

Original Article

# Research of the Physico-Chemical Properties of Antipyrene-Antiseptics, Which Contain Nitrogen, Sulfur

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**Abstract** - As a result of the interaction of nitrogen, -phosphorus, -sulfur-containing compounds, new multifunctional oligomeric antipyretic antiseptics were synthesized and their physicochemical properties were determined. Sulfur and nitrogen group oligomers were obtained, and their antiseptic properties, mechanisms of synergistic action, physical and chemical properties, and density were studied. By forming special coatings on the surface of wooden building materials, we have synthesized sulfur and nitrogen group composites, which provide physical and mechanical properties and properties of modified wood material at a high level of temperature, pressure, oxidation and corrosion stability. A number of ongoing studies, as a result of the addition of special chemicals to the composition of building materials, structurally strengthening the gardens, make it possible to ensure the stability of its mechanical properties and chemical and thermal properties.

**Keywords** - Nitrogen, Phosphorus, Sulfur, Oligomer, Antiperin-antiseptic, IR spectroscopy, TG and DTA analyzes.

## 1. Introduction

The process of formation of carbon disulfide bonds as a result of the addition of organic resins to sulfur compounds consists of the interaction of unsaturated hydrocarbon-based alkenes with sulfur, which form a very small amount of oil-gas components. Since the structure of organic tars mainly consists of aromatic and heterocyclic rings connected by short aliphatic bonds, they are compounds with functional properties that can react with sulfur [1, 2, 3].

Interaction processes of sulfur with organic binders can proceed through ionic and radical mechanisms as a result of the decomposition of sulfur at high temperatures. In the first case, when the ring opens, electron pairs can remain on the sulfur atom, and as a result, a lack of electrons is formed at the other end of the resulting chain. In the second case, each sulfur atom can attach one electron [4, 5].

As a result of sulfur-based reactions, there is a decrease in the amount of organic resins and an increase in the dispersed phase of high-molecular compounds; it is important to increase the role of coagulation walls in the formation of the properties of oil-sulfur binders. As a result of the interaction of sulfur with oil, hydrogen sulfide is obtained. Then various sulfur-containing organic compounds (first mercaptans, with their subsequent decomposition and transformation into sulfides) are formed [6, 7].

Conducted theoretical studies allow us to assess the sufficient complexity of this mechanism, as well as the

interaction of sulfur with organic binders, and the areas of application of the resulting compounds are an important area of research. On the basis of this research, the possibility of using directly usable organic sulfur compounds obtained during the processing of oil products and polysulfide and sulfur composites obtained as a result of the interaction reaction of organic compounds with sulfur was analyzed [8, 9].

Elemental sulfur reacts with organic compounds to form various elemental-organic compounds [5]. Reactions with organic substances in the presence of sulfur have been mentioned in several literatures; they can form sulfur-containing organic compounds by forming an active reaction process in several directions simultaneously [10, 11].

The synthesis of SN-functional derivatives of hydrocarbons is convenient and is carried out based on elemental sulfur. At the same time, the possibility of obtaining valuable products with a wide range of sulfur content is an important direction in modern organic chemistry [12, 13].

Sulfur-based polymers, oligomers, and low-molecular-weight organic compounds are widely used in almost all industries. For example, its use in medicine, agriculture, animal feed additives, paints, corrosion inhibitors, surkov oils, natural gas safety mercaptans, sorbents, rubber vulcanizing additives, composites, chemical industry,



construction and road construction are very cost-effective and environmentally friendly. The results of scientific research show that [14, 15].

## 2. Research Methods

Infrared spectroscopy (IR). The IR spectra of the original and synthesized compounds were obtained on UR – 20 and UR – 75 spectrophotometers. Samples in powder form were obtained by adding potassium bromide.

### 2.1. Differential-Thermogravimetric Method

Differential-thermogravimetric analysis of the synthesized briquettes was performed on a derivatograph operating in the Paulik F, Paulik I, Erdey L system. This method is based on the change of thermal effects of compounds in the temperature range of 293-793 K when the temperature rise rate is 2-5 K / min.

The Freeman and Carroll method used TGA data to calculate the effective kinetic parameters of the destruction of stabilized samples. The decomposition rate of the polymer is as follows:

$$dW/dt = (A_0/RH)e^{-E/RT}W^n \quad (1)$$

Where RH-heating rate, W-polymer size, (mass),  $A_0$ -predispositional multiplication, the effect of n-reaction composition, and effective activation energy of E-polymer thermodestruction.

The order of the reaction n is found from the equation of the tangent angle of deviation of the graph of dependence in the logarithmic coordinates, and the effective activation energy of the thermodilution is determined from the intersecting segment on the ordinate axis [16,17].

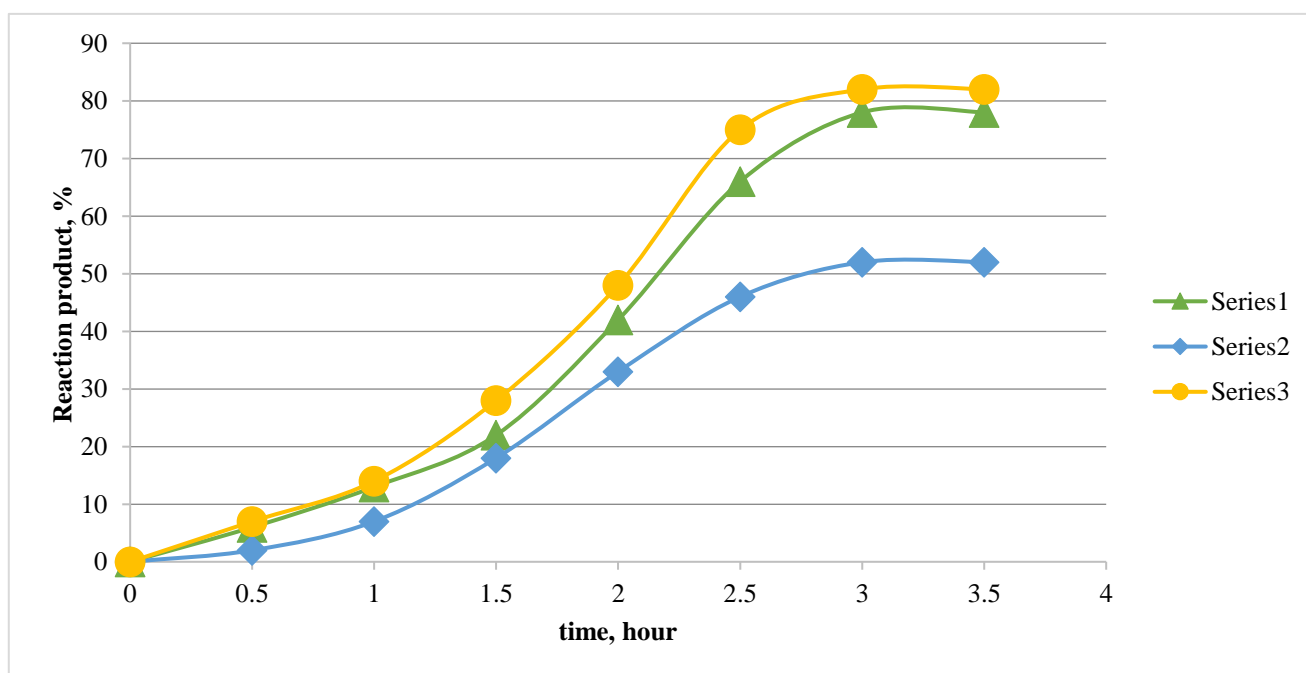
## 3. Experimental Part

New multifunctional oligomeric flame retardant antiseptics were synthesized as a result of the interaction of nitrogen-, phosphorus-, and sulfur-containing compounds.

In the synthesis of these substances, 20 g of orthophosphoric acid and 5 g of sulfur were added to a 250 ml flask equipped with a stirrer, reverse coolant, and thermometer. The temperature of the mixture was then raised to 80–90 °C, neutralized with 20 g of ammonia solution and 10 urea, and stirred for 1.5 h. An aqueous solution of adduct containing sulfur was prepared, and the reaction with 2 g of urotropin at 90-100 °C was continued for 1-1.5 hours until the medium was dehydrated. The result was a light yellow solid with a yield of 78% [18, 19].

During the reaction, several synthesis processes were carried out under different conditions and environments, and the ratio of time, temperature and starting materials to the yield of synthesized oligomeric flame retardant antiseptics and the effect of catalyst species were studied. Based on the above, the optimal conditions for obtaining synthetic antipyretic antiseptics were found [20, 21].

Figure 2 below analyzes the reactions of sulfur-retaining oligomers in different proportions. Accordingly, the ratio of orthophosphoric acid: sulfur: ammonia solution: urea: urotropins was 1: 0.25: 1: 0.5: 0.25 and the temperature was 100°C, and the duration of the process was 3 hours (Fig. 1). In Figure 2, the temperature dependence of these reaction processes was determined, and the reaction products at different temperatures in the ratio 1: 0.25: 1: 0.5: 0.25, which achieved the highest yield among all the ratios, were studied. The best results were obtained at 90-100 °C as these reaction processes were mainly carried out in aqueous solutions.



1). 1:0,25:1:0,5:0,25; 2). 0,5:0,5:0,5:0,25:0,25; 3). 1:0,25:1:0,5:0,5.

Fig. 1 Dependence of the reaction yield on the ratio of starting materials and time in obtaining organic compounds containing sulfur

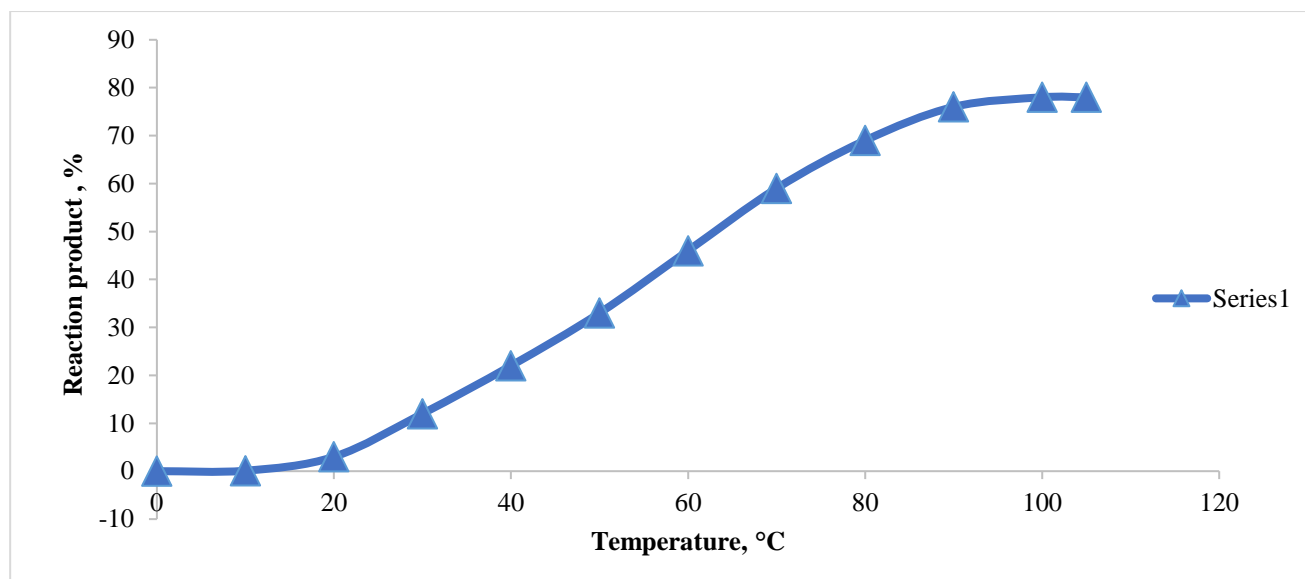


Fig. 2 The product of the reaction temperature dependence, Temperature dependence of reaction yield in obtaining organic compounds containing sulfur

Table 1. Physicochemical properties of sulfur-containing organic oligomer flame retardant antiseptics

Naming of oligomer	Raw materials	Relative (mol)	Amount %	Aggregate status	pH	Density, g/cm <sup>3</sup>
Organic compounds that store sulfur	orthophosphoric acid:	1:0,25:1:0,5:0,25	78	Light yellow	7,5	1,2
	sulfur: ammonia solution:	0,5:0,5:0,5:0,25:0,25	52	viscous	6,0	1,1
	urea: urotropin	1:0,25:1:0,5:0,5	82	substance	7,5	1,3

Table 2. Orthophosphoric acid: sulfur: ammonia solution: urea: sensitivity of urotropins to solvents of oligomeric compounds obtained in different proportions

T/p	Solvents	Effect of oligomers obtained in different proportions on solvents	
		1:0,25:1:0,5:0,25	1:0,25:1:0,5:0,5
1	Water	15% (22°C)	1-1,5% (80°C)
2	Transformer oil	0,15% (100°C)	1,2% (100°C)
3	Dimethyl sulfoxide	8% (55°C)	12% (55°C)
4	Benzene	1,2% (60°C)	7,4% (60°C)

During this reaction, it was found that mainly sulfur and urotropin greatly affect the yield of compounds formed by changing the ratios. That is when the ratio of the element sulfur is increased, it is found that as a result of the interaction of sulfur with orthophosphoric acid, some sulfur is released from the reaction process. The effect of changes in the amount of urotropin on the molecular mass of the synthesized substances and various solvents was determined on the basis of experimental results.

The physicochemical properties of the synthesized substance are given in Table 1.

It has been studied that the physicochemical properties of sulfur-containing organic oligomeric flame retardants differ in part depending on their proportions. The main reason for these results was that the amount of urotropin varied significantly.

Accordingly, the sensitivity of orthophosphoric acid: sulfur: ammonia solution: urea: urotropins to solvents of compounds obtained in different proportions is given in Table 2.

IR spectroscopy of organic oligomeric compounds containing sulfur obtained in two different ratios (1: 0.25: 1: 0.5: 0.25 and 1: 0.25: 1: 0.5: 0.5) consisting of the same synthesized ingredients when the analyzes are studied, it can be seen that the main absorption lines are close to each other [22, 23]. IR spectroscopy analyzes have wavelengths in the 2889–2850 cm<sup>-1</sup> domains and absorption lines in the 1633–1600 cm<sup>-1</sup> domains, confirming close absorption in Figures 1 and 2 of the -SN<sub>2</sub> groups. However, although the presence of -SN<sub>2</sub> groups in the IR spectra shown in both images has been identified, the absorption areas can be clearly seen in Figure 2, where the urotropin ratio is high.

Absorption lines in the IR spectrum (Figures 1 and 2) were typically found to have absorption lines in the 3300–3500 cm<sup>-1</sup> range (-CONHR) with almost identical absorption lines in both images. In addition, in the range of 800-550 cm<sup>-1</sup>, there are absorption lines containing carbon and sulfur and sulfur and sulfur bonds characterizing the -CS- and -SS- groups, and in the range of 950 - 1350 cm<sup>-1</sup> (P = O) and the presence of (-P - O - C) phosphorus groups can be seen in a broadly intensive state [24, 25].

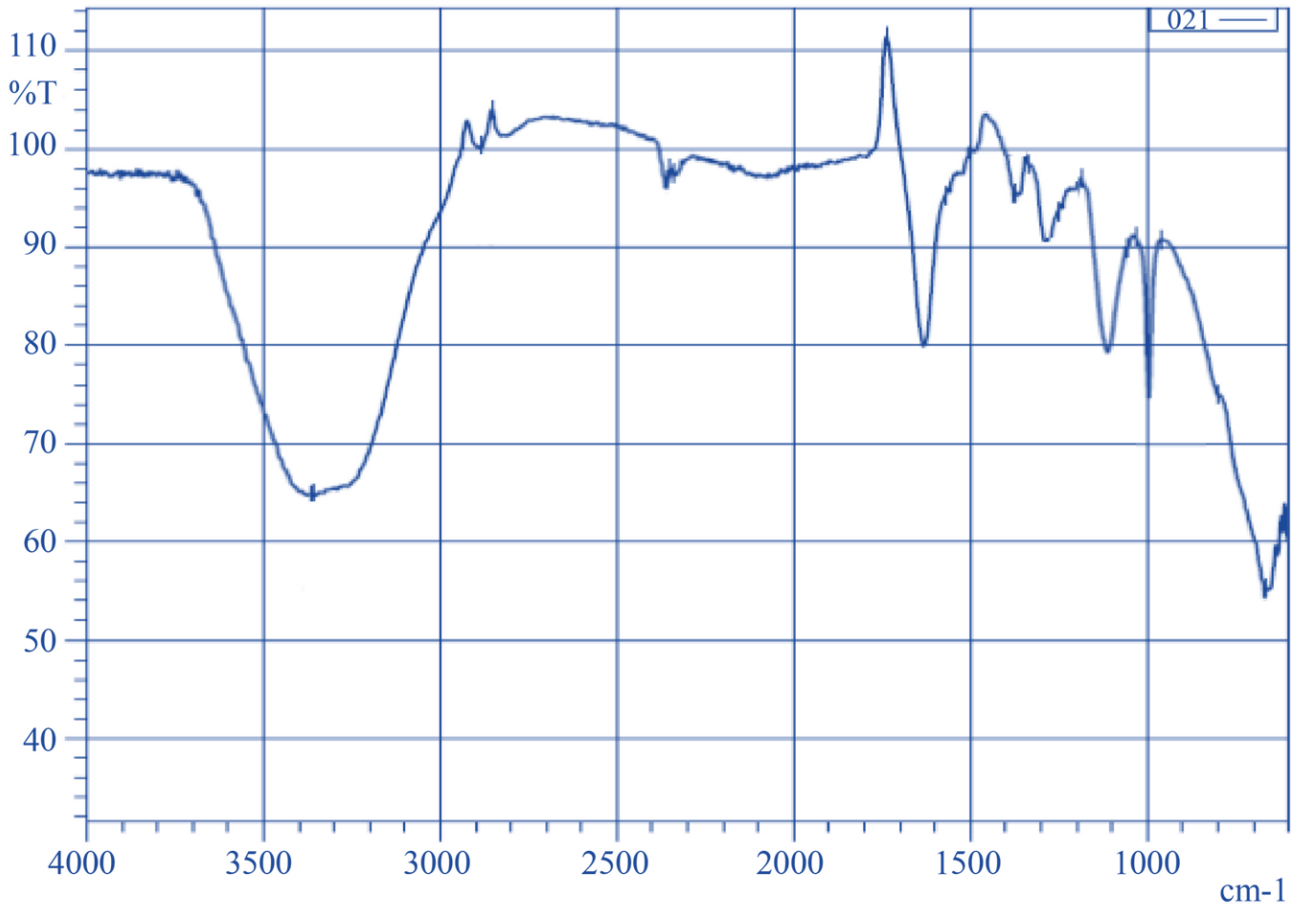


Fig. 3 IR spectroscopy analysis of nitrogen synthesized based on elemental sulfur, organic oligomeric compounds containing sulfur

In our subsequent studies, the effect of increasing the amount of urotropin on the characteristic viscosity by measuring the viscosity of oligomeric compounds obtained in different proportions of orthophosphoric acid: sulfur: ammonia solution: urea: urotropins was determined [26, 27].

The viscosity of organic oligomeric compounds containing sulfur obtained in two different ratios (1: 0.25: 1: 0.5: 0.25 and 1: 0.25: 1: 0.5: 0.5) consisting of the same ingredients synthesized for the measurement, we used a method that was determined by measuring the flow time of aqueous solutions of oligomers containing sulfur by Ubellode viscometer (diamert-0.56mm) and different concentrations. In this method, first of all, 1%, 0.5% and

0.25% aqueous solutions of the obtained substances were prepared, and the experimental processes were carried out at a temperature of 22 °C.

The results of the experimental tests are given in Table 3 and characterize the organic oligomeric compounds containing sulfur obtained in different ratios (1: 0.25: 1: 0.5: 0.25 and 1: 0.25: 1: 0.5: 0.5). Viscosity was determined using the graphs shown in Figures 2 and 3. The overall results showed that an increase in the amount of urotropin in the process of obtaining oligomeric compounds obtained in different proportions had an effect on increasing the characteristic viscosity.

Table 3. Orthophosphoric acid: sulfur: ammonia solution: urea: urotropins in different proportions (1: 0.25: 1: 0.5: 0.25 and 1: 0.25: 1: 0.5: 0.5) measuring the viscosity of solutions

#№	The ratio of oligomers	Concentration of solution%	$\eta_{отн}$	$\eta_{уд}$	$\eta_{ур}$	$\eta_{лог}$	$\eta_{хв}$
1	1:0,25:1:0,5:0,25	1	1,05	0,05	0,05	0,048	0,049
		0,5	1,03	0,03	0,06	0,720	
		0,25	1,02	0,02	0,08	1,410	
2	1:0,25:1:0,5:0,5	1	1,06	0,06	0,06	0,058	0,059
		0,5	1,042	0,042	0,081	0,734	
		0,25	1,025	0,025	0,10	1,411	

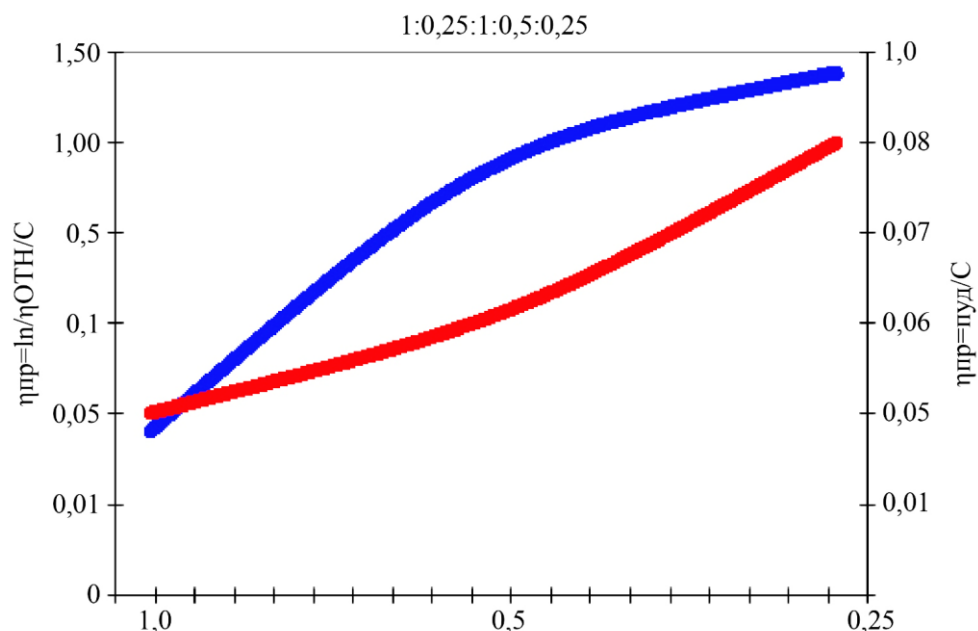


Fig. 4 Graphical determination of the characteristic viscosity of oligomers obtained in the following ratios (1: 0.25: 1: 0.5: 0.25)

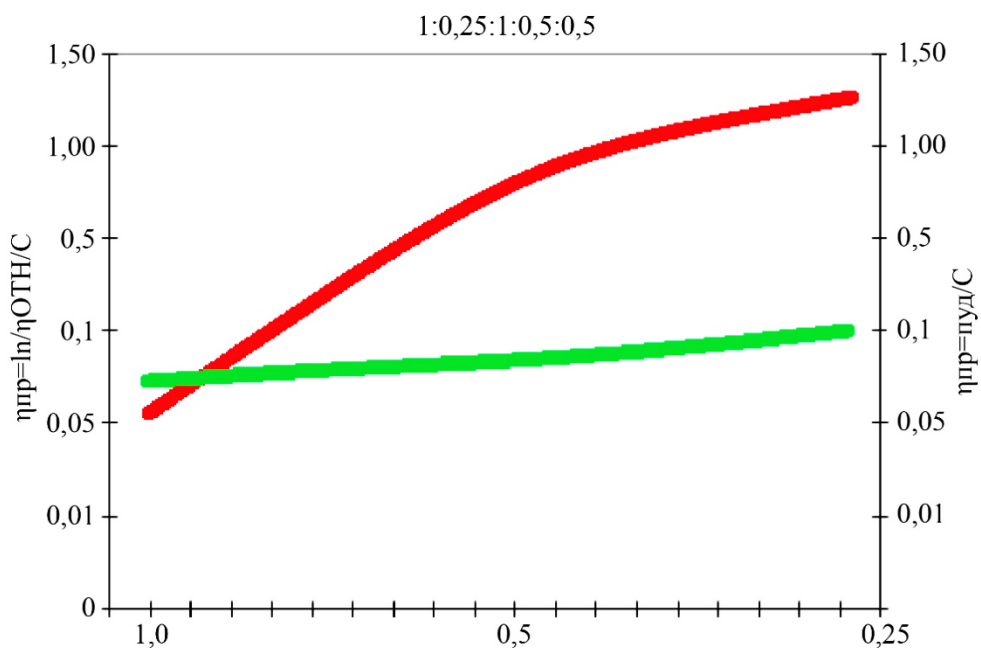


Fig. 5 Graphical determination of the characteristic viscosity of oligomers obtained in the following ratios (1: 0.25: 1: 0.5: 0.5)

The orthophosphoric acid we synthesized: sulfur: ammonia solution: urea: urotropins with different ratios of 1: 0.25: 1: 0.5: 0.25 and 1: 0.25: 1: 0.5: 0.5 studied the thermal analysis of compounds [2, 28].

TG and DTA analyzes of the obtained oligomers were found to be close to each other when compared with the literature. Basically, the characteristics of these sulfur-retaining oligomers were analyzed.

It was found that the mass loss of oligomers obtained in different proportions was slightly higher than that of the abyssals. It has also been found that the presence of endothermic and exothermic peaks in the DTA lines results

in chemical changes as a result of heating the sample. DTA analyzes of oligomers obtained in different ratios include:

Based on these ratios of -1: 0.25: 1: 0.5: 0.25, the DTA curves of the oligomers were determined to have two endothermic effects at 140 and 278 °C and four exothermic effects at 338, 458, 598 and 733 °C.

In oligomers obtained on the basis of ratios of -1: 0.25: 1: 0.5: 0.5, the DTA curves were relatively low, with two endothermic effects at 126 and 269 °C and four exothermic effects at 296, 426, 604 and 718 °C.

TG analyzes of oligomers obtained at different ratios were analyzed as follows. The mass loss of sulfur-containing

oligomers under the influence of temperature is shown in Figure 5 [30, 31].

Table 1 examines the mass loss of sulfur-containing oligomers under the influence of temperature, and relatively direct oxidation can be observed in the early stages of the process.

These oligomers can be classified as thermally stable based on the results obtained. If attention is paid to mass loss, the data presented in Table 1 can be used to analyze the 50%

mass loss of sulfur oligomers obtained at a ratio of 1: 0.25: 1: 0.5: 0.25 at a temperature of 550 °C.

If we analyze the second ratio 1: 0.25: 1: 0.5: 0.5, they show that the ratio of organic compounds in the composition of 500 °C 50% mass loss is higher. In general, both ratios can be observed to lose basic masses at temperatures of 250–650 °C. The main reason for this can be explained by the conversion of nitrogen, phosphorus and sulfur into volatile substances as a result of oxidation under the influence of temperature [27, 29].

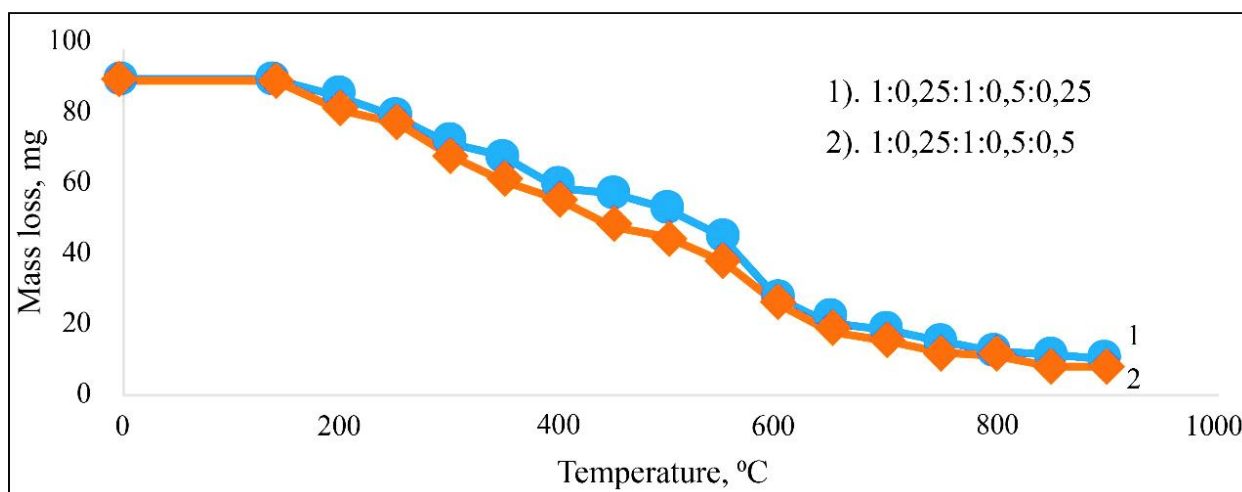


Fig. 6 Mass loss kinetics of sulfur-storing oligomers under the influence of temperature.

Table 4. The effect of temperature on the mass loss of oligomers containing sulfur was obtained in different proportions

Temperature range, °C	Weight loss, mg	The average rate of mass loss is mg/min $v_m = \Delta m / \Delta \tau$
<b>1:0,25:1:0,5:0,25</b>		
140-250	10 mg	0,8
250-450	22 mg	1,76
450-650	36 mg	2,88
650-900	12 mg	0,96
<b>1:0,25:1:0,5:0,5</b>		
140-250	12 mg	0,96
250-450	30 mg	2,40
450-650	30 mg	2,40
650-900	10 mg	1,25

The average rate of mass loss ( $v_m$ ),  $\Delta m$ — mass loss, mg;  $\Delta \tau$ —min, time.

#### 4. Conclusion

In world practice, the study of organic compounds containing nitrogen and sulfur opens up opportunities. As a result of the interaction of nitrogen, phosphorus, and sulfur-containing compounds, new multifunctional oligomeric flame retardant antiseptics were synthesized, and their physicochemical properties were studied. The effect of temperature, duration of synthesis, and the ratio of raw materials on the physico-chemical properties of obtained

oligomers was determined in preparing various brands of oligomeric flame retardants-antiseptics intended for deep penetration into wooden building materials.

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