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- (54) Water based functional fluids.
- (a) A functional fluid composition for use as a hydraulic fluid, lubricant, cutting fluid or drilling fluid which comprises
  - (a) from 20 to 98% by weight water
  - (b) from 0 to 80% by weight of a glycol, and
  - (c) from 0.5 to 25% by weight of a thickening agent prepared by (a) reacting one mole of a monofunctional active-hydrogen-containing compound having at least 10 carbon atoms with between 20 and 400 moles of one or more alkylene oxides and (b) thereafter reacting the product of step (a) with a diepoxide in an amount such that the molar ratio of diepoxide to hydroxyl groups in the product of step (a) is between 0.2:1 and 5:1. The fluid may further comprise functional components for preventing wear and corrosion and also 0.5 to 15% by weight of a non-ionic, cationic, anionic or amphoteric surfactant.

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#### WATER BASED FUNCTIONAL FLUIDS

The present invention relates to the use of water based functional fluids which can be used for example as hydraulic fluids, lubricants, drilling or cutting fluids.

Functional fluids based upon mixtures of water and polyether glycols are well known and are used in a wide variety of applications eg as cutting fluids, hydraulic fluids and the like.

Typical formulations of water-based hydraulic fluids (known as HF-C fluids) comprise approximately 15-30% of a high-viscosity polyether glycol, and 35-45% water, with the balance (up to 50%) being simple glycol and small amounts of additives known to the skilled man. Typically these hydraulic fluids have a viscosity of 32-68 cSt at  $40\,^{\circ}$  C.

There is a growing trend however to reduce the polyether glycol content of such functional fluids. For example it would ultimately be desirable to reduce the polyether glycol content of hydraulic fluids to 10% or possibly even less. There is also a trend to produce what can be described as High Water Based Fluids (HWBFs), containing typically 50-98 percent water, by additionally removing some or all of the glycol.

A problem arises when attempts are made to reduce the polyether glycol content of hydraulic fluids in that the viscosity of the hydraulic fluid is reduced. This arises because standard polyether glycol fluids do not possess sufficient thickening power. Simply increasing the viscosity of the polymer does not achieve the required effect, because the increase in solution viscosity becomes marginal, and the polymer becomes unstable under conditions of high shear.

It has previously been proposed for example in US 4 767 555 and US 4 288 639 to achieve the required viscosity of such hydraulic fluids by incorporation of small amounts of materials which (a) are water compatible and (b) increase the viscosity of the hydraulic fluids by intermolecular association in solution. Such materials are known as associative thickeners.

The hydraulic fluids previously described as high-water-based fluids (HWBFs), usually incorporating either no simple glycol, or only small amounts as antifreeze, have a number of disadvantages when compared to HFC water glycol fluids, viz

- i) Although the HWBFs normally exhibit 'permanent shear stability', that is the viscosity of the fluid remains stable over a long period of operation, they none-the-less exhibit temporary loss of viscosity in high shear zones, as evidenced by loss of flow rate and pump efficiency in hydraulic systems, in other words they lack 'temporary shear stability'.
- ii) Under such conditions of temporary viscosity loss, hydrodynamic lubricity of the fluid also suffers. Lubrication is maintained only by additives which provide boundary lubrication (EP additives). Despite such additives being highly developed, these conditions can result in enhanced wear. Additionally the best of these additives in many instances are toxic, hydrolytically unstable and environmentally undesirable.
  - iii) The viscosity index of such fluids is often poor which can give problems with pump start-up.
  - iv) HWBF's are sensitive to water loss, which has a dramatic and undesirable effect on fluid viscosity.

US 3538033 discloses a family of polyether derivatives of diepoxides which can be used as thickeners in textile printing emulsions, cosmetic emulsions, aqueous pigment suspensions and the like.

The problem to be solved is therefore to produce functional fluids, suitable for the applications described above, which have good permanent and temporary shear stability, contain low concentrations of the associative thickeners, have good resistance to water loss in terms of viscosity change, have good apparent viscosity indices, and which maintain hydrodynamic lubrication under high shear conditions.

According to the present invention there is provided a functional fluid composition for use as a hydraulic fluid, lubricant, a cutting fluid or a drilling fluid which comprises

(a) from 20 to 98% by weight water

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- (b) from 0 to 80% by weight of a glycol and
- (c) from 0.5 to 25% by weight of a thickening agent prepared by (a) relatively reacting one mole of a monofunctional, active-hydrogen- containing compound having at least 10 carbon atoms with between 20 and 400 moles of one or more alkylene oxides and (b) thereafter reacting the product of step (a) with a diepoxide in an amount such that the molar ratio of diepoxide to hydroxyl groups in the product of step (a) is between 0.2:1 and 5:1.

The present invention solves the problem defined above by employing a thickening agent of the type defined in US 3538033 in the applications described above, together with the formulation technology which is detailed below.

Considering the water component of the composition first, it is preferred that this comprises between 36 and 95% of the functional fluid most preferably between 45 and 80%.

As regards the glycol, this can be in principle any glycol which is miscible with water and includes

monoethylene glycol and monopropylene glycol and low molecular weight oligomers thereof (ie having up to 6 ethylene and/or propylene glycol units). Preferably the glycol is selected from monoethylene glycol, diethylene glycol and triethylene glycol. It is preferred that the glycol comprises between 20 and 60% of the functional fluid.

The first stage of preparing the thickening agent involves reacting the monofunctional active hydrogen containing compound with one or more alkylene oxides. By the term 'active-hydrogen- containing compound' is meant one which contains hydrogen measurable by the Zerewitinoff active hydrogen test. Such compounds include alcohols, phenols, thiols, fatty acids and amines. The preferred compounds are  $C_{16}$  to  $C_{20}$  monohydric alcohol or thiols,  $C_{16}$  to  $C_{20}$  alkyl phenols and  $C_{16}$  to  $C_{20}$  aliphatic amines.

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The active hydrogen containing compound is alkoxylated with one or more alkylene oxides using either a base or a Lewis acid as a catalyst. Typically the alkylene oxide is one or more of ethylene oxide, propylene oxide or the isomers of butylene oxide. Suitably between 20 and 400 moles of the alkylene oxide are added to the active hydrogen containing compound of which 20 to 100 mole % (most preferably 65 to 85 mole %) is ethylene oxide and 0 to 80% (most preferably 15 to 35%) is propylene and/or butylene oxide.

In the second stage of manufacture the product of the first stage is reacted with a diepoxide. The diepoxide can in principle by any organic compound having two epoxide groupings. Suitable diepoxides are those having between 4 and 30 carbon atoms and include vinylcyclohexene diepoxide, bisphenol A/epichlorohydrin condensate and the like. It is preferred to use the diepoxide in amounts such that the molar ratio of epoxide groups to hydroxyl groups (in the product of stage 1) is in the range 0.2 to 1 to 5 to 1. The second stage of the process can also be catalysed by either a base or a Lewis acid.

It is preferred that the thickening agent comprises between 2 and 10% by weight of the functional fluid composition. It is also preferred that the thickener itself should have a neat viscosity in excess of 4000 cSt at 40°C.

Concerning the antiwear components, these are typically metal or amine salts of organo sulphur, phosphorus or boron derivatives, or carboxylic acids. Typically these include salts of  $C_1$  to  $C_{22}$  carboxylic acids, aliphatic or aromatic; sulphur acids such as aromatic sulphonic acids, phosphorus acids, for example acid phosphate esters and analogous sulphur compounds, eg thiophosphoric and dithiophosphoric acid. Many further antiwear agents are suitable, and known to the skilled man.

Inhibitors for corrosion of metals can be organic or inorganic, for example metal nitrites, hydroxyamines, neutralised fatty acid carboxylates, phosphates, sarcosines and succinimides etc. Most useful are amines such as alkanolamines, e.g. ethanolamine, diethanolamine and triethanolamine. Non-ferrous metal inhibitors include for example aromatic triazoles.

It is preferred to include in the formulation 0.5 to 15% of a surfactant as a co-thickening agent, which may be non-ionic, cationic, anionic or amphoteric. Examples of suitable surfactants include linear alcohol alkoxylates, nonylphenol ethoxylates, fatty acid soaps, amine oxides etc. Most preferred are linear secondary alcohol alkoxylates, eg those commercially available from BP Chemicals under the registered trade mark Softanol. The surfactants behave synergistically with the associative thickener, such that a given viscosity can be achieved with a lower total thickener content of the blend compared to use of the associative thickener alone.

In addition to the above the functional fluid composition may also contain optional components such as extreme pressure additives, antifoams, antimicrobials and the like which are well known to the skilled man. It is also possible to add further known thickening agents and co-thickening agents if desired.

The functional fluid compositions of the present invention may be prepared by mixing the four main components and any additional materials which are required in a vessel of suitable size.

In the preferred embodiments of the invention, that is those formulations containing 20 or more percent of glycol, it has been found that temporary shear stability is conferred to the fluids. This enhances the capability of the fluids to provide hydrodynamic lubrication under high shearing conditions. The presence of the 20 or more percent glycol, and of the surfactant cothickener also improves the apparent viscosity index of the fluids when compared against fluids containing no glycol or surfactant.

In those embodiments of the invention containing less than 20 percent glycol, the problem of temporary shear is also addressed, by use of associative thickeners of the present invention with high inherent polymer viscosity (eg greater than 20,000 cSt at 40°C). In high shear environments, thickening achieved by association is lost when the fluids shear temporarily. However, the viscosity of the fluid is maintained at an intermediate level by the normal thickening of a high viscosity polymer, typically to a level of 10-20 cSt at 50°C. This limits the loss of pump efficiency, and ensures that hydrodynamic lubricity is retained. This approach also enhances the characteristics of the fluid concerning viscosity change on water loss and viscosity index.

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The functional fluid compositions of the present invention are particularily useful as hydraulic fluids in piston, gear or vane hydraulic pumps, motors and general hydraulic systems, eg hydraulic rams, robots and the like.

The invention is now illustrated by the following examples:

Example 1

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A commercial sample of oleyl/cetyl alcohol (85/15) was catalysed with potassium hydroxide (0.3% by weight on the target alkoxylate), dried, and reacted with a 70/30 (weight/weight) mixture of ethylene oxide and propylene oxide at 115-125°C to produce an alkoxylate having an experimentally determined molecular weight of 6000 (by hydroxyl determination).

500 grams of the above un-neutralised alkoxylate was reacted at 130°C with 15.9 grams bisphenol A/epichlorohydrin condensate (Epikote 828 - ex Shell) for three hours. The residual catalyst was removed by treatment with magnesium silicate to leave an associative thickener with a neat viscosity of 5000 cSt @ 40°C. A 10% aqueous solution viscosity was determined as 193 cSt @ 40°C.

# Example 2

500 grams of the above-mentioned un-neutralised alkoxylate from example 1 was reacted at 130°C with 31.8 grams of bisphenol A/epichlorohydrin condensate for 3 hours, the catalyst was removed with magnesium silicate treatment, to leave an associative thickener with a neat viscosity of 17,300 cSt @ 40°C. A 10% aqueous solution viscosity was determined as 2100 cSt @ 40°C.

Example 3

A commercial sample of oleyl/cetyl alcohol (85/15) was reacted in the manner described in example 1 with an 80/20 mixture of ethylene oxide and propylene oxide to produce an alkoxylate having a molecular weight of 6000.

300 grams of the above-mentioned un-neutralised polyalkylene glycol was reacted with 14.0 grams of vinylcyclohexene diepoxide for three hours at 140-150°C. The resulting associative thickener had a neat viscosity of 4,900 cSt @ 40°C and a 10% aqueous solution viscosity of 1427 cSt @ 40°C.

#### Example 4

A commercial sample of oleyl/cetyl alcohol (85/15) was reacted in the manner described in example 1 with an 75/25 mixture of ethylene oxide and propylene oxide to produce an intermediate having a molecular weight of 2950, then with further ethylene oxide to produce an alkoxylate having a molecular weight of 3100.

2000 grams of the above mentioned un-neutralised polyalkylene glycol was reacted with 175 grams of vinylcyclohexene diepoxide at 140-150°C for 6 hours to yield an associative thickener with a 10% aqueous solution viscosity of 1742 cSt.

### Examples 5 - 8

Dodecan-1-ol, Tetradecan-1-ol, Hexadeca-1-ol and Octadecan-1-ol were reacted under base catalysis with 80/20 blends of ethylene oxide/propylene oxide to produce alkoxylates having molecular weights of 6000. 200 grams of each alkoxylate was reacted with 9.4 grams of vinylcyclohexane diepoxide in the manner described for example 3 to yield four associative thickeners. The products were used to provide a comparison of solution viscosity versus hydrophobe chain length.

EXAMPLE	Comparison	5	6	7	8
PRODUCT Viscosity, neat, cSt @ 40°C	Breox 75W18000* 18.000	C-12 22,400	C-14 10,400	C-16 27,400	C-18 21,400
Viscosity, 10%, cSt @ 40° C	1	117	461	4,680	34,000

<sup>\*</sup> standard polyglycol thickener.

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#### Example 9

The thickener (8%) from example 1 was formulated into a hydraulic fluid of 46 cSt at 40°C containing water (36%), diethylene glycol (50%), a fatty acid and morpholine. This was subjected to a four ball test (IP239) at 40 kg for 1h, and gave a wear scar of 0.56 mm, a result similar to that of a similarly formulated hydraulic fluid using 15% of a conventional polyglycol thickener.

The same thickener at a level of 7% in a high water based fluid of 46 cSt at 40 °C was subjected to 50 passes in a Kurt Orban shear stability tester (IP294) and suffered no permanent loss of solution viscosity.

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## Example 10

The thickener from example 4 was formulated at a level of 8% into a hydraulic fluid containing 30% diethylene glycol and 58% water, the balance being an antiwear and anticorrosion package. The said hydraulic fluid was circulated over 24 hours in a 7 kW Vickers PFB-5 axial piston pump at 50°C and 170 bar. The flow rate, which can be directly related to the fluid viscosity, was monitored to indicate viscosity in the high shear regions of the pump. The said hydraulic fluid was compared to a similar hydraulic fluid containing only 10% diethylene glycol and 80% water, and with a further hydraulic fluid, based on a commercially available, low-viscosity, associative thickener, a fatty alcohol ethoxylate capped with an olefin epoxide, and containing no diethylene glycol.

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	Absolute	Apparent Viscosity in	Apparent Viscosity in % Retained Viscosity	
	Viscosity cP @	Shear Zone cP	under High Shear	Flow I/m
	47 C			
Example 10	38	31	81%	14.6
10% DEG Fluid	27	12	44%	14.11
Fluid from commercial low viscosity Associative Thickener	21	3.4	16%	12.46

### Example 11

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The thickener from example 1 (6.2%) and a 5 mole ethoxylate of a linear secondary C-12/14 alcohol (1.55%) (Softanol 50 - ex BP Chemicals) were blended with diethylene glycol (40%) and water (50%), the balance consisting of functional antiwear, anticorrosion and antifoam agents (2.25%) to give a hydraulic fluid of 45.5 cSt @ 40° C. The following data were determined.

Four ball wear scar, mm (1h, 40kg, IP239): 0.61 Shear Stability (IP294), % viscosity change: -7

### Example 12

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The associative thickener from example 3 (3.6%) and a 5-mole ethoxylate of a linear secondary C-12/14 alcohol (0.9%) were blended with diethylene glycol (20%) and water (73.4%), the balance being additives (2%), to give a hydraulic fluid of 49.3 cSt @ 40° C. The following data were determined.

Four ball wear scar, mm (1h, 40kg, IP239): 0.62

Shear Stability (IP294), % viscosity change: +5

### Example 13

LXample 1

The associative thickener from example 3 (5.4%) and a 12 mole ethoxylate of a linear secondary C-12/14 alcohol (3.6%) (Softanol 120 - ex BP Chemicals) were blended with diethylene glycol (39%) and water (50%), the balance consisting of antiwear anticorrosion, antifoam additives (2%), to give a hydraulic fluid of 46.8 cSt @ 40° C. The following data were determined.

Four ball wear scar, mm (1h, 40kg, IP239): 0.61

Shear Stability (IP294), % viscosity change: +2.9

#### Example 14

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The thickener from example 2 (5.0%) and a 12 mole ethoxylate of a linear, secondary, C12/14 alcohol (1.25%) were blended with diethylene glycol (20%) and water (70%), the balance consisting of antiwear, anticorrosion and antifoam agents (3.75%) to give a hydraulic fluid of 46.1 cSt @ 40° C. The following data were determined.

Four ball wear scar, mm (1h, 40kg, IP239): 0.68

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#### Example 15

To demonstrate the effect of diethylene glycol on apparent viscosity index two ISO 68 fluids were prepared from the thickener from example 3. Fluid 1 containing 6.0 percent of thickener, 30 percent of diethylene glycol and 64 percent of water. Fluid 2 containing 5.0 percent thickener and 95 percent water. The combined effect of the presence of diethylene glycol and the slightly increased thickener requirement in fluid 1 results in a significantly lower viscosity fluid at 20°C than that observed for fluid 2, demonstrating that the apparent viscosity index of fluid 1 is higher than that of fluid 2.

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	Visc. @ 40° C, cSt	Visc. @ 20 °C, cSt
Fluid 1	72.5	585
Fluid 2	71.5	985

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#### Example 16

To demonstrate the effect of co-thickeners on apparent viscosity index, two fluids of 550 cSt @ 40°C were prepared as follows: Fluid 1 contained 6.0 percent of thickener from example 2, 4.0 percent of a 12 mole ethoxylate of a C-12/14 linear secondary alcohol, 10 percent diethylene glycol and 80 percent water. Fluid 2 contained 10.0 percent thickener from example 2, 10 percent diethylene glycol and 80 percent water. The presence of surfactant in fluid 1 and reduced thickener requirement results in a significantly lower viscosity fluid at 20°C than observed for fluid 2, demonstrating that the apparent viscosity index of fluid 1 is higher than that for fluid 2.

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	Visc. @ 40°C, cSt	Visc. @ 20°C, cSt
Fluid 1	550	4000
Fluid 2	550	5500

### Claims

- 1. A functional fluid composition for use as a hydraulic fluid, lubricant, cutting fluid or drilling fluid which comprises
  - (a) from 20 to 98% by weight water
  - (b) from 0 to 80% by weight of a glycol, and
- (c) from 0.5 to 25% by weight of a thickening agent prepared by (a) relatively reacting one mole of a monofunctional active-hydrogen-containing compound having at least 10 carbon atoms with between 20 and 400 moles of one or more alkylene oxides and (b) thereafter reacting the product of step (a) with a diepoxide in an amount such that the molar ratio of diepoxide to hydroxyl groups in the product of step (a) is between 0.2:1 and 5:1.
- 2. A functional fluid composition as claimed in claim 1 further comprising functional components for preventing wear and corrosion.
  - 3. A functional fluid composition as claimed in claim 1 comprising from 35 to 95% by weight water.
  - 4. A functional fluid composition as claimed in claim 1 comprising from 20 to 60% by weight glycol.
- 5. A functional fluid composition as claimed in claim 1 comprising 2 to 10% by weight of the thickening agent
- 6. A functional fluid as claimed in claim 1 or claim 4 further comprising 0.5 to 15% by weight of a non-ionic, cationic, anionic or amphoteric surfactant.
- 7. A functional fluid composition as claimed in claim 1 comprising 0 to 20% by weight glycol and 0.5 to 25% by weight of a thickening agent having a neat viscosity greater than 20,000 cSt at 40° C.
- 8. A process for preparing a functional fluid as defined in claim 1 which comprises mixing the required amounts of water, glycol and thickening agent.
- 9. A process as claimed in claim 8 in which functional components for preventing wear and corrosion are added during mixing.

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# **EUROPEAN SEARCH REPORT**

EP 89 31 1655

	DOCUMENTS CONSI	DERED TO BE RELEVAN	VT.	
Category		dication, where appropriate,	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.5)
D,X	US-A-4 767 555 (E. * Abstract; column 2, line 50; example column 7, lines 1-7	1, line 9 - column s II,III; table III;	1,3-5,7	C 10 M 173/02
Υ	column /, lines 1-/	, Claims 4,5	2,6	
A	GB-A-1 182 448 (KA * Page 1, line 9 - examples 1-12; clai 538 033 (N. HAYASHI	page 2, line 126; ms 1-12 * & US-A-3	1,7,8	
Υ	US-A-4 312 775 (E. * Whole document *	J. PANEK et al.)	2,6	
A	" who le document "		1,3-5,7 -9	
	& US-A-4 411 819			
A	US-A-4 481 125 (R. 	V. HOLGADO)		
				TECHNICAL FIELDS SEARCHED (Int. Cl.5)
				C 10 M
	<u>.</u> -			
	The present search report has b	een drawn up for all claims		
TH	Place of search E HAGUE	Date of completion of the search 08-12-1989	FIS	Examiner CHER W.H.F.
	CATEGORY OF CITED DOCUME	NTS T: theory or princ E: earlier patent	ciple underlying the	e invention lished on, or

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X: particularly relevant if taken alone
Y: particularly relevant if combined with another document of the same category
A: technological background
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P: intermediate document

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D: document cited in the application

L: document cited for other reasons

& : member of the same patent family, corresponding document