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Thermoresistive properties of (Copper, Neodymium) Acetylacetonate

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A new material tetrakis-μ3-(methoxo) (methanol)-pentakis (acetylacetonate) (tricuprum (II), neodymium (III)) methanol (I) was synthesized as $[Cu_3Nd(AA)_5(OCH_3)_4CH_3OH]$ CH_3OH, where HAA = $H_3C–C(O)–CH_2–C(O)–CH_3$. Based on the data of elemental analysis and physical-chemical research methods, it was found that the obtained coordination complex (I) contains atoms of Copper (II) and Neodymium (III) in the ratio Cu:Nd = 3:1, and its composition corresponds to the gross formula: $Cu_3NdO_{16}C_{31}H_{55}$. The measurement of electrical conductivity of the obtained material was performed in the compressed form. For the coordination complex (I), the number of valence electrons in one molecule was calculated to be 270; the mass of one molecule was calculated to be $163.65 \cdot 10^{-20}\ kg$; the total number of molecules in the volume of a cylindrical sample weighing 0.125 g and having volume of $17.74 \cdot 10^{-9}$ m^3 was calculated to be $7.638 \cdot 10^{13}\ molec$; and the total number of valence electrons as $20.6232 \cdot 10^{15}$. In the temperature range of 303 – 423 K, the specific resistance of the compressed sample decreases from $2 \cdot 10^{12}$ to $5 \cdot 10^{4}$ Ohm·cm, which confirms that the isolated compound is a semiconductor with a band gap of 1.6125 eB. The electrical conductivity properties of the coordination complex as a thermosensitive element were studied; for this purpose we used an experimental sample of compressed material with geometric dimensions of $1 \cdot 10^{-3}$ m×$0.5 \cdot 10^{-3}$ m×$0.5 \cdot 10^{-3}$ m.

Keywords: temperature, thermistor, concentration, semiconductor, electrical conductivity properties, coordination complex.

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Introduction

A thermistor is one of the products of electronic engineering with electrical resistance to depend greatly on temperature. Many systems of remote and centralized temperature measurement and control, thermal control of machines and mechanisms, fire alarm, temperature compensation circuit of various electric circuit elements, high frequency radiation power measurement, liquid and gas velocity measuring devices, etc. are developed and operate using thermistors [1-6]. Most industrial types of thermistors with a negative temperature coefficient of resistance (NTCR) are manufactured using semiconductor materials with oxides of 3d-metals [1-6]. In industry, such semiconductor materials are obtained by co-precipitation and further wet mixing of raw materials with subsequent grinding in ball mills and long-term high-temperature sintering of the main components [5, 6].

However, such oxide semiconductor materials have a number of disadvantages: compositional change due to electric current flow, chemical instability of the material in the operating temperature range and high sensitivity to dopants that may occur in the manufacture of thermistors, as well as low ability to reproduce initial parameters (nominal resistance values and NTCR). In addition, practical use of oxide semiconductors does not enable to obtain materials with a wide range of resistivity, which limits the ability to manufacture products of the same dimensions with a wide range of nominal resistance values.

Consequently, the synthesis of new semiconductor materials with significant conductivity and high values of

NTCR is relevant as they will allow to develop thermistors with high sensitivity to temperature changes.

In the field of new functional materials development an important role belongs to coordination complexes in which β-diketone is a chelating and in some cases a bridging ligand [7-14]. Nowadays, the spheres of practical use of functional materials containing β-diketonates of metals are constantly expanding. In particular, they are used in gas sensors, molecular thermometers, in the production of optical fiber and light-converting materials as well as they are the source for obtaining the materials with valuable electrical, optical, catalytic and other properties [7-14]. Of particular interest among this class of coordination complexes belongs to heterometallic β-diketonates which have semiconductor properties [15,16].

I. Theoretical and experimental research

The purpose of this research is the synthesis of heterometallic (copper, neodymium) acetylacetonate and the study of its thermoresistive properties. It is known [15,16] that heterometallic β-diketonates have semiconductor properties, the performance of which depends on the nature of both metals and ligands, as well as on the stereochemistry of the coordination complex.

In order to find new heterometallic β-diketonates with semiconductor properties, a method for the synthesis of a tetrakis-μ3-(methoxo) (methanol) (methanol)-pentakis (acetylacetonate) (tricuprum (II), neodymium (III)) methanol coordination complex was developed, which has a composition:

$$[Cu_3Nd(AA)_5(OCH_3)_4CH_3OH] \cdot CH_3OH$$

where HAA = $H_3C–C(O)–CH_2–C(O)–CH_3$.

Tetrakis - μ3-(methoxo) (methanol) pentakis (acetylacetonate) (tricuprum (II), neodymium (III)) methanol (I) was obtained by the following method: 3.62 g (15 mmol) salt weights of copper (II) nitrate trihydrate and 2.19 g (5 mmol) of neodymium (III) nitrate hexahydrate were dissolved in 130 ml of methyl alcohol absolute containing 40 ml of orthoformate and kept for 2 hours at room temperature in a hermetically sealed conical flask. Next, 2.7 ml (25 mmol) of acetylacetone was added to the reaction mixture, the conical flask was closed with a reversible water cooler and placed on a heated magnetic stirrer. Then, with continuous stirring and heating (~ 50 ºC), piperidine was gradually introduced into the reaction mixture to pH = 8 with the heating and stirring for one hour. After cooling, a homogeneous blue fine-crystalline sediment precipitated, which was filtered on a glass filter, washed with a small amount of absolute methanol, diethyl ether and dried in a vacuum desiccator over silica gel.

The practical yield is 4.07 g, which is 80% of theoretical one. The isolated coordination complex (I) is a fine-crystalline powder, which is poorly soluble in dimethylformamide, preferably in dimethyl sulfoxide, practically insoluble in alcohols, chloroform, benzene, acetone, and decomposes in water.

For the synthesized coordination complex (I) in a dry powder state, elemental analysis was performed and it was found that it contains:

Cu – 18.65%; Nd – 14.25%; C – 36.68%; H – 5.24%.

The presented data indicates that the ratio of metals in complex (I) is Cu:Nd = 3:1, and its composition corresponds to the following gross formula: $Cu_3NdO_{16}C_{31}H_{55}$. In addition, magnetochemical, IR spectroscopic and thermogravimetric studies were performed for the isolated heterometallic complex (I) [17].

A detailed analysis of the obtained experimental data of physical-chemical research methods allows us to propose the following scheme of arrangement of chemical bonds for coordination complex (I) (Fig. 1) [17]:

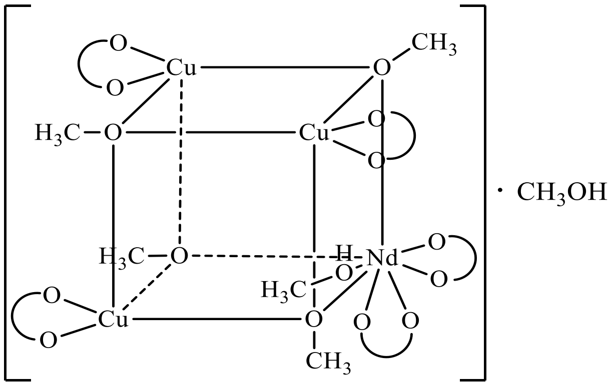

Fig. 1. The scheme of arrangement of chemical bonds in tetrakis-μ3-(metokso) (methanol) pentakis (acetylacetonate) (tricuprum (II), neodymium (III)) methanol (I).

Since the obtained complex $[Cu_3Nd(AA)_5(OCH_3)_4CH_3OH] \cdot CH_3OH$ (I) contains crystallization molecules of methyl alcohol, which are easily cleaved by heating, the study of its electrical properties was performed after preheating in an oven at 383ºK until termination mass changes.

To measure the electrical conductivity of the isolated complex in the form of powder, we used a specially designed pressing device, which was used to make a cylindrical sample weighing 0.125 g with a volume of $17.74 \cdot 10^{-9}$ m^3. Based on these data according to formula (1), the density of the manufactured experimental sample of the test material in the compressed state is calculated as:

$$\rho = m/v = 7.046 \cdot 10^3 kg/m^3, \quad (1)$$

where ρ stands for the density of substance; m – for the mass of experimental sample; v – for the volume of experimental sample.

For coordination complex $[Cu_3Nd(AA)_5(OCH_3)_4CH_3OH]$ (I) molar mass was calculated which equals to 985.5 *g/mol* and the number of valence electrons in one molecule is 270.

To find the mass of one molecule, we used formula (2)

$$m_0 = M/N_A = 163.65 \cdot 10^{-20} kg, \quad (2)$$

where M stands for molar mass of the complex (I); m_0 – for the mass of one molecule of complex (I); N_A – for Avogadro constant.

The total number of molecules in the volume of a cylindrical sample weighing 0.125 g with a volume of 17.74·10^{-9} m^3 was calculated by formula (3):

$$N_{\text{мол}} = \frac{m}{m_0} = 7.638 \cdot 10^{13} molec., \qquad (3)$$

where $N_{мол}$ stands for the total number of molecules; m_0 – for the mass of one molecule of complex (I); m – for the mass of the experimental sample.

The total number of valence electrons is:

$$N = 270 \cdot N_{molec} = 20.6232 \cdot 10^{15}. \qquad (4)$$

The experiment showed that in the temperature range of 303 – 423 K the resistivity of the pressed sample of the test material of complex (I) decreases from 2·10^{12} to 5·10^{4} *Ohm·cm*, therefore, the isolated complex is a semiconductor.

Based on experimental data, the specific conductivity of the complex for these temperatures was calculated. For $T_1 = 303$ K – $\sigma_1 = 5.0 \cdot 10^{-11}$ $(Ohm \cdot m)^{-1}$, and for $T_2 = 423$ K – $\sigma_2 = 2.0 \cdot 10^{-3}$ $(Ohm \cdot m)^{-1}$. According to these data, the band gap was calculated as:

$$\Delta E = \frac{k \ln\frac{\sigma_1}{\sigma_2}}{\left(\frac{1}{T_2} - \frac{1}{T_1}\right)} = 2.58 \cdot 10^{-19} J = 1.6125 eV, \qquad (5)$$

where ΔE stands for the band gap of coordination complex [$Cu_3Nd(AA)_5(OCH_3)_4CH_3OH$] (I); $\acute{k}$ – for Boltzmann constant; T – for absolute temperature; and σ– for specific conductivity of material.

The calculations confirm that this material is actually a semiconductor with current carriers of both signs.

To use this complex as a thermosensitive element, the test sample was pressed in the form of an 0402 type SMD thermistor with geometric dimensions 1·10^{-3} m×0.5·10^{-3} m×0.5·10^{-3} m.

With a given width of a semiconductor band gap, using the formula for the concentration of charge carriers dependence on temperature, we obtained a graphical dependence of the concentration of charge carriers on temperature, which is shown in Fig.2.

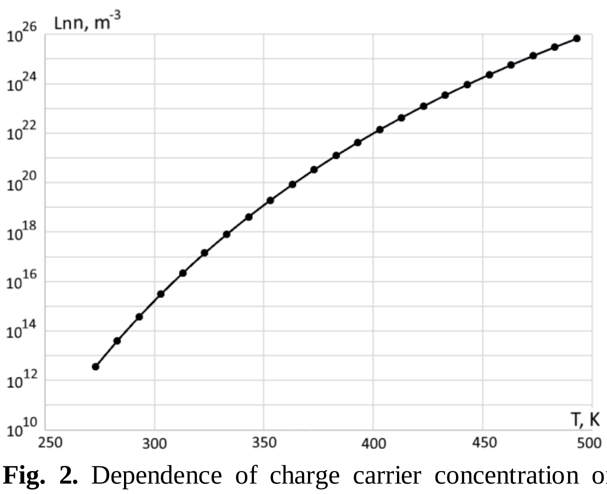

Fig. 2. Dependence of charge carrier concentration on temperature.

As it can be seen from Fig. 2, the concentration of charge carriers in the temperature range of 273 ÷ 493 K increases from 3.53·10^{12}m^{-3} до 6.61·10^{25}m^{-3}. The calculations for the obtained coordination complex (I) and the study of the effect of temperature on the electrical parameters of this substance show the following results.

The graph shows that at 273 K the specific conductivity was 5.67·10^{-14} $(Ohm \cdot m)^{-1}$, and at 493 K it was 1.063 $(Ohm \cdot m)^{-1}$. The graph of dependence of resistance change of the material being studied on temperature is given in fig. 5.

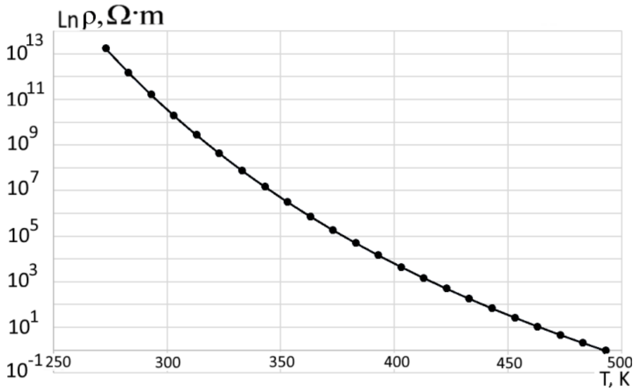

Fig. 3. Dependence of specific resistance on temperature.

As we can see from Figure 3, in the temperature range from 273 K to 493 K the specific resistance changed from 1.76·10^{13} *Ohm·m* to 0.94 *Ohm·m*.

The graph of the dependence of specific conductivity on temperature is given in Fig. 4.

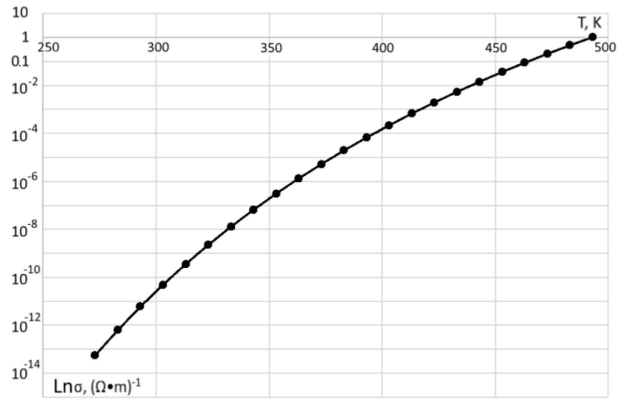

Fig. 4. Dependence of specific conductivity of semiconductor material on temperature.

As we can see from Figure 5, the resistance of the sample decreases rapidly, so at a temperature of 273 K it is equal to 7.04·10^{16} *Ohm*, and at 303 K it is 7.99·10^{13} *Ohm*, while at 493 K it equals to 3763.02 *Ohm*. The difference in 13 items indicates that this material can be used to create thermosensitive resistors or to be the basis to create more complicated devices that will operate in a wide temperature range.

Having applied the law of dependence of material resistance and charge carrier concentration on temperature, the dependence of the amount of current passing through the test sample on temperature is obtained. Graphical dependence of current on temperature at voltage U = 10 V is shown in Figure 6.

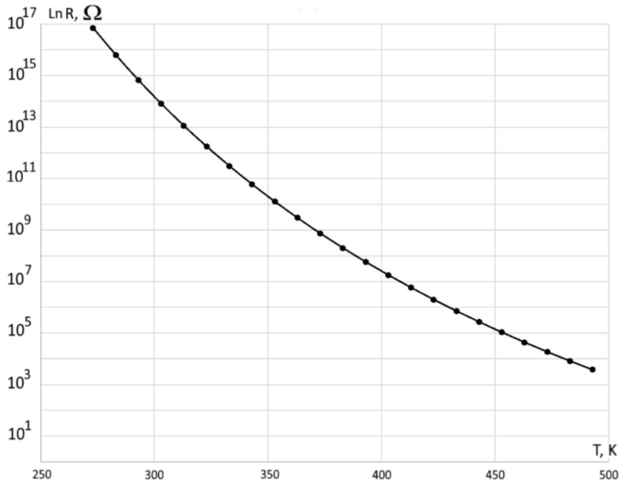

Fig. 5. Dependence of resistance of material with geometric sample size $1 \cdot 10^{-3}$ m×$0.5 \cdot 10^{-3}$ m×$0.5 \cdot 10^{-3}$ m on temperature.

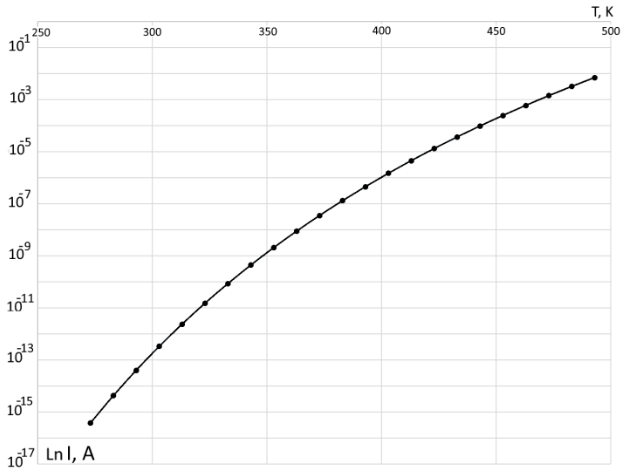

Fig. 6. Dependence of current on temperature at voltage U = 10 V.

The graph shows that at a supply voltage of 10 V and a temperature of 273 K the current value is $I = 1.42 \cdot 10^{-16} A$, and at a temperature of 493 K $I = 2.65 \cdot 10^{-3} A$.

Therefore, the nominal supply voltage for such material will be tens of volts.

The logarithmic dependence of current density on temperature at supply voltages of 1 V and 10 V is shown in Fig.7.

The graph (Fig. 7) shows that the current density reaches zero at a temperature of 333 K. The value of current density varies from $1.9 \cdot 10^{-19} A/m^2$ to 35432.5 A/m^2 within temperature shift from 273 K up to 493 K.

Based on the obtained dependences we can say that the use of synthesized material to create thermosensitive elements is quite perspective.

The calculation of the Hall constant at 30 °C shows the following results:

$$R_H = 1/nq = 2009.6 m^3 \cdot C^{-1}, \qquad (6)$$

where *n* stands for charge carriers concentration; and *q* – for electron charge.

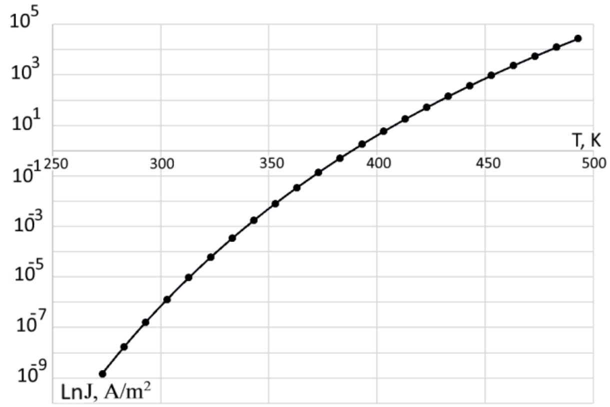

Fig. 7. The dependence of current density on temperature at voltage U = 10 V.

The calculation of the quantum Hall constant was performed by formula (7):

$$R_{\text{кв } H} = -3\pi/8nq = -2366.39 m^3 \cdot C^{-1}, \qquad (7)$$

Using formula (7) and the equation of the dependence of charge carriers concentration on temperature we obtained formula (8) which shows the dependence of the Hall constant on temperature:

$$R_{\text{кв } H} = -\frac{3\pi}{8nq_0} \cdot e^{\frac{\Delta E}{kT}} \; . \qquad (8)$$

According to formula (8), the logarithmic dependence of the quantum Hall constant on the temperature is constructed, which is given in Fig. 8

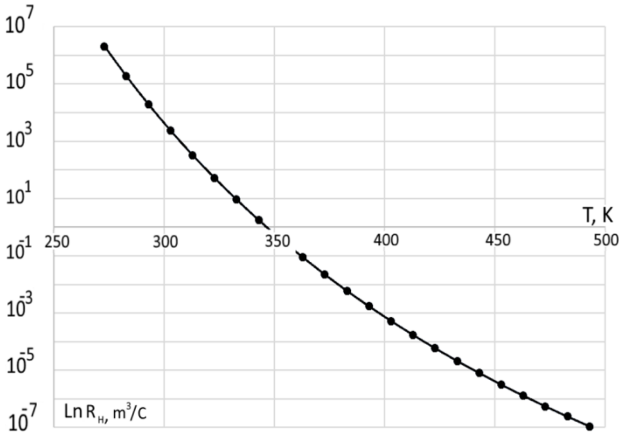

Fig. 8. Dependence of the quantum Hall constant on temperature.

As it can be seen from Fig. 8, the value of the quantum Hall constant for such a material, with increasing temperature from 273 K to 493 K, decreases from $2.08 \cdot 10^{6}\, m^3 \cdot C^{-1}$ to $1.1 \cdot 10^{-7}\, m^3 \cdot C^{-1}$.

To find the mobility of charge carriers from the experimental data of resistivity at 303 K and $\rho = 2 \cdot 10^{12} Ohm \cdot cm$, the specific conductivity $\sigma = 5.0 \cdot 10^{-11}$ *sim/m* was calculated.

$$\mu_n = R_H \cdot \sigma \; . \qquad (9)$$

Let us determine the mobility of charge carriers for the quantum case:

$$\mu_n = R_{кв}\, H \cdot \sigma = 1.183 \cdot 10^{-7} m^3 \cdot (V \cdot sec)^{-1}. \quad (10)$$

Substituting the dependences of the Hall constant on temperature and the specific conductivity on temperature, it was determined that the mobility of charge carriers is a constant value $\mu = 1.183\times10^{-7}$ $м^3/(V \cdot s)$ and does not depend on temperature.

Conclusions

A new thermosensitive element based on the synthesized semiconductor material has been developed. The study of the conductive properties of heterometallic (copper, neodymium) acetylacetonate in a compressed form in the temperature range of 273 – 493 K showed that with increasing temperature the resistivity decreases sharply from $1.76 \cdot 10^{13}$ to 0.94 *Ohm·m*, which is typical for semiconductor materials, and the resistance of the sample decreases rapidly so that at a temperature of 273 K it is equal to $7.04 \cdot 10^{16}$ *Ohm*, and at 303 K it is $7.99 \cdot 10^{13}$ *Ohm*, while at 493 K it equals to 3763.02 *Ohm*, as well as at 273 K specific conductivity was $5.67 \cdot 10^{-14}$ $(Ohm \cdot m)^{-1}$, while at 493 K it was 1.063 $(Ohm \cdot m)^{-1}$.

At a supply voltage of 10 V and at a temperature of 273 K the current value is I = $1.42 \cdot 10^{-16}$ *A*, and at a temperature of 493 K I = $2.65 \cdot 10^{-3}$ *A*. The value of current density varies from $1.9 \cdot 10^{-19}$ A/m^2 to 35432.5 A/m^2 within the temperature shift from 273 K to 493 K. The range of operating temperatures is from +30 to + 220°C, and the decomposition of the chemical complex occurs from 250°C, the concentration of charge carriers increases from $3.11 \cdot 10^{15}$ m^3 at 30°C to $6{,}61 \cdot 10^{25}$ m^{-3} at 220°C; at this condition the Hall constant with increasing temperature from 30 to 220°C decreases from 2366.39 $m^3 \cdot C^{-1}$ to $1.11 \cdot 10^{-7}$ $m^3 \cdot C^{-1}$.

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Терморезистивні властивості (купрум, неодим) вмісного ацетилацетонату

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Синтезовано новий матеріал тетракіс-μ3-(метоксо) (метанол)-пентакіс (ацетилацетонато) (трикупрум (II), неодим(III)) метанол (I), такого складу: $[Cu_3Nd(AA)_5(OCH_3)_4CH_3OH] \cdot CH_3OH$, де НАА = $H_3C–C(O)–CH_2–C(O)–CH_3$. На основі даних елементного аналізу та фізико-хімічних методів дослідження встановлено, що отримана комплкексна сполука (I) містить атоми Купруму(II) та Неодиму(III) у співвідношенні Cu:Nd = 3:1, а її склад відповідає брутто-формулі: $Cu_3NdO_{16}C_{31}H_{55}$. Вимірювання електропровідності отриманого матеріалу проводили в пресованому вигляді. Для комплексної сполуки (I) розраховано кількість валентних електронів в одній молекулі – 270; масу однієї молекули - $163{,}65 \cdot 10^{-20}$ кг,; загальну кількість молекул в об’ємі циліндричного зразка масою 0,125 г та об’ємом 17,74·10-9 м3 - $7{,}638 \cdot 10^{13}$ молек.; загальну кількість валентних електронів - $20{,}6232 \cdot 10^{15}$. В інтервалі температур 303 – 423 К питомий опір пресованого зразка зменшується від 2·1012 до 5·104 Ом·см, це підтверджує, що виділена сполука є напівпровідником, з шириною забороненої зони 1,6125 еВ. Досліджено електропровідні властивості комплексної сполуки, як термочутливого елементу, для цього використовували експериментальний зразок спресованого матеріалу з геометричними розмірами $1 \cdot 10^{-3}$ м×$0{,}5 \cdot 10^{-3}$ м×$0{,}5 \cdot 10^{-3}$ м.

Ключові слова: температура, терморезистор, концентрація, напівпровідник, електропровідні властивості, комплексна сполука.