7]{C_3H_7(OCH_2CHCH_3)_{10}-(OCH_2CH_2)_{10}O}_3-B$ " should read
-- $\sqrt[7]{C_3H_7(OCH_2CHCH_3)_{10}-(OCH_2CH_2)_{10}O}_3-B$ ---.

Col.3, line 64, that portion of the formula reading
"and $C_2H_5(OCH_2CH_2)_2-$ " should read -- $C_2H_5(OCH_2CH_2)_2-$ ---

Col.5, line 74, "500°F." should read --550°F.---

Col.6, line 22, "field" should read --fluid---

Col.12, line 43, Example XXVII under column of percent by weight,
"7.500" should read --75.00---

Col.13, line 10, Example XXX, " $\sqrt[7]{C_2H_5(OCH_2H_2)_3O}_3-B$ " should read
-- $\sqrt[7]{C_2H_5(OCH_2CH_2)_3O}_3-B$ ---.

Col.15, line 15 in Table 1, under column of test results..of Example I,
"7.2" should read --15.4---

Col.15, line 21 in Table 1, under column of SAE 70R3 specification,
"." should read --1---

Col.15, line 33 in Table 1, under column of test results..of Example I,
"0. 5" should read --0.05---

See attached sheet also..

continued Certificate of Correction for U.S. Patent 3,625,899

Col.20, Claim 24, line 38, " $(R_1-(OCH_2CHR_2)_n(OCH_2CHR_3)_nO)_s-B$ "
should read $--(R_1-(OCH_2CHR_2)_n(OCH_2CHR_3)_nO)_s-B---$.

Col.20, Claim 24, line 50, " $(-(OCH_2CH_2)_r, (OCH_2CHCH_3)_s-),$ "
should read $--\sqrt{-(OCH_2CH_2)_r, (OCH_2CHCH_3)_s-}---$.

Signed and sealed this 1st day of May 1973

(SEAL)
Attest:

EDWARD M. FLETCHER, JR.
Attesting Officer

ROBERT GOTTSCHALK
Commissioner of Patents