# Sperm Oil Replacements: Synthetic Wax Esters from Selectively Hydrogenated Soybean and Linseed Oils

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# ABSTRACT AND SUMMARY

Synthetic wax esters with properties similar to those of sperm whale oil have been prepared entirely from soybean and linseed oils. The synthesis required: (a) selective hydrogenation of the oils with copper-on-silica gel catalyst, (b) hydrogenolysis of fatty acids to fatty alcohols with copper-cadmiumchromium catalyst, and (c) esterification of hydrogenolysis products to yield predominantly long chain fatty esters which contained unsaturation in both the alcohol and acid moieties. Similarity of physical and chemical properties indicate that these wax esters are possible replacements for sperm oil. After sulfurization, the wax esters also have potential as extreme pressure lubricant additives.

# INTRODUCTION

In 1970, the United States placed the sperm whale on the endangered species list and, in 1971, banned the import of its products. For this reason and because of shortages and increased prices of petrochemicals, we have investigated the preparation of sperm whale oil replacements from renewable agricultural sources.

Previously (1,2) we described esters of commercial partially hydrogenated soybean acids and of selectively hydrogenated soybean (SHSBA) and linseed acids (SHLSA) as possible replacements for sperm whale oil in lubricants. The saturated alcohols used in the preparation of these esters were derived mostly from petrochemicals.

This paper reports the preparation of wax esters entirely from soybean and linseed oils. The synthesis required the selective hydrogenation of the oils with copper-on-silica gel catalyst (3) at 170 C and 600 psi pressure followed by hydrogenolysis of the fatty acids, derived from the selectively hydrogenated oils (2), at 300 C and 2500-3000 psi pressure with a copper-cadmium-chromium catalyst. Conversion of fatty acids to fatty alcohols was 35-99+% with less than 5% increase in saturates and with 0-53% of diene reduced to monoene. The main side product of the hydrogenolysis was fatty alcohol-acid esters. Esterification of reaction product with a calculated amount of the corresponding fatty alcohols or fatty acids produced all wax esters. Kinematic viscosity data and smoke, flash, and fire points indicate that the wax esters from SHSBA (WESHSBA) and wax esters from SHLSA (WESHLSA) would be possible replacements for sperm oil. After sulfurization, these wax esters have potential as extreme pressure (EP) additives. EP additives prevent destructive metalto-metal contact in lubrication at either high pressure or temperature or both. WESHSBA and WESHLSA were sulfurized, tested, and evaluated as EP and antiwear additives.

## EXPERIMENTAL PROCEDURES

# Materials and Methods

Refined and bleached soybean and linseed oils came from commercial sources. Fatty acid compositions of oils as determined by gas liquid chromatography (GLC) of methyl esters were soybean: 10.7%, C16:0; 3.6%, C18:0; 25.2%, C18:1; 53.2%, C18:2; and 7.5%, C18:3; and linseed: 6.7%, C16:0; 3.7%, C18:0; 23%, C18:1; 15.6%, C18:2; and 51%, C18:3. A 15% copper-on-silica gel catalyst was prepared with copper nitrate trihydrate and heat activated as outlined by Koritala (3).

SHSBA and SHLSA were obtained from saponification of the parent selectively hydrogenated oils.

## Other Reagents

Girdler (Chemetron Corp., Louisville, KY) T-1057 catalyst (ca. 40.8% CuO, 19.7% CdO, and 14.3%  $Cr_2O_3$ ) was used for hydrogenolysis. Topaz S105 paraffin oil (Topaz S105) is produced by Atlantic Richfield Co., Philadelphia, PA.

Engine crankcase base oil (AA), automatic transmission base fluid (BB), RGO-100 gear lubricant (CC), 100/100 viscosity solvent-extracted neutral oil (DD), and commercial sperm oil replacements were provided by Southwest Research Institute (SWRI), San Antonio, TX.

# Hydrogenation

Soybean and linseed oils were selectively hydrogenated with the copper-on-silica gel catalyst at 170 C and 600 psi pressure as described in a previous publication (2). Selectively hydrogenated soybean oil (SHSBO) contained 10.2%, C16:0; 4.2%, C18:0; 76.2%, C18:1, 9.4%, C18:2; 0.0%, conjugatable C18:3; and 37.3% isolated *trans* double bonds. Selectively hydrogenated linseed oil (SHLSO) contained 6.2%, C16:0; 4.6%, C18:0; 37.6%, C18:1; 48.0%, C18:2; 3.6%, C18:3; 5.0%, conjugatable C18:2; 1.5% conjugatable C18:3; and 44.4% isolated *trans* double bonds.

#### Hydrogenolysis

For a typical hydrogenolysis, a 1000 ml stainless-steel Magne-Dash autoclave was charged with 600 ml of SHSBA and 30 g of Girdler T-1057 catalyst per 100 ml acids. After the vessel was purged with nitrogen and pressurized with hydrogen to 3000 psi at room temperature, the charge was heated with stirring to 300 C. Hydrogen pressure was then maintained at 3000 psi for 5 hr. At this stage, hydrogen uptake was nil over a period of 0.25 hr. After cooling the autoclave to 80 C, the batch was filtered with filter aid and the product was analyzed. When necessary, trace amounts of metals (from catalyst) were removed by vacuum bleaching with 1% activated clay (4). Analysis of product (experiment 1, Table I) showed 97.9% fatty alcohols, 0.2% free fatty acids, and 1.9% wax esters. This standard procedure (SP) was followed in all hydrogenolyses.

# Esterification

A portion of the above hydrogenolysis product (472.3 g, 1.74 mole alcohols) and 446 g (1.67 mole) precursory SHSBA were refluxed in the presence of 2 g of a catalyst consisting of three parts by weight of calcium acetate and one part of barium acetate with 500 ml of xylene. Theoretical amounts of water of esterification were removed by a Dean-Stark trap. The reaction mixture was washed with water, dried, and stripped of solvent. Infrared (IR) analysis

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		ND30		1.45	1.46	1.45		1.46	1.46	1.46	1.45	1.4573		
		Iodine value	86.2	91.4	92.3	90.2	125.7	104.5	109.9	104.1	113.0	108.0		
	trans	Isolated (%)	37.3	37.5	40.2	37.5	44.4	50.4	51.5	53.5	54.3	44.5		
	2	Acids (%)	9.4	1	9.2	5.5	48.0	27.3	27.6	25.0	I	28.7		
	C18:2	Alcohols (%)	I	10.2	9.6	10.0	I	21.5	27.1	23.0	31.8	31.2		
	1	Acids (%)	76.2	74.5	75.6	75.0	37.6	62.2	60.6	64.7	ł	59.7		
alysis	C18:1	Alcohols (%)		74.3	74.2	75.0		63.2	58.6	62.9	54.9	52.7		
GLC analysis	0	Acids (%)	4.2	10.5	4.9	10.6	4.6	5.3	5.1	5.2	I	4.2		
	C18:0	Alcohols (%)		4.9	5.6	4.7		8.7	7.3	5.0	6.1	6.2		
	0	Acids (%)	10.2	15.0	10.3	8.9	6.2	6.2	6.7	5.1	1	7.4		
	C16:0	Alcohols (%)		10.6	10.6	10.3		6.5	6.9	9.1	7.2	6.6		
	Conversion acids	to alcohols (%)		6.79		95.6	I	35.0	ł	38.2	+66	48.8		
		as oleic (%)	100	0.23	0.58	1.3	100	39.6	1.3	24.0	0.0	7.2	bean acids.	
	Hours	at 300 C		5.0		4.5	I	4.5		6.5	6.75	4.5	ogenated sov	
	Catalvst	(g/100 ml acids)		Ś		S	I	1		1	10	s	aSHSBA: Selectively hydrogenated soybean acids	
	Acids	experiment no.	SHSBA <sup>a</sup>	SHSBA <sup>b</sup> 1	WESHSBAC	SHSBA <sup>b</sup> 2	SHLSAd	SHLSA <sup>e</sup> 3	WESHLSA	SHLSA <sup>d</sup> 4	SHLSA <sup>d</sup> 5	SHLSA <sup>b</sup> 6	aSHSBA: Sel	

dSHLSA: Selectively hydrogenated linseed acids containing 3.6% triene. cWESHSBA: Wax esters from selectively hydrogenated soybean acids. psi pressure. <sup>e</sup>Conducted at 2500

WESHLSA: Wax esters from selectively hydrogenated linseed acids.

fatty acids, isolated fatty alcohols, and acetate esters of isolated fatty alcohols were compared for absorbance at 3310 cm<sup>-1</sup> (OH stretching); 1055 cm<sup>-1</sup> (C-O stretching for alcohol); 1735 cm<sup>-1</sup> (C-O stretching); and 1170 cm<sup>-1</sup> (C-O ester stretching). Conversion of acids to alcohols was calculated from weight percent of isolated acids and alcohols. For GLC analysis, acid fractions were converted to ethyl esters with 5% HCl in anhydrous ethanol and alcohol fractions were acetylated with acetic anhydride-pyridine reagent. Esters were analyzed on a 12ft-1/8 in. OD stainlesssteel column packed with 10% EGSS-X on Gas Chrom. P, 100-120 mesh (organosilicon polyester packing, Applied Science Laboratories, Inc., State College, PA) in a Model 1625 C Varian gas chromatograph or in a Model 5750 Hewlett-Packard gas chromatograph. The column was held at 170 C with a helium flow of 35 ml/min. IR spectra were recorded with a Beckman IR 8 spectrophotometer, both for thin films of 100% material and for carbon disulfide solutions of compounds. Ultraviolet (UV) spectra were obtained on a Beckman DB spectrophotometer. Iodine value (IV) was calculated from GLC analysis or determined by official AOCS method Cd 1-25 (6). Acid value (AV) and percentage-free fatty acids were determined by official AOCS methods Da 14-42 and Da 4a-42 (6), respectively.

Nonconjugatable diene and triene were determined by the difference between total diene and triene by GLC analyses and conjugatable diene and triene by UV analysis. Percentage of isolated trans double bonds was determined by AOCS Official Method Cd 14-61 (6).

Viscosities of wax esters were taken in Cannon-Fenske-Ostwald viscometers. The viscosity indices were obtained from viscosities at 100 and 210 F by ASTM Method D 2270 (7). The kinematic viscosity was converted to saybolt universal viscosity (SUS) according to ASTM Method D 2161 (7).

Smoke, flash, and fire points were measured by the Cleveland open flash cup, ASTM D 92-33 (7) and AOCS Official Method Cc 9a-48 (6).

# **Evaluation of Sulfurized Sperm Oil Candidates**

As we reported previously (2), sperm oil candidates were sulfurized and evaluated under a research contract with SWRI. Although SWRI had overall responsibility for sulfurizing and evaluating our sperm oil candidates, part of the work was performed at the Eastern Regional Research Center, such as the four-ball EP test ASTM Method 2596 (7), four-ball wear test ASTM Method D 2266-67 (7), and freezing and pour points ASTM Method D 97-57 (7).

EP tests were made on a Precision Scientific four-ball EP tester (1440 rpm) in which loads were successively increased first in 20- and then 10-kg increments until an immediate seizure occurred, representing the weld point. Scar diameters were determined with a Precision four-ball wear tester. Samples were run for 1 hr at 600 rpm at 120 C and under a 50-kg load with and without additive. After the balls were cleaned with naphtha and hexane, scar diameters were measured under a microscope assembly #73607, with

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For acid-alcohol analysis (Table I), wax esters and hydrogenolysis products were saponified with ethanolic potassium hydroxide and isolated, as detailed by Miwa (5). The reaction mixture was diluted with water and the fatty alcohol fraction was extracted with ethevl ether. The acid salts were acidified with dilute HCl, and the free fatty acids

were extracted with ethyl ether. Isolation of fatty acids from fatty alcohols was essentially complete as determined by GLC and IR analysis. IR spectra of neat hydrogenolysis products, isolated fatty acids, methyl esters of isolated

## Analytical Methods

#### TABLE II

Physical Properties of Wax Esters of Selectively Hydrogenated Soybean and Linseed Acids

		Visco SUS		Viscosity	Poin	ts (F)	
Wax esters <sup>a</sup>	IV	100	210	index	Smoke	Flash	Fire
WESHSBA	112.6	135.7	47.8	211	320	536	698
WESHLSA	91.4	92.4	42.0	207	311	482	770
Sperm oil <sup>c</sup>	82.0	109.0	44.8	223	275- 325	490	655- 675

<sup>a</sup>Wax esters: WESHSBA, wax esters from selectively hydrogenated soybean acids; WESHLSA, wax esters from selectively hydrogenated linseed acids.

bSUS = Saybolt Universal viscosity.

<sup>c</sup>Winterized at 45 F.

measuring grid (Precision Scientific).

The following tests were made by SWRI: Sulfur analysis, base oil solubility test, copper strip corrosion test ASTM Method D 130 (7), API gravity at 60 F ASTM Method D 287 (7), lead corrosion test FTM 5321 (8), foam test ASTM D 892 (7), emulsion test ASTM D 1401, N-pentane and benzene insolubles ASTM Method D 893-52 T (7), viscosity and viscosity index ASTM Method 2270 (7), and thermal stability test FTM 2504-1 (8).

### **RESULTS AND DISCUSSION**

Previously described, selectively hydrogenated soybean and linseed acids containing increased amounts of monoene and nonconjugatable diene have unusual thermal and oxidative stability and yet retained sufficient reactivity to permit sulfurization (2). These acids were reduced to the corresponding alcohols in 35-99% yield with less than 5% increase in saturates and with 0-53% reduction of diene to monoene. Analysis of hydrogenolysis products, SHSBA, SHLSA, WESHSBA, and WESHLSA are given in Table I. The main side product of the hydrogenolysis was fatty alcohol-acid esters. GLC analysis of products showed the formation of trace amounts of hydrocarbons. However, head gases were not collected and analyzed for hydrocarbons. Because the selectivity of copper-cadmium catalyst in the reduction of unsaturated acids to unsaturated alcohols has been reported extensively (9-13), a comprehensive study of the reaction conditions was not made. The aim of this work was to prepare samples of wax esters entirely from soybean and linseed acids for comparison with sperm whale oil. Results of this work indicated that optimum conditions for hydrogenolysis was 5 g Girdler T-1057 catalyst per 100 ml of fatty acids, hydrogen pressure of 3000 psi, and a temperature of 300 C for 5 hr.

WESHSBA was obtained by the esterification of a calculated amount of SHSBA with reduction products from experiment 1. WESHLSA was obtained by the esterification of a calculated amount of alcohols from experiment 5 with reduction products from experiment 4. Also shown in Table I are the hydrogenolysis products from experiments no. 2, 3, and 6. These products were not used in evaluation work. Reduction products from experiment 6 contained 7.2% free fatty acids, 4.0% free fatty alcohols, and 88.8% wax esters. When this mixture was refluxed with xylene, and water of esterification was azeotropically removed, a product containing 97% wax esters and 3% Fatty acids was obtained.

The unusual chemical composition and physical properties of sperm oil make it useful in such diverse applications as cutting oils, fine cosmetics, leather-softening agents, and spinning lubricants in the textile industry. One large commercial source of oleyl alcohol in this country has been from the saponification of sperm whale oil. Oleyl alcohol

TABLE III

Physical	Properties	of	Sulfurized	Wax	Estersa
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Property	WESHSBA	WESHLSA	sso
Sulfur, %	10.5	10.8	11
Pour point, F	82	73	64
Freezing point, F	77	68	59
Flash point, F	468	460	464
Fire point, F	514	525	536
Saponification number	97.1	98.4	166.8
Neutral number	3.25	5.46	3.05
Viscosity at 210 F, SUS <sup>b</sup>	243	537	331

<sup>a</sup>Sulfurized wax esters: WESHSBA, wax esters from selectively hydrogenated soybean acids; WESHLSA, wax esters from selectively hydrogenated linseed acids; SSO, sulfurized sperm oil.

<sup>b</sup>SUS = Saybolt Universal viscosity.

has many established uses and many more potential uses could be realized if it were more readily available on a large scale (13, 14).

In particular, oleyl alcohol is an intermediate for biodegradable detergents and in different types of surface active and detergent applications for household or industrial use. Although unsaturated fatty alcohols described in this work have geometrical and positional isomers, they should be considered as a source of monounsaturated alcohols. Physical properties of WESHSBA and WESHLSA (not winterized) were studied and compared with sperm oil winterized at 45 F (Table II). WESHSBA have smoke, flash, and fire points higher than those of sperm oil. WESHLSA have smoke, flash, and fire points comparable to those of sperm oil. WESHSBA and WESHLSA also have high viscosity indices (uniform viscosity over a broad temperature range).

In the lubrication of certain gear elements in automotive vehicles and various industrial machines, high pressure can cause a film of lubricant to rupture with subsequent damage to the machinery. For this reason, EP lubricants are fortified with additives to augment lubricity at either high pressures or temperatures, or both. EP lubricants should have good lubricity, good cooling properties, high film strength, good load-bearing ability, and miscibility with the usual types of base oils. Sulfurized sperm oil (SSO) satisfies these requirements and has been used extensively in EP additives. For this reason, WESHSBA and WESHLSA was sulfurized for evaluation as EP and antiwear additives.

The pour and freezing points of the sulfurized wax esters were higher than those of SSO (Table III); however, these values are comparable with those of several commercial sulfurized sperm oil replacements and may be improved by winterization.

The sulfurized wax esters were evaluated in base oils used in EP automotive and industrial applications. Performance of SSO replacements were compared (Table IV)

(kg)         star (nm)         corresion         (mg/n <sup>2</sup> )         100         210         index y           140         0.535         18         -         13.57         119         13.57         119           260         0.533         1A         30.5         133.48         15.20         119           260         0.553         1A         30.5         134.19         13.67         111           280         0.553         1A         30.5         134.19         13.67         111           280         0.553         1A         30.5         137.80         13.60         1118           280         0.593         1A         37.80         36.02         1113         113           280         0.591         1B         2.7         549.00         35.37         103         112           280         0.593         3A         36.1         20.07         113         20.3           280         0.593         3A         36.1         20.93         21.7         112           280         0.663         3A         24.7         549.00         35.37         113           280         0.603         3A         24.7 </th <th>additividy         (b)         set frame         controls         (b)         set frame         (c)         (c)&lt;</th> <th></th> <th>Sulfurized</th> <th>Extreme pressure Weld point</th> <th>Wear Average wear</th> <th>Connerc</th> <th>Lead</th> <th>Kinematic viscosityd Cs at F</th> <th>atic ityd F</th> <th>Viscosity</th> <th>API<sup>e</sup> gravity (deoree API</th> <th></th> <th>Emulsion test (ml)</th> <th>5-</th> <th>Fo</th> <th>Foam test<sup>f</sup> (ml)</th> <th>(Jn</th>	additividy         (b)         set frame         controls         (b)         set frame         (c)         (c)<		Sulfurized	Extreme pressure Weld point	Wear Average wear	Connerc	Lead	Kinematic viscosityd Cs at F	atic ityd F	Viscosity	API <sup>e</sup> gravity (deoree API		Emulsion test (ml)	5-	Fo	Foam test <sup>f</sup> (ml)	(Jn
100       0453       18       - </th <th>100       045       18       -<th>Base oil<sup>a</sup></th><th>additive<sup>b</sup></th><th>(kg)</th><th>scar (mm)</th><th>corrosion</th><th><math>(mg/in^2)</math></th><th>100</th><th>210</th><th>index</th><th>60 F)</th><th>lio</th><th>H<sub>2</sub>O</th><th>Emul</th><th>_</th><th>Π</th><th>Ξ</th></th>	100       045       18       - <th>Base oil<sup>a</sup></th> <th>additive<sup>b</sup></th> <th>(kg)</th> <th>scar (mm)</th> <th>corrosion</th> <th><math>(mg/in^2)</math></th> <th>100</th> <th>210</th> <th>index</th> <th>60 F)</th> <th>lio</th> <th>H<sub>2</sub>O</th> <th>Emul</th> <th>_</th> <th>Π</th> <th>Ξ</th>	Base oil <sup>a</sup>	additive <sup>b</sup>	(kg)	scar (mm)	corrosion	$(mg/in^2)$	100	210	index	60 F)	lio	H <sub>2</sub> O	Emul	_	Π	Ξ
200         0573         3A         0.2         13.57         119         28.8         1         4         75         0.0         100         200           200         0573         1A         3.5         13.57         119         28.8         0 <td>240         0575         AA         0.2         132.42         11.9         23.0         1         4         75         0.0         100           210         0575         AA         3.5         131.19         11.37         11.9         23.0         11.9         73.0         0.00         100         200         <td< td=""><td>AA</td><td>None</td><td>140</td><td>0.635</td><td>18</td><td>I</td><td> </td><td>1</td><td>  1</td><td></td><td></td><td></td><td></td><td></td><td></td><td>1</td></td<></td>	240         0575         AA         0.2         132.42         11.9         23.0         1         4         75         0.0         100           210         0575         AA         3.5         131.19         11.37         11.9         23.0         11.9         73.0         0.00         100         200 <td< td=""><td>AA</td><td>None</td><td>140</td><td>0.635</td><td>18</td><td>I</td><td> </td><td>1</td><td>  1</td><td></td><td></td><td></td><td></td><td></td><td></td><td>1</td></td<>	AA	None	140	0.635	18	I		1	1							1
20         0.53         3A         3B         15.2         119         27.8         1         8         70         0.00         200	200         0.530         3A         3B         15.20         119         27.8         1         8         70         0.00         200           200         0.535         1A         33         12.51         13.11         15.27         119         27.6         0.00         20		10% WESHSBA	240	0.575	3A	0.2	132.42	13.57	119	28.0	-	4	75	0-0	10-0	0.0
300         0.333 $1\Lambda/B$ 323         [131,19]         11,57         101         77.8         1         0         79         900         900           200         0.373         1         3         127.97         11,30         11,31         17,7         75         0         79         900         900           200         0.575         1         3         127.97         11,30         113         77.7         75         0         79         900         900           200         0.593         1         2         33.1         13.1         13.57         12         23.1         11         93.57         9         900         100         900         900         100         900         900         100         9	300         0.333         1.0, B         7.3         1.13         1.37         1.13         7.73         1.1         0         7.9         0.0         0.0           200         0.637         1.1         3.3         1.13.0         1.13         7.73         1.1         0         7.9         0.0         0.0           200         0.637         1.1         3.5         1.13.0         1.13         7.73         5.3         0.0		10% WESHLSA	260	0.550	ЗA	38.6	153.18	15.20	119	27.8	1	œ	70	0-0	20-0	0-0
200         0.430         1A         30.3         134.19         11.30         11.3         77.6         7         9         7.9         9.00         9.00           200         0.635         1B         -	200         0.430         1A         30.3         134.19         11.30         11.1         77.6         7         9         4.9         2.00           200         0.575         1A         30.3         173.10         11.1         77.6         0         79         4.90         200           200         0.575         1B         5         35.3.0         11.3         77.7         7         0         79         4.90         200           200         0.593         1A         12.53         55.30         50.09         113         77.5         7         0         79         4.9         200         000           200         0.593         1A         7.14         112         25.3         13.9         14.9         14.1         112         25.3         6         19         200         000         100           200         0.603         3A         4.1         24.79         50.79         111         25.3         6         13         200         100         100         200         100         200         200         100         200         200         200         200         200         200         200         200		10% SSO	300	0.583	1A/B	22.5	131.19	13.57	101	27.8	T	0	79	0-0	40-0	0-0
2400.5751A3517.7913.2011377.7750791002002800.033282853.4611325.3212525244700002800.033185.553.4611325.321252526002800.033182.7543.5364.0711325.79244700002800.033182.7543.5310325.3710325.3792470002800.033182.7543.5320.325.3710325.3792470002800.033342.126.32611225.379112261935.071002800.0403828.620.7520.7511125.1935901002800.0453820.311125.1911261930.001002800.0553820.711125.191135.2901002002800.0563820.724.7720.7311325.29901002002800.0563820.720.7711125.231.4117261202001002800.0563820.720.755.7<	240         0.575         1A         35         17.79         13.20         117.97         13.20         117.97         13.20         117.97         13.20         13.2		10% Com Sub A	280	0.480	٩V	30.3	134.19	13.61	111	27.6	1	0	79	45-0	30-0	0-0
2100.6251B5538.635311241002000.0531A12753.3011223.112244100002000.0531A12753.3011123.71020000002000.0531A77.453.0311123.740200002000.0633A7.753.0311123.74020002000.0603A36.120.011125.1999901002000.0603A36.120.320.7711025.1999901002000.0633A3A34.420.0321.1125.1999901002000.0633A3A34.420.9720.9311125.224.7250.001002000.0633B20.027.645.0311125.224.7250.02001002000.0633B20.027.645.0311125.224.7250.02001002000.0633B20.027.645.0311125.22427.002001002000.0633B20.027.645.0311125.224.725.002001002000.053 <td< td=""><td>210         0.035         1B         5         53.6         5.3.0         11         5.3.3         21         5         34         41         0         0         0           220         0.533         13         5.3.4         4.1.4         112         5.3.3         13         23         34         41         0         0         00           230         0.531         13         2.4.3         54.3.0         112         55.3         13         23         34         0         0         00         00           230         0.531         13         2.4.3         54.3.0         11         25.3         6         13         0         0         00<td></td><td>10% Com Sub B</td><td>240</td><td>0.575</td><td>14</td><td>3.5</td><td>127.97</td><td>13.20</td><td>113</td><td>27.7</td><td>75</td><td>0</td><td>79</td><td>10-0</td><td>20-0</td><td>20-0</td></td></td<>	210         0.035         1B         5         53.6         5.3.0         11         5.3.3         21         5         34         41         0         0         0           220         0.533         13         5.3.4         4.1.4         112         5.3.3         13         23         34         41         0         0         00           230         0.531         13         2.4.3         54.3.0         112         55.3         13         23         34         0         0         00         00           230         0.531         13         2.4.3         54.3.0         11         25.3         6         13         0         0         00 <td></td> <td>10% Com Sub B</td> <td>240</td> <td>0.575</td> <td>14</td> <td>3.5</td> <td>127.97</td> <td>13.20</td> <td>113</td> <td>27.7</td> <td>75</td> <td>0</td> <td>79</td> <td>10-0</td> <td>20-0</td> <td>20-0</td>		10% Com Sub B	240	0.575	14	3.5	127.97	13.20	113	27.7	75	0	79	10-0	20-0	20-0
240 $0.675$ 28 $5.36, 5.35, 6.34, 112$ $5.31, 31$ $21, 25, 34$ $41, 00$ $00$ 280 $0.593$ 1A $12.7$ $537, 80$ $50.2$ $112$ $52.3$ $24$ $47$ $00$ $00$ 280 $0.593$ 1A $12.7$ $537, 80$ $50.2$ $112$ $52.7$ $9$ $24$ $47$ $00$ $00$ 280 $0.603$ 1B $7.7$ $549, 00$ $55.7$ $112$ $25.7$ $9$ $24$ $47$ $00$ $100$ 280 $0.603$ 3A $36.1$ $236.3$ $21.57$ $112$ $25.1$ $9$ $24$ $47$ $00$ $100$ 280 $0.603$ 3B $36.1$ $236.3$ $21.57$ $112$ $25.7$ $9$ $24$ $47$ $00$ $100$ 280 $0.603$ 3B $36.1$ $236.3$ $21.57$ $112$ $25.3$ $96.9$ $190$ $100$ 280 $0.665$ 3B $20.0$ $27.78$ $5.03$ $21.73$ $112$ $22.2$ $47$ $500$ $100$ 280 $0.670$ 3A $21.2$ $237.73$ $21.73$ $31.73$ $112$ $22.2$ $47$ $500$ $200$ 280 $0.670$ 3A $21.2$ $27.73$ $5.73$ $113$ $21.2$ $31.67$ $112$ $22.6$ $400$ 280 $0.670$ $34$ $21.2$ $27.73$ $5.73$ $113$ $21.2$ $21.2$ $21.2$ $21.2$ $21.2$ $21.2$ $21.2$ $21.2$ $21.2$ <td< td=""><td>240         0675         2.B         5.3         53.4         1.1         5.3         2.1         2.5         3.4         0.0         0.0           280         0.593         1.A         7.7         549.4         56.0         111         2.5         3         4         4         0.0         0.0           280         0.593         1.A         7.7         549.0         55.7         112         2.5         3         3         3         0.0         0.0           280         0.693         3.A         5.1         5.3         0.3         3         3         0.0         0.0           280         0.683         3.A         5.0         3.5         112         5.7         112         5.7         12         5.0         10.0         0.0</td><td>BB</td><td>None</td><td>120</td><td>0.625</td><td>1B</td><td>1</td><td>i</td><td>ļ</td><td>l</td><td>ł</td><td>l</td><td>I</td><td>ł</td><td>I</td><td>-</td><td>I</td></td<>	240         0675         2.B         5.3         53.4         1.1         5.3         2.1         2.5         3.4         0.0         0.0           280         0.593         1.A         7.7         549.4         56.0         111         2.5         3         4         4         0.0         0.0           280         0.593         1.A         7.7         549.0         55.7         112         2.5         3         3         3         0.0         0.0           280         0.693         3.A         5.1         5.3         0.3         3         3         0.0         0.0           280         0.683         3.A         5.0         3.5         112         5.7         112         5.7         12         5.0         10.0         0.0	BB	None	120	0.625	1B	1	i	ļ	l	ł	l	I	ł	I	-	I
2000.590382.5364.1441.141122.51192.4410.00.02000.6631A12.754.3011325.345.31326.01002000.6631A27.454.3051.3710325.345.91010.02000.6631A3.154.120.011325.545990.02000.6633A3.17.11122.6.12.0.19960102000.6633A3.1.42.1.571112.6.12.6.19960112000.6633A3.1.42.2.592.0.751132.6.26132.901002000.6133A3.1.42.2.592.7.545.0.751132.6.26132.9.2001002000.6653A2.1.52.1.51112.6.26132.6.22.0010020110.00.6153A2.1.52.1.52.1.531.12.6.22.02.0010020110.00.6153A2.1.52.1.531.12.6.23.02.6.22.001002020.6553A2.0.22.0.311731.12.6.23.1.42.6.22.002.002030.6563A2.0.22.0.22.0.22.0.22	200         0.590         3B         33:3         43:44         41.14         112         35:7         15         44         47         00         00           200         0.633         1A         7:4         54:93         56:09         112         35:7         1         57         56:09         113         35:7         56:09         121         35:7         56:09         121         35:7         57         60         00         00           200         0.603         3A         36:1         25:3         55:09         121         35:7         57         9         37         3         00         100           200         0.603         3A         11.0         23:7         112         35:7         9		<b>10% WESHSBA</b>	240	0.675	2B	5.8	538.62	35.30	118	25.3	21	25	34	0-0	0-0	0-0
2000.6381A1A17,453,736.0211325,79244700002000.6331A27,753,730.000.6333790.00.02000.6033A36,125,0321,5711226,126193590.00.002000.6633B26,6230,3021,5711026,1996220,00.002000.6633B24,7720,9011325,26193570.00.002000.6633B24,7720,9011325,262020,01002000.6733B24,7720,9011325,262020,01002000.6353B24,7720,9011325,262020,01002000.6353B24,7720,9011325,262020,01102000.6353B20,020,111731,1322222000.6353B21,227,685,0911331,13222222000.6353B11227,685,0912531,11332222222000.63511812,627,685,0912531,1	280         0638         1A         77,7         537,20         5.007         113         25,7         9         24         47         00         00           280         0.653         1A         77,7         537,20         5.607         113         25,7         9         24         47         00         00           280         0.6501         1B         27,4         543,00         55,37         103         25,7         40         37         3         00         100           280         0.660         3B         26,1         250,3         21,57         110         25,1         8         11         61         510,0         100         100           200         0.615         3B         23,47         20,35         573         113         25,2         6         13         61         100         100           200         0.651         1B         12,4         23,475         20,35         113         25,2         6         13         61         100         100         100         100         100         100         100         100         100         100         100         100         100         100 <td< td=""><td></td><td><b>10% WESHLSA</b></td><td>260</td><td>0.590</td><td>3B</td><td>25.3</td><td>643.44</td><td>41.14</td><td>112</td><td>25.1</td><td>15</td><td>24</td><td>41</td><td>0-0</td><td>0-0</td><td>0-0</td></td<>		<b>10% WESHLSA</b>	260	0.590	3B	25.3	643.44	41.14	112	25.1	15	24	41	0-0	0-0	0-0
32006533A2754.5.33.0912125.953.2330.00.001300.6033 $2.7$ $5.3.7$ 103 $25.7$ 40 $27$ $37.3$ $3$ $0.0$ $0.00$ 2000.6033 $2.7$ $5.1$ $2.03$ $21.57$ $112$ $26.1$ $26$ $37$ $37$ $30.0$ $1000$ 2000.6063 $28.6$ $273.59$ $21.57$ $110$ $26.1$ $26$ $9$ $9$ $62$ $20.0$ $100$ 2000.6763 $3$ $3.4$ $4.6$ $237.7$ $20.94$ $110$ $26.2$ $6$ $12$ $20.0$ $100$ 2000.6663 $3$ $2.46$ $5.04$ $117$ $26.2$ $6$ $12$ $26.0$ $200$ 2000.6663 $2.23.77$ $20.75$ $118$ $26.2$ $6$ $12$ $26.0$ $200$ $100$ 2000.65018 $12.6$ $23.77$ $3.27.6$ $5.04$ $117$ $31.1$ $3$ $27$ $260$ $200$ 2000.65018 $12.6$ $27.78$ $5.06$ $123$ $31.4$ $12$ $27.60$ $200$ $200$ 2000.65018 $12.6$ $27.78$ $5.06$ $123$ $31.4$ $12$ $26$ $200$ $200$ 2000.65018 $12.6$ $5.778$ $5.06$ $2102$ $31.4$ $12$ $26$ $200$ $200$ 200 <td< td=""><td>200         0.651         3A         27.4         54.30         1.21         2.5.7         4.5         2.3         0.0         0.00           200         0.661         13         2.7.4         54.30         57         103         2.5.7         4.5         2.5.7         4.0         0.00         100           200         0.660         13         2.5.1         2.0.1         2.0.1         2.0.0         100           200         0.663         3A         36.1         2.7.5.9         2.1.57         112         2.5.1         2         4.6         2.0.0         100           200         0.663         3A         1.4         2.7.5.9         2.1.77         112         2.5.1         6         110.20         110.0         117         111         2.5.2         6         110.20         110.0         111         <td< td=""><td></td><td>10% SSO</td><td>280</td><td>0.628</td><td>1A</td><td>12.7</td><td>537.80</td><td>36.02</td><td>113</td><td>25.7</td><td>6</td><td>24</td><td>47</td><td>0-0</td><td>0-0</td><td>0-0</td></td<></td></td<>	200         0.651         3A         27.4         54.30         1.21         2.5.7         4.5         2.3         0.0         0.00           200         0.661         13         2.7.4         54.30         57         103         2.5.7         4.5         2.5.7         4.0         0.00         100           200         0.660         13         2.5.1         2.0.1         2.0.1         2.0.0         100           200         0.663         3A         36.1         2.7.5.9         2.1.57         112         2.5.1         2         4.6         2.0.0         100           200         0.663         3A         1.4         2.7.5.9         2.1.77         112         2.5.1         6         110.20         110.0         117         111         2.5.2         6         110.20         110.0         111 <td< td=""><td></td><td>10% SSO</td><td>280</td><td>0.628</td><td>1A</td><td>12.7</td><td>537.80</td><td>36.02</td><td>113</td><td>25.7</td><td>6</td><td>24</td><td>47</td><td>0-0</td><td>0-0</td><td>0-0</td></td<>		10% SSO	280	0.628	1A	12.7	537.80	36.02	113	25.7	6	24	47	0-0	0-0	0-0
240         0.591         1B         4.7         549.0         55.37         103         25.7         40         37         3         0.0         100           280         0.603         3         56.1         250.30         21.57         112         26.1         25         0.0         100           280         0.603         3A         36.1         250.30         21.57         110         26.1         2         37         3         0.0         100           280         0.603         3A         34.6         230.30         21.57         112         26.1         2         37         3         0.0         100           280         0.605         3A         24.5         20.3         113         2.53         6         13         37         60         100           200         0.605         3A         24.5         20.3         113         21.3         25.3         0.0         100         0.0         0.0         0.0         0.0         0.0         0.0         0.0         0.0         0.0         0.0         0.0         0.0         0.0         0.0         0.0         0.0         0.0         0.0         0.0 <t< td=""><td>240         0.591         1B         4.7         54.0         5.37         103         2.57         40         37         3         0.0         100           200         0.600         3         36.1         26.0         24.7         25.1         26.1         26         9         55         0.0         100           200         0.603         3         36.1         26.3         21.77         112         26.1         26         9         9         61         510.0         100           200         0.613         3         3         11.0         25.1         6         15         51         61         510.0         100         100         100         100         110         111         25.1         66         150.0         100         110         100         100         100         100         100</td><td></td><td>10% Com Sub A</td><td>320</td><td>0.653</td><td>ЗA</td><td>27.4</td><td>543.52</td><td>36.09</td><td>121</td><td>25.9</td><td>S</td><td>22</td><td>53</td><td>0-0</td><td>0-0</td><td>0-0</td></t<>	240         0.591         1B         4.7         54.0         5.37         103         2.57         40         37         3         0.0         100           200         0.600         3         36.1         26.0         24.7         25.1         26.1         26         9         55         0.0         100           200         0.603         3         36.1         26.3         21.77         112         26.1         26         9         9         61         510.0         100           200         0.613         3         3         11.0         25.1         6         15         51         61         510.0         100         100         100         100         110         111         25.1         66         150.0         100         110         100         100         100         100         100		10% Com Sub A	320	0.653	ЗA	27.4	543.52	36.09	121	25.9	S	22	53	0-0	0-0	0-0
200 $0.003$	200       0.603		10% Com Sub B	240	0.591	1B	4.7	549.00	35.37	103	25.7	40	37	e	0-0	10-0	0-0
2400.4003A36.135.235.131.225.1996350.00.02600.6603A3A34.720.311025.19996330.00.02700.6133A3A4.6239.7620.7511325.199954510.01102800.6653B20.027.645.0411731.0362020.01002800.6653B20.027.645.0411731.0362020.020.02800.6673A21.237.735.3313531.11376010020.01003800.6673A21.237.2614031.331.11376010020.01003800.6701B12.227.735.5313531.113766230.020.01003000.943 <td>200         0400         3A         36.1         25.30         21.57         112         26.1         26         9         9         9         9         9         0         000         100           200         0640         3A         36.1         250.3         21.57         110         26.1         26         19         35         00         100           200         0647         3A         19.6         239.77         20.39         111         25.3         6         15         60         100         100           200         0667         3A         21.2         337.75         50.3         111         25.3         6         53.0         160         100</td> <td>22</td> <td>None</td> <td>130</td> <td>0.603</td> <td>i</td> <td>I</td> <td>I</td> <td>ł</td> <td>ł</td> <td>I</td> <td>1</td> <td>ł</td> <td>1</td> <td>I</td> <td>I</td> <td>I</td>	200         0400         3A         36.1         25.30         21.57         112         26.1         26         9         9         9         9         9         0         000         100           200         0640         3A         36.1         250.3         21.57         110         26.1         26         19         35         00         100           200         0647         3A         19.6         239.77         20.39         111         25.3         6         15         60         100         100           200         0667         3A         21.2         337.75         50.3         111         25.3         6         53.0         160         100	22	None	130	0.603	i	I	I	ł	ł	I	1	ł	1	I	I	I
260 $0.660$ 3B $28.6$ $2.72.59$ $2.071$ $110$ $26.1$ 962 $20.0$ $000$ 270 $0.0613$ 3A $240.77$ $20.971$ $110$ $25.1$ 96 $230.0$ $1600$ $111$ 270 $0.0613$ 3A $240.77$ $20.971$ $20.901$ $113$ $25.2$ $6$ $530.0$ $1600$ $110$ 270 $0.0613$ 3A $240.77$ $20.971$ $20.931$ $110$ $25.2$ $6$ $230.0$ $1600$ $110$ 280 $0.665$ 3B $21.0$ $27.64$ $5.04$ $117$ $31.0$ $36$ $20$ $240.7$ 280 $0.677$ 1B $12.9$ $27.764$ $5.03$ $117$ $31.1$ $3$ $12$ $27.00$ $200$ 280 $0.637$ 1B $12.9$ $27.764$ $5.03$ $31.4$ $12$ $27.200$ $200$ 280 $0.637$ 1B $12.9$ $27.764$ $5.03$ $31.3$ $31.3$ $3$ $3$ $3$ $30.0$ $210$ 290 $0.637$ $110$ $12.9$ $27.64$ $5.03$ $31.3$ $31.3$ $31.3$ $32.200$ $2000$ $200$ 200 $0.637$ $110$ $12.9$ $27.64$ $5.03$ $31.3$ $31.3$ $31.3$ $32.200$ $2000$ $200$ 210 $0.637$ $0.794$ $-100$ $0.637$ $0.637$ $0.637$ $0.637$ $0.637$ $0.637$ 210 $0.642$ $-100$ $0.636$ $0.637$	260         0.660         3B         28.6         72.59         2.07         110         2.6.1         9         6         2         200         0.00           260         0.645         3A         19.0         240.77         20.00         113         25.2         6         15.0         0.00         15.0         1600         1600         15.0         1600         110         2500         200         1600         170         31.1         31.0         31.0         300         111         31.1 <td< td=""><td></td><td><b>10% WESHSBA</b></td><td>240</td><td>0.400</td><td>3A</td><td>36.1</td><td>250.30</td><td>21.57</td><td>112</td><td>26.1</td><td>26</td><td>19</td><td>35</td><td>0-0</td><td>10-0</td><td>0-0</td></td<>		<b>10% WESHSBA</b>	240	0.400	3A	36.1	250.30	21.57	112	26.1	26	19	35	0-0	10-0	0-0
2600.6423A19.024.730.0911036.281161061061061001001002700.6133A4.6239.7620.731133.536156400800111010.000.6633B20.0027.445.0411731.05.65.0080012800.6673A21.233.025.7313531.11322222800.6701B12.927.735.0417731.231.113222222800.6701B12.927.735.0512031.11322222222222800.6701B10.827.665.0912531.11333	260         0.642         3A         19.0         240.77         50.4         110         25.2         8         11         6         15         60.0         110         25.2         8         11         6         30.0         110         25.2         6         15         60.0         100         100         100         110         25.2         6         15         60         30.0         11         110         110         25.2         6         200         100         110         25.2         6         15         60         200         100         110         25.2         6         200         100         110         25.2         6         200         100         110         25.2         31.1         13         21.2         32.02         31.3         31.1         13         2         20         200         200         200         200         200         200         200         200         200         200         21.2         32.02         31.3         31.3         31.3         31.3         31.3         31.3         31.3         31.3         31.3         31.3         31.3         31.3         31.3         30.0         30.0         30.0         <		<b>10% WESHLSA</b>	260	0.660	3B	28.6	272.59	22.77	110	26.1	6	6	62	20-0	0-0	0-0
280 $0.675$ 3B $31.4$ $24.79$ $20.76$ $113$ $25.2$ $6$ $12$ $6$ $42.0$ $16.00$ $11$ 280 $0.665$ $33$ $2$ $   -$ <td>270         0.675         3B         31.4         244.79         20.0         113         35.9         6         20         54         420.0         160.0         11           270         0.613         3A         4.6         239.76         20.75         113         36.2         6         15         6         15         6         15         6         15         90.0         160.0         160.0         160.0         160.0         11         17         31.1         13         2         6         20         24         40.0         160.0         11         2         25.2         4         5.04         17         31.1         13         3         13         3         13         3         13         3         13         3         13         3         13         3         13         3         13         3         13         3         13         3         13         3         13         3         13         3         13         3         13         3         13         3         13         3         13         13         3         13         3         13         3         13         13         13         13</td> <td></td> <td>10% SSO</td> <td>260</td> <td>0.642</td> <td>ЗA</td> <td>19.0</td> <td>240.77</td> <td>20.94</td> <td>110</td> <td>26.2</td> <td>80</td> <td>11</td> <td>61</td> <td>510-20</td> <td>150-0</td> <td>180-0</td>	270         0.675         3B         31.4         244.79         20.0         113         35.9         6         20         54         420.0         160.0         11           270         0.613         3A         4.6         239.76         20.75         113         36.2         6         15         6         15         6         15         6         15         90.0         160.0         160.0         160.0         160.0         11         17         31.1         13         2         6         20         24         40.0         160.0         11         2         25.2         4         5.04         17         31.1         13         3         13         3         13         3         13         3         13         3         13         3         13         3         13         3         13         3         13         3         13         3         13         3         13         3         13         3         13         3         13         3         13         3         13         3         13         13         3         13         3         13         3         13         13         13         13		10% SSO	260	0.642	ЗA	19.0	240.77	20.94	110	26.2	80	11	61	510-20	150-0	180-0
2700.6133A4.6239.7620.7511826.261560530.080.0110 $1020$ $3H$ $21.20$ $27.64$ $5.04$ $117$ $31.0$ $36$ $20$ $24$ $55.0$ $200$ 280 $0.6570$ $3H$ $21.29$ $27.64$ $5.04$ $117$ $31.0$ $36$ $200$ $200$ $200$ 300 $0.670$ $1H$ $12.2$ $27.78$ $5.03$ $133$ $31.4$ $12$ $27$ $66$ $1000$ $200$ 300 $0.673$ $1H$ $12.6$ $27.78$ $5.09$ $125$ $31.1$ $13$ $7$ $66$ $1000$ $200$ 300 $0.673$ $1H$ $12.6$ $27.78$ $5.09$ $125$ $31.1$ $13$ $3$ $24$ $2200$ $200$ $200$ $0.673$ $27.68$ $5.09$ $125$ $31.1$ $31$ $3$ $34$ $2200$ $300$ $11$ $280$ $0.675$ $27.68$ $5.09$ $125$ $31.1$ $3$ $3$ $2$ $2$ $2$ $280$ $0.675$ $27.68$ $5.09$ $125$ $31.1$ $3$ $3$ $200$ $300$ $300$ $300$ $280$ $0.675$ $0.675$ $0.675$ $0.675$ $0.675$ $0.675$ $0.675$ $0.675$ $280$ $0.676$ $0.675$ $0.675$ $0.675$ $0.675$ $0.675$ $0.675$ $0.675$ $200$ $0.676$ $0.676$ $0.675$ $0.675$ $0.675$ </td <td>270       0.613       3A       4.6       239.76       20.75       118       2.6.2       6       15       60       530-0       80-0       1         110       1.0265       3       2       2.0       17       31-0       35       20       27-0       29-0         280       0.657       3A       2.12       32.02       5.73       135       31.1       13       7       60       100-0       20-0<!--</td--><td></td><td>10% Com Sub A</td><td>280</td><td>0.675</td><td>3B</td><td>31.4</td><td>244.79</td><td>20.90</td><td>113</td><td>25.9</td><td>9</td><td>20</td><td>54</td><td>420-0</td><td>160-0</td><td>120-0</td></td>	270       0.613       3A       4.6       239.76       20.75       118       2.6.2       6       15       60       530-0       80-0       1         110       1.0265       3       2       2.0       17       31-0       35       20       27-0       29-0         280       0.657       3A       2.12       32.02       5.73       135       31.1       13       7       60       100-0       20-0 </td <td></td> <td>10% Com Sub A</td> <td>280</td> <td>0.675</td> <td>3B</td> <td>31.4</td> <td>244.79</td> <td>20.90</td> <td>113</td> <td>25.9</td> <td>9</td> <td>20</td> <td>54</td> <td>420-0</td> <td>160-0</td> <td>120-0</td>		10% Com Sub A	280	0.675	3B	31.4	244.79	20.90	113	25.9	9	20	54	420-0	160-0	120-0
110 $1.020$ $3$ $20.0$ $27.64$ $5.04$ $117$ $31.0$ $36$ $20$ $20-0$ $20-0$ 280 $0.657$ $3A$ $21.2$ $27.08$ $5.03$ $117$ $31.1$ $36$ $20-0$ $20-0$ 280 $0.670$ $1B$ $12.6$ $27.78$ $5.05$ $122$ $31.3$ $12$ $26$ $200-0$ $20-0$ 270 $0.670$ $1B$ $12.6$ $27.78$ $5.05$ $122$ $31.3$ $31.2$ $26$ $200-0$ $20-0$ 280 $0.794$ 280 $0.642$ 280 $0.642$ 280 $0.658$ $5.09$ $126$ $31.3$ $31.3$ $31.3$ $32.200$ $30.0$ $11$ 280 $0.658$ $0.658$ $0.658$ $0.658$ $0.658$ $0.656$ $0.656$ $0.656$ 290 $0.553$ $0.558$ $0.558$ $0.558$ $0.556$ $0.566$ $0.523$ $0.523$ 200 $0.523$ $0.5670$ $0.5670$ $0.5670$ $0.5670$ $0.5670$ $0.5670$ 200 $0.5670$ $0.5670$ $0.5670$ $0.5670$ $0.5670$ $0.590$ $0.570$ 200 $0.5884$ $0.5884$ $0.6870$ $0.5884$ $0.6870$ $0.670$ $0.670$ 200 $0.5670$	110       1.020       3       20.0       27.64       5.04       117       31.0       36       20       24       25.0       40.0         280       0.6570       3A       21.2       27.04       5.04       117       31.0       36       20       24.0       20.0		10% Com Sub B	270	0.613	ЗA	4.6	239.76	20.75	118	26.2	9	15	60	530-0	80-0	110-0
280       0.665       3B       20.0       27.64       5.04       117       31.0       36       27       66       25.0       20.0       11.1       31.3       31.3       31.3       31.3       31.3       31.3       31.3       21.0       20.0       20.0       20.0       20.0       10.0       10.0       10.0       10.0       10.0       10.0       10.0       20.0       11.1       31.1       31.3       31.1       31.3       31.3       31.3       21.0       20.0       21.	280         0.665         3B         20.0         27.64         5.04         117         31.0         36         27         66         25.00         20.0           300         0.697         1B         12.2         37.78         5.05         12.3         31.1         13         3         13         3         13         23         29.0         20.0         21.0         20.0         100         11         13         7         66         20.0         20.0         11           270         0.673         5.09         12.6         5.773         5.09         12.5         31.1         13         3         13         3         13         230.0         20.0         10         1           2700         0.573         5.09         12.5         5.09         12.5         31.1         13         3         13         3         3         230.0         0.0         1         1         230.0         10         1         2         2         1         1         1         1         1         1         2         2         1         1         1         1         1         1         1         1         1         1         1	DD	None	110	1.020	I	ł	I	}	ļ	I	t	I	I	I	I	1
280         0.670         3A         212         32.02         5.73         135         31.1         13         7         60         1000         200           300         0.630         1B         12.9         37.78         5.05         122         31.3         31.3         31.3         31.3         32         36         2300         200         11           270         0.630         1B         12.9         37.78         5.05         122         31.3         31.3         32         36         2300         200         1           280         0.753         5.09         125         31.1         5         0         7         280         30.0         1           280         0.673         - <td< td=""><td>280         0.670         3A         212         32.02         5.73         135         31.1         13         7         60         100-0         20-0           360         0.637         1B         12.9         27.68         5.09         122         31.3         3         13         3         3         2         60         100-0         20-0         1           270         0.642         -         <td< td=""><td></td><td>10% WESHSBA</td><td>280</td><td>0.665</td><td>3B</td><td>20.0</td><td>27.64</td><td>5.04</td><td>117</td><td>31.0</td><td>36</td><td>20</td><td>24</td><td>25-0</td><td>40-0</td><td>0-0</td></td<></td></td<>	280         0.670         3A         212         32.02         5.73         135         31.1         13         7         60         100-0         20-0           360         0.637         1B         12.9         27.68         5.09         122         31.3         3         13         3         3         2         60         100-0         20-0         1           270         0.642         - <td< td=""><td></td><td>10% WESHSBA</td><td>280</td><td>0.665</td><td>3B</td><td>20.0</td><td>27.64</td><td>5.04</td><td>117</td><td>31.0</td><td>36</td><td>20</td><td>24</td><td>25-0</td><td>40-0</td><td>0-0</td></td<>		10% WESHSBA	280	0.665	3B	20.0	27.64	5.04	117	31.0	36	20	24	25-0	40-0	0-0
300         0.697         1B         12.9         27.78         5.05         123         31.4         12         250.0         20.0         12           270         0.6713         1B         12.6         27.73         5.05         140         31.3         33         13         34         20.0         20.0         10           120         0.773         1B         12.6         27.73         5.05         120         31.3         33         13         34         20.0         10         1           280         0.643         - <td>300         0.697         18         12.9         37.78         5.05         123         31.4         12         5.500         2000         12           270         0.610         18         12.6         27.72         5.26         140         31.1         3         13         3         3         3         2000         2000         1           270         0.620         18         12.6         27.72         5.25         140         31.1         3         13         3         3         300         2000         2000         1           280         0.643         -</td> <td></td> <td>10% WESHLSA</td> <td>280</td> <td>0.670</td> <td>3A</td> <td>21.2</td> <td>32.02</td> <td>5.73</td> <td>135</td> <td>31.1</td> <td>13</td> <td>2</td> <td>60</td> <td>100-0</td> <td>20-0</td> <td>95-0</td>	300         0.697         18         12.9         37.78         5.05         123         31.4         12         5.500         2000         12           270         0.610         18         12.6         27.72         5.26         140         31.1         3         13         3         3         3         2000         2000         1           270         0.620         18         12.6         27.72         5.25         140         31.1         3         13         3         3         300         2000         2000         1           280         0.643         -		10% WESHLSA	280	0.670	3A	21.2	32.02	5.73	135	31.1	13	2	60	100-0	20-0	95-0
360       0.713       1B       12.6       27.72       5.26       140       31.3       33       13       34       220-0       20-0       1         270       0.620       1B       16.8       27.68       5.09       125       31.1       5       0       75       280-0       30-0       1         280       0.675       - <td>360       0.713       1B       12.6       27.72       5.26       140       31.3       33       13       34       220.0       20.0       1         270       0.6520       1B       16.8       27.68       5.09       125       31.1       5       0       75       280.0       30.0       1         280       0.6573       -<!--</td--><td></td><td>10% SSO</td><td>300</td><td>0.697</td><td>1B</td><td>12.9</td><td>27.78</td><td>5.05</td><td>122</td><td>31.4</td><td>12</td><td>7</td><td>66</td><td>250-0</td><td>20-0</td><td>80-0</td></td>	360       0.713       1B       12.6       27.72       5.26       140       31.3       33       13       34       220.0       20.0       1         270       0.6520       1B       16.8       27.68       5.09       125       31.1       5       0       75       280.0       30.0       1         280       0.6573       - </td <td></td> <td>10% SSO</td> <td>300</td> <td>0.697</td> <td>1B</td> <td>12.9</td> <td>27.78</td> <td>5.05</td> <td>122</td> <td>31.4</td> <td>12</td> <td>7</td> <td>66</td> <td>250-0</td> <td>20-0</td> <td>80-0</td>		10% SSO	300	0.697	1B	12.9	27.78	5.05	122	31.4	12	7	66	250-0	20-0	80-0
270       0.620       1B       16.8       27.68       5.09       125       31.1       5       0       75       280-0       30-0       1         280       0.673       - <td>270       0.620       1B       16.8       27.68       5.09       125       31.1       5       0       75       280-0       30-0       1         280       0.673       -<td></td><td>10% Com Sub A</td><td>360</td><td>0.713</td><td>1B</td><td>12.6</td><td>27.72</td><td>5.26</td><td>140</td><td>31.3</td><td>33</td><td>13</td><td>34</td><td>220-0</td><td>20-0</td><td>100-0</td></td>	270       0.620       1B       16.8       27.68       5.09       125       31.1       5       0       75       280-0       30-0       1         280       0.673       - <td></td> <td>10% Com Sub A</td> <td>360</td> <td>0.713</td> <td>1B</td> <td>12.6</td> <td>27.72</td> <td>5.26</td> <td>140</td> <td>31.3</td> <td>33</td> <td>13</td> <td>34</td> <td>220-0</td> <td>20-0</td> <td>100-0</td>		10% Com Sub A	360	0.713	1B	12.6	27.72	5.26	140	31.3	33	13	34	220-0	20-0	100-0
120       0.794       -         280       0.642       -         280       0.675       -         280       0.668       -         280       0.553       -         280       0.553       -         280       0.553       -         230       0.553       -         300       0.553       -         300       0.553       -         320       0.506       -         230       0.506       -         230       0.506       -         231       0.500       -         230       0.500       -         230       0.500       -         230       0.500       -         230       0.500       -         230       0.500       -         230       0.500       -         230       0.500       -         230       0.500       -         230       0.500       -         230       0.500       -         230       0.500       -         230       0.500       -         230       0.504 <td< td=""><td>120       0.794       -         280       0.642       -         280       0.675       -         280       0.675       -         280       0.668       -         280       0.553       -         280       0.553       -         280       0.553       -         300       0.553       -         300       0.553       -         300       0.553       -         300       0.553       -         320       0.500       -         220       0.500       -         230       0.500       -         230       0.500       -         230       0.500       -         230       0.500       -         230       0.500       -         280       0.500       -         280       0.570       -         280       0.570       -         280       0.570       -         280       0.570       -         280       0.570       -         280       0.581       -         3807       -       -&lt;</td><td>E</td><td>10% Com Sub B</td><td>270</td><td>0.620</td><td>1B</td><td>16.8</td><td>27.68</td><td>5.09</td><td>125</td><td>31.1</td><td>5</td><td>0</td><td>75</td><td>280-0</td><td>30-0</td><td>100-0</td></td<>	120       0.794       -         280       0.642       -         280       0.675       -         280       0.675       -         280       0.668       -         280       0.553       -         280       0.553       -         280       0.553       -         300       0.553       -         300       0.553       -         300       0.553       -         300       0.553       -         320       0.500       -         220       0.500       -         230       0.500       -         230       0.500       -         230       0.500       -         230       0.500       -         230       0.500       -         280       0.500       -         280       0.570       -         280       0.570       -         280       0.570       -         280       0.570       -         280       0.570       -         280       0.581       -         3807       -       -<	E	10% Com Sub B	270	0.620	1B	16.8	27.68	5.09	125	31.1	5	0	75	280-0	30-0	100-0
120       0.794       -         280       0.642       -         280       0.675       -         280       0.658       -         280       0.553       -         280       0.553       -         280       0.553       -         230       0.558       -         300       0.553       -         310       0.558       -         320       0.500       -         230       0.500       -         230       0.500       -         230       0.500       -         230       0.500       -         230       0.500       -         230       0.500       -         230       0.500       -         230       0.500       -         230       0.500       -         230       0.500       -         230       0.500       -         230       0.500       -         258A, wax esters from selectively hydrogenated so       -         ASTM Method D 130-65: 1, slight tarmish; 2, medi       -	120       0.794       -         280       0.642       -         280       0.642       -         280       0.658       -         280       0.658       -         280       0.553       -         280       0.568       -         280       0.553       -         280       0.553       -         300       0.558       -         310       0.506       -         280       0.500       -         280       0.500       -         280       0.500       -         280       0.500       -         280       0.500       -         280       0.670       -         280       0.670       -         280       0.670       -         280       0.671       -         281       wax esters from selectively hydrogenated so         4STM Method D 130-65: 1, slight tarnish; 2, medi         10.7       -         803       -         2803       -         2803       -	Topaz	2														
280 0.042 280 0.675 280 0.553 280 0.553 300 0.558 300 0.503 320 0.500 230 0.596 230 0.596 230 0.596 28A, wax esters from selectively hydrogenated so SBA, wax esters from selectively hydrogenated so ASTM Method D 130-65: 1, slight tarnish; 2, medi	<ul> <li>280 0.675</li> <li>280 0.678</li> <li>280 0.553</li> <li>280 0.553</li> <li>280 0.553</li> <li>280 0.523</li> <li>300 0.523</li> <li>200 0.506</li> <li>320 0.506</li> <li>320 0.596</li> <li>280 0.506</li> <li>280 0.506</li> <li>280 0.670</li> <li>280 0.670</li> <li>281 vax esters from selectively hydrogenated so</li> <li>4STM Method D 130-65: 1, slight tarnish; 2, medi</li> <li>finstitute.</li> </ul>	\$010	None	071	0.794	i	I	ł	١	١	I	l	I	I	I	I	I
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oub b, commercial substitute b. Copper strip corrosion test ASTM Method D 130-65: 1, slight tarnish; 2, medium tarnish; 4, corrosion. dCs, Centistokes. <sup>e</sup> API = American Petroleum Institute.	oud b, commercial substitute b. Copper strip corrosion test ASTM Method D 130-65: 1, slight tarnish; 2, medium tarnish; 4, corrosion. dCs, Centistokes. eAPI = American Petroleum Institute. froam test ASTM Method D 807 securation of highhing 5 min and settling 10 min: 1 at 75 F after collansing the foam	<sup>a</sup> Base oi oil; Topaz S <sup>b</sup> Sulfuri	ils: AA, Southwest I 105, Topaz S105 pa zed additives: WESH	Research Instit raffin oil (simil ISBA, wax este	ute's (SWRI) crai llar to 102 paraffi ers from selectivel	nkcase base c n oil). ly hydrogenat	il; BB, SWRI ed soybean ac	transmissi ds; WESH	on base flu LSA, wax e	iid; CC, SWRI esters from sele	(RGO-100) gear 1. ectively hydrogen.	ubricant; I ated linsee	DD, Mayco ed acids; Co	's (100/100 2m Sub A, e	0 vis) solven commercial	t extracted substitute	l neutral A; Com
dCs, Centistokes. <sup>e</sup> API = American Petroleum Institute.	dCs, Centistokes. <sup>e</sup> API = American Petroleum Institute. <sup>f</sup> Fram test ACTM Method D 807 servience of hithhline 5 min and settline 10 min: 1 at 75 F after collansine the fram	Copper	strip corrosion test	ASTM Method	l D 130-65: 1, slig	ht tarnish; 2,	medium tarn	ish; 3, dark	: tarnish; 4,	corrosion.							
<sup>e</sup> API = American Petroleum Institute.	<sup>e</sup> API = American Petroleum Institute. Froam test ASTM Method D 802 servinges of highling 5 min and settling 10 min : 1 at 75 F : 11 at 75 F after collansing the fram	<sup>d</sup> Cs, Cer	ıtistokes.														
	fFrom test ASTM Method D 802 services of highling 5 min and settling 10 min : 1 at 75 F: 11 at 76 F after collansing the from	f = IdVa	American Petroleum	Institute.													

TABLE IV

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in base oils AA, BB, CC, DD, and Topaz S 105. SSO and two commercial SSO substitutes (Com Sub A, Com Sub B) are included for further comparison. Most commercial sulfurized replacements are sold as "packages" containing a number of additives, such as viscosity improver, metal deactivator, antioxidants, and EP agents. The sulfurized wax esters from hydrogenated fatty acids contained no additives nor were they winterized before sulfurization. SSO replacements were added to each base oil at 10% by weight concentration levels. The blended oils were stored for 24 hr at 35 F, 24 hr at room temperature, 24 hr at 35 F, and then 1 mo at room temperature. All sulfurized materials had good solubilities in all the base oils.

Both wear and EP test data were obtained with sulfurized WESHSBA and WESHLSA as 10% by weight concentration in all base oils. Wear and EP tests for SSO, Com Sub A, and Com Sub B on the basis of 10% by weight concentration in all the base oils are given for comparison. Although data suggest that Topaz S 105 was a reasonable choice for screening candidate materials, replacements performed differently in each of the base oils; therefore, the choice of a given additive will depend on its intended application. All sulfurized products showed both EP characteristics and antiwear properties. At 5% concentration in Topaz S105, sulfurized additives WESHSBA and WESHLSA exhibited EP properties better than those of SSO or of Com Sub A and B. SSO, Com Sub A and B showed better antiwear properties than the wax esters.

Although the sulfurized wax esters appeared to be effective EP agents, they gave a copper corrosion test of 2B to 3B. EP additives should have a copper corrosion rating of 2C or better. The higher than desired corrosion ratings and antiwear values may have been caused either by a too high sulfurization level or by sulfur-contained impurities, or both. Copper corrosion tests run with wax esters as 10% blend in base oils AA, BB, CC, and DD showed near acceptable values as did SSO in base oil CC, and Com Sub A in base oils AA, BB, and CC. Perhaps with proper adjustment of either sulfur concentration or addition of metal-deactivators, or both, the additives would possibly improve greatly in antiwear, and anti-copper corrosion properties.

Sulfurized WESHLSA had higher than expected lead corrosion in base oils AA, BB, and CC; comparable to that of Com Sub A. In base oils AA and BB, sulfurized WESHSBA exhibited lead corrosion tendencies much less than did SSO or Com Sub A and B.

Values of kinematic viscosity data, viscosity indices, and API gravities of all materials tested as 10% blends in the four base oils are within most industrial and military specification for lubricants containing EP additives.

In summarizing emulsion test data of additives as 10% blends in the four base oils, all additives form stable emulsions with the four base oils and are suitable in this regard for marine engine lubrication and cutting oils. Candidate additives in base oils BB and CC exhibit excellent deemulsification properties and should find application in forced-feed circulating lubrication systems, provided that the other physical and chemical properties required of such systems are also met.

Foam test data of additives as 10% blends in the four base oils demonstrate that all candidate additives met test requirements. Sulfurized WESHSBA and WESHLSA showed less foaming tendencies than did SSO or Com Sub A and B in base oils AA, CC, and DD. In base oil BB, sulfurized WESHSBA and WESHLSA, SSO and Com Sub A showed no foam tendencies.

Thermal stability test FTM 2504-1 was made with 10% by weight additives concentration in base oil CC. The sulfurized WESHSBA blend failed the test because the increase in viscosity was higher than permissible (100%). The sulfurized WESHLSA blend polymerized after 40 hr of test. "Although this material is apparently not suitable for engine gear application, it did seem to perform reasonably well otherwise and may find application as an industrial lubricant requiring lower than 300 F operating temperature or thermal stability.

Since most lubricants are formulated with a number of additives, each having certain performance characteristics, sperm oil replacement candidates could not be expected to meet all lubricant specifications. However, the sulfurized candidate additives have good EP properties and are superior to SSO and commercial additives in foaming tendencies. Thermal stability and copper and lead corrosion tendencies exhibited by the wax esters are less than desired, but can be improved by including either an appropriate metal deactivator or antioxidant, or both.

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