Esters of Aromatic Alcohols and Dibasic Acids as Base Stocks for Nonspreading Oil Compositions

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The need for a stable nonspreading lubricant for military instruments intended for use at low temperatures stimulated experimental work leading to the development of stable nonspreading organic fluids (*1–4,12*). At present only one material, benzyl phenylundecanoate (*4*), has been found promising for this purpose, and it requires a diluent in order to be useful at even moderately low temperatures. Additional work on the development of low temperature nonspreading fluids was undertaken in this laboratory in order to make available additional compounds and to attempt to obtain lubricants with improved low temperature properties over blends containing benzyl phenylundecanoate.

From the information available on diesters and nonspreading compounds, it seemed likely that improved nonspreading

Table I. Intermediates

Material	Source	Grade	Boiling Pt. Range, °C.	Pressure, Mm.	Melting Point, °C.	Refractive Index, n_D^{20}
Ethylene glycol phenyl ether	Dow Chemical Co.	Special[a]	132.0–133.0	18	...	1.53873
Octylphenol	Rohm and Haas Co.	Commercial	...	...	84.0–85.0	...
Propylene glycol phenyl ether	Dow Chemical Co.	Special	128.2–130.0	19	...	1.52397
Propylene glycol *p-tert*-butylphenyl ether	Dow Chemical Co.	Special	134.0–135.0	2.8	43.7–45.8	...
Ethylene glycol *p-tert*-butylphenyl ether	Dow Chemical Co.	Special	146.0–149.0	5	...	1.51866
Adipic acid	Distillation Prod. Ind.	Eastman	...	...	149.6–150.6	...
Azelaic acid	Emery Ind., Inc.	Commercial	...	...	104.0–105.2	...
Pimelic acid	Distillation Prod. Ind.	Eastman	...	...	105.1–105.6	...
Suberic acid	Bios Laboratories	...	...	...	139.8–140.8	...
3-Methyladipic acid	Matheson Coleman and Bell	Research	...	...	94.5–96.0	...
p-Isopropylbenzyl alcohol	Distillation Prod. Ind.	Eastman	134.5–136.2	19	...	1.52096
Benzyl alcohol	Merck & Co.	Reagent	104.0–105.0	20	...	1.53940
Propylene glycol *p-sec*-phenyl ether	Dow Chemical Co.	Special	123.0–124.0	2.2	...	1.50734
Dipropylene glycol phenyl ether	Dow Chemical Co.	Special	125.0–127.0	2.3	...	1.50327
m-Methylbenzyl alcohol	Matheson Coleman and Bell	Research	67.9–68.0	1.2	...	1.53684
DL-1-Methylbenzyl alcohol	Distillation Prod. Ind.	Eastman	50.0–50.3	0.85	...	1.52835
2-Phenylpropanol	Delta Chemical Works	...	81.5–82.2	1.2	...	1.52573

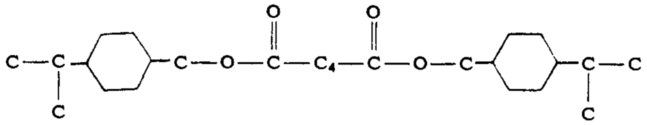
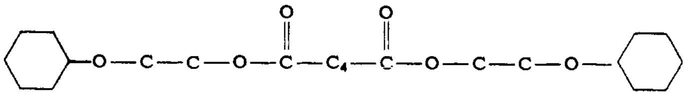
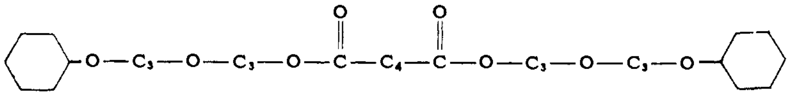
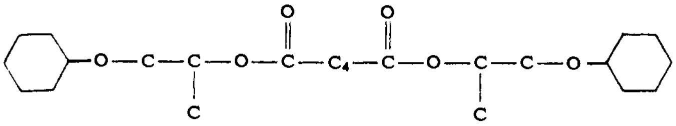
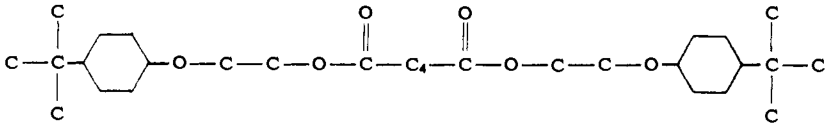
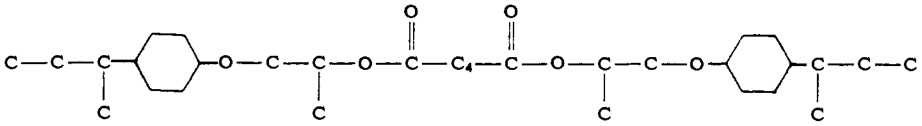
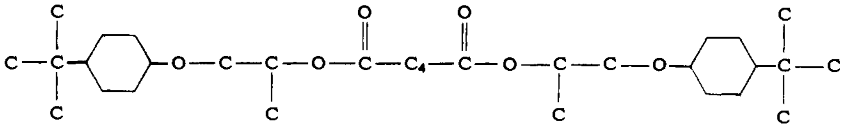
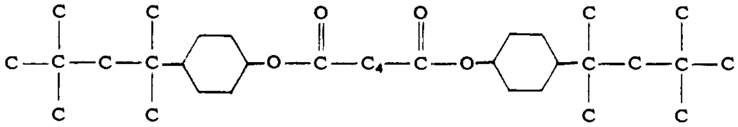
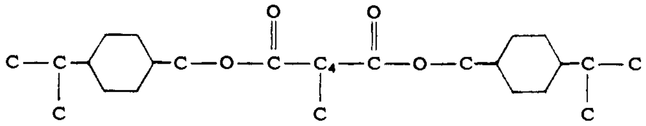
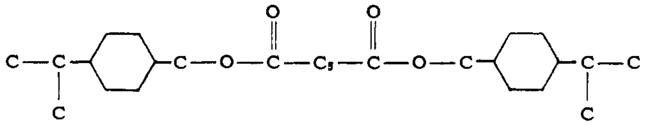
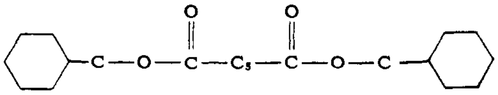
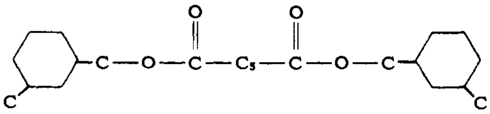
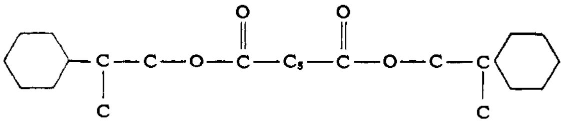
[a] All Dow Chemical Co. compounds, Special Grade, are single compounds of 98% purity or better.

Table II. Preparation of Esters

Adipates	Alcohol, Moles	Acid, Moles	Reflux Time, Hr.	H_2O Collected, Moles	Saponification Equivalent		Carbon, %		Hydrogen, %	
					Found	Calcd.	Found	Calcd.	Found	Calcd.
Bis(*p*-isopropylbenzyl) adipate	0.50	0.20	10	0.42	207.4	205.3	76.30	76.06	8.07	8.35
Bis(2-phenoxyethyl) adipate	1.50	0.65	10	1.28	194.8	193.2	68.40	68.37	6.46	6.78
Bis[3-(3-phenoxypropoxy)propyl] adipate	0.50	0.20	16	0.42	265.5	265.3	68.13	67.92	8.09	7.97
Bis(1-methyl-2-phenoxyethyl) adipate	1.50	0.65	20	1.25	203.2	207.3	69.80	69.54	7.01	7.29
Bis(2-*p-tert*-butylphenoxyethyl) adipate	1.10	0.50	10	0.97	253.8	249.4	72.49	72.25	8.07	8.49
Bis[1-methyl-2(*p-sec*-butylphenoxy) ethyl] adipate	1.00	0.40	15	0.78	273.0	263.4	73.14	72.96	8.90	8.81
Bis[1-methyl-2(*p-tert*-butylphenoxy) ethyl] adipate	0.50	0.20	15	0.42	266.5	263.4	73.12	72.96	9.02	8.81
Bis[*p*-(1,1,3,3-tetramethylbutyl) phenyl] adipate	0.48	0.20	50	0.31	258.6	261.4	78.46	78.11	9.37	9.64
Bis(*p*-isopropylbenzyl)3-methyladipate	0.50	0.21	4	0.42	210.5	212.3	76.35	76.38	8.48	8.55
Pimelates										
Bis(*p*-isopropylbenzyl) pimelate	0.90	0.30	5	0.67	221.6	212.3	76.90	76.42	8.28	8.55
Dibenzyl pimelate	0.40	0.15	4	0.31	171.3	170.2	74.11	74.09	7.03	7.11
Bis(*m*-methylbenzyl) pimelate	0.75	0.30	4	0.61	188.3	184.3	75.65	74.95	7.75	7.66
Bis(2-phenylpropyl) pimelate	0.55	0.25	55	0.50	197.2	198.3	75.80	75.72	8.19	8.14
Suberates										
Bis(*p*-isopropylbenzyl) suberate	0.60	0.25	5	0.56	221.6	219.3	76.70	76.67	8.63	8.73
Dibenzyl suberate	0.45	0.20	5	0.41	182.5	177.2	74.58	74.55	7.26	7.39
Azelates										
Bis(*p*-isopropylbenzyl) azelate	0.30	0.13	4	0.26	226.3	228.9	76.95	76.96	9.02	8.90
Dibenzyl azelate	1.50	0.65	6	1.43	189.2	184.3	74.98	74.97	7.69	7.66
Bis(*m*-methylbenzyl) azelate	0.80	0.28	4	0.57	205.1	198.3	75.78	75.73	7.90	8.14
Bis(2-phenylpropyl) azelate	0.55	0.25	5	0.50	215.7	212.3	76.58	76.38	8.60	8.55
Bis(1-methylbenzyl) azelate	0.82	0.35[a]	...	...	199.5	198.3	75.65	75.72	7.98	8.14
Bis(1-methyl-2-phenoxyethyl) azelate	1.50	0.65	12	1.33	226.3	228.3	71.24	71.02	7.98	7.95
Bis(2-*p-tert*-butylphenoxyethyl) azelate	0.50	0.23	15	0.43	278.9	270.4	73.52	73.28	9.01	8.95
Bis[1-methyl-2(*p-sec*-butylphenoxy) ethyl] azelate	0.24	0.10	8	0.20	283.1	284.4	74.14	73.91	9.25	9.21
Bis[1-methyl-2(*p-tert*-butylphenoxy) ethyl] azelate	0.45	0.18	8	0.36	285.7	284.4	73.98	73.91	9.26	9.21

[a] Azelayl chloride.

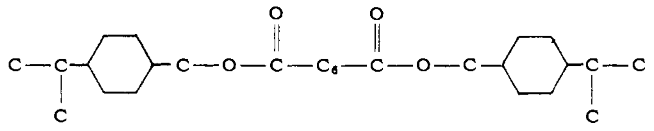
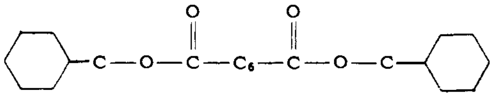
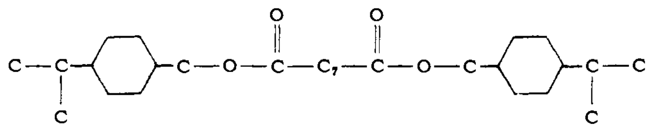
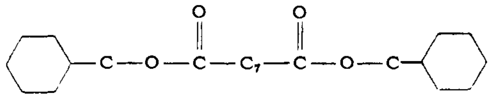
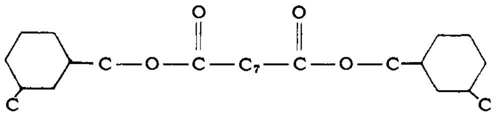
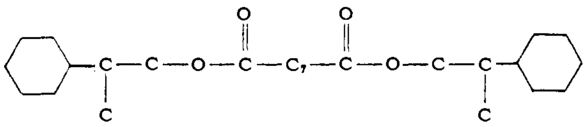
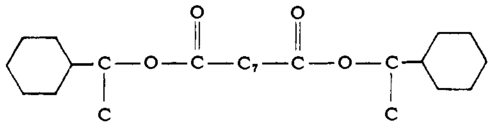
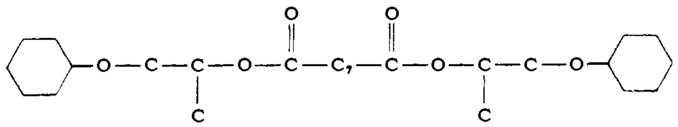
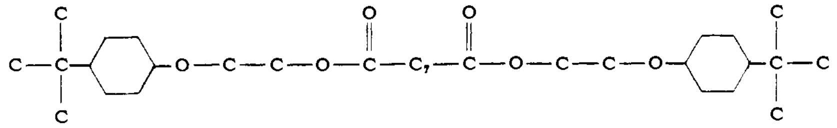
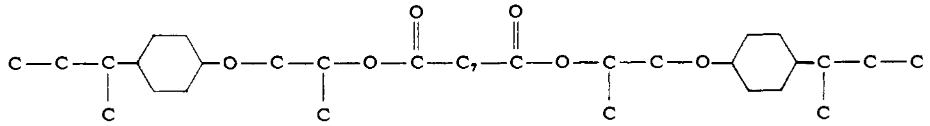
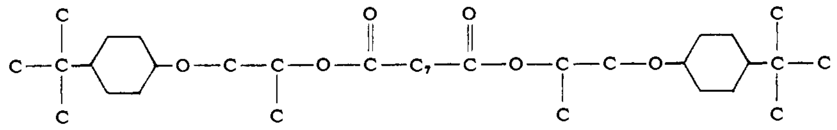
Table III.

Adipates	Structure
Bis(*p*-isopropylbenzyl) adipate	
Bis(2-phenoxyethyl) adipate	
Bis[3-(3-phenoxypropoxy)propyl] adipate	
Bis(1-methyl-2-phenoxyethyl) adipate	
Bis(2-*p*-*tert*-butylphenoxyethyl) adipate	
Bis[1-methyl-2-(*p*-*sec*-butylphenoxy)ethyl] adipate	
Bis[1-methyl-2-(*p*-*tert*-butylphenoxy)ethyl] adipate	
Bis[*p*-(1,1,3,3-tetramethylbutyl)phenyl] adipate	
Bis(*p*-isopropylbenzyl)3-methyladipate	
Pimelates	
Bis(*p*-isopropylbenzyl) pimelate	
Dibenzyl pimelate	
Bis(*m*-methylbenzyl) pimelate	
Bis(2-phenylpropyl) pimelate	

Properties of Esters

Calculated Molecular Weight	Neut. No.	Refractive Index, n_D^{20}	Density, d_4^{25}	Surface Tension, Dynes/Cm.	Melting Point, °C.	Pour Point, °F.	Viscosity, Cs.				% Spreading after	
							100° F.	0°F.	−30° F.	−40°F.	7 days	30 days
410.5	0.00	...	...	...	29.2–30.3	...	...	...	...	...	...	...
386.4	0.00	...	...	...	92.2–92.9	...	...	...	...	...	...	...
530.5	0.08	1.50498	...	...	...	−5	66.1	Viscous	...	...	9	30
414.5	0.00	...	...	...	83.0–84.2	...	...	...	...	...	...	...
498.7	0.00	...	...	...	57.7–59.1	...	...	...	...	...	...	...
526.7	0.08	1.50816	...	...	...	0	161.0	Viscous	...	...	1	8
526.7	0.17	...	...	...	77.3–78.3	...	...	...	...	...	...	...
522.8	0.00	...	...	...	73.4–74.2	...	...	...	...	...	...	...
424.6	0.08	1.51988	1.0325	36.6	...	−15	34.9	5,170	Viscous	...	5	115
424.5	0.08	1.52083	1.0334	37.5	...	−35	30.2	2,060	39,200	...	0	1
340.4	0.02	1.52978	1.0947	44.0	...	−30	13.6	360	Solid	...	0	0
368.5	0.01	1.52939	1.0729	42.3	...	−50	20.5	1,310	...	70,000	2	3
396.5	0.04	1.51816	1.0419	43.4	...	−55	24.3	928	8,580	21,900	0	0

Table III.

Suberates	Structure
Bis(*p*-isopropylbenzyl) suberate	
Dibenzyl suberate	
Azelates	
Bis(*p*-isopropylbenzyl) azelate	
Dibenzyl azelate	
Bis(*m*-methylbenzyl) azelate	
Bis(2-phenylpropyl) azelate	
Bis(1-methylbenzyl) azelate	
Bis(1-methyl-2-phenoxyethyl) azelate	
Bis(2-*p*-*tert*-butylphenoxyethyl) azelate	
Bis[1-methyl-2(*p*-sec-butylphenoxy) ethyl] azelate	
Bis[1-methyl-2(*p*-*tert*-butylphenoxy) ethyl] azelate	

base fluids could be achieved by the introduction of suitable aromatic groups in the diester molecules. Accordingly, a series of diesters were prepared from adipic, pimelic, suberic, and azelaic acids and aromatic alcohols, such that the diesters possess a "dumbbell" structure—i.e., a benzene ring at each end of the longest straight chain. Most of these compounds were found to be nonspreading.

The explanations for nonspreading have been discussed (*7,10,14*) and more recently by Bielak and Mardles (*5*) and Zisman and others (*8*). There appears to be agreement that a lower energy film must first be adsorbed upon the metal surface which retards further spreading. The minute traces of adsorbable impurities present or formed in the fluid upon contact with the metal surface can form this film. Zisman and others (*8*) specifically treat some "dumbbell"-type esters and conclude that these esters will produce, in effect, a mixed molecular film of phenyl groups and dibasic acid and that further spreading is retarded because the surface tension of the oil exceeds the critical surface tension—i.e., the surface tension below which spreading will

(Continued)

Calculated Molecular Weight	Neut. No.	Refractive Index, n_D^{20}	Density, d_4^{25}	Surface Tension, Dynes/Cm.	Melting Point, °C.	Pour Point, °F.	Viscosity, Cs.				% Spreading after	
							100° F.	0° F.	−30° F.	−40° F.	7 days	30 days
438.6	0.07	1.51780	1.0267	37.8	...	−40	31.5	2,010	>30,000	...	0	0
354.4	0.06	1.52622	1.0804	43.2	...	+20	14.7	Solid	...	...	0	0
452.6	0.02	1.51503	1.0185	37.5	...	−50	29.8	1,240	12,200	...	0	0
368.5	0.02	1.52361	1.0702	42.3	...	+ 5	15.3	Solid	...	...	0	0
396.5	0.08	1.52308	1.0511	41.5	...	−55	23.1	1,180	...	44,400	0	0
424.6	0.02	1.51394	1.0326	42.8	...	−55	28.0	1,020	7,900	18,900	0	0
396.5	0.06	1.51460	...	...	...	−35	25.4	713	9,970	...	4	4
456.6	0.00	1.51380	...	...	...	− 5	80.0	Viscous	...	...	1	1
540.8	0.00	1.51339	...	...	...	+5	314.0	Viscous	...	...	1	1
568.8	0.02	1.55167	...	...	...	+30	195.0	Viscous	...	...	0	0
568.8	0.05	1.50645	...	...	...	+50	470.0	Viscous	...	...	1	1

occur on this film. In conformance with the hypothesis proposed by Zisman (*8*), because the most adsorbable products of the hydrolysis of the diesters described in this report are the alcohol, the free acid, and the mono ester, the spreadability of the diester will depend on its ability to spread over a layer of the aforementioned materials. The critical surface tension of the low energy film formed by adsorption of the products of hydrolysis is estimated as 35 dynes per cm. by Zisman and others. As the surface tensions of the low temperature fluids reported here (Table III) are between 36.6 and 44.0, they should be nonspreading fluids. The starting materials were selected with a small degree of branching in order to achieve low pour or freezing points and minimum temperature coefficients of viscosity in addition to nonspreading.

MATERIALS

The source and grades of intermediates are in Table I. Pimelic and 3-methyladipic acids were both recrystallized from benzene. The azelaic acid was recrystallized from benzene, then from water using Nuchar, and finally from water alone. The adipic acid and the octylphenol were recrystallized, respectively, from water and low boiling petroleum ether. The propylene glycol *p-tert*-butylphenyl ether and all of the other liquid materials were fractionally distilled before use. The suberic acid was used as received. Melting and boiling point ranges given in Table I are of the purified materials except that of suberic acid, which represents that of the material as received.

PREPARATION OF ESTERS

All of the diesters, except bis(1-methylbenzyl) azelate, were prepared in the same manner. A typical preparation is that of bis(*p*-isopropylbenzyl) azelate. Forty-five grams (0.3 mole) of *p*-isopropylbenzyl alcohol, 25 grams (0.13 mole) of azelaic acid, 100 ml. of toluene, and 0.5 gram of *p*-toluenesulfonic acid monohydrate were placed in a 500-ml., 3-necked flask fitted with a stirrer, water trap, and reflux condenser. This mixture was refluxed for 4 hours, during which time 4.7 ml. of water were collected. Toluene and excess alcohol were then removed by distillation using a water aspirator, then an oil pump. The reaction mixture was mixed with 2 grams of powdered anhydrous C.P. potassium carbonate, warmed to 50° C. for several hours with occasional shaking, permitted to stand at room temperature overnight, and filtered. It was then twice distilled in a short path still at 20 microns.

Bis(1-methylbenzyl) azelate was prepared by the reaction of azelayl chloride and 1-methylbenzyl alcohol at 0° C. To a 500-ml., 3-necked flask fitted with a stirrer, dropping funnel, and a reflux condenser with a drying tube were added 79.7 grams (0.35 mole) of azelayl chloride (prepared from azelaic acid and thionyl chloride). Maintaining the flask at 0° C. with an ice-water bath, 100 grams (0.8 mole) of 1-methylbenzyl alcohol were added dropwise over a period of 6 hours. Thirty milliliters of dry pyridine were then slowly added over a period of 2 hours while the temperature was maintained at 0° C. Excess alcohol and pyridine were removed by successive distillations using a water aspirator and a vacuum pump. Two hundred milliliters of ether were added and the ether solution was washed once with 2% sodium hydroxide and then with water until neutral. The ether layer was dried with anhydrous sodium sulfate and distilled, using a water aspirator. Two grams of powdered anhydrous potassium carbonate were then added, and the mixture was warmed for several hours with occasional shaking at 50° C., permitted to stand at room temperature overnight, and filtered.

Attempts to determine saponification equivalent by the usual techniques using ethyl alcohol or diethylene glycol for preparing the potassium hydroxide reagent were unsuccessful. The following procedure was used. Approximately 2 grams of ester were weighed in a 125-ml. Erlenmeyer flask; 20 ml. (accurately measured) of 1*N* potassium hydroxide dissolved in ethylene glycol were added; a reflux condenser was placed on the flask and the mixture was refluxed gently for 45 minutes. Two drops of phenolphthalein indicator solution were added and the contents of the flask were titrated with 0.25*N* hydrochloric acid. All determinations were run in duplicate and blank determinations were made simultaneously. Neutralization numbers were determined by ASTM method D 974-51T.

PHYSICAL PROPERTIES OF ESTERS

Physical characteristics of the diesters are in Table III. Pour point and viscosity were determined in accordance with ASTM methods D 97-47 and D 439-40T, respectively. Density was determined in accordance with ASTM method D 941-47T. Surface tension was measured using Sugden's maximum bubble pressure method (*13*). Spreading of the oil was determined in the following manner: One-inch diameter disks of WD 52-100 steel hardened to Rockwell C62 were polished successively with 0, 2/0, 3/0, and 4/0 emery paper, and washed under warm tap water while being scrubbed with cotton gauze, then washed successively with distilled water and redistilled methanol. The disks were dried by clean air. A drop of redistilled methanol was placed on the surface of each disk. Complete and uniform evaporation indicated acceptable surfaces. A drop of oil, 1 to 2 mm. in diameter, was placed on each disk by means of a platinum wire. The degree of spreading was obtained by measuring the change in diameter after 7 and 30 days, respectively, using a traveling microscope.

The data in Table III show that all of the pimelates, suberates, and azelates spread not more than 4% in 30 days. The adipate esters are mostly solid at room temperature; the few which are fluid are very viscous and exhibit some tendency to spread. Comparison of the data on dibenzyl and bis(*p*-isopropylbenzyl) suberate with analogous azelates and pimelates indicates that better low temperature properties can be attained in diesters prepared from dibasic acids having an odd number of carbon atoms in the longest aliphatic chain. The melting points of the acids themselves are in accordance with the generalization that compounds with an odd number of carbon atoms in an aliphatic homologous series possess lower melting points, and this trend also appears in the pour points of the esters.

Data available in the literature (*6,11,12*) indicate that ether linkages in a molecule do not materially affect the viscosity-temperature characteristics of a compound—i.e., the ether oxygen atom behaves very much like a carbon atom in a straight chain. Based upon this consideration and because they were readily available, the phenyl ether alcohols were chosen for some of the work described here. It was found, however, that compounds containing these alcohols have higher viscosities and pour points than when the oxygen is replaced by a carbon atom. For example, bis(*p*-isopropylbenzyl) azelate and bis(2-phenylpropyl) azelate have markedly lower pour points and viscosities than bis(1-methyl-2-phenoxyethyl) azelate, although they are structurally similar and have comparable molecular weights.

Table IV. Composition of Blends Tested

A	% by Weight
Bis(*p*-isopropylbenzyl) azelate	59.95
Diethylene glycol dicaproate	39.45
Dodecyl piperidine stearate	0.40
p-tert-Butyl catechol	0.20
B	
Bis(*p*-isopropylbenzyl) azelate	50.00
Diethylene glycol bis(3,5,5-trimethyl hexanoate)	49.30
Phenyl-1-naphthylamine	0.30
Dodecyl piperidine stearate	0.40
C	
Bis(2-phenylpropyl) azelate	59.85
Diethylene glycol bis(3,5,5-trimethyl hexanoate)	39.45
Dodecyl piperidine stearate	0.40
Phenyl-1-naphthylamine	0.30
D	
Bis(2-phenylpropyl) pimelate	59.85
Diethylene glycol bis(3,5,5-trimethyl hexanoate)	39.45
p-tert-Butyl catechol	0.30
Dodecyl piperidine stearate	0.40
E	
Benzyl phenyl undecanoate	59.95
Diethylene glycol dicaproate	39.45
Dodecyl piperidine stearate	0.40
p-tert-Butyl catechol	0.20

Table V. Properties of Blends

	A	B	C	D	E
Spreading, %					
After 7 days	0.0	0.0	0.0	0.0	0.0
After 30 days	0.0	0.0	0.0	0.0	0.0
Pour point, °F.	−65	−65	−65	−50	−65
Viscosity, cs.					
At 100°F.	13.9	19.7	19.5	18.2	9.8
At 0°F.	256	753	592	550	184
At −40°F.	2,990	35,400	15,000	23,200	2,240
Neutralization number	0.06	0.05	0.03	0.04	0.10
Oxidation characteristics					
Change in neutralization number	+0.04	+0.07	+0.07	+0.06	+0.08
Volatile acids formed, mg. KOH/g. oil	0.03	0.03	0.01	0.03	0.16
Change in viscosity at 100°F., cs.	+0.14	+0.30	+0.31	+0.18	+0.50
Change in wt. of copper strip, mg.	−1.1	−0.8	−1.0	−2.6	−5.4
Condition of copper strip	Stained	Stained	Stained	Stained	Dark and stained
Change in wt. of steel strip, mg.	−0.1	−0.1	−0.1	−0.1	+0.2
Condition of steel strip	Clean	Clean	Clean	Clean	Clean
Color of oil before oxidation	Light amber	Amber	Amber	Amber	Light amber
Color of oil after oxidation	Dark amber	Dark amber	Dark amber	Very dark amber	Very dark amber
Amount of sediment formed	None	None	None	None	None
Evaporation, %	0.02	0.24	0.15	0.05	1.2

EVALUATION OF BLENDS

The low temperature viscosities and pour points of the nonspreading diesters are too high for use as low temperature instrument oils. Diluents must be used to lower the viscosity and pour point to such an extent that fluidity persists at the required low temperatures and that mechanisms employing this lubricant are operable. Also, of paramount importance, the blend must retain nonspreading properties, in order that the minute droplet of oil used on jewel bearings will remain in place to provide adequate lubrication.

Bis(*m*-methylbenzyl) azelate, bis(*p*-isopropylbenzyl) azelate, bis(2-phenylpropyl) pimelate, and bis(2-phenylpropyl) azelate were selected for further evaluation in blends. These esters were blended with glycol esters of aliphatic acids selected so as to yield blends most likely to have good low temperature properties and not interfere with nonspreading characteristics. The diluents were spreading compounds and in general have much lower viscosities at low temperatures than the nonspreading components. It was found that the proportion of diluent which could be tolerated before spreading occurred, varied between 30 and 50%. The use of a larger proportion of the nonspreading component in these blends did not improve the spreading characteristics significantly. Too large a proportion of nonspreading component gave blends which were only slightly better in pour point and viscosity than the unblended nonspreading diester. The only two diluents which gave nonspreading blends were diethylene glycol dicaproate and diethylene glycol bis(3,5,5-trimethyl hexanoate). However, at lower temperatures crystallization occurs in blends consisting of 60% bis(2-phenylpropyl) pimelate or bis(2-phenylpropyl) azelate using diethylene glycol dicaproate as the diluent. No blend using bis(*m*-methylbenzyl) azelate was found to be nonspreading.

Nonspreading blends which displayed low pour point and low temperature viscosity are in Table IV. These blends were subjected to a more complete evaluation, the results of which are given in Table V. Blend E (*4*) (oil meeting Specification MIL-L-3918, lubricating oil, instrument, jewel bearing, nonspreading, low temperature) was included for comparative purposes.

Oxidation stability was determined by means of a dynamic oxidation test using 25-gram samples of oil (9). Neutralization number of the oil before and after oxidation was determined by ASTM method D 974-51T. Evaporation was determined by ASTM method D 972-51T.

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