

INVESTIGATION OF THERMAL PROPERTIES OF COORDINATION COMPOUNDS OBTAINED FROM IMMOBILIZED LIGANDS

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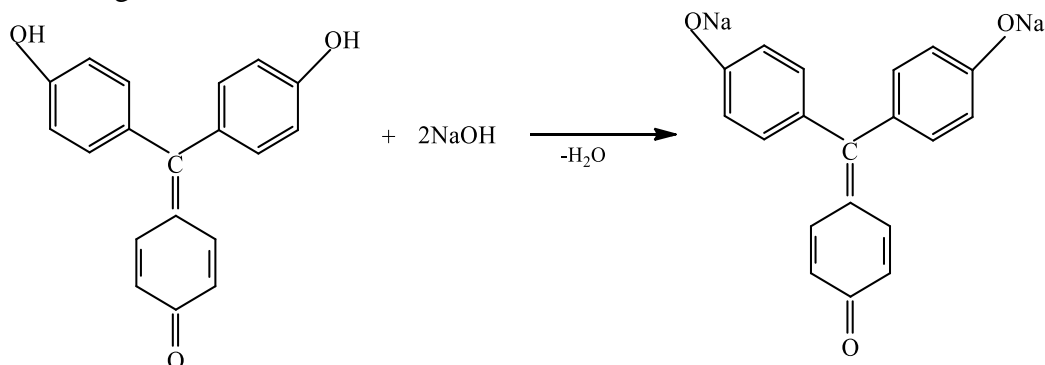
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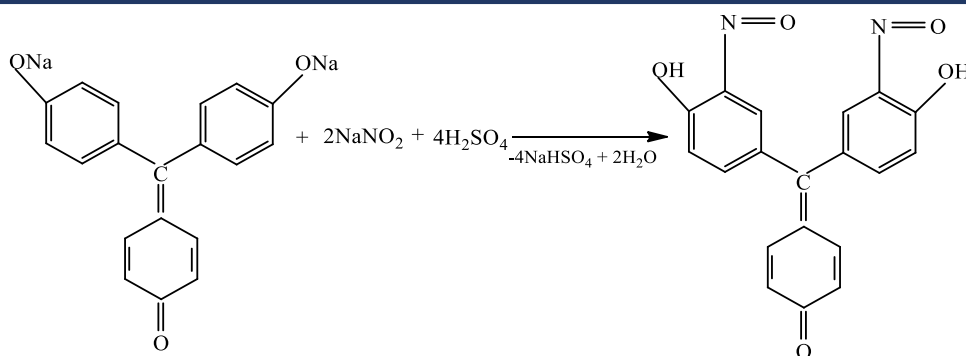
Abstract. In the practical experiments of this study, immobilized ligands were obtained in the presence of nitro- and sulfo- compounds of aurene. The thermal stability of the obtained ligands was studied using a laboratory device. In addition, in this laboratory test experiment, a comparative analysis of the thermal stability of immobilized ligands and the coordination compounds that they form was carried out.

Keywords: immobilized ligand, thermal destruction, derivatogram indicators, sulfo - and nitro - compounds of aurene.

Introduction. In the practical experiments of this research work, immobilized ligands with the participation of nitro- and sulfo compounds of aurene were obtained. The kinetics of thermal degradation was studied using a laboratory instrument of the obtained ligands. In the course of this laboratory experiment, a comparative analysis of the thermal stability of immobilized ligands and the coordination compounds formed by them was also carried out. As the basis of the immobilized ligands, the initial chemical compositions consisting of the nitro compound of Aurene and the sulfo compound of Aurene were obtained. The obtained coordination compounds were investigated using a laboratory instrument (DTG-60/ (Shimadzu)) in differential thermogravimetric kinetics of thermo-oxidative destruction.

Methods and materials. For practical experiments, a test tube equipped with a dropper, a mechanical mixer and a thermometer obtained. The test tube was heated to a temperature of 20°C and a solution of 3 g of aurene, 0.8 g of NaOH in 7.2 ml of water was added dropwise. In addition, during the reaction, 30 ml of NaNO₂ solution in water was added dropwise, as well as a 20% H₂SO₄ solution with a volume of 20 ml. All solutions cooled, stirring, for an hour after addition. The precipitate formed as part of the resulting solution washed and dried in the open air. The resulting product was 3.5 g.





Results and discussion. The thermal stability of coordination compounds formed by nitro- and sulfur compounds of aurene (immobilized ligands) obtained during practical experiments was studied using comparative analysis.

In particular, on the basis of immobilized ligands, the sample of the aurene nitro compound is given a derivatographic representation consisting of 2 curves. Three endothermic effects were detected on the derivatogram curve (DTA) at 238°C, 323.1°C and 378°C, and no exothermic effect was observed.

When analyzing the thermogravimetric curve (TGA), the TGA curve is mainly realized in 3 temperature ranges of intensive decomposition. The decay range 1 occurred in the temperature range 23.42-187.73°C with a loss of 0.14 mg or 4.67% of the mass. The second decay was observed at intermediate temperatures of 187.73-376.29°C. During this decay, it was found that 0.85 mg or 28.34% of the mass was lost.

Finally, the third decay occurred in the temperature range of 376.29-601.23 oc. In this case, an increase in weight by 1.39 mg or 46.34% is appropriate. In the temperature range of 23.42-601.23 oc, it was found that the total weight loss was 2.38 mg or 79.35%, which took 60.24 minutes (fig 1).

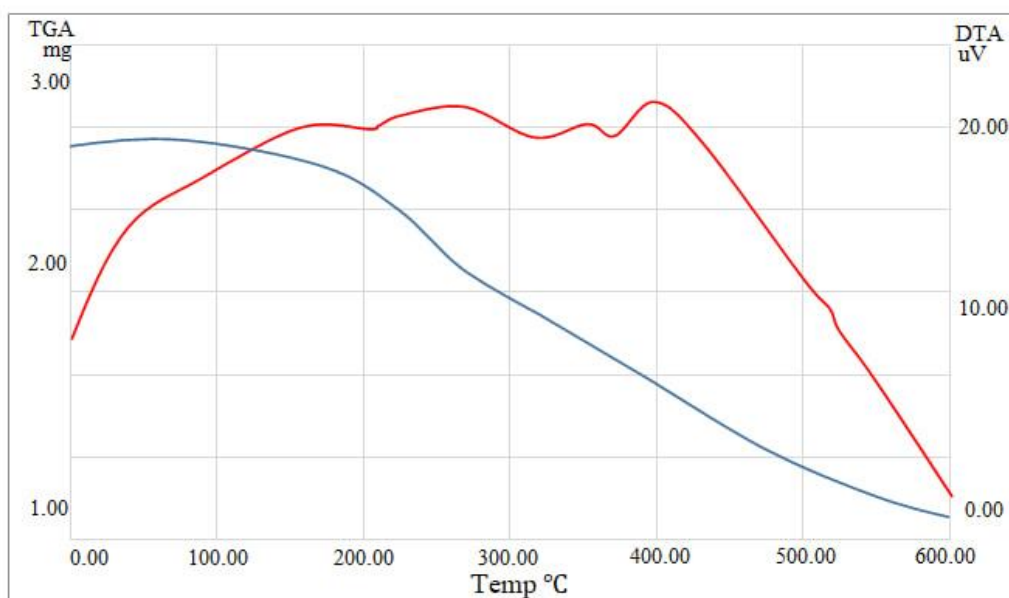


Figure 1. Derivatographic indicators of the sample of the aurene nitro compound based on immobilized ligands

(On the first - the curve of thermogravimetric analysis (TGA); on the second - the curve of differential thermal analysis (DTA))

The analysis of the thermogravimetric analysis curve and the differential thermal analysis curve is shown in table first below. It can be seen from the table that the greatest mass loss is observed during the third intermediate decomposition. That is, it is 46.34% of the mass in this range (table 1).

Table 1

Analysis of the thermogravimetry (TGA) curve

o/n	Temperature (°C)	Time (minute)	Weight (mg)	Mass lost (%)
1	23,42-187,73	15,20	0,14	4,67
2	187,73–376,29	19,15	0,85	28,34
3	376,29-601,23	25,29	1,39	46,34

In this study, the properties of thermo-oxidative degradation of a sample of an aurene nitro compound based on an immobilized ligand were study based on experimental data obtained on the kinetics of processes in the temperature range 50 - 601.23°C.

Derivatives of a sample of a sulfo compound of immobilized aurene based on a ligand also studied (fig 1).

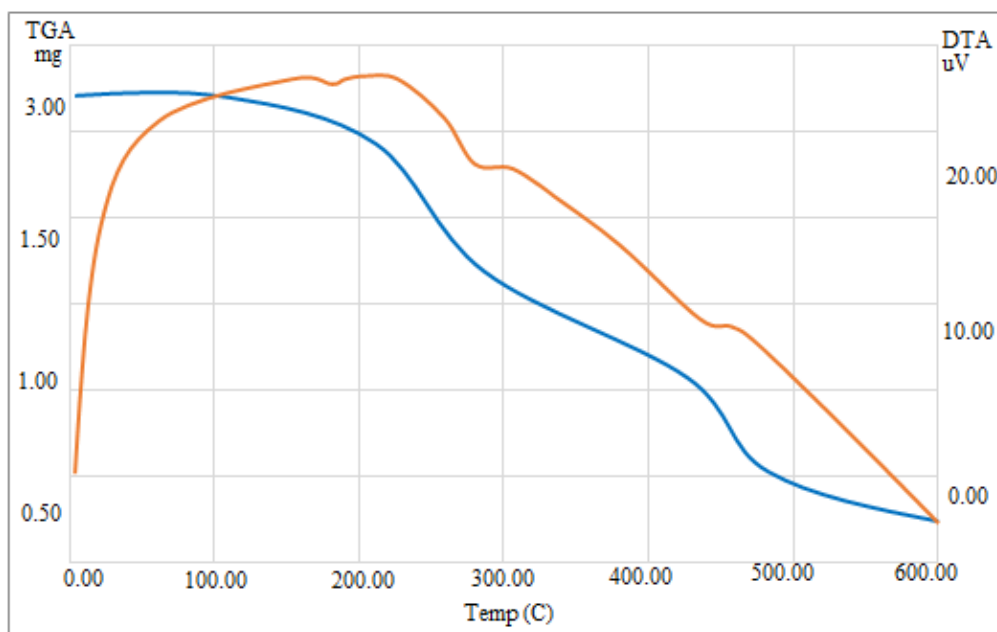


Figure 2. Derivatographic indicators of the sample of the aurene sulfo compound based on immobilized ligands

(On the first - the curve of thermogravimetric analysis (TGA); on the second - the curve of differential thermal analysis (DTA))

In particular, three endothermic effects were detect on the derivatogram curve (DTA) at 188,17°C, 302,54°C, 348°C and 450,85°C and no exothermic effects were observe. Analysis of the thermogravimetric curve (TGA) shows that the TGA curve is mainly performed in 3 temperature ranges of intensive decomposition. The first decay occurred in the temperature range of 27.67-163.52°C and lost 0.250 mg or 4.710% mass. The second decay was observed in the temperature range of 163.52-371.17°C and the mass loss was 1,830 mg or 51.69%. The third decay occurred in the temperature range of 371.17-601.62°C, and the mass was 0.451 mg or 20.120%. At the same

time, it was found that the total weight reduction in the temperature range of 27.67 – 601.62°C was 2.531 mg or 76.52%, and the time consumption was 61.152 minutes.

The indicators of the thermogravimetric analysis curve and the differential thermal analysis curve are presented in the table below. In the table, the greatest mass loss during the second intermediate decay is 51.69% (table 2).

Table 2

Analysis of the thermogravimetry (TGA) curve

o/n	Temperature (°C)	Time (minute)	Weight (mg)	Mass lost (%)
1	27,67-163,52	15,35	0,250	4,710
2	163,52–371,17	21,42	1,830	51,69
3	371,17-601,62	25,73	0,451	20,120

According to the results of the study, kinetic parameters for various temperature ranges of the process were determined. Its advantage was to carry out a number of measurements and calculate kinetic properties over the entire temperature range of reactions from a single sample. The degree of mass loss was determined by graphical differentiation of the TGA curve.

A detailed analysis of the thermogravimetric analysis curve and the differential thermal analysis curve of aurene sulfo- compounds presented in the table below (table 3).

Table 3

The effect of temperature on the mass loss of an immobilized compound based on the aurene sulfo- ligand

o/n	dw 2.01	1/T	dw/dT	mg	minute	T°+K
1	1.91	0.0026	0.0131	0.1	7.61	373
2	1.80	0.0021	0.0119	0.21	17.63	473
3	1.39	0.0017	0.0224	0.62	27.61	573
4	0.76	0.0014	0.0332	1.25	37.63	673
5	0.54	0.0012	0.0308	1.47	47.63	773
6	0.45	0.0011	0.0270	1.56	57.61	873
7	0.42	0.0011	0.0266	1.59	59.61	893

Thus, in the course of the research, the properties of thermo-oxidative degradation of nitro- and sulfonic compounds of immobilized ligands based on Auren were studied on the basis of experimental data obtained on the kinetics of processes in the temperature range from 293 to 862 K. In the subsequent research work, it is advisable to investigate the application areas of nitro- and sulfo compounds of Aurene based on the thermal parameters identified in the material under consideration.

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