These data strongly suggest the reaction of iron and silicon to form a low-melting, ductile film on the bearing surface that is much more inert to further chemical reaction than the original steel surface.

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Diarylalkanes as High Temperature, Nuclear Radiation–Resistant Hydraulic Fluids

D. R. WILGUS, A. C. ETTLING, and M. A. PINO
California Research Corp., Richmond, Calif.

THE DESIRABLE characteristics of conventional hydraulic fluids include adequate lubricating properties, low volatility, low corrosivity, low flammability, a small change of viscosity over a wide temperature range (high viscosity index), and stability to oxidation, shear, and hydrolysis. A number of structurally different materials are satisfactory lubricants over a relatively narrow temperature range of application. Representative classes of these base stocks are petroleum fractions, polyglycol ethers, phosphates, silicates, and dibasic acid esters. Such materials are inadequate for future military aircraft which require fluids capable of withstanding high temperature and nuclear radiation environments.

Organic compounds vary widely in their ability to resist thermal and radiolytic change. Unsubstituted polyphenyls are known to have good radiation resistance and outstanding thermal stability (*2*). The chief deficiencies of the polyphenyls in hydraulic fluid applications are liquid range and viscosity index. A program was undertaken to improve these latter properties and retain the desirable radiation and thermal resistance of the aromatic ring by suitable "tailoring" of alkylaromatic materials. Such a combination of qualities is found in the α,ω-diarylalkanes.

A variety of diarylalkane derivatives was synthesized and screened as potentially stable hydraulic fluids. A correlation was made of molecular structure, pertinent physical properties, and thermal and radiation stability.

EXPERIMENTAL

Syntheses. Attachment of the paraffin bridge to the phenyl rings in α,ω-diphenylalkanes was in all cases at primary carbon atoms. Diphenylmethane and bibenzyl (1,2-diphenylethane) were commercial products (Distillation Products); other unsubstituted α,ω-diphenylalkanes were prepared by acylation of benzene with the appropriate aliphatic dibasic acid chlorides, followed by Wolff-Kishner reduction of the respective diketones (*3*). Over-all yields from the dibasic acids were 50 to 60%. Symmetrically substituted α,ω-diarylalkanes were obtained by the same procedure using selected aromatics (diphenyl ether, biphenyl, Tetralin, butylbenzenes). Difficulty was encountered in the synthesis of the three 1,9-bis(butylphenyl)-nonanes. Products were contaminated with about 5% of unreduced ketone. Attempts to effect complete purification were unsuccessful.

In general, the synthesis of unsymmetrically substituted α,ω-diarylalkanes was carried out by the alkylation of specific diarylalkane intermediates with 1-olefins using either chloroform-modified aluminum chloride or hydrogen fluoride (HF) as catalysts. Under the conditions employed, the point of attachment may be any carbon other than the terminal one. No attempt was made to determine the degree of concurrent meta and para substitution of the alkyl groups. The degree of disproportionation and isomerization occurring during alkylation was not well established. Boiling ranges, viscosities, refractive indices, and infrared spectral analyses were used in the characterization of products.

Phenyl(*n*-nonylphenyl)isodecane was prepared by acylation of diphenylisodecane (derived from isosebacic acid, U.S.I. Chemicals) with pelargonyl chloride and subsequent Wolff-Kishner reduction of the ketone.

Diphenylmethane derivatives were synthesized by an HF alkylation procedure using 1-olefins. Attempted alkylations of diphenylmethane by aluminum chloride catalysis yielded primarily low boiling, disproportionated materials.

Dicyclohexylisodecane was obtained by the catalytic hydrogenation of diphenylisodecane at 390° F. and 2200 p.s.i. over Raney nickel.

1,6-Bis(phenoxy)hexane was prepared by refluxing an isopropyl alcohol solution of sodium phenolate with hexamethylene dibromide.

Test Methods. Unless otherwise stated, physical and chemical properties were measured by standard methods.

Pour point determinations were made with about 5 grams of material. The sample was charged to an 8-dram vial equipped with a low temperature thermometer. The vial was placed in a dry ice-acetone bath, and the temperature was gradually lowered. The sample was stirred intermittently with the thermometer, and the temperature at which the material ceased to flow was observed. The sample was allowed to liquefy and the procedure repeated. The reproducibility of pour points was ±2° F. The contribution of impurities to observed pour points and the probability of supercooling were not fully explored.

Thermal stability screening tests were conducted as follows: Ten milliliters of test fluid was placed in a Type 410 stainless steel capsule (*8*) under a helium atmosphere. The capsule was welded shut and heated for 20 hours in a muffle furnace at 700° F. Gas evolution was measured by venting the gas to a vacuum flask at the conclusion of the test. The change in viscosity of the fluid was determined.

Irradiation of exploratory base stocks was performed at the Materials Testing Reactor (MTR) Canal Gamma Source, Arco, Idaho. The dose rate varied from 10^7 to 10^9 ergs per gram of carbon per hour. Ten milliliters of fluid was exposed in sealed, stainless steel capsules under a helium atmosphere. Each fluid was irradiated at about 60° F. at two dosage levels. Viscosity changes at 100° and 210° F. and the volume of gas evolved were measured after each exposure.

Oxidation-corrosion tests were conducted at 500° F. in an apparatus designed to one fourth the scale of Federal Standard Method No. 5308.4. Twenty-five milliliters of test fluid containing 2% of oxidation inhibitor was charged to a glass vessel equipped with an air bubbling tube. Various metal specimens in the form of washers were fitted about the bubbling tube and separated by glass collars. The vessel was heated for 48 hours in an aluminum block at 500° F. An air flow rate of 1.25 liters per hour was maintained. Standard inspections were made on end products and metal weight changes were determined.

A modified version of the Mean Hertz load test was used for screening purposes in evaluating the lubricating properties of selected base fluids. The procedure, equipment, and techniques were similar to those described in Federal Standard Method No. 6503. The load in kilograms required to give a 1.0-mm. scar was determined. Correlation of the screening procedure with full-scale Mean Hertz load tests had been previously established (*3*).

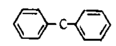
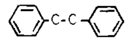
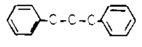
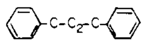
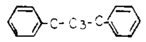
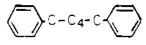
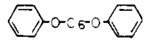
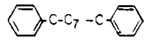
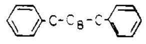
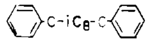
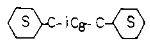
A hydraulic pump test was run on one fluid based on a substituted α,ω-diarylalkane. A New York Air Brake 66WA300 pump was used. The test was run at 3450 r.p.m. and 3000-p.s.i. output pump pressure for 250 hours (150 hours at 160° F., 100 hours at 275° F.). The test loop consisted of a pump, a Fulflo 10-micron, sintered stainless steel filter, a pressure-regulating valve, a heat exchanger, and a nitrogen-pressurized fluid reservoir. Pump inspections, including weighing of pump parts, were made at 0-, 150-, and 250-hour points. The weights of 1-inch metal specimens suspended in the reservoir were also determined.

STRUCTURE-PROPERTY CORRELATIONS

Unsubstituted α,ω-Diphenylalkanes. The effect of molecular structure on particular physical properties of unsubstituted α,ω-diphenylalkanes is shown in Table I. Certain generalizations are readily apparent. A linear relationship exists between viscosity and the number of carbon atoms of the normal paraffin bridge. Variable effects are observed in viscosity-temperature characteristics (V.I. and ASTM slope) and pour points. The V.I. decreases progressively as the number of coupling carbon atoms increases from one to four. A point of inversion occurs at 1,4-diphenylbutane; and higher molecular homologs exhibit a V.I. increase with increasing number of carbon atoms in the paraffin chain. Discontinuities in V.I. and in ASTM slope are illustrated in Figure 1.

Branching of the coupling paraffin at carbon atoms nonadjacent to the phenyl ring gives pronounced improvement in low temperature properties, but with an attendant sacrifice of V.I. Replacement of carbon with oxygen—e.g., 1,6-bis(phenoxy)-hexane—degrades both pour and V.I.

Table I. Physical Properties of α,ω-Diphenylalkanes

α,ω-Diphenylalkane	Viscosity, Cs., 210° F.	ASTM Viscosity Index	ASTM Slope (100–210° F.)	Pour Point, ° F.	Ref.
	0.97	116	0.873	77(m.p.)	(*7*)
	1.14	110[a]	0.862[a]	124(m.p.)	(*6, 7*)
	1.31	85	0.878	−5	(*7*)
	1.49	46[a]	0.909[a]	126(m.p.)	(*7*)
	1.64	90	0.836	18	(*7*)
	1.81	98	0.813	5–10	(*1, 7,* [b])
	2.62	−51[a]	0.928[a]	183(m.p.)	([b])
	2.36	125	0.745	20	([b])
	2.57	128	0.749	55–64	(1,[b])
 [c]	2.51	58	0.835	−70	([b])
	3.63	78	0.791	<−60	([b])

[a] Viscosity at 100° F. was extrapolated.

[b] Present work.

[c] Derived from commercial isosebacic acid (U.S.I.), approximate composition: 72–80% 2-ethylsuberic acid, 12–18% 2,5-diethyl adipic acid, 6–10% sebacic acid.

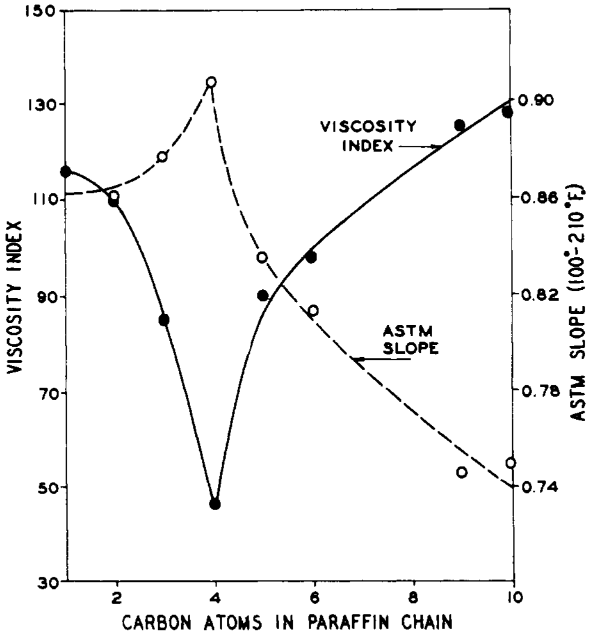
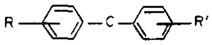


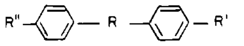
Figure 1. Viscosity index and ASTM slope as a function of *n*-paraffin chain length in α,ω-diphenylalkanes

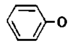
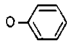
Table II. Influence of Alkyl Groups on Properties of Alkylated Diphenylmethanes



R	*R′*	Viscosity, Cs., 210° F.	ASTM Viscosity Index	ASTM Slope (100–210° F.)	Pour Point, ° F.
H	*sec*-C_{16}	4.46	115	0.743	−22
sec-C_4	*sec*-C_{12}	3.37	72	0.802	−58
sec-C_8	*sec*-C_8	2.66	84	0.800	−65
Mixed *sec*- C_6, C_8, C_{10}	Mixed *sec*- C_6, C_8, C_{10}	3.14	72	0.805	−60

Table III. Physical Properties of Symmetrically Substituted α,ω-Diphenylalkanes



R′′	R	R′	Viscosity, Cs., 210° F.	ASTM Viscosity Index	ASTM Slope (100°–210° F.)	Pour Point, ° F.
H	*n*-C_9	H	2.36	125	0.745	20
C_1	*n*-C_9	C_1	2.76	121	0.754	92(m.p.)
H	—C-isoC_8-C-	H	2.51	58	0.835	−70
C_1	—C-isoC_8-C-	C_1	3.76	14	0.846	−20
n-C_4	*n*-C_9	*n*-C_4	7.53[a]	127	0.678	<−60
sec-C_4	*n*-C_9	*sec*-C_4	7.72[a]	91	0.750	−35
tert-C_4	*n*-C_9	*tert*-C_4	8.44[a]	78	0.764	−30
Fused C_4[b]	*n*-C_9	Fused C_4[b]	11.4	57	0.786	−20
sec-C_6[c]	*n*-C_6	*sec*-C_6[c]	8.77	42	0.809	−25
	*n*-C_9		12.4	91[d]	0.714[d]	162(m.p.)
	*n*-C_9		10.6	97[d]	0.710[d]	104(m.p.)

[a] Fluid contaminated with ketone impurity (ca. 5%).
[b] Diarylalkane derived from Tetralin.
[c] Uncertain orientation.
[d] Viscosity at 100° F. was extrapolated.

Saturation of terminal aromatic rings (dicyclohexylisodecane) raises the viscosity and improves V.I.

Viscosity indices as high as 128 were obtained with the α,ω-diphenylalkanes. The relatively high aromatic content of these hydrocarbons made them promising radiation-resistant materials and prompted a further study of α,ω-diphenylalkane derivatives. A study was made of the influence of specific substituents on physical properties and on stability to various types of degradative processes.

Particular emphasis was placed on the improvement of low temperature properties of these dumbbell-type molecules.

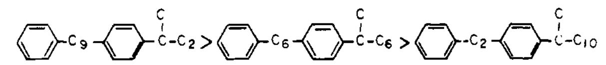
Alkylated Diphenylmethanes. Some alkylated diphenylmethanes were investigated and are shown in Table II along with certain physical properties. For a given alkyl-aryl carbon content, the dialkyl derivatives were superior in pour point, but inferior in V.I. to the monoalkyldiphenylmethane. Utilization of mixed alkyl groups did not materially alter the physical properties of the dialkylated base stocks.

Symmetrically Substituted α,ω-Diphenylalkanes. The viscosities, viscosity indices, and pour points of symmetrically substituted α,ω-diphenylalkanes which were examined are given in Table III. Methyl substituents raised the pour point and viscosity level without improving the V.I. Isomeric butyl groups on 1,9-diphenylnonane gave derivatives with apparently improved low temperature properties. (Ketonic impurities in these materials may have influenced observed pour points.) The pour point of the *n*-butyl homolog is indeed surprising. The descending order of V.I. and pour for the butyl series is:

$$nC_4 > sec\text{-}C_4 > tert\text{-}C_4 > \text{fused } C_4$$

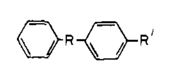
Phenyl and phenoxy substituents raised the pour point, as expected. Interest in these derivatives stemmed from their high aromatic content.

Unsymmetrically Substituted α,ω-Diphenylalkanes. In an attempt to develop fluids with improved low temperature properties, α,ω-diphenylalkanes were alkylated with several olefins. A comparison was made of alkylated products with approximately the same alkyl-aryl carbon content by varying the chain lengths of the alkylene bridge and the alkyl substituent. Beneficial effects were realized not only with respect to pour properties, but also to viscosity-temperature characteristics for the majority of alkylated diarylalkane derivatives under study. Improvement in these properties appeared to be a function of length of the alkylene bridge. The viscosity level and pour points increased and viscosity indices decreased in the following order:



These effects are summarized in Table IV along with properties of other alkylated derivatives. Insufficient data preclude a precise interpretation of structural transformations that have occurred. It is likely that disproportionation reaction products contribute to observed physical properties. The good viscosity indices, low pour points, and viscosities of isopropyl- and butyl-1,9-diphenylanonanes

Table IV. Influence of Alkyl Groups on Physical Properties of Unsymmetrically Substituted α,ω-Diphenylalkanes



R	*R′*	Viscosity, Cs., 210° F.	ASTM Viscosity Index	ASTM Slope (100–210° F.)	Pour Point, ° F.
n-C_2	H	1.14	110[a]	0.862[a]	124(m.p.)
n-C_2	*sec*-C_{12}	7.19	111	0.717	−35
n-C_6	H	1.81	98	0.813	5
n-C_6	*sec*C_8	4.13	112	0.750	−62
n-C_9	H	2.36	125	0.745	20
n-C_9	isoC_3	3.46	136	0.726	−70
n-C_9	*sec*-C_4	3.81	143	0.713	−68
n-C_9	*tert*-C_4	4.46	159	0.685	−70
-C-isoC_8-C-	H	2.51	58	0.835	−70
-C-isoC_8-C-	*n*-C_9	5.48	126	0.720	−35
-C-isoC_8-C-	*sec*-C_{14}	7.31	110	0.714	−50

[a] Viscosity at 100° F. was extrapolated.

make these materials particularly attractive as radiation-resistant hydraulic fluids.

EVALUATION OF DIARYLALKANE DERIVATIVES

Thermal and radiation stability screening tests were conducted on selected structural types of exploratory base fluids. Oxidation-corrosion and lubricating property tests were performed on compounds of special interest. A hydraulic pump test was run on one of the more promising α,ω-diarylalkane derivatives.

Thermal Stability. Gas evolution and viscosity change of fluids heated at 700° F. for 20 hours were used as a measure of relative thermal stability. The method used had several inherent disadvantages, but it was an expedient means for the preliminary evaluation of materials. Viscosity change was chosen as the main criterion for intercomparison of fluids.

Thermal stability data are summarized in Table V. Certain trends are indicated in thermal characteristics and structural differences, although precise correlations were not established. Branching or an increase in chain length of the aromatic-coupling paraffin had a deleterious effect on thermal properties. Marked improvement was observed in stability when the aromatic rings of diphenylisodecane were hydrogenated. The saturated derivative, dicyclohexylisodecane, was equivalent in stability to 1,10-diphenyldecane. Further measurable improvement was observed for the diphenylisodecane with an incorporated *n*-nonyl alkyl group. Variation in thermal properties was apparent in disubstituted (C_4) 1,9-diphenylnonanes. The *tert*-butyl and fused C_4 groups substantially increased the thermal stability of the parent 1,9-diphenylnonane. Normal and *sec*-butyl substituents decreased thermal stability. The order of stability based on per cent viscosity change was as follows: 1,9-bis(*tert*-butylphenyl)nonane > 1,9-bis(1,2,3,4-tetrahydronaphthyl)nonane > 1,9-diphenylnonane > 1,9-bis(*n*-butylphenyl)nonane > 1,9-bis(*sec*-butylphenyl)nonane. Phenyl- and phenoxy-substituted analogs were nearly equivalent in stability, but were inferior to the parent compound.

Radiation Stability. The majority of fluids irradiated in an inert atmosphere at about 60° F. showed less than a 10% viscosity gain at 100° F. after a dosage of 1.0×10^{10} ergs per gram of carbon. Substantial differences in viscosity change were observed with additional exposure up to 6.0×10^{10} ergs per gram of carbon. Gas evolution was a function of dosage level as well. The average volume of gas evolved for diarylalkanes exposed to 1.0×10^{10} ergs per gram of carbon was about 1 ml. per gram of fluid; exposure to 4.0 to 6.0×10^{10} ergs per gram of carbon, about 3 ml. per gram of fluid. Dicyclohexylisodecane, on the other hand, gave excessive gas (16 ml. per gram of fluid) at 5.8×10^{10} ergs per gram of carbon.

Since test samples had different initial viscosities and received variable radiation dosages, an index of damage, η_{10} (*4*) was adopted to permit an intercomparison of the relative radiation resistance of materials at a given dosage level. The index of damage is defined as the viscosity in centistokes at that temperature, t_{10}, at which the viscosity of the original material was 10 centistokes. Figure 2 depicts the manner in which these values were obtained from measured viscosities at 100° and 210° F. for both irradiated and nonirradiated fluids. Coordinates of the plot are those used in ASTM Chart D 341 for petroleum products. Individual η_{10} values at two dosages were determined for the compounds tested. Logarithmic plots of these values as a function of dosage were made, and a dosage of 5.0×10^{10} ergs per gram of carbon was chosen for the interrelation of all fluids. Linearity was previously shown in a similar plot

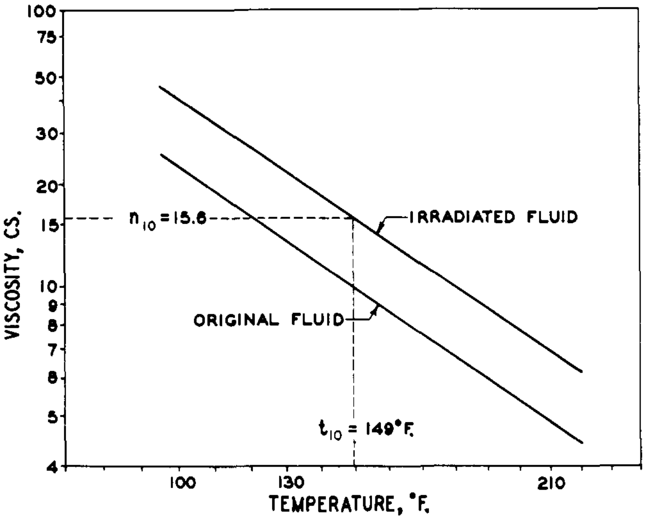


Figure 2. Determination of η_{10} for irradiated diarylalkanes of different initial viscosity

Table V. Thermal and Radiation Stability of Diarylalkane Base Fluids

Base Fluid	Thermal Stability, Capsule, 20 Hr. at 700° F.			Radiation Stability, Dosage 5.0×10^{10} Ergs/G. Carbon, Index of Damage[a]
	% Viscosity loss		Gas evolved, ml./g. fluid	
	100° F.	210° F.		
1,9-Bis(*tert*-butylphenyl)nonane	5	2	17.0	12.8
1,6-Diphenylhexane	6	3	0.3	12.2
1,9-Bis(1,2,3,4-tetrahydronaphthyl)nonane	16	9	1.5	12.7
Phenyl(*n*-nonylphenyl)isodecane	21	12	1.2	13.8
Isopropyl-1,9-diphenylnonane	26[b]	15[b]	1.0[b]	13.5
1,10-Diphenyldecane	29	16	4.2	14.1
Dicyclohexylisodecane	31	16	1.5	16.0
1,9-Diphenylnonane	38	25	2.3	12.4
1,9-Bis(*n*-butylphenyl)nonane	48	36	60.0	11.2
1,9-Bis(phenoxyphenyl)nonane	53	37	1.7	11.8
1,9-Bis(biphenylyl)nonane	56	44	3.5	10.2[c]
1,9-Bis(*sec*-butylphenyl)nonane	74	54	8.0	12.3
Diphenylisodecane	79	55	16.0	11.2
1,6-Bis(*sec*-hexylphenyl)hexane	89	63	33.0	12.6
Phenyl(*sec*-tetradecylphenyl)isodecane	91	76	14.0	12.1

[a] Viscosity (cs.) at that temperature (t_{10}) at which the viscosity of the original material was 10 cs.

[b] Six-hour test.

[c] Solid at exposure temperature.

Table VI. Oxidation-Corrosion Characteristics of Diarylalkanes

(48 hr., 500° F.; 1.25 l. air/hour)

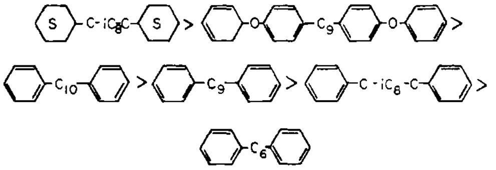
DBPC = Di-*tert*-butyl-*p*-cresol TS = Tridecyl selenide

Diarylalkane	Inhibitor Type	Inhibitor %	Viscosity Change, % 100° F.	Viscosity Change, % 210° F.	Neutralization No.	Metal Weight Change, Mg./Sq. Cm. Al	Steel	Cu	Ag	Be-Cu	Insolubles, %	Evaporation, %
1,6-Diphenylhexane	DBPC	2	...	+3060	32.3	−0.06	−0.06	−2.42	0.00	−1.77	...	5.38
1,9-Diphenylnonane	DBPC	2	+1720	+448	17.2	0.00	−15.1	−0.96	−0.03	−0.51	14.6	4.31
1,10-Diphenyldecane	DBPC	2	+178	+90.0	8.23	0.00	−3.75	−0.96	−0.06	+0.03	0.84	1.27
Diphenylisodecane	DBPC	2	+6380	+933	14.4	−0.09	+7.40	−0.87	−0.16	−0.45	13.3	19.1
Dicyclohexylisodecane	DBPC	2	+9.68	+7.17	6.24	−0.16	+0.03	−0.09	−0.16	−0.06	7.97	0.49
1,9-Bis(biphenylyl)nonane	DBPC	2	(Solid)	+52.4	4.95	−0.09	−0.12	−0.32	−0.06	−0.03	2.68	0.00
1,9-Bis(phenoxyphenyl)nonane	(None)	...	(Solid)	(Solid)	...	...	...	...	...	...	...	1.20
	TS	5	−21.2	−10.8	1.27	0.00	−0.71	−26.8	−86.0	−19.9	1.80	0.57
1,9-Bis(1,2,3,4-tetrahydronaphthyl)nonane	DBPC	2	+216	+83.3	3.61	0.00	−0.03	−0.06	−0.03	0.00	1.90	0.00
1,9-Bis(*n*-butylphenyl)nonane	DBPC	2	+212	+93.0	11.0	−0.13	−0.10	−0.13	0.00	+0.29	(Trace)	...
1,9-Bis(*tert*-butylphenyl)nonane	DBPC	2	...	+961	15.5	−0.03	−3.29	−0.25	−0.22	+0.16	0.50	1.50
Isopropyl-1,9-diphenylnonane	(None)	...	+2440	+597	11.9	−0.31	−5.30	−4.03	+0.62	−2.46	0.90	8.4
	DBPC	2	+613	+222	10.6	0.00	−0.30	−2.17	+1.24	+7.44	0.40	0.7

for butylbenzene, within the region of dosage of current concern (*5*).

The comparative radiation stability of test fluids is shown in Table V. The lower the η_{10} value, the greater the resistance to radiation damage. The apparent superiority of 1,9-bis(biphenylyl)nonane must be qualified, since this material was solid at the exposure temperature. The stability of diphenylisodecane was significantly depreciated by the conversion of aromatic to naphthenic rings. Relative resistance to radiation damage was not strictly a matter of per cent aromatic content. Factors such as position and type of alkyl attachments and molecular configuration are also important.

Oxidation-Corrosion. Quarter-scale oxidation-corrosion tests at 500° F. were performed only on selected fluids. Di-*tert*-butyl-*p*-cresol and tridecyl selenide were used as oxidation inhibitors. In general, corrosion of metals was low; viscosity changes were large. Test results are shown in Table VI. Oxidative stability of the diarylalkanes decreased with decreasing chain length of the coupling paraffin. Terminal phenoxy substituents on 1,9-diphenylnonane substantially increased oxidative stability. Hydrogenation of aromatic rings in diphenylisodecane likewise enhanced the stability of the diarylalkane. The observed order of decreasing oxidation stability of the inhibited fluids was as follows:



Lubricating Properties. Modified Mean Hertz load screening test results on the dumbbell-type hydrocarbons indicated low-to-good film strength characteristics. Values (determined from the load in kilograms required to give a 1.0-mm. scar) ranged from 10 to 23 with 1,9-bis(phenoxyphenyl)-nonane showing the best lubricating properties of fluids

Table VII. Wear Data of Isopropyl-1,9-Diphenylnonane Formulation in New York Air Brake Hydraulic Pump Test Model 66WA300

(Pressure, 3000 p.s.i.; full delivery, 3450 r.p.m.)

Pump Part	Wear (Weight Loss, Mg.) after: 150 hr., 160° F.	100 hr., 275° F.
Nutating plate	2.5	4.5
Nutating plate pivot	2.0	0.3
Creep plate	1.4	1.1
Bronze creep plate bearing	162.2	10.5
Nine pistons		
Av.	2.7	4.2
Max.	6.0	7.9
Nine piston return collars		
Av.	0.5	1.0
Max.	0.7	1.7
Nine piston sleeves		
Av.	0.5	0.3
Max.	0.7	0.9
Nine check valves		
Av.	0.6	1.0
Max.	0.8	2.0
Corrosion specimens in reservoir		
Copper	0.3	0.3
Beryllium-copper	0.4	0.3
Steel	0.4	0.0
Aluminum	0.4	(+0.8)
Silver	0.6	0.5

Table VIII. Physical Properties and Stability Data of Structurally Different Base Stocks

Fluid	Viscosity, Cs.		ASTM Viscosity Index	Pour Point, ° F.	Thermal Stability, 20 Hr., 700° F.		Oxidation Corrosion 48 Hr., 500° F., % Change, V_{100}	Radiation Stability, 8.7×10^{10} Ergs/G. Carbon % Gain, V_{100}
	100° F.	210° F.			% loss, V_{100}	Gas evolved, ml./g.		
Disiloxane[a] (Oronite 8200)	31.6	11.0	179	<−80	90	74	−36.6	510[b]
Naphthenic white oil	70.5	7.44	65	−30	67	5.4	(Solid)	1070
Polyglycol (UCON DLB 144E)	30.5	7.10	167	−65	(Very large)	(Very high)	−18.0	1000
Di-2-ethylhexyl sebacate	12.7	3.34	155	<−80	(Solid)	...	+1180	220[c]
Fluorolube	81.4	5.40	−200	...	...	...	[d]	330(*4*)
$C_{16\text{-}18}$ *sec*-alkylbiphenyl	36.5	5.27	77	−60	79	8.9	+208	110
$C_{14\text{-}16}$ *sec*-alkyldiphenyl ether	26.8	4.85	115	−25	77	4.0	+229	140
Isopropyl-1,9-diphenylnonane[e]	23.1	4.88	150	−65	16[f]	0.9	531[g]	140

[a] Compounded fluid.

[b] Extrapolated value.

[c] Contained 2% dodecyl selenide.

[d] Not measured, but assumed to be small.

[e] Contained disproportionated polymer.

[f] Six-hour test.

[g] With 95% N_2, 5% O_2.

tested. Nonadditive petroleum oils fall in this same range.

Four-ball wear tests (2 hours, 10 kg., 1200 r.p.m.) were conducted on one of the more promising α,ω-diarylalkane derivatives. Wear values for isopropyl-1,9-diphenylnonane were found to be less than 0.5 mm. at 167° and 275° F., but over 0.9 mm. at 400° F. Introduction of 2% of tricresyl phosphate into the fluid lowered the wear at 400° F. to about 0.5 mm.

A New York Air Brake 66WA300 pump test was performed on a fluid formulation based on isopropyl-1,9-diphenylnonane. The fluid tested contained 5% of a polyalkylaromatic viscosity-temperature improver, 2% of di-*tert*-butyl-*p*-cresol oxidation inhibitor, and 0.001% of a silicone antifoam agent. Table VII summarizes the wear data from the run. Buna N O-rings and all parts of the pump were in excellent condition. A 30% viscosity loss at 210° F. was observed within the first 50 hours of pump operation; little viscosity change occurred in the succeeding 200 hours of the test. The initial drop in viscosity is attributed primarily to shear breakdown of the viscosity-temperature improver.

COMPARISON OF SYNTHETIC FLUIDS

Certain diarylalkane derivatives, particularly unsymmetrically substituted diphenylalkanes, possess many functional properties desirable in hydraulic fluids. Table VII compares these materials with other types of synthetic oils.

Disiloxane and polyglycol fluids have superior viscosity-temperature characteristics and pour properties and fair-to-good oxidation stability. Under the extreme conditions of high radiation dosage or high temperature, they are susceptible to breakdown. Gas formation at 700° F. is excessive.

Naphthenic white oil (a highly purified petroleum fraction) has a comparatively low V.I., but good thermal stability. Oxidation and radiation stability are poor.

The dibasic acid ester, di-2-ethylhexyl sebacate, has good physical properties, but is deficient in thermal and oxidation stability.

The fluorolube, poly(trifluorovinyl chloride), has fair radiation stability and probably good resistance to oxidation. Its viscosity-temperature relationship is unattractive for hydraulic fluid applications.

$C_{16\text{-}18}$ *sec*-alkylbiphenyl, $C_{14\text{-}16}$ *sec*-alkyldiphenyl ether, and isopropyl-1,9-diphenylnonane are fluids with relatively high aromatic content. The alkyl substituted biphenyl has good stability to radiation damage, oxidation, and thermal degradation; but its viscosity-temperature characteristics are poor. The diphenyl ether derivative has comparable stability and improved V.I. properties. Its pour point is high, however. The isopropyl-1,9-diphenylnonane fluid is somewhat less stable to oxidation than the above two materials, but compares favorably in thermal and radiation stability. The V.I. characteristics and low temperature properties are superior.

CONCLUSIONS

The preliminary evaluation of pertinent properties and stabilities of fluids with diverse molecular structures establishes the substituted diarylalkanes as very promising base stocks for hydraulic fluids. In addition to having desirable physical properties, they possess a high order of oxidation, thermal, and radiation stability. A formulated fluid based on isopropyl-1,9-diphenylnonane operates satisfactorily in an aircraft piston pump. This fluid is expected to have an applicable temperature range from −20° to 500° F. and to be usable for short periods of time up to 700° F., although it has been tested in mechanical systems operating at the extremes of temperature. It is capable of withstanding gamma dosages as high as 5 to 10 × 10^{10} ergs per gram of carbon without major change. The evaluation of this fluid in dynamic in-source tests over the indicated temperature range is desirable to qualify further its usefulness as a high temperature, nuclear radiation-resistant hydraulic fluid.

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