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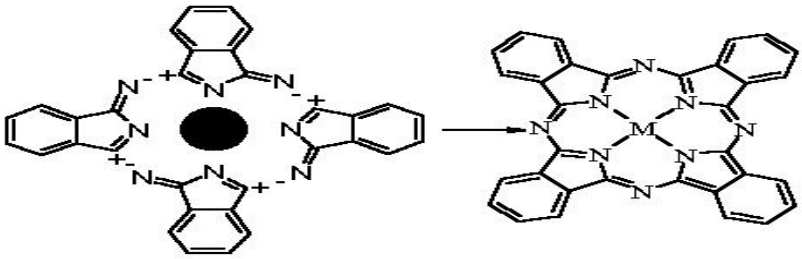
# OBTAINING PIGMENTS CONTAINING COPPER AND ANALYSIS OF THEIR PROPERTIES

**Abstract:** A technology for obtaining copper-preserving phthalocyanine pigment by heating in the presence of urea, phthalic anhydride, metal salts and a catalyst was proposed. The properties of CuPc pigments were studied using thermal analysis and X-ray phase analysis methods. An infrared spectroscopy analysis of the obtained new CuPc pigment was carried out. Based on the IR analysis, it was found that the newly obtained substance contains important functional groups. The volume and density of the obtained sample was determined. A thermogravimetric analysis of copper-containing pigment was carried out. In the thermal analysis, it was determined that the obtained pigment undergoes a mass change in three stages.

**Key words:** Chemistry, phthalocyanine, pigment, dye, sulfuric acid, urea, phthalic anhydride, copper, calcium, element, central atom, temperature, matter.

Currently, more than 70 different phthalocyanines are known. Phthalocyanines - phthalodinitrile in its mesomeric form begin to group around the metal ion, which again leads to the formation of a macrocycle. A flat aromatic macroring highly resistant to aggressive reagents is tetramerized at the atom of metal 4 molecules of phthalodinitrile [1]. The nature of the metal ion at the center of the phthalocyanine ring has a significant effect on the physical and chemical properties. For example, when a metal ion is replaced by a complex, the redox properties of the macrocyclic ring or the nature of the photochemically excited state change dramatically.




**1-rasm. Flat aromatic macroring tetramerization at the metal 4-molecule phthalonitrile atom**

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Phthalocyanines are tetraazobenzororphyrins, highly heterocyclic compounds, consisting of an isoindole ring interconnected by structural sp2-hybridized nitrogen atoms with natural porphyrins. However, unlike porphyrins, phthalocyanines do not occur in nature, they are completely synthetic compounds.

The main difference between the phthalocyanine macrocycle and the porphyrin system is the presence of four phenylene rings and nitrogen atoms in the meso positions instead of carbon [2]. Phthalocyanine complexes of various metals can also be used to create complex composite materials. It was shown in the work that it is possible to create composite structures based on copper phthalocyanine and polystyrene using the laser coating method [3]. A detailed study of carbon compounds such as phthalocyanine can be complicated because the central metal atom makes a dominant contribution to the dispersion of metal phthalocyanines due to the difference in atomic weight [4].

**Materials and Methods.** For synthesis at high temperature by heating method, a special container made of high temperature, acid-resistant stainless steel metal with a capacity of 1000 ml, a furnace capable of obtaining high temperature, a glass tube, a rubber hose and materials are needed. Urea, phthalic anhydride, copper (II) chloride and orthoboric acid were mixed in a special container. The mixture was placed in an oven at 250 0C for one hour. For the gases released as a result of the reaction and phthalimide, a gas outlet pipe was installed on the top of the furnace, and the hose connected to the pipe was immersed in a container of water.

As a result, a porous purple substance was formed in the vessel. The resulting substance was cooled to 100 °C and mixed with 20 ml of concentrated (90%) sulfuric acid, resulting in a dark green solution. The resulting solution was cooled to 50°C and mixed with boiling water. The solution melted and a blue precipitate was formed, and the separated yellowish-brown liquid was carefully poured into another container and disposed of. Distilled water was added to the precipitate and it was filtered using a vacuum pump in a Buchner funnel. The dark blue filtrate was copper phthalocyanine, which was dried in an oven at 100°C for two hours. The dried copper phthalocyanine was crushed in a mortar and sieved.

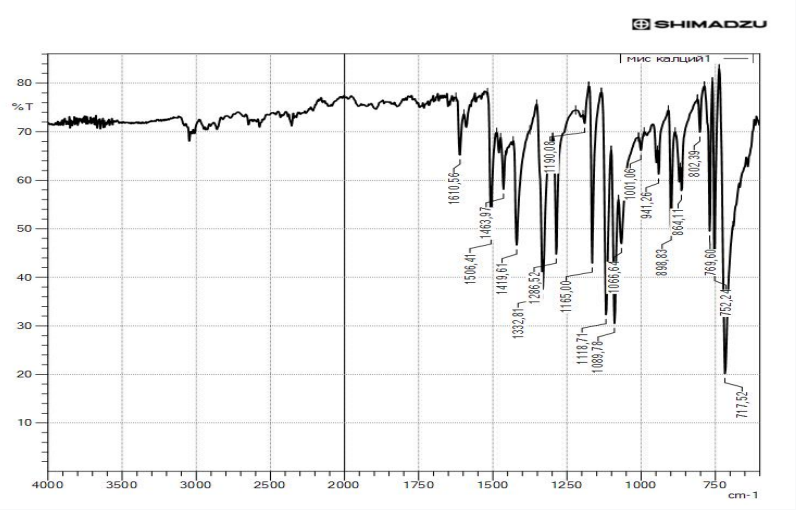

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**Figure 3. IR-spectroscopy analysis of the obtained copper-containing pigment**

**Results and Discussion.** The reaction of the synthesis of the obtained substance was expressed as shown in Fig. 2, based on the analysis of the IR- spectrum analysis of the substance. Formation of phthalocyanine rings in copper-calcium phthalocyanine is seen in the absorption region of 752 cm-1, formation of C-H+ in the isoindole plane is seen in the absorption region of 1165 cm-1, pyrrole rings are seen in the absorption region of 1332 cm-1 , pyrrole nitrogen atoms appear in the absorption region of 1419 cm-1, isoindole 1463 cm-1 appears in the absorption region, -N= 1506 cm-1 appears in the absorption region

**Determination of volume and density.**

The analysis was performed by a standard method [5]. A sample of 1.0 g (with an accuracy of 0.01 g) is placed in a cylinder with a capacity of 25 $cm^3$ with a funnel at a distance of 20 mm from the upper edge of the cylinder walls without shaking using a stationary. The surface of the sample was then carefully leveled with a spatula and the pigment volume was set on a cylinder measuring scale.

Mass volume ($V_{omma}$) was determined according to the formula

$$V_{\text{омма}} = \frac{V'}{m_1 - m_0}, \quad (2.1)$$

where: V'-pigment volume (cm3), m0, m1-mass of empty and filled cylinders (g).

After determining the bulk volume, the cylinder was shaken for 1 min to cover the sample, and the value of the compressed volume was determined by the measured volume. If there is no more than 0.2 cm3 between the last V values, shaking is stopped.

The exact density of the pigment (color) was determined by the formula

$$\rho_{\text{аник}} = \frac{m_1 - m_0}{V''}, \quad (2.2)$$

where: V"-volume of pigment after compression ($cm^3$), $m_0$, $m_1$-mass of empty and filled cylinders (g).

**Thermal stability of copper-containing pigments.** The thermal analysis of the obtained organic pigments with a new composition was carried out in the temperature range of 600°C. All samples of the thermal analysis of the mentioned pigments were carried out in a dynamic mode at a speed of 10 degrees/min in an aluminum mortar. In addition, endothermic and exothermic points of organic pigment were proved.

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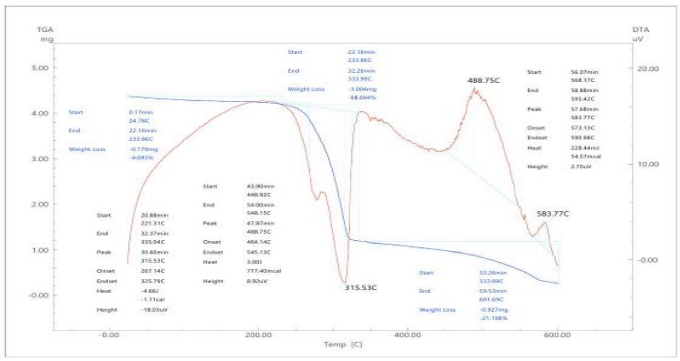


**Figure 3. Derivatogram of new organic pigments containing copper**

The maximum temperature of 600°C was selected for the newly synthesized copper-containing organic pigment in dry mass presented in Figure 3, and the results of the pigment analysis were studied according to the given thermogravimetric derivatogram (TG) and differential thermogravimetric analysis.

Two exothermic effects were observed at 488.75 and 583.77°C and one endothermic effect at 315.53°C. 4,373 mg of organic pigment was taken in an open-mouth crucible made of aluminum resistant to temperature of 600°C, and the temperature was gradually increased starting from 20°C.

According to the analysis of the thermogravimetric curve of the organic pigment, the TG curve mainly takes place in the temperature range of 3 intensive mass losses. Mass-loss range 1 corresponds to 24.78 - 233.86°C, mass-loss range 2 corresponds to 233.86-333.99°C, and mass-loss range 3 corresponds to 333.99-600°C .

The analysis shows that the 1st mass-loss interval has a mass loss of 0.179 mg, i.e. 4.093%, while the second mass-loss is the most intense decay. The main amount of mass loss in this decay is 3.004 mg, i.e. 68.694%. In the third mass loss interval, the mass loss is 0.927 mg, which is 21.198%.

The best temperature for the synthesis of copper phthalocyanine pigment was found to be 250 0C. Based on the experiments, copper phthalocyanine pigment gives the highest result at 250 0C. Among the main characteristics, it was observed that the intensity of the pigment is better than that obtained at low temperature

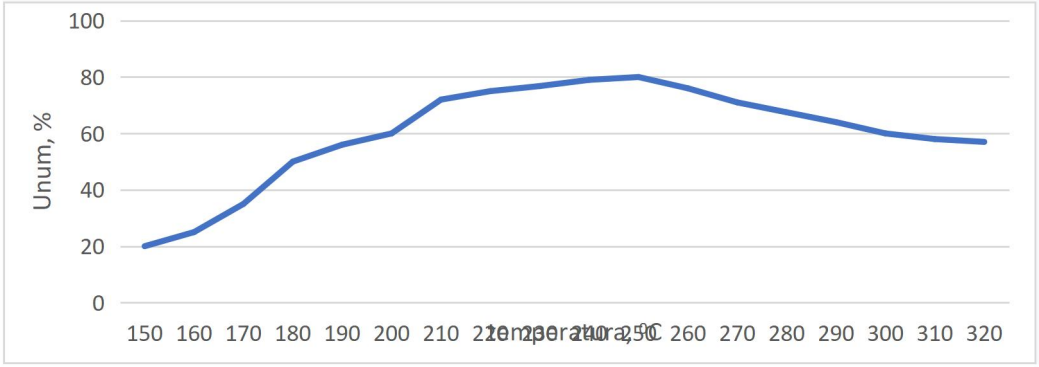

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**Figure 4. Effect of temperature on product yield during synthesis of copper phthalocyanine pigment in dry mass at high temperature**

**Conclusion.** A technology for obtaining copper-preserving phthalocyanine pigment by heating in the presence of urea, phthalic anhydride, metal salts and a catalyst was proposed. A method of changing the synthesized CuPc pigments into a and b modification was developed. The properties of CuPc pigments were studied using thermal analysis and X-ray phase analysis methods. The cost-effectiveness indicators of using synthesized CuPc pigment as an import substitute pigment were calculated, according to which the cost-effectiveness was 2451614000 soums for CuPc, 3794221750 soums for CuPc. Thus, CuPc pigment production passed the tests successfully.

The newly synthesized CuPc pigment showed good results according to GOST 6465-76 based on the standard requirements for PF-115 enamel of different colors. PF-115 enamel prepared by adding CuPc pigment was recommended to be used mainly as a coating for construction materials such as iron, wood, etc.

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