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FOREIGN TECHNOLOGY DIVISION

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by

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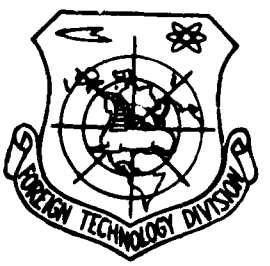

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ABSTRACT

In order to develop antioxidant motor-oil additives whose composition includes phosphorus and sulfur, monochlorides of cyclohexyl- and methylcyclohexylphosphorous acids were prepared. The chlorides obtained were subjected to condensation reactions with various alkyl phenols (C_3-C_{10}) and sulfide-disulfide alkyl phenols. The effect of the synthesized compounds on the performance characteristics of D-11 oil was investigated. Tests of these compounds for the oxidation resistance, thermal stability, and anticorrosive properties of the diesel oil showed that the induction period of the oxidation, determined by the method of the AzNII, increases from 30 to 70-100 min, and the time of absorption of 10 ml of oxygen, from 240 to 340-355 min. The anticorrosive properties were tested under drastic conditions (method of NAMI); in the presence of the additives, the corrosion decreases from 360 to 1-13 g/m^2. The thermal stability of D-11 oil increases from 25 to 60-105 min. Phosphorus- and sulfur-containing compounds in compounds in combination with SB-3 and BFK additives have practically no effect on the wetting properties of the oils.

STUDY OF ADDITIVES SYNTHESIZED FROM DERIVATIVES OF NAPHTHENIC HYDROCARBONS

I. M. Orudzheva and Sh. M. Novruzov

To obtain antioxidant additives to motor oils we synthesized P- and SP-containing compounds. Cyclohexanol and methylcyclohexanol were taken as the initial raw materials. The reaction of these alcohols with phosphorus trichloride produced the corresponding monochlorides of cyclohexyl and methylcyclohexyl phosphorous acids.

The synthesized acid chlorides were subjected to condensation with various alkyl phenols (C_3–C_{10}), and as a result a series of triply substituted phosphites was obtained.

These phosphites contain 5.8–9.2% phosphorus. We obtained P-containing additives, for which the monochlorides of dicyclohexyl phosphorous acid were reacted with sulfide-disulfide alkyl phenols. The characteristics of the obtained P- and PS- containing additives are given in Table 1.

We studied the effect of these compounds on the performance characteristics of D-11 oil. Mixed with the new additives, D-11 oil was tested for stability, thermal stability and anticorrosion properties.

The results of these tests are given in Tables 2 and 3.

From the data of Table 2 it is evident that in small concentrations (about 0.1%) the phosphites very effectively improve the stability of oils (as determined by the AzNII method) at 175°C. The induction period of oxidation increases from 30 to 70–100 min, and the time for absorption of 10 ml of oxygen, from 240 to 340–355 min.

All the synthesized compounds increase the stability of D-11 oil identically. An analogous picture is observed in the tests of these for anticorrosion properties and thermal stabiltiy (Table 3).

Table 1. Elementary Composition of Compounds Synthesized from Naphthenic Hydrocarbons

Proposed Formula	Content, %			
	Carbon	Hydrogen	Phosphorous	Sulfur
$(C_6H_{11}O-)_2POC_6H_4C_3H_7$	69.37	8.95	9.20	——
$(CH_3C_6H_{10}O-)_2POC_6H_4C_3H_7$	70.68	9.83	7.56	——
$(CH_3C_6H_{10}O-)_2POC_6H_4C_4H_9$	70.48	9.23	7.89	——
$(C_6H_{11}O-)_2POC_6H_4C_5H_{11}$	70.62	9.54	7.90	——
$(C_6H_{11}O-)_2POC_6H_4C_{10}H_{21}$	73.02	10.36	5.84	——
$(C_6H_{11}O-)_2P(O)-C_6H_3(C_3H_7)-S-C_6H_3(C_3H_7)-P(O)(-OC_6H_{11})_2$	66.91	7.90	8.54	4.83
$(C_6H_{11}O-)_2P(O)-C_6H_3(C_4H_9)-S-C_6H_3(C_4H_9)-P(O)(-OC_6H_{11})_2$	66.79	8.10	7.32	3.94
$(C_6H_{11}O-)_2P(O)-C_6H_3(C_5H_{11})-S-C_6H_3(C_5H_{11})-P(O)(-OC_6H_{11})_2$	66.97	9.42	6.90	3.51
$(C_6H_{11}O-)_2P(O)-C_6H_3(C_4H_9)-S_2-C_6H_3(C_4H_9)-P(O)(-OC_6H_{11})_2$	64.86	8.50	8.06	6.94
$(C_6H_{11}O-)_2P(O)-C_6H_3(C_5H_{11})-S_2-C_6H_3(C_5H_{11})-P(O)(-OC_6H_{11})_2$	65.83	8.00	7.69	7.00

Table 2. Stability of D-11 Oil Mixed with Different Antioxidant Additives by the AzNII Method

Sample	Quantity of Additive, %	Induction Period, min	Time for Absorption of 10 ml of Oxygen, min
Oil Without Additive	—	30	240
Oil + Additive No. 1	0.1	63	335
Oil + Additive No. 2	0.05	93	354
Oil + Additive No. 2	0.3	95	355
Oil + Additive No. 3	0.1	108	341
Oil + Additive No. 4	0.1	77	341
Oil + Additive No. 5	0.1	76	354
Oil + Additive No. 5	0.05	61	321

Table 3. Effect of P- and S-Containing Compounds on the Anticorrosion and Thermal-Stability Properties

Sample	Quantity of Additive, %	Thermal Oxidation Resistance by the Papok Method, min	Index of Lacquer Deposition, K_1	Lacquer Residue, %	Corrosion by the NAMI Method, 25 h, g/m^2
Oil Without Additive	—	25	0.89	22	360
Oil + Additive No. 1	0.5	50	0.19	9.5	—
Oil + Additive No. 1	1.0	98	0.08	8	27.0
Oil + Additive No. 4	0.5	57.5	0.24	14	1.65
Oil + Additive No. 4	1.0	105	0.05	6	4.65
Oil + Additive No. 5	0.5	54.2	0.25	14	5.0
Oil + Additive No. 5	1.0	94	0.09	9	8.7
Oil + Additive No. 6	1.0	74	0.13	13	6.35
Oil + Additive No. 7	1.0	75	0.12	12	6.0
Oil + Additive No. 8	1.0	73	0.14	10.5	14
Oil + Additive No. 9	1.0	53	0.17	9	3.9
Oil + Additive No. 10	1.0	49	0.18	9	8.4

Table 4. Wetting Properties of D-11 Oil with Additives by the PZV Method

Sample	Wetting Properties, Units
Oil Without Additive	5 to 5.5
Oil + 5% SB-3	1 to 1.5
Oil + 6% BFK	0.5 to 1.0
Oil + 5% SB-3 + 1% Additive No. 8	0.5
Oil + 6% BFK + 1% Additive No. 8	0.5
Oil + 6% BFK + 1% Additive No. 4	0 to 0.5

The anticorrosion properties of D-11 oil were tested under drastic conditions by the NAMI method for 25 h in the presence of a catalyst, copper petronate. When 0.5–1.0% of the P-containing compounds are added to D-11 oil corrosion of the oil is reduced from 360 to 1–13 g/m^2. The phosphites also effectively increase the thermal stability of D-11 oil and reduce the coefficient of laquer deposition (Table 3).

P- and S-containing compounds produced from cyclohexanol and containing 6.9–8.5% P and 3.5–6.9% S incrase the thermal stability of D-11 oil and improve its anticorrosion properties (Table 3). In combination with SB-3 and BFK additives these compounds do not worsen the wetting properties of diesel oil (Table 4).

Thus, from derivatives of naphthenic hydrocarbons we synthesized a number of new S- and P-containing compounds that in pure form and in combination with other known multipurpose additives improve the performance characteristics of oils.