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EFFECT OF ADDITIVE CONCENTRATION ON THE ANTIWEAR AND ANTISEIZING PROPERTIES OF OIL

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Foreign Technology Division
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13. ABSTRACT

Min. effective concns. (2.5, 0.3, 2.5, and 5.0%, resp.) of a sulfurized olefin tetramer (OTP), ethylene diisopropyl xanthate (LZ-23K), $CCl_3PO(OBU)2$ (Khloref-40), and $(CCl_3(Ch_2)4)2S$ (Sul'fol) for reducing wear and seizing in lubricating oil IS-50 were ~~detd.~~ at sliding rates smaller than or equal to 9.5 m/sec and loads of 7.5 to 75 kg in a 4 ball friction machine. The antiseizing efficiency of LZ-23K was even impaired at higher concns. and that of $(RO)2P(S)SCH2CHMeCCl_3$ (LZ-309,2) and (ME(CH2)3CHETCH2O)2PSSZN-SSP$(OCH2CHMe_2)2$ (DF-11) was not appreciable. [AT1206806]

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14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Lubricating Oil Lubricant Additive Chlorinated Organic Compound Ether Ester Organic Phosphorus Compound Antiseize Additive/(U)OPT Lubricant Additive (U)LZ-23K Lubricant Additive (U)Khloref 40 Lubricant Additive (U)DF-11 Lubricant Additive (U)LZ-309,2 Lubricant Additive (U)IS-50 Lubricating Oil						

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EFFECT OF ADDITIVE CONCENTRATION ON THE ANTIWEAR AND ANTISEIZING PROPERTIES OF OIL

[Article by Yu.Ya. Podol'skiy, G.V. Shipilov, and I.G. Tsurkan; Moscow, Trudy Vsesoyuznogo Ordena Trudovogo Krasnogo Znameni Nauchno-Issledovatel'skogo Instituta Zheleznodorozhnogo Transporta, Russian, No 400, 1970, pp 12-20]

Because of the sharply heightened operating speeds and the unit contact loads experienced by friction surfaces employing boundary lubrication, lubricating agents possessing superior antiwear and antiseizing properties have come to be widely used. These properties are attained by introducing chemically active additives into oil. Sulfur, phosphorus, and chlorine-containing additives, as well as multiple action compounds containing two or several active elements, have received the widest application. Numerous investigations into compounds of this type have had the primary purpose of discovering their mechanism of action,[1-10] while the question of effective additive concentration, one of no less practical importance, has as yet been almost untouched. The few papers treating this question [8,9,11] indicate that the dependence of additive effectiveness on concentration is highly specific. This specificity is predicated not only by the differences in additive activity, but also by the fact that the effective concentration of additives on friction surfaces can differ significantly from their concentration in the lubricant in bulk form.[10] From economic considerations the additive concentration in oil must be at a minimum. On the other hand it must be sufficient to ensure both high additive activity and the necessary safe life of the assembly experiencing friction.

This investigation had the purpose of trying to determine the minimum concentrations of some antiwear and antiseizing additives below which they have no effect. The experiments were conducted under conditions modeling high-load, high-speed friction assemblies. Such conditions were achieved with a four-ball bearing friction machine belonging to the laboratory of polymer rheology under the USSR Academy of Sciences Institute of Petrochemical Synthesis.[12] Table 1 provides a list of the substances investigated -- typical representatives of the antiseizing and antiwear additives produced by domestic industry before 1970, and experimental additives. IS-50 (GOST [All-Union State Standard] 1707-51) oil with a viscosity of 46.5 cs [centistoke] at 50°C was used as the base oil. Solutions of the additives in this oil at concentrations of 10, 5, 1.25, 2.5, 0.62, 0.31, and 0.15 percent by weight were studied. The experiments were conducted with known techniques [12] that can be summarized as follows.

Each experiment was conducted at a constant load but with gradual increase -- linearly with respect to time -- of the speed of relative movement of the friction bodies (standard 12.7 mm diameter bearing balls made of ShKh-15 steel with a hardness rating HRC 60-62). Friction was recorded continuously during the experiment, and the diameter of wear depressions formed on the three motionless balls as a result of friction was measured at the end of the experiment with a microscope. This way we found the dependence of the coefficient of friction μ on speed v, and the degree of wear and speed value at which seizing -- a sharp change in the curve $\mu(v)$ -- occurs at a particular load. The load was increased from one experiment to the next in 7.5 kg stages from 7.5 to 75 kg. The maximum slip speed was 26,400 rpm, which is 9.5 meters/second when recomputed for point contact of the balls. In this case acceleration was 0.105 meters/sec^2. Each experiment was started with new balls or with unworn parts of the ball surfaces. All experiments were conducted at room temperature within the friction assembly.

Let us begin our examination of the experimental data with the sulfur-containing additives OTP (sulfurated tetramers of C_3 and C_5 olefin hydrocarbons) and LZ-23k (ethylene di-isopropylxanthate).

The dependence of the coefficient of friction on speed for solutions of the first additive is given in Figure 1. We obtained 80 friction graphs (eight concentrations and 10 loading stages) for each of the additives investigated. Only 12 of the most typical graphs are shown in Figure 1. Here each column of graphs corresponds to 7.5, 45, and 75 kg loads. They are grouped horizontally with respect to different concentrations of the additive in the base oil. Numerical concentration values are indicated by the digits to the right.

Table 1

Additive code name	Additive name	Chemical formula	Concentration of chemically active elements
OTP	Sulfurated tetramers of olefin hydrocarbons	—	15-25% S
LZ-23k	Ethylene di-isopropyl-xanthate	$C_3H_7-OC(=S)-S-CH_2-CH_2-S-C(=S)O-C_3H_7$	42.7% S
Khloref-40	Dibutyl ester of tri-chlormethylphosphonic acid	$CCl_3PO(OC_4H_9)_2$	34% Cl, 10% P
Sul'fol	Di-1,1,1-trichlorpen-tylsulfide	$[CCl_3(CH_2)_4]_2S$	8% S, 55.8% Cl
LZ-300/2	Chlorbutylene-di-iso-propyldithiophosphate	$(RO)_2P(=S)SCH_2-CHCCl_3-CH_3$	19.6% S, 9.36% Cl, 10.5% P
DF-11	Zinc dialkyldithio-phosphate	$[CH_3-(CH_2)_3-CHC_2H_5-CH_2O]_2PSSZnSSP[OCH_2CH(CH_3)_2]_2$	4.5% P, 5.28% Zn, 5.5% S

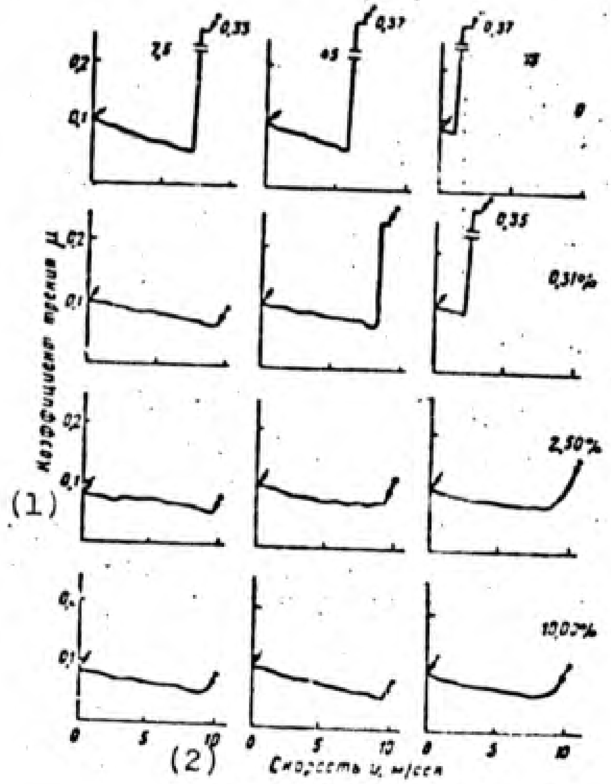
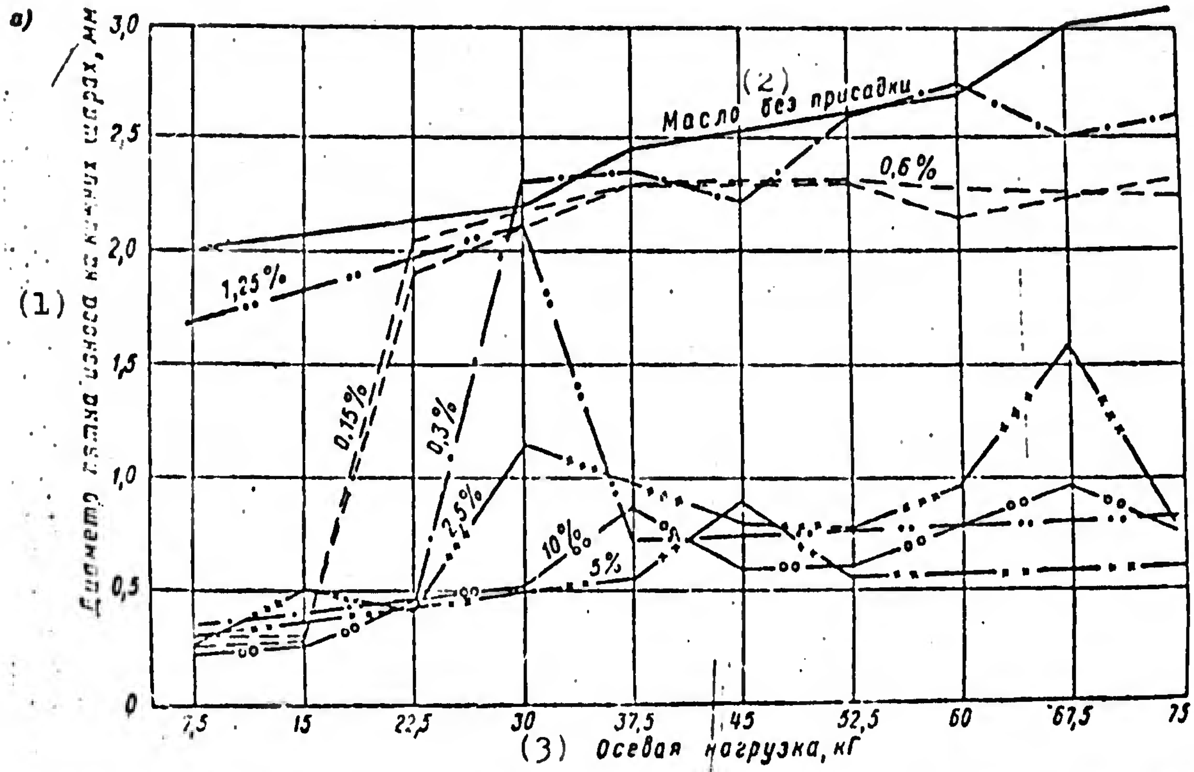


Figure 1. Typical Friction Graphs for Oil Solutions of OTP Additive at Different Concentrations and Loads (numbers after the curve salient points indicate maximum values of the coefficient of friction, arrows indicate slip starting points, arrows with circles indicate termination of slip)

Key:

1. Coefficient of friction, μ
2. Speed, v, meters/sec

As is evident from the data presented, when there is friction between steel surfaces coated with oil without additives (top horizontal row of graphs), seizing occurs at the minimum load of 7.5 kg. When the load is increased seizing occurs at lower and lower slip speeds. Addition of OTP additive to the oil at 0.31 percent by weight prevents seizing at the minimum load and makes it possible to significantly increase the speeds at which seizing does occur in the presence of higher loads. At as low a concentration as 2.5 percent by weight, seizing is prevented throughout the whole range of loads investigated.



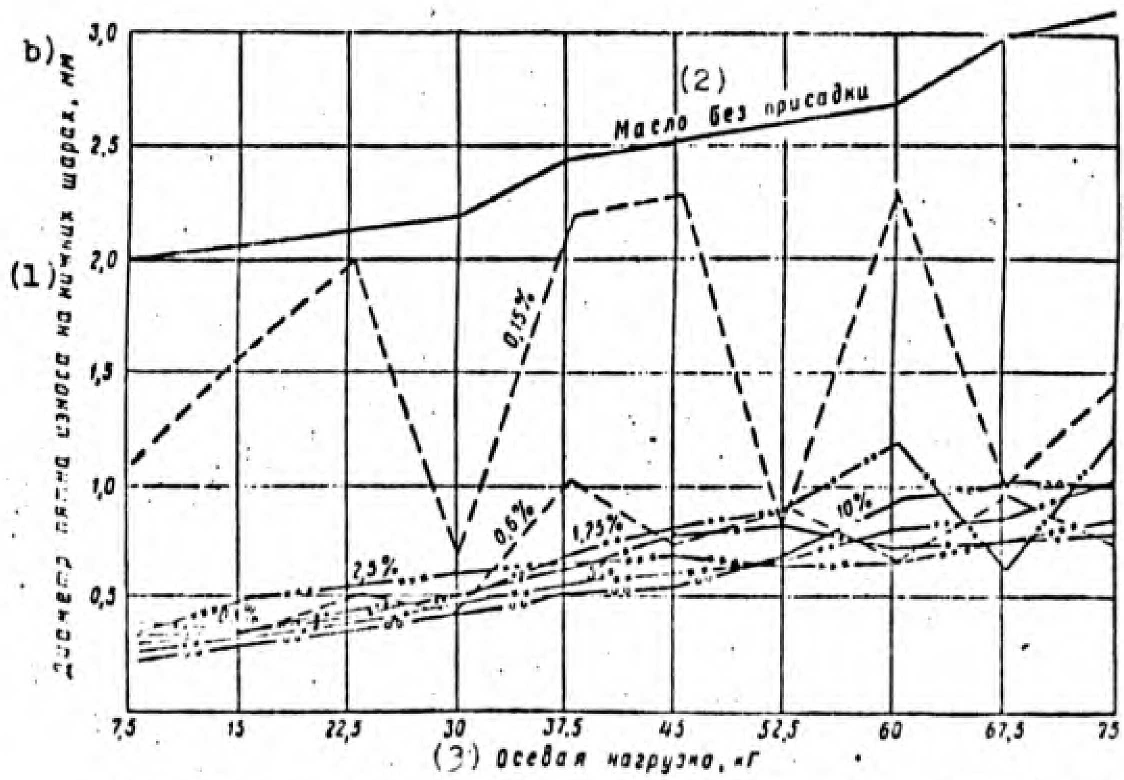


Figure 2. Dependence of Wear on Load at Maximum Speed (or at Seizing Speed) for OTP (a) and LZ-23k (b) Additive Solutions in Oil (numbers by [continued on following page]

curves indicate additive concentration in oil by weight)

Key:
1. Diameter of wear points on lower balls, mm
2. Oil without additive
3. Axial load, kg

A further increase in concentration up to 10 percent does not contribute significant changes to the nature of the oil's action.

The dependence of wear measured at the end of the experiment on the load (Figure 2) confirms the conclusion above and visually demonstrates the sharp drop in wearing intensity when oil containing over 2.5 percent additive by weight is used. The dependence of wear on load for a 1.25 percent additive solution, as seen in this figure, is of interest. In this case the high wear at light loads is succeeded by moderate and low wear when the load is increased. This indicates that the additive activity increases as the friction conditions become more severe because of increased heat evolution at the area of contact.

Similar dependencies for LZ-23k additive are shown in Figure 2b and Figure 3.

From the data in the figures it is evident that there is a certain reduction of wear even at the minimum concentrations investigated (see Figure 2b). At as low a concentration as 0.31 percent by weight the additive prevents seizing throughout the whole range of loads and speeds. An increase of the concentration to 2.5 percent by weight, however, causes reappearance of seizing in the high load range (see Figure 3). This is consistent with data indicating a reduction of the loads at which seizing of steel occurs when the concentration of dibenzyldisulfide in the nonpolar naphthene-paraffin fraction of residual oil is increased.[3] When the additive concentration is increased further, seizing becomes more and more clearly expressed at these loads. Although seizing at these concentrations is subdued and is not accompanied by any noticeable increase in wear (see Figure 2b), the fact itself of the appearance of seizing as the concentration of the antiseizing additive is increased earns special consideration, and it indicates that there is a certain optimum concentration. Both a higher and a somewhat lower concentration are equally undesirable.

The concentration effects of compounds containing the trichlormethyl group of Khloref-40 (dibutyl ester of trichlormethyl phosphonic acid) and of Sul'fol (di-1,1,1-trichlorpentyl sulfide) are quite monotonic in the sense that the antiseizing action of their oil solutions intensifies gradually as the additive concentration increases. The minimum effective concentrations of these two additives are 2.5 and 5 percent by weight for Khloref-40 and Sul'fol respectively.

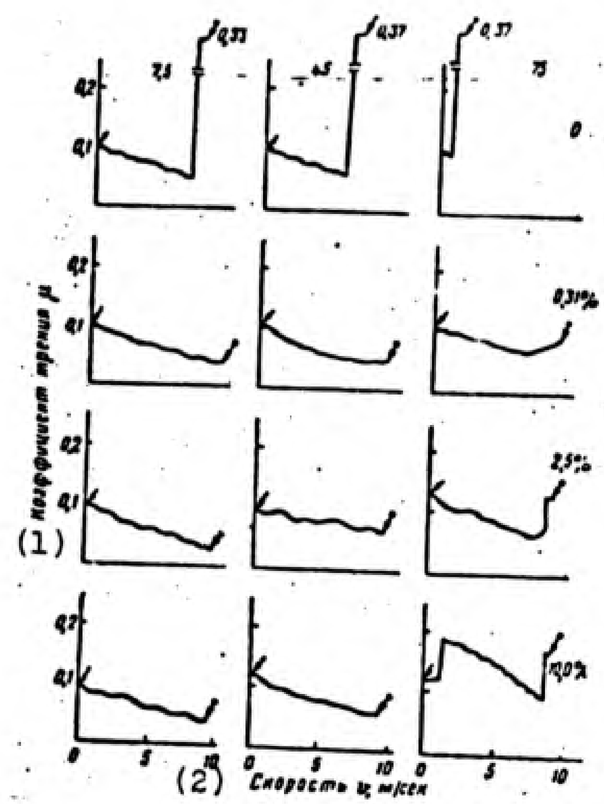


Figure 3. Typical Friction Graphs for Oil Solutions of LZ-23k Additive at Different Concentrations and Loads (see remarks in the legend for Figure 1)

Key:

1. Coefficient of friction, μ
2. Speed, ν, meters/sec

Table 2 provides the minimum concentrations of the four additives examined above, below which they are ineffective as agents that prevent seizing of steel at high contact loads and slip speeds. As far as the two remaining additives are concerned (DF-11 and LZ-309/2), under these conditions they did not display a noticeable antiseizing action throughout the whole range of concentrations studied (up to 10 percent by weight).

Table 2

Additive code name	Minimum effective concentration of				
	Additive		Active elements, moles/1,000 gm of oil		
	% by weight	Moles/ 1000 gm oil	S	Cl	P
OTP	2.5	0.151	0.086	—	—
LZ-23k	0.3	0.089	0.006	—	—
Khloref-40	2.5	0.096	—	0.042	0.038
Sul'fol	5.0	0.156	0.057	0.075	—

As can be seen from Table 2, the minimum effective concentrations for different additives, with respect to both weight and moles, differ significantly from each other. The same can be said for the concentrations of individual active elements in these additives. This means that the total concentration of active components can in no way serve as the basis by which to select the needed additive concentration.

Conclusions

1. At high slip speeds and unit loads, the sulfur-containing additives LZ-23k and OTP have the greatest effectiveness from the point of view of preventing seizing of steel surfaces and reducing their wear.

2. An example with LZ-23k additive shows that the antiseizing properties of oil can worsen as the concentration of this compound within it is increased.

3. Selection of the minimum effective concentration must be made for each additive individually in each specific case, inasmuch as the concentration of active elements in the oil cannot serve as the basis for evaluating the oil's antiseizing or antiwear effectiveness.

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