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THE SYNTHESIS OF PHOSPHORUS- AND SULPHUR-CONTAINING COMPOUNDS AND A STUDY OF THEIR ACTION ON THE QUALITY OF LUBRICATING OILS

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Compds. contg. both S and P, giving good anticorrosion and detergency properties to lubricating oils and having good compatibility with other additives, were prepd. by treating dialkyl dithiophosphates ((RO)2P(S)SH) with unsatd. compds., e.g. MeCN, Me methacrylate, or vinyl acetate to give trialkyl dithiophosphates; by polymg., generally in the presence of $AlCl_3$, paraffin or kerosene thermal cracking products, the residue from petroleum cracking, low mol. wt. polymers, or isobutylene, treating the polymers with P_2S_5, and converting the products to their Ca, Ba, or Zn salts; and by converting mixts. of dithiophosphate and dithiophosphonate diesters prepd. from P_2S_5 and octyl alc., alkylphenols, and (or) polymer distillates to their An or Ba salts. [AT1206758]

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Organic Phosphorus Compound						
Anticorrosive Additive						
Detergent Additive						
Thermal Cracking						
Petroleum Product						
Calcium						
Barium						
Zinc						

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THE SYNTHESIS OF PHOSPHORUS- AND SULFUR-CONTAINING COMPOUNDS AND A STUDY OF THEIR ACTION ON THE QUALITY OF LUBRICATING OILS

R. K. Mamedova, S. M. Abutalybova

At the present time there are a limited number of antioxidizing additives recommended to improve the stability of motor oils. Of these, phosphorus- and sulfur-containing additives are some of the most effective.

This article will review our experiments with the synthesis and study of various organic compounds which contain phosphorus and sulfur.

Studies of the synthesis of these compounds were conducted in two directions:

1) the synthesis of dithio-phosphoric acid triesters;

2) the production of dialkenyl-dithiophosphine salts and diester-dithiophosphoric acids.

The combination reaction of dithiophosphoric acid esters to unsaturated compounds was used to synthesize dithiophosphoric acid triesters.

Trialkyl-dithiophosphates and combined dithiophosphoric acid esters were produced.

Trialkyl-dithiophosphates were produced [1, 2] by adding dialkyl-dithiophosphoric acids to various unsaturated hydrocarbons according to the equation

$$(RO)_2 PSSH + C_nH_{2n} \longrightarrow (RO)_2 PSSR',$$

were R is the alkyl radical from $C_1 - C_4$,

and R' is the alkyl radical from $C_6 - C_{10}$, cyclohexane and methylcyclohexane.

[illegible] ters of co[illegible]inc[illegible] dit[illegible]iophosphoric acid were produced by adding [illegible]iophosphoric acid to acrylnitryl, acrylic acid methyl ether and [illegible] tate, and [illegible]signated SA-1, SA-2, and SA-3, respectively.

Analysis of the esters produced is given in Table 1.

TABLE 1

Name of ester	Found, %		Calculated, %	
	S	P	S	P
SA-1	14,92	7,0	16,0	7,8
SA-2	12,63	5,56	14,3	6,7
SA-3	12,84	5,87	14,6	7,2

The dithiophosphoric acid esters produced were tested with D-11 oil. It was established that trialkyl-dithiophosphates have high anti-corrosion properties and the ability to increase the strength of the oil film. Testing according to the Central Scientific Research Institute of Automobiles and Automobile Engines (NAMI) method under severe conditions has shown that the addition of 2% trialkyl-dithiophosphates is sufficient to eliminate corrosion of D-11 oil.

SA type esters have greater anti-corrosion properties than trialkyl-dithiophosphates. When 1% of these additives is added to D-11 oil, its corrosion is completely eliminated.

To determine the compatability of type SA additives with SB-3 additives, samples were prepared with D-11 oil and subjected to preliminary laboratory studies. Table 2 shows the results of the tests.

TABLE 2. RESULTS OF LABORATORY STUDIES OF TYPE SA ADDITIVES

Sample	Corrosion according to (severe) NAMI method, g/m²	Detergent properties according to PZV* points
D-11 oil, selective refinement	360	5.5
D-11 oil with additives:		
1% SA-1	0.25	–
1% SA-2	0.45	–
1% SA-3	2.90	–
3% SB-3 + 2% SA-1	0.85	3.5
3% SB-3 + 2% SA-2	0.65	2.5
3% SB-3 + 2% SA-3	10.85	2.5 - 3.0
5% SB-3 + 1% SA-1	0.5	1.0
5% SB-3 + 1% SA-2	1.0	–
5% SB-3 + 1% SA-3	7.9	1.0 - 1.5

*This designates ignition delay period.

As is evident from the data in Table 2, the best results with respect to detergent and anticorrosion characteristics were obtained by mixing 5% SB-3 and 1% SA-2.

The positive results obtained in the preliminary laboratory studies of D-11 oil containing 5% SB-3 and 1% SA-2 were verified in tests in a GAZ-51 engine for 150 hours.

We also synthesized phosphorus- and sulfur-containing organic compounds based on reaction products of phorphorus pentasulfide with unsaturated hydrocarbons and their polymers.

Raw materials containing unsaturated hydrocarbons which were used included fractions of paraffin thermal cracking, residue of petroleum thermal cracking, low-molecular polymers and polymer distillate produced in the polymerization of a butane-butylene fraction.

By treating ten-degree paraffin-cracking fractions with phosphorus pentasulfide and zinc oxide, we produced zinc salts with a 16 - 18% ash content, containing 6 - 9% sulfur and 2 - 8% phosphorus.

To produce phosphorus- and sulfur-containing additives on a base of low-molecular polymers, we polymerized a broad fraction of distillate of paraffin thermal cracking, boiled off at 140 - 250°, kerosene of mazut thermal cracking, boiled at 120 - 170°, as well as isobutylene.

Broad fractions of the distillate of thermal cracking of paraffin and kerosene were polymerized in the presence of aluminum chloride at a temperature of -10°. After appropriate processing, polymers with a 28 - 40% yield were produced.

Conditions were found to polymerize isobutylene in the presence of aluminum chloride, providing poly-isobutylene with a molecular weight of 600 - 1400 and yields of 71 - 87% [3].

These polymers were reacted with phosphorus pentasulfide at temperatures of 140 - 220° C.

The optimum amount of phosphorus pentasulfide for treating the polymer was 15%, as increasing the amount of phorphorus pentasulfide from 15 to 20% did not significantly increase the amount of sulfur and phosphorus in the phosphorosulfated polymer.

The latter was subsequently neutralized to produce various salts, whose quality is given in Table 3.

TABLE 3. THE QUALITY OF SALTS PRODUCED ON A BASE OF PHOSPHORUS- AND SULFUR-CONTAINING POLYMERS

Product	Molecular weight of original polymer	Ash content %	Concentration, %	
			Phorphorus	Sulfur
Na-salt of phosphorus- and sulfur containing polyisobutylene	826	7.8	1.45	3.55
" "	703	6.2	1.79	3.91
" "	1035	6.0	1.81	4.70
-salt of phosphorus- and sulfur-containing polyisobutylene	1035	10.5	1.78	4.60
SA-salt " "	1035	—	1.53	4.05
VA-salt of phosphorus- and sulfur-containing polymer produced from distillate of paraffin heat-cracking	570	8.5	2.30	3.20
-salt " "	570	9.8	2.20	2.10
Na-salt of phosphorus and sulfur-containing polymer produced from kerosene distillate of mazut heat cracking	—	9.1	2.70	3.10
-salt " "	—	7.8	2.80	2.90

Polymer distillate was also used as a source material for phosphoro-sulfation.

Salts produced from phosphorus-sulfated polymer-distillates were unstable. Therefore, the effect of catalyst $AlCl_3$ and steam on the quality of salts was studied.

The phosphoro-sulfation of the polymer distillate was conducted:

a) in the presence of catalyst $AlCl_3$;

b) by treating the reaction product with superheated steam without the catalyst;

c) in the presence of catalyst $AlCl_3$ with subsequent treatment of the reaction product with superheated steam.

Barium, calcium and zinc salts were synthesized from phosphoro-sulfated polymer distillate produced by the three methods. Results of the analysis of these salts are given in Table 4.

TABLE 4. RESULTS OF ANALYSIS OF DIALKENYL-DITHIOPHOSPINE ACID SALTS

Salts	$AlCl_3$	Steam	Analysis		
			P, %	S, %	Ash, %
Va	+		[illegible]	5,1	24,4
Sa	+		[illegible],45	[illegible],9	14,7
	+		8,13	9,5	18,1
Va		+	[illegible]	5,9	15,0
Sa		+	5,61	8,0	27,0
		+	7,73	8,9	22,7
Va	+	+	7,73	14,9	18,0
Sa	+	+	10,96	10,9	22,0
	+	+	7,24	8,6	27,0

It is evident from the table that the analyses of additives produced by the three methods differ very little from each other.

However, the additives produced by the third method are clearer, lighter and more stable than the additives produced by the other methods. Barium salt, produced by the third method, is called additive IKhP 2/11.

Phosphorus- and sulfur containing additives were also synthesized on the base of the distillate residue of petroleum heat cracking, boiled off at a temperature above 240°. For this the distillate residue was treated with 15% phosphorus pentasulfide at temperatures of 130 - 140°. The reaction product was diluted with AS-6 oil in a 1 : 1 ratio and neutralized with baric hydroxide at temperatures of 80 - 120° C.

The additive produced was called INKhP-40, and was of the following quality:

Ash content	5.5%
Sulfur content	5.0%
Phosphorus content	2.25%

All the phosphorus- and sulfur-containing compounds synthesized on a base of unsaturated hydrocarbons were tested as motor oil additives.

When 1% zinc salts of phosphoro-sulfated fractions of paraffin heat cracking products were added to D-11 oil, its corrosion was reduced to 0.5 - 1.0 g/m^2.

Mixtures of SU machine oil with 1% and 3% of various salts of phosphorus- and sulfur-containing polymers were tested for corrosion by the NAMI method and for heat stability by the Papok method at temperatures of 250° C. Test results are given in Table 5.

TABLE 5. LABORATORY TEST RESULTS OF SU MACHINE OIL COMBINED WITH VARIOUS SALTS OF PHOSPHORUS- AND SULFUR-CONTAINING POLYMERS

Sample	Molecular weight of original polymer	Thermal stability at 250° C, min	Coefficient of lacquer formation $K_{lacq.}$	Corrosion according to NAMI method, g/m²	
				10 hrs.	25 hrs.
Machine oil SU	-	17	0,90	25	360
Same + 1% Va-salt of phosphorus- and sulfur-containing poly-isobutylene	703	36	0,22	2,4	[illegible]
Same + 1% Va-salt -"-	[illegible]	37	0,36	[illegible]	[illegible]
Same + 1% Va-salt -"-	1035	50	0,10	1,5	14
Same + 1% Sa-salt -"-	1035	41	0,12	32	-
Same + 1% Sa-salt -"-	1035	31	0,25	[illegible]	2
Same + 3% Va-salt -"-	1035	51	0,15	none	2
Same + 3% Sa-salt -"-	1035	44	0,13	2	16
Same + 3% Sa-salt -"-	1035	36	0,23	none	none
Same + 3% Va-salt of phosphorus- and sulfur-containing polymer produced from paraffin-cracking distillate	-	78	0,21	8	-
Same + 3% -salt -"-	-	35	0,56	3	27
Same + 3% Va-salt of phosphorus- and sulfur-containing polymer produced from kerosene cracking distillate	-	92	0,12	6	35
Same + 3% -salt -"-	-	51	0,33	2	22

Good results with respect to reducing corrosion from 360 to 0, determined according to the severe NAMI method, were obtained with zinc salt of phosphorus- and sulfur-containing poly-isobutylene with a molecular weight of 1035.

An effective increase in heat stability is observed by adding barium salts of polymers produced from cracking-product distillates.

Of the salts of dialkenyl-dithiophosphine acids produced from polymer distillate, the best anti-corrosion properties belonged to additive IKhP 2/11, which was given more detailed study.

To reveal the compatability of additive IKhP 2/11 with sulfonate additive SB-3, the following two samples were prepared and subjected to laboratory study:

	Detergent properties, according to PZV method, points	Corrosion according to severe method, g/m^2
D-11 oil + 3% SB-3 + 2% IKhP 2/11	2.0	20.6
D-11 oil +5% SB-3 + 1% IKhP 2/11	1.5	2.9

As is evident from these data, additive IKhP 2/11 combines well with sulfonated additive SB-3.

Preliminary laboratory tests established that additive INKhP-40 has high anti-oxidizing and anti-corrosion properties. Additives were composed on a base of additives INKhP-40 and SB-3, with D-11 oil, which was subjected to laboratory testing as well as brief tests in a GAZ-51 engine for 150 hours.

The test results were compared with those of two samples taken as the standard:

1) AS-9, 5NKZ with 2.6% OLOA-2054 and 0.6% OLOA-267;

2) D-11 with 2.6% OLOA-2054 and 0.6% OLOA-267.

To compare the effectiveness of additive INKhP-40 with additive OLOA-267 of the Orobis firm, tests were made of D-11 oil with 5% additive SB-3 and 0.6% OLOA-267.

The results from tests of these samples are given in Tables 6 and 7, from which it is evident that compositions based on additives SB-3 and INKhP-40 with group B oils, according to preliminary laboratory and engine testing, differ little from the composition of additives based on those of the Orobis firm.

TABLE 6. RESULTS OF LABORATORY TEST OF OILS WITH ADDITIVE COMPOSITION

Sample Name	Thermal stability according to Papok method, at 250° C min	Corrosion according to severe (NAMI) method, g/m^2	Detergent properties according to PZV method, points	Alkalinity mg KOH	
				pH to 9	pH to 4
Oil AS-9, 5 NKZ + 2.6% OLOA-2054 + 0.6% OLOA-267	41	5.2	–	0.26	2.0
Diesel oil D-11 + 2.6% OLOA-2054 + 0.6% OLOA-267	46	0.5	0	0.68	2.2
Diesel oil D-11 + 5% SB-3 + 0.6% OLOA-267	41	1.4	1.0	0	0.57
Diesel oil D-11 + 5% SB-3 + 1% INKhP-40	65	–	1.5	0	0.19

A comparison of the results of testing the anti-oxidizing additives INKhP-40 and OLOA-267 shows that additive INKhP-40 is on practically the same level of effectiveness with additive OLOA-267.

Studies were also conducted on producing new, more effective anti-oxidizing additives for lubricating oils. These additives were synthesized, using more readily-available compounds as the initial material — namely, the chemical industry's alkylphenol, octyl alcohol and polymer distillate.

TABLE 7. RESULTS OF 150-HOUR TESTS OF OILS WITH ADDITIVE COMPOSITIONS IN GAZ-51 ENGINE

Sample / Index	Oil AS-9, 5NKZ + 2.6% OLOA-2054 + 0.6% OLOA-267 (standard)	Oil D-11 + 2.6% OLOA-2054 + 0.6 % OLOA-267	Oil D-11 + 5% SB-3 + 0.6% OLOA-267	Oil D-11 + 5% SB-3 + 1% INKhP-40
Piston ring wear by weight, mg				
1st ring	100	225	137	94
2nd ring	100	167	118	110
3rd ring	100	206	167	127
Connecting rod bearing wear, mg				
Upper	100	113	113	40
Lower	100	96	90	72
Ring mobility	100	100	100	100
Cleanness of skirt points	100	108	194	228
Carbon deposit, g				
From rings	100	133	206	141
From grooves	100	216	1000	580
Total: from rings & grooves	100	148	430	252
From side surface	100	141	95	248
From bottom	100	100	–	78

These compounds are treated with phosphorus pentasulfide and subsequently neutralized with zinc oxide and baric hydroxide.

Additives were produced in two ways:

1) by combined neutralization of dithiophosphoric and dithiophosphinic acid;

2) by neutralization of the phosphoro-sulfated mixture of initial products.

In the first case, the reaction of phosphorus pentasulfide with alkylphenol or with octyl alcohol produced esters of dithiophosphoric acid, and the reaction of phosphorus pentasulfide with a polymer distillate produced esters of dithiophosphinic acid. Esters of dithiophosphoric and dithiophosphinic acids were combined in a 1 : 1 ratio and neutralized with zinc oxide.

To produce anti-oxidizing additives by the second method, the initial products (alkylphenol + polymer distillate or octyl alcohol + polymer distillate) were first mixed in a 1 : 1 ratio (mole); then the mixture was treated with phosphorus pentasulfide and neutralized with the metal oxide.

The additives produced are analyzed in Table 8.

The additive produced by the second method (No. 1) from a phosphoro-sulfurated mixture of normal primary octyl alcohol and polymer distillate, is called IKnP 2/12, and that from a phosphoro-sulfated mixture of alkylphenol and polymer distillate — IKhP 2/13 (No. 2). We must note that additives IKhP-2/12 and IKhP 2/13 differ very little in their ash, sulfur and phosphorus content. However, according to mobility and solubility in the oils, additive IKhP-2/12 is better.

TABLE 8. ANALYSIS OF ADDITIVES PRODUCED

No.	Additives	Means of production	Analysis		
			Ash, %	Phosphorus %	Sulfur, %
1.	$\left[\begin{pmatrix} C_8H_{17}O \\ C_nH_{2n-1} \end{pmatrix}_2 PS_2\right]_2 Zn$	II	28,7	8,13	10,95
2.	$\left[\begin{pmatrix} RC_6H_4O \\ C_nH_{2n-1} \end{pmatrix}_2 PS_2\right]_2 Zn$	II	24,3	6,49	11,35
3.	$\begin{matrix} (C_8H_{17}O)_2\ PS_2 \\ (C_nH_{2n-1})_2\ PS_2 \end{matrix} > Zn$	I	35,0	5,50	12,57
4.	$\begin{matrix} (RC_6H_4O)_2\ PS_2 \\ (C_nH_{2n-1})_2\ PS_2 \end{matrix} > Zn$	I	24,5	6,51	11,59
5	$\left[\begin{pmatrix} RC_6H_4C \\ C_nH_{2n-1} \end{pmatrix}_2 PS_2\right]_2 Ba$	I	21, 7	6,05	7,12

$R = C_8 - C_{12}$

All the additives were added to oil D-11 in the amount of 1%, and their corrosion was determined by the NAMI method in the presence of copper naphthenate. The compatability of these additives with sulfonated additive SB-3 was also studied. Table 9 gives the results of these tests.

TABLE 9. RESULTS OF LABORATORY TESTS OF OIL D-11 WITH VARIOUS ANTIOXIDANTS

Composition of sample	Additive index	Test results: Detergent properties according to PZV method, points	Test results: Corrosion, according to (severe) NAMI method, g/m²
Д-11 + 1% $\left[\begin{pmatrix} C_8H_{17}O \\ C_nH_{2n-1} \end{pmatrix}_2 PS_2\right] Zn$	IKhP-2/12	-	6,2
Д-11 + 3% СБ-3 + 2% $\left[\begin{pmatrix} C_8H_{17}O \\ C_nH_{2n-1} \end{pmatrix}_2 PS_2\right] Zn$	-"-	2	2,1
Д-11 + 1% $\begin{matrix} (RC_6H_4O)_2\ PS_2 \\ (C_nH_{2n-1})_2\ PS_2 \end{matrix} > Zn$	4	-	0,9
Д-11 + 3% СБ-3 + 2% $\begin{matrix} (RC_6H_4O)_2\ PS_2 \\ (C_nH_{2n-1})_2\ PS_2 \end{matrix} > Zn$	4	3	0,1
Д-11 + 1% $\left[\begin{pmatrix} RC_6H_4O \\ C_nH_{2n-1} \end{pmatrix}_2 PS_2\right]_2 Ba$	5	-	17,[illegible]
Д-11 + 3% СБ-3 + 2% $\left[\begin{pmatrix} RC_6H_4O \\ C_nH_{2n-1} \end{pmatrix}_2 PS_2\right]_2 Ba$	5	2,5-3,0	0,65
Д-11 + 1% $\begin{matrix} (C_8H_{17}O)_2\ PS_2 \\ (C_nH_{2n-1})_2\ PS_2 \end{matrix} > Zn$	3	-	1,0
Д-11 + 3% СБ-3 + 2% $\begin{matrix} (C_8H_{17}O)_2\ PS_2 \\ (C_nH_{2n-1})_2\ PS_2 \end{matrix} > Zn$	3	3	0,5
Д-11 + 1% $\left[\begin{pmatrix} RC_6H_4O \\ C_nH_{2n-1} \end{pmatrix}_2 PS_2\right]_2 Zn$	IKhP-2/13	-	0,4
Д-11 + 3% СБ-3 + 2% $\left[\begin{pmatrix} RC_6H_4O \\ C_nH_{2n-1} \end{pmatrix}_2 PS_2\right]_2 Zn$	-"-	2,5-3,0	0,1

TABLE 10. RESULTS OF LABORATORY TESTS OF OIL D-11 WITH VARIOUS ADDITIVES

Sample	pH	Total alkalinity	Corrosion at 140°C, g/m^2	Corrosion at 160°C, g/m^2	After oxidation 100 cSt	Residue, %	Detergent Potential %
D-11 + 2.7% BFK + 1.3% SB-3 + 1.2% INKhP-21 standard	9.4	1.73	125.0	316.5	64.88	32.2	53
D-11 + 2.7% BFK + 1.3% SB-3 + 1% IKhP 2/12	7.1	2.24	21.9	213.1	122.0	–	53
D-11 + 2.7% BFK + 1.3 SSK + 1.2% INKhP-21	8.9	1.53	159.4	302.5	indist.	33.8	53
D-11 + 2.7% BFK + 1.3% SSK + 1% IKhP 2/12	7.2	2.0	16.2	246.5	indist	37.3	53
D-11 + 1.7% BFK + 1.3% SSB + 1.2% INKhP-21	8.7	1.57	146.7	302.4	45.6	26.7	48
D-11 + 2.7% BFK + 1.3% SSB + 1% IKhP 2/12	7.2	2.1	18.2	197.7	145.4		53
D-11 + 4% SSK = 1.2% INKhP-21	8.6	0.53	22.6	141.6	no movement	32.0	58
D-11 + 4% SSK + 1% IKhP 2/12	6.3	1.03	1.8	39.0	" "	37.0	53
D-11 + 4% SSB + 1% IKhP 2/12	7.5	1.1	1.95	18.4	–	–	53

As can be seen from the data in Table 9, all the additives have high anticorrosion properties and combine well with sulfonated additive SB-3.

Considering its high solubility and mobility, additive IKhP 2/12 was selected for further study. The possibility of replacing various series of antioxidizing additive INKhP-21 in the additive composition with additive IKhP 2/12 was studied. Results of these studies are given in Table 10.

As is evident from the data in Table 10, replacing antioxidizing additive INKhP-21 with the new additive IKhP 2/12, sharply improves the effectiveness of the additive composition, increases total alkalinity and reduces corrosion, determined at temperatures of 140 and 160° C.

Thus, additive IKhP 2/12, a zinc salt of the phosphoro-sulfurated combination of octyl alcohol and polymer distillate, both in pure form and in combination with compositions of other additives, surpasses the effectiveness of the well-known antioxidizing additive INKhP-21.

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