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INVESTIGATION OF PHTHALOCYANINE DIAMIDOPHOSPHATE-COPPER BY THERMAL ANALYSIS

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Abstract: The article presents the results of a study of the synthesis of a new type of pigment - copper phthalocyanine diphosphate (DAPCuPc), which contains macroheterocyclic compounds. The influence of the method of obtaining a pigment in the liquid and solid phases on its physicochemical properties and intensity has been studied. The gross formula of the pigment is given and the areas of its practical application are shown. Thermal analysis on a Setaram LabSys Evo instrument (France) was used to study the thermal stability of a highly intense pigment of the organic copper phthalocyanine pigment, which was compared with the closest analog, the copper phthalocyanine pigment. The results of the mass

loss of the pigment upon heating are presented. It is shown that, due to the content of phosphorus-containing compounds, the synthesized pigment has high heat resistance and intensity. Thermal studies of exothermic and endothermic processes were carried out in the temperature range of 100-500 °C.

Keywords Phthalocyanine pigment, thermal analysis, exothermic, endothermic, copper diamidophosphate, phthalic anhydride, intensity.

1. Introduction. Today, there is a scientific basis in the world for solving a number of problems to improve the properties of phthalocyanine-based pigments and their effective use, including: simplification and safety of methods for the synthesis of phthalocyanine pigments; creation of new types of phthalocyanine pigments soluble in water and solvents; increased production of phthalocyanine pigments. It is necessary to find and expand the scope of new compositions that can purposefully change the anticorrosion, static and dynamic strength properties of phthalocyanine pigments [1].

At present, advanced technologies are being developed in the world, such as the production of electrical and optical materials based on phthalocyanine pigments, solar cells, and chemical sensors. An important advantage of phthalocyanine pigments is their very low toxicity; they are used in the production of packaging materials for food products, children's toys, and medicines. Also, phthalocyanines and their derivatives play an important role in obtaining light and heat resistant dyes, which are characterized by high chemical resistance and color stability [2].

About 25% of the volume of organic pigments produced are phthalocyanine pigments. In this regard, phthalocyanines containing nitrogen and phosphorus-containing groups are of particular scientific and practical interest. In this case, it is possible to obtain varnish coatings in the presence of phthalocyanine pigments [3].

Phthalocyanine is a planar 18 π-electron heterocyclic aromatic system with an alternating nitrogen and carbon ring structure derived from porphyrin. Phthalocyanines have been known for over 70 years and are widely used as dyes. Moreover, they have generated interest for various applications such as liquid crystals, photosensitizers, non-linear optics, solar cells, catalysis, and various chemical sensor applications. The peripheral and non-peripheral positions of the benzene ring of phthalocyanines can be replaced by many other molecules to impart new properties[4].

Probably, there is no such area of modern science in which the possibilities of using the properties of phthalocyanines would not be investigated, as evidenced by a huge number of publications [5].

The most interesting and in demand are the photophysical properties of Pcs [6]: high extinction and, in some cases, intense luminescence, the possibility of controlling the position of absorption/emission maxima, nonlinear optical characteristics, etc. Their combination with high (thermo)chemical stability and the ability to sublimate without decomposition, which makes it possible to obtain mechanically strong layers up to 100 μm thick with reproducible electrophysical characteristics, makes it easy to obtain various optoelectronic devices based on Pcs: in materials for creating charge memory in the manufacture of CD / DVD - discs, photosensitizers, chemosensors, light emitters [7], optical limiters, and photorefractive materials [8]. A number of semiconductor structures based on Pcs have a significantly increased photocurrent signal in the near IR spectral region and open up prospects for creating new generation solar energy devices [9]. Water-soluble phthalocyanines are used as sensitizers in photodynamic therapy of oncological diseases [10], in coatings and matrices of conducting polymers and artificial lipids immobilized on electrodes [11].

Complexes of phthalocyanines with transition metals have found application as

catalysts for the purification of hydrocarbons from sulfur compounds and in the neutralization of toxic effluents. Heterogeneous catalysts obtained by fusing a mixture of CoPc with salts of metals of variable valence onto a polymer matrix are highly efficient [12].

Phthalocyanine derivatives with mesomorphism can serve as "structural action" additives in the composition of tribologically efficient and environmentally friendly lubricants. Promising is the use of Pcs in the composition of solid layered lubricants (TLS) and plastic lubricants (PS), which have a long service life at temperatures above 180°C, high moisture resistance, and good colloidal stability. On the basis of Pcs, it is planned to create permanent triboactive lubricating compositions for friction units [13].

Thus, due to the unique combination of valuable properties, Pcs derivatives are widely used in many branches of modern materials technology (Figure 1.).

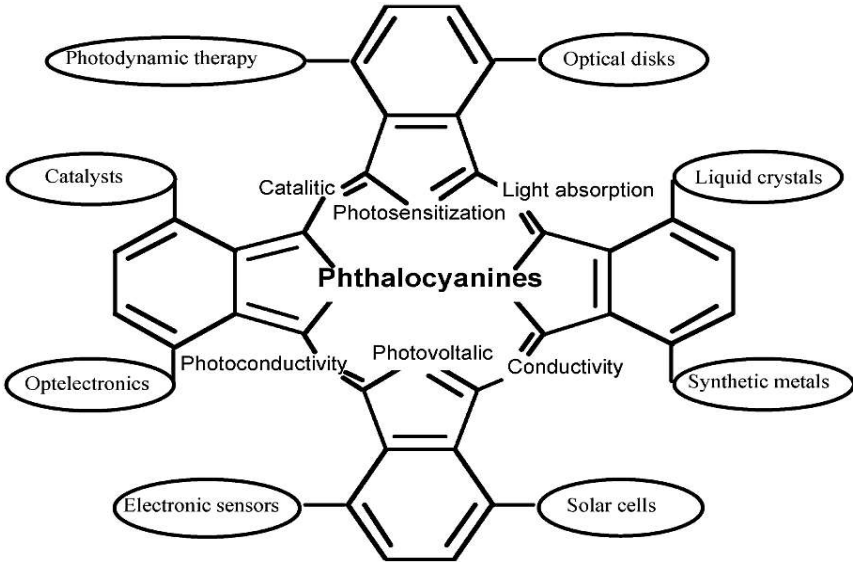

Figure 1. Modern applications of phthalocyanines. Further, aspects of the use of phthalocyanines that are directly related to this work are considered in more detail

2. Experimental Methodology. Synthesis of copper phthalocyanine (DAPCuPc) based on diamidophosphate. The synthesis was carried out in two ways: in a solvent medium and by heating at high temperatures [3].

1-way. Synthesis was carried out at high temperature by heating. 9.8 g (0.1 mol) of phosphoric acid and 18 g (0.3 mol) of urea were loaded into a beaker, stirred with a glass rod until urea was completely dissolved in phosphoric acid at 130 °C, then a mixture of the remaining ingredients was added, which consist from: 7 g (0.04 mol) copper(II) chloride, 6 g (0.1 mol) urea, 24 g (0.17 mol) phthalimide. After adding all the reagents, the reaction mass was stirred and the catalyst was added in an amount of 1 % by weight of phthalimide. The reaction mass is mixed until homogeneous (red). The reaction mixture is heated to 260 °C for 3 hours. The resulting powder reaction mixture is cooled to 50 °C and 85 % sulfuric acid is added in the amount necessary to completely dissolve the resulting powder. Further, hot water is added to this mixture, while the pigment is deposited on the bottom of the vessel, the liquid is decanted, the pigment is washed with hot water and filtered. The resulting pigment is dried in a drying cabinet at 50-60 °C until completely dry. Product yield 80%.

2-way. For this, 73.6 g (0.4 mol) of diamidophosphate, 24 g (0.4 mol) of urea,

235.6 g (1.6 mol) of phthalimide, 70.8 g (0.4 mol) of copper(II) chloride (II). To the mass of copper acetate and phthalimide add 1% ammonium heptamolybdate and 550 ml of dimethyl sulfoxide (DMSO) as a solvent. The reaction is carried out at a temperature of 150-180 °C for 3-4 hours with stirring. Upon completion of the reaction, the reaction mass is cooled and filtered on a Buchner funnel, and the dark turquoise pigment remaining in the funnel is again washed with distilled water. The washed product is dried in an oven at 50 °C. Product yield 93%.

3. Results and its discussion. The new pigment diamidophosphate-copper-phthalocyanine (DAP-CuPc) was obtained in two different ways. Based on the results obtained, the second method was chosen. The proposed reaction mechanisms and the formula of the resulting substance are based on elemental analysis and IR spectral analysis:

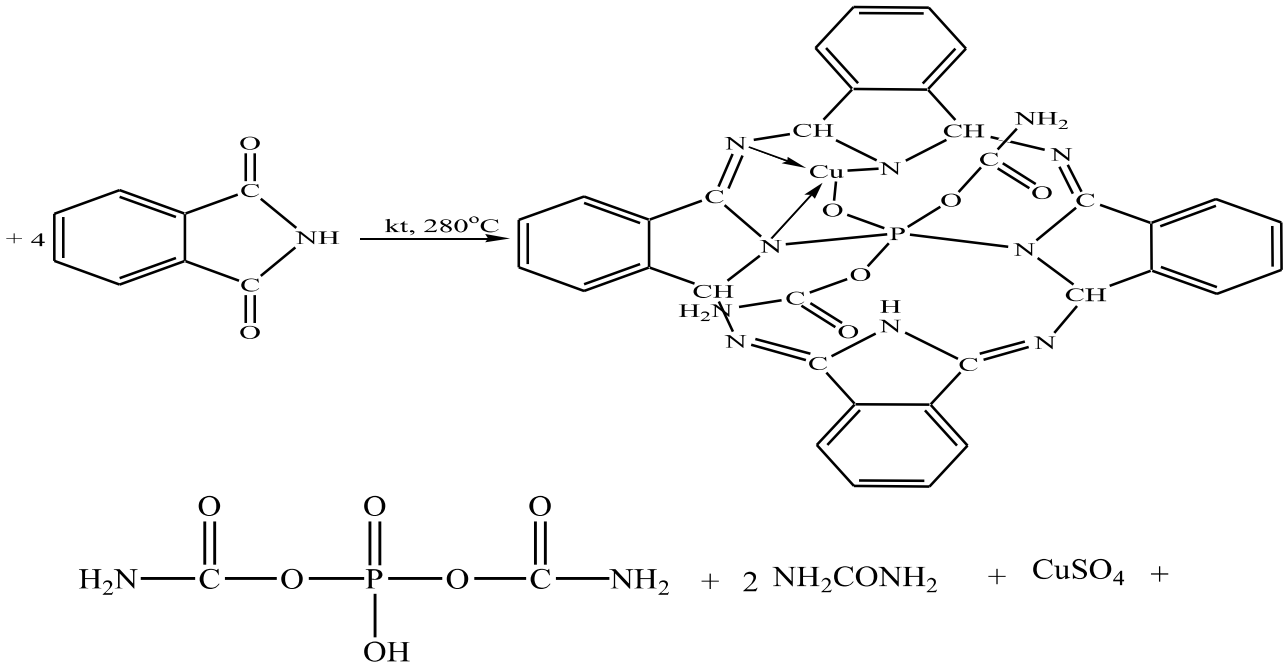

Figure 2. New pigment, diamidophosphate-copper phthalocyanine (DAPCuPc)

To study the thermal properties of the resulting pigment, thermal analysis was carried out, the result of the derivatorgaf is shown in Figure 3

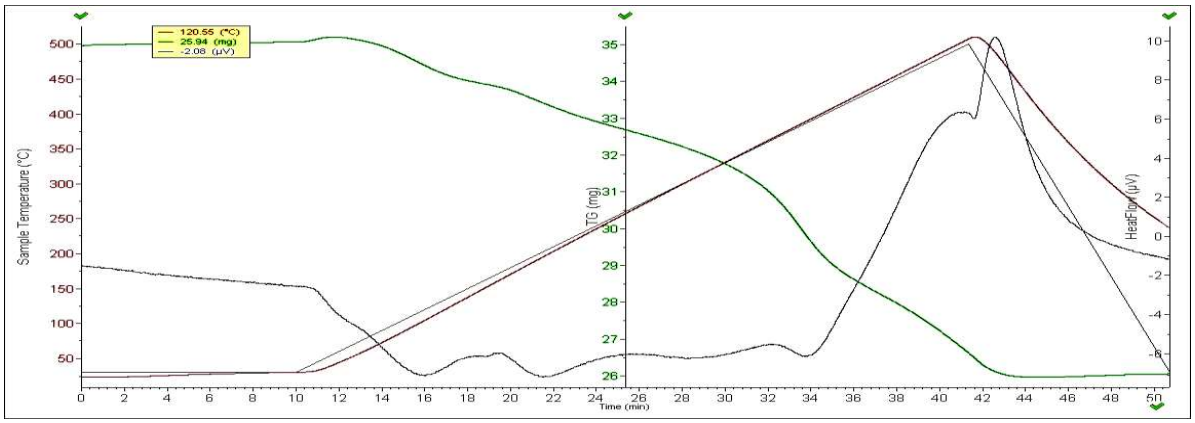

Figure 3. Derivatogram of the DAP-CuPc pigment

For the DAPCuPc pigment, a temperature above 500 °C was chosen, and as a result of pigment analysis, four endothermic effects were observed at temperatures of 30, 45, 51, 375 °C and three exothermic effects at 53, 390, 510 °C, the mass of the DAPCuPc pigment decreases due to residual moisture and adsorbed water of the internal structure of the complex. A subsequent mass loss was observed at 300 °C, the initial mass was 35 mg, and the amount of energy consumed at this temperature was 5.05 μV·s/mg. The decrease in mass is associated with the decomposition of nitrogen derivatives in the DAPCuPc pigment and carbon-bound compounds in the ring. Thermal analysis was carried out up to 500 °C and the total mass loss was calculated for each temperature separately. The DAPCuPc pigment retains a residual weight of 26,5 mg due to molecules that form a bond with phosphorus, and the weight of the pigment is reduced by 9.5 mg from the total weight obtained by keeping it at this high temperature for 37 minutes. According to the results of the analysis, the newly synthesized DAPCuPc pigment has the form of randomly arranged particles; amorphous. Table 1 shows a comparison of the thermal analysis of the DAPCuPc pigment and the copper phthalocyanine pigment.

Table 1

Comparative thermal analysis of the DAPCuPc pigment and the copper phthalocyanine pigment obtained as a control

№	Temperature °C	Residual mass, mg	Lost weight, mg	Lost weight, %	Power consumption (μV*s/mg)
		received 35 mg of pigment DAP-CuPc with a total mass			
1	100	34.4	0.6	1.71	2.6
2	200	33.1	1.9	5.41	3.38
3	300	31.0	4	11.4	5.89
4	400	28.2	6.8	19.4	4.02
5	500	26.5	8.5	24.2	6.18
		received 24 mg of copper phthalocyanine pigment with a total mass			
1	100	23.8	0.2	0.83	2.45
2	200	20.6	3.4	14.2	1.91
3	300	15.4	8.4	35.0	3.09
4	400	9.8	14.2	59.2	4.08
5	500	5.1	18.9	78.8	5.93

The results of this derivatogram show that the main mass loss of the synthesized DAPCuPc pigment occurs in the range of 150-470 °C. And in the range of 50-150°C, the mass loss of the DAPCuPc pigment is negligible.

4. Conclusion. A new type of pigment, copper diphosphate phthalocyanine (DAPCuPc), was synthesized in two ways: in the liquid phase in the presence of dimethyl sulfoxide and in the solid phase by heating the components at a high temperature. The pigment yield during synthesis in a liquid medium was 93%, but its intensity turned out to be low, and under the conditions of synthesis in the solid phase, a high pigment intensity was achieved at its 80% yield. The physicochemical properties of the new pigment synthesized in various aggregative states are compared with those of the closest analogue, copper phthalocyanine. The high thermal stability of the new pigment was discovered and possible areas of its practical application in the national economy were proposed.

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