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FIRE-RESISTANT FUNCTIONAL FLUIDS

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No Drawing. Continuation-in-part of application Ser. No. 601,335, Dec. 13, 1966. This application Mar. 13, 1969, Ser. No. 807,074

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U.S. Cl. 252—49.9 12 Claims

ABSTRACT OF THE DISCLOSURE

Homogeneous fire-resistant, low cost, functional fluids are disclosed which are composed of 40 to 50% highly chlorinated hydrocarbons containing at least 48% by weight chlorine, 20 to 30% petroleum-base oil, 5 to 20% liquid triaryl phosphate, 8 to 35% trialkyl phosphate which serves as a mutual solvent for the other ingredients and stabilizing amounts of a triorganic phosphite and an epoxy plasticizer.

CROSS REFERENCE TO RELATED APPLICATIONS

This application is a continuation-in-part of United States patent application No. 601,335, filed Dec. 13, 1966, now abandoned.

BACKGROUND OF THE INVENTION

Field of the invention

Fire-resistant functional fluids.

Description of the prior art

Many fire-resistant functional fluids are known. Desirably a functional fluid should have viscosity characteristics such that it may be used over a wide temperature range; it must not adversely affect the materials of construction of the system in which it is used; it must possess adequate lubricity and mechanical stability in order that it may be used in valves, pumps, self-lubricated pumps and other components of hydraulic power systems; and it should not be abnormally toxic or otherwise harmful to personnel who come in contact with it.

Suitable fire-resistant hydraulic fluids and lubricants consisting essentially of a mixture of 20–80% triaryl phosphate, 80–20% chlorinated biphenyl, and 0.2–10% polyalkyl methacrylate as a viscosity index improver are disclosed in United States Patent No. 3,136,726 to D. H. Moreton. Fluids composed of a mixture of primarily triaryl and trialkyl phosphates, and minor proportions of a flame-proofing agent and mineral oil are reported in United States Patent No. 2,549,270, issued to Watson, July 19, 1948. These patented fluids are expensive, because they contain major proportions of expensive ingredients and very little of the cheap mineral oil.

Petroleum-base oils, though good lubricants and economically desirable, are not used in high proportions in fire-resistant functional fluids because of their flammability. Chlorinated hydrocarbons are good flame retardants for petroleum-base oils. The higher the combined chlorine content is, the more effective the chlorinated hydrocarbons are as flame retardants. However, the more highly chlorinated hydrocarbons, those containing at least 48% combined chlorine, are not compatible with mixtures of triaryl phosphates and petroleum-base oils.

Triaryl and trialkyl phosphates are widely used in fire-resistant functional fluids to provide flame resistance and lubricity. Triaryl phosphates are preferred to trialkyl phosphates in functional fluids for anti-wear properties because they cost less than trialkyl phosphates.

SUMMARY OF THE INVENTION

We have now discovered that inexpensive homogeneous fire-resistant functional fluids can be prepared which contain major amounts of the inexpensive highly chlorinated liquid hydrocarbons and petroleum-base oils, together with minor amounts of triaryl phosphates, by including in the fluid a minor portion of a trialkyl phosphate as a mutual solvent for the other ingredients, and a heat stabilizing amount of a mixture of a triorganic phosphite and an epoxy plasticizer. Fluids containing (a) 35 to 50 parts by weight of a highly chlorinated liquid hydrocarbon, (b) 20 to 30 parts by weight of a petroleum-base oil, (c) 5 to 20 parts by weight of a triaryl phosphate, and (d) 8 to 40% by weight of a trialkyl phosphate based on the total weight of the other ingredients are homogeneous, compatible, fire-resistant functional fluids. The stabilizing phosphite is added to the functional fluids in an amount of about 0.1 to 1 part by weight per 100 parts by weight of fluid composed of components (a) (b) (c) and (d). The stabilizing epoxy plasticizer is added to the functional fluids in an amount of about 0.5 to about 2 parts by weight per 100 parts by weight of fluid composed of components (a) (b) (c) and (d). Surprisingly, the mixed stabilizers have a stabilizing effect greater than that due to the additive effect of the stabilizers.

DESCRIPTION OF THE INVENTION AND THE PREFERRED EMBODIMENTS

Triaryl phosphates suitable for the purposes of this invention are those phosphates containing 3 hydrocarbon radicals, where the aromatic nucleus may contain 0 to 3 alkyl substituents, and the alkyl substituents may contain one to three carbon atoms. These triaryl phosphates particularly include tricresyl phosphate, cresyl-diphenyl phosphate, xylyl-diphenyl phosphate, phenyl-dixylyl phosphate, trixylyl phosphate, isopropylphenyl diphenyl phosphate, diisopropylphenyl monophenyl phosphate, tris isopropylphenyl phosphate and mixtures thereof, and liquid mixtures of triphenyl phosphate, and tricresyl phosphate, etc. The aromatic phosphate esters particularly suitable for the purpose of this invention may be represented by the formula:

$$\overset{\mathrm{O}}{\overset{\|}{\mathrm{P}}}\!-\!\left(\mathrm{OR}\right)_3$$

where R is selected from the group consisting of phenyl, cresyl and xylyl radicals. Trixylyl phosphate was found to be slightly more compatible with the other ingredients of these functional fluids than the other triaryl phosphates listed above, and is therefore the slightly preferred aryl phosphate for use in this invention.

The chlorinated hydrocarbons useful in this invention may be any of the commercially available highly chlorinated paraffins or chlorinated biphenyls. The chlorinated hydrocarbon should be liquid and contain enough chlorine to impart flame resistance to the formulation. Compounds containing at least 48% by weight of combined chlorine are desirable for their flame-resistant properties.

The chlorinated hydrocarbons generally have poor heat stability, but this may be overcome by incorporation of any of a sizeable array of heat stabilizers. The type of heat stabilizers normally used in polyvinyl chloride compositions consisting of metal-organic compounds containing barium, cadmium, zinc, tin, lead, epoxy compounds and organic phosphites may be used. Combinations of organo-phosphites and epoxidized oils were found to be the preferred heat stabilizers for these functional fluids.

The most preferred stabilizer found is a combination of phenyl didecyl phosphite and epoxidized linseed oil containing 9.5% oxirane oxygen.

The petroleum-based oils suitable for the purposes of this invention are mineral oils with a Saybolt Universal Viscosity (ASTM Saybolt Method D–88) in the range of 32–200 SUS at 100° F. and petroleum-base hydraulic oil with a Saybolt Universal Viscosity in the range of about 140–400 SUS at 100° F. The apparently wide viscosity ranges provide final formulations which meet the different viscosity requirements of various hydraulic pumps, fluid motors, and other components of hydraulic power systems.

The trialkyl phosphate useful for inclusion in the present compositions are those in which the alkyl radical has from 4–12 carbon atoms each and include the following: tributyl phosphates, triamyl phosphates, trihexyl phosphates, triheptyl phosphates, trioctyl phosphates, trinonyl phosphates, tridecyl phosphates, tridodecyl phosphates and particularly branched homologs such as tris(2-ethylhexyl) phosphate, tris(2-ethylbutyl) phosphate and tris (3,5,5-trimethylhexyl) phosphate, and the like.

Heat stabilizing triorganic phosphites useful in practicing this invention include trialkyl phosphites, triaryl phosphites, alkyl-diaryl phosphites, tri(alkylatedaryl) phosphites and mixtures of these phosphites. The organophospite is added in the amount of about 0.1 to about 1, and preferably about 0.4 to 0.6, part by weight per 100 parts by weight of fluid containing major amounts of the highly chlorinated liquid hydrocarbon, petroleum-based oil, triaryl phosphate and trialkyl phosphate. Suitable phosphites include triphenyl phosphite, phenyl-didecyl phosphite, trioctyl phosphite and the like.

Heat stabilizing epoxy plasticizers, of the type used in stabilizing poly(vinyl chloride) compositions, useful in practicing this invention include epoxidized oils such as epoxidized soybean oil and epoxidized linseed oil, and epoxidized esters such as butyl epoxy tallate, octyl epoxy tallate, butyl epoxy stearate, and the like. The epoxy compounds are added in the amount of about 0.5 to about preferably about 1 to about 1.5 parts by weight per 100 parts by weight of functional fluid composed principally of a highly chlorinated hydrocarbon, petroleum-base oil and trialkyl phosphate. Epoxidized compounds with high oxirane oxygen contents are preferred to epoxy compounds with low oxirane oxygen contents.

The novel functional fluids of this invention can be compounded together with conventional functional fluid additives such as heat stabilizers, anti-oxidants, rust preventatives, viscosity index improvers, detergent-dispersion additives and the like.

The following typical examples of formulations prepared in accordance with this invention are given by way of illustration and not by limitation. The examples were all prepared by simply stirring the ingredients listed in each example together. All parts and percentages are by weight.

EXAMPLE 1

	Parts by weight
Chlorinated paraffin (50% chlorine)	49
Petroleum oil	29
Trixylyl phosphate	13
Tris(2-ethylhexyl) phosphate	8.4
Epoxidized linseed oil (9.5% oxirane oxygen)	0.6
Triphenyl phosphite	0.25

This sample possesses good lubricity, mechanical stability and is not abnormally toxic or harmful. This sample showed only slight discoloration after 60 minutes in a 400° F. heat test. The petroleum oil used in this example had a viscosity at 100° F. of 358 SUS and a specific gravity at 20° C. of 0.871. This example was repeated substituting chlorinated biphenyl containing 50% chlorine for the chlorinated paraffin and similar results were obtained.

EXAMPLE 2

	Parts by weight
Chlorinated paraffin (50% chlorine)	49
Petroleum oil	29
Trixylyl phosphate	13
Tris(2-ethylhexyl) phosphate	8.4
Epoxidized linseed oil (9.5% oxirane oxygen)	0.6

This sample is a homogeneous solution with good lubricity, mechanical stability and low toxicity. This sample turned significantly darker than Example 1 after 60 minutes in a 400° F. heat test. The petroleum-base oil of Example 1 was used in this example. This example was repeated substituting chlorinated biphenyl described in Example 1 for the chlorinated paraffin and similar results were obtained.

EXAMPLE 3

	Parts by weight
Chlorinated paraffin (50% chlorine)	49
Petroleum oil	29
Trixylyl phosphate	13
Tributyl phosphate	9
Epoxidized linseed oil (9.5% oxirane oxygen)	0.6
Triphenyl phosphite	0.25

This sample possesses good lubricity, mechanical stability and is not abnormally toxic or hamful. The sample showed only slight discoloration after 60 minutes in a 400° F. heat test. The petroleum oil of Example 1 was used in this example. This example was repeated substituting chlorinated biphenyl containing 50% chlorine for the chlorinated paraffin with similar results.

EXAMPLE 4

	Parts by weight
Chlorinated paraffin (50% chlorine)	49
Mineral oil	29
Trixylyl phosphate	13
Tris(2-ethylhexyl) phosphate	9
Epoxidized linseed oil	0.6
Triphenyl phosphite	0.25

This sample possesses good lubricity, mechanical stability and is not abnormally toxic or harmful. The sample showed only slight discoloration after 60 minutes in a 400° F. heat test. The mineral oil used in this example had a viscosity at 100° F. of 180–190 SUS and a specific gravity at 15.6° C. of 0.820 to 0.880. This example was repeated substituting chlorinated biphenyl (50% chlorine) for the chlorinated paraffin with similar results.

EXAMPLE 5

	Parts by weight
Chlorinated paraffin (50% chlorine)	49
Mineral oil	29
Trixylyl phosphate	13
Tributyl phosphate	9
Epoxidized linseed oil	0.6
Triphenyl phosphite	0.25

This sample is a homogeneous solution with good lubricity, mechanical stability and is not abnormally toxic or harmful. The sample discolored very slightly in a 60 minute 400° F. heat test. The mineral oil used in this example had a viscosity at 100° F. of 180–190 SUS and a specific gravity of 15.6° C. of 0.820 to 0.880. This example was repeated substituting chlorinated biphenyl (50% chlorine) for the chlorinated paraffin with similar results.

EXAMPLE 6

	Parts by weight
Chlorinated paraffin (50% chlorine)	40
Mineral oil	29
Trixylyl phosphate	20
Tris(2-ethylhexyl) phosphate	11
Epoxized linseed oil	0.6
Triphenyl phosphite	0.25

This sample possesses good lubricity, mechanical stability and is not abnormally toxic or harmful. The sample showed only slight discoloration and a very slight increase in acidity after 60 minutes in a 400° F. heat test. The mineral oil used in this example had a viscosity at 100° F. of 180–190 SUS and a specific gravity at 15.6° C. of 0.820 to 0.880. This example was repeated substituting chlorinated biphenyl (50% chlorine) for the chlorinated paraffin with similar results.

EXAMPLE 7

	Parts by weight
Chlorinated paraffin (50% chlorine)	40
Mineral oil	20
Trixylyl phosphate	30
Tris(2-ethylhexyl) phosphate	10
Epoxized linseed oil	0.6
Triphenyl phosphite	0.25

This sample possesses good lubricity, mechanical stability and is not abnormally toxic or harmful. The sample showed only slight discoloration and a very slight increase in acidity after 60 minutes in a 400° F. heat test. The mineral oil used in this example had a viscosity at 100° F. of 180–190 SUS and a specific gravity at 15.6° C. of 0.820 to 0.880. This example was repeated substituting chlorinated biphenyl (50% chlorine) for the chlorinated paraffin with similar results.

EXAMPLE 8

A series of functional fluids were prepared and evaluated to determine the effectiveness of various epoxy and phosphite heat stabilizers in functional fluids. The composition details of the fluids are set forth in Table I and the results of a heat test are listed in Table II.

TABLE I

	a	b	c	d	e	f
Chlorinated paraffin (50% chlorine)	49	49	49	49	49	49
Petroleum oil	29	29	29	29	29	29
Trixylyl phosphate	13	13	13	13	13	13
Tris(2-ethylhexyl) phosphate	8.4	8.4	8.4	8.4	8.4	8.4
Epoxidized linseed oil (9.5% oxirane oxygen)		0.6	------	0.6	------	0.3
Triphenyl phosphite			0.25	0.25	0.6	0.3

Fifty gram samples of the functional fluids as shown above were heated at 400° F. and observed at intervals of 20, 30, 40, 50 and 60 minutes. The samples were removed from the oven after each time interval and arranged according to color with number 1 having the lightest color and number 6 having the darkest color. The initial color of the test fluids was amber. After 20 minutes, sample *a* was only slightly darker than the original, *b* was only very slightly darker, and the remaining four samples had little or no observable color change. Obvious color differences began to show after 30 minutes. The arrangement of the samples for each time interval is recorded in Table II.

TABLE II

	30 min.	40 min.	50 min.	60 min.
(a)	6 (dark brown)	6 (v. dark brown)	6 (black)	6 (black).
(b)	5	4	4	3 (brown).
(c)	4	5	5	5 (v. dk. brown).
(d)	2	2	2	1 (dk. amber).
(e)	3	3	3	4 (dark brown).
(f)	1 (slightly darker than original)	1 (slight darkening)	1 (slight darkening)	2 (light brown).

The above data indicate that sample *d* is much better than either *b* or *c* and sample *f* is much more resistant to color change than *b* or *e*. The fact that *f* is much more resistant to color change than either *b* or *e* clearly shows

that the stabilizing effect of the epoxidized linseed oil and triphenyl phosphite is greater than that due to the additive effect of these two stabilizers.

EXAMPLE 9

A series of fire-resistant functional fluids were prepared to evaluate the effectiveness of typical epoxy compounds and organo-phosphites as heat stabilizers in fire resistant functional fluids. The following test composition was used in this series of tests:

	Parts by weight
Chlorinated paraffin (50% chlorine)	49
Petroleum oil	29
Trixylyl phosphate	13
Tris(2-ethylhexyl) phosphate	9

All the samples in the "phosphite tests" contained 0.6 part by weight of expoxidized linseed oil per 100 parts of the test composition and the phosphite and phosphite amount were varied as indicated in Table III. All the samples in the "epoxy tests" contained 0.25 part by weight of triphenyl phosphite per 100 parts by weight of the test composition and the epoxy plasticizers and epoxy plasticizer amount were varied in the test composition as indicated in Table IV.

Fifty gram samples of the test compositions with the various heat stabilizer compositions shown in Table III were heated at 400° F. in an oven and observed for color changes at intervals of 10, 20, 30, 45 and 60 minutes and 50 gram samples of test composition with the various heat stabilizers shown in Table IV were also heated at 400° F. in an oven, but observed for color change at intervals of 15, 30, 45 and 60 minutes.

TABLE III.—EFFECT OF PHOSPHITES ON HEAT STABILITY

	Parts by weight	Minutes at 400° F.				
		10	20	30	45	60
a. Triphenyl	0.1	9	9	7	7	7
b. Triphenyl	0.25	No change	5	4	5	4
c. Triphenyl	0.5	No change	3	2	2	2
d. Phenyl-didecyl	0.1	No change	7	6	8	8
e. Phenyl-didecyl	0.25	No change	2	3	4	5
f. Phenyl-didecyl	0.5	No change	1	1	1	1
g. Trioctyl	0.1	No change	8	9	9	9
h. Trioctyl	0.25	No change	6	8	6	6
i. Trioctyl	0.5	No change	4	5	3	3
j. Control, no phosphite	---------	No change	10	10	10	10

The samples were removed from the oven after each time interval and arranged according to color. The data above indicate the rank of the samples with respect to color, #1 being lightest and #10 darkest.

Sample "f" was best in all cases—initially amber and turning to a dark amber in 60 minutes.

Sample "j" was worst in all cases—initially amber and turning dark brown, nearly black, in 60 minutes.

TABLE IV.—EFFECT OF EPOXIES ON HEAT STABILITY

	Parts by weight	Minutes at 400° F. 15	30	45	60
k. Epoxy linseed [1]	0.3	No change	10	11	9
l. Epoxy linseed	0.6	No change	3	4	5
m. Epoxy linseed	1.0	No change	1	1	1
n. Epoxy soya [2]	0.3	No change	12	12	10
o. Epoxy soya	0.6	No change	4	6	6
p. Epoxy soya	1.0	No change	2	2	2
q. Octyl epoxy tallate [3]	0.3	No change	9	9	12
r. Octyl epoxy tallate	0.6	No change	8	8	8
s. Octyl epoxy tallate	1.0	No change	6	5	4
t. Butyl epoxy stearate [4]	0.3	No change	11	10	11
u. Butyl epoxy stearate	0.6	No change	7	7	7
v. Butyl epoxy stearate	1.0	No change	5	3	3
w. Control, no epoxy		No change	13	13	13

[1] Epoxidized linseed oil, 9.5= oxirane oxygen.
[2] Epoxidized soybean oil, 7.2= oxirane oxygen.
[3] Epoxidized octyl tallate, 4.5= oxirane oxygen.
[4] Epoxidized butyl oleate, 3.9= oxirane oxygen (butyl epoxy stearate).

The samples were removed from the oven after each time interval and arranged according to color.

The data above indicate the rank of the samples with respect to color, #1 being lightest and #13 darkest.

Sample "m" was consistently best, initially amber and turning to a darker amber color in 60 minutes.

Sample "w" was consistently worst, initially amber and turning dark brown to black in 60 minutes.

The test results in Tables III and IV show that greater stability was obtained with higher levels of stabilizer in both cases. Expoxidized soybean oil and epoxidized linseed oil were clearly the best heat stabilizers. The mixed aryl-dialkyl phosphite was superior to the other two phosphites.

As will be apparent to those skilled in the art, numerous modifications and variations of the invention illustrated above may be made without departing from the spirit of the invention.

What is claimed is:

1. A liquid fire-resistant functional fluid composition which comprises: (a) 60 to 80% of a mixture of liquid chlorinated hydrocarbons and petroleum oils, the petroleum oil comprising 20 to 30% of the total composition; (b) 20 to 40% of a mixture of liquid triaryl phosphate and trialkyl phosphate, the triaryl phosphate comprising at least 5% of the total composition, and the trialkyl phosphate comprising at least 8% of the total composition; the liquid chlorinated hydrocarbon being selected from the group consisting of chlorinated paraffins containing at least about 48% by weight chlorine and chlorinated biphenyl containing at least about 48% combined chlorine; the petroleum oil being selected from the group consisting of mineral oils having a viscosity of about 30 to 200 SUS and petroleum base hydraulic oils having a viscosity of about 140 to 400 SUS; the liquid triaryl phosphate being selected from the group consisting of triaryl phosphates represented by the formula

$$\overset{O}{\overset{\|}{P}}\!-\!\left(OR\right)_3$$

where R is selected from the group consisting of phenyl, cresyl, and xylyl radicals and the liquid mixtures of these triaryl phopshates; and the trialkyl phosphate being selected from the group consisting of trialkyl phosphates in which each alkyl radical has from 4 to 12 carbon atoms; (c) 0.1 to 1 part by weight, per 100 parts by weight of the total of (a) and (b), of a triorganic phosphite; and (d) 0.5 to 2 parts by weight per 100 parts by weight of the total of (a) and (b) of an epoxy plasticizer.

2. A fire-resistant functional fluid according to claim 1 wherein the chlorinated paraffin has a molecular weight of at least 560 and contains at least 48% combined chlorine.

3. A fire-resistant functional fluid according to claim 1 wherein the chlorinated biphenyl contains at least 48% combined chlorine.

4. A fire-resistant functional fluid according to claim 1 wherein the trialkyl phosphate is tris(2-ethylhexyl) phosphate.

5. A fire-resistant functional fluid according to claim 1 wherein the trialkyl phosphate is tributyl phosphate.

6. A fire-resistant functional fluid according to claim 1 wherein the triaryl phosphate is trixylyl phosphate.

7. A fire-resistant functional fluid according to claim 1 consisting of the following ingredients:

Ingredients:	Parts by weight
Chlorinated paraffin (50% chlorine)	49
Petroleum oil	29
Trixylyl phosphate	13
Tris(2-ethylhexyl)phosphate	8.4
Epoxidized linseed oil (9.5 oxirane oxygen)	0.6
Triphenyl phosphite	0.25

8. A fire-resistant functional fluid according to claim 1 consisting of the following ingredients:

Ingredients:	Parts by weight
Chlorinated paraffin (50% chlorine)	49
Petroleum oil	29
Trixylyl phosphate	13
Tributyl phosphate	9
Epoxidized linseed oil (9.5 oxirane oxygen)	0.6
Triphenyl phosphite	0.25

9. A fire-resistant functional fluid according to claim 1 consisting of the following ingredients:

Ingredients:	Parts by weight
Chlorinated paraffin (50% chlorine)	49
Petroleum oil	29
Trixylyl phosphate	13
Tributyl phosphate	9
Epoxidized linseed oil (9.5 oxirane oxygen)	0.6
Phenyl didecyl phosphite	0.25

10. The fire-resistant functional fluid of claim 1 in which the triorganic phosphite is selected from the group consisting of triaryl phosphites, trialkyl phosphites and aryl dialkyl phosphites.

11. The fire-resistant functional fluid of claim 1 in which the triorganic phosphite is selected from the group consisting of triaryl phosphites, trialkyl phosphites and phite and trioctyl phosphite.

12. The fire-resistant functional fluid of claim 1 in which the epoxy compound is selected from the group consisting of epoxidized soybean oil, epoxidized linseed oil, butyl epoxy tallate, butyl epoxy stearate and epoxidized octyl tallate.

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UNITED STATES PATENT OFFICE

CERTIFICATE OF CORRECTION

Patent No. 3,496,107 Dated February 17, 1970

Inventor(s) D. A. Lima, et al

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

Column 4, line 25, "hamful" should read --harmful--.

Column 4, line 72, "epoxized" should read --epoxidized--.

Column 5, line 15, "epoxized" should read --epoxidized--.

Column 7, line 18-21, "=" should read --%--.

Claims 7, 8, & 9, line 9, "9.5" should read --9.5%--.

Column 8, line 49 & 50, "triaryl phosphites, trialkyl phosphides and phite and troctyl phosphite" should read --triphenyl phosphite, phenyl didecyl phosphite and trioctyl phosphite--.

Signed and sealed this 4th day of January 1972.

(SEAL)
Attest:

EDWARD M.FLETCHER,JR.
Attesting Officer

ROBERT GOTTSCHALK
Acting Commissioner of Patents