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CHLORINATION OF PHTHALIC ANHYDRIDE

Vohid Akhmedov

Professor of the Bukhara Institute of Engineering and Technology, Uzbekistan, Bukhara E-mail: <u>chemistry2927@mail.ru</u>

Zukhriddin Rayimov

Docent of the Bukhara Institute of Engineering and Technology, Uzbekistan, Bukhara

Farkhod Kurbanov

Docent of the Bukhara Institute of Engineering and Technology, Uzbekistan, Bukhara

ХЛОРИРОВАНИЕ ФТАЛЕВОГО АНГИДРИДА

Ахмедов Вохид Низомович

профессор, Бухарского инженерно-технологического института, Республика Узбекистан, г. Бухара

Райимов Зухриддин Хайриддин угли

доцент, Бухарского инженерно-технологического института, Республика Узбекистан, г. Бухара

Курбанов Фарход Пулатович

стажёр Бухарского инженерно-технологического института, Республика Узбекистан, г. Бухара

ABSTRACT

Currently, scientific research is being conducted to create a new generation of additional reagents used in oil and gas processing, as well as to improve production technology. In order to prevent wear and tear of chemical industry equipment and ensure the stability of its operation, special attention is paid to the production of competitive alternative products containing halogen-preserving organic compounds and meeting regulatory requirements for quality indicators. This article presents the results of research on obtaining a chlorine derivative of phthalic anhydride.

АННОТАЦИЯ

В настоящее время проводятся научные исследования по созданию нового поколения дополнительных реагентов, используемых при переработке нефти и газа, а также по совершенствованию технологии производства. В целях предотвращения износа оборудования химической промышленности и обеспечения устойчивости его работы особое внимание уделяется производству конкурентоспособной альтернативной продукции, содержащей галогенсохраняющие органические соединения и отвечающей нормативным требованиям по показателям качества. В данной статье представлены результаты исследований по получению хлорпроизводного фталевого ангидрида.

Keywords: halogenation, solvent, dimethyl sulfoxide (DMSO), dimethylformamide (DMFA), phthalic anhydride, chlorination, catalytic system

Ключевые слова: галогенирование, растворитель, диметилсульфоксид (ДМСО), диметилформамид (ДМФА), фталевый ангидрид, хлорирование, каталитическая система

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Introduction. Since the coupling reaction of halogens to acid anhydrides has not been studied, it is important to obtain halogenated derivatives of these anhydrides. Such substances occupy an important place in the chemical industry and agriculture. The synthesis of halogenated derivatives of phthalic anhydride using solvent (DMSO) was investigated in our research. From the analysis of the studied literature, it can be seen that the solvent dissolves well in DMSO. This makes it possible to increase the yield of the chlorinated derivative of phthalic anhydride [1-3].

Phthalic anhydride is a weak acid. When phthalic anhydride reacts with chlorine gas under high pressure, a chlorine compound of phthalic anhydride is formed. As part of the research, the following experiments were carried out:

1. Chlorine gas was synthesized from hydrochloric acid.

2. Chlorine gas was passed through a solution of phthalic anhydride and dimethylsulfoxide (DMSO).

As part of the research, the halogenating agent chlorine was synthesized from hydrochloric acid. For this, potassium permanganate was treated with hydrochloric acid. The reaction proceeds as follows[4-6]:

$$2\text{KMnO}_4 + 16\text{HCl} \longrightarrow 2\text{KCl} + 2\text{MnCl}_2 + 5\text{Cl}_2 + 8\text{H}_2\text{O}$$

Materials and methods. The laboratory device for obtaining chlorine gas consists of a round bottom flask with two necks and a separating funnel installed in it (Fig. 1). First, 20 g of potassium permanganate was placed in a round-bottom flask with two necks. 30 ml of hydrochloric acid was added to the separatory funnel installed in the flask. The separator was filled drop wise with hydrochloric acid using a funnel separator. At this time, chlorine gas began to be released. The generated

chlorine was directed to the next reaction stage using a gas discharge nozzle. The resulting chlorine gas was passed through a catalytic system consisting of phthalic anhydride, DMSO and AlCl₃. The introduction of a halogen atom into the aromatic ring is based on the Friedel-Crafts reaction, and the process proceeds in the electrophilic exchange mechanism. AlCl₃ acts as a catalyst in this reaction.



1-Double-necked round bottom flask, 2-Separating funnel, 3-Stand, 4-Gas outlet nozzle

Figure 1. Chlorine gas extraction laboratory device

As a result of the reaction of the addition of a chlorine atom to phthalic anhydride, it was found that

a monochlorinated compound of phthalic anhydride is formed. The reaction can be summarized as follows:



The interaction of phthalic anhydride and chlorine atom takes place in a heterogeneous system. The procedure was carried out in a laboratory device consisting of a specially equipped glass jar (Fig. 2). This glass is equipped with a thermometer (7), a gas distributor (6) and nozzles (8) that evenly distribute chlorine gas in the form of small bubbles on the surface in order to increase the contact surface of the gas and liquid system.



1-Round-bottomed two-necked flask, 2-Separating funnel, 3-Separating funnel, 4-Gas transfer nozzle, 5-Beaker, 6-Gas distributor, 7-Thermometer, 8-Nozzle, 9-Gas discharge nozzle, 10-Electric oven

Figure 2. A laboratory device for obtaining a monochlorinated compound of phthalic anhydride

An electric heater (10) is installed under the glass. A large number of small gas bubbles supplied through the gas distributor also perform the function of forced convection of the heat coming from the heater and mixing of the multi-component system. The liquid reaction system was prepared as follows: 100 ml of DMSO and 7.5 g of AlCl₃ were added to a 300 ml conical flask and the mixture was stirred while heating at 40-45 °C. After complete dissolution of AlCl₃, 75 g of phthalic anhydride was added to the flask and stirred. After a homogeneous system was formed in the flask, the mixture was cooled to a temperature of 25 °C and placed in a glass (5). After that, in order to carry out the halogenation reaction, the liquid in the glass was heated to a temperature of 80 °C using an electric heater (10).

Chlorine gas synthesized in a round-bottomed twonecked flask (1) was transferred from the lower part of the 5th glass through the 6th gas distributor using the 4th gas transfer nozzle. After 3 hours, the synthesis process was stopped and the substances were cooled.

Results and discussion. The synthesized substance was analyzed by IR-spectroscopy. In order to identify the functional groups in 5-chloroisobenzofuran-1,3-dione, it was determined using IR spectroscopic analysis method. The following absorption areas can be seen in the IR spectrum of 5-chloroisobenzofuran-1,3-dione (Fig. 3).

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Figure 3. Results

1703.17 cm⁻¹ -C=O carbonyl group valence vibrations, 1254.44 cm⁻¹ carboxyl group valence vibrations, and 3003.48 symmetric vibrations for the C–H bond in the aromatic ring, 1254.44; Corresponds to deformational, symmetric vibrations of the chlorine bond in the aromatic ring in the 1131.14 and 1002.12 cm⁻¹ fields. This means that it corresponds to the composition of the synthesized substance.

It is known that solvents are important in the course of chemical reactions. In this case, factors such as properties and nature of the solvent affect the yield of the product formed during the reaction. The reaction of chlorine gas with phthalic anhydride was carried out in the presence of various solvents. Solvents were analyzed depending on the yield of 5-chloroisobenzofuran-1,3dione. Reactions were carried out for 3 hours. In the reactions, the catalyst AlCl₃ was 10% by mass of phthalic anhydride, and the temperature was 80 °C.

In our research, we found that DMSO is the most important solvent. In our experiment, when DMSO was used as a solvent, the product yield was 40.3%. In order to study the effect of the nature of different solvents on the chemical reaction, it was 37% in the presence of DMFA at a temperature of 25 °C. At the same time, it was 20% in water. Among the solvents used in the synthesis process, it was observed that the addition of chlorine gas to phthalic anhydride is more active than other solvents in DMSO.

When the temperature of the reactor in which the reaction is progressing is increased, the speed of movement of the particles of the reacting substances increases, and this situation increases the number of collisions between the particles. As a result, it causes an increase in product yield. Also, decomposition of organic substances is observed at extremely high temperatures. Therefore, it is important to find the optimal temperature value of the reaction under investigation. Taking this into account, the effect of temperature on the exchange reaction of phthalic anhydride with chlorine gas was studied. Catalyst AlCl3 and solvent DMSO were used in the reaction. The temperature was carried out between 30-100°C to obtain the monochloro derivative of the product phthalic anhydride. The obtained results were recorded in Table 1.

Table 1.

The effect of temperature on the reaction of phthalic anhydride with chlorine gas, the duration of the reaction is 3.5 hours

N⁰	Temperature, °C	Yield, %
1	30	12,4
2	40	18,6
3	50	24,3
4	60	29,8
5	70	35,7
6	80	40,3
7	90	36,8
8	100	29,6

As can be seen from Table 1, the reaction was carried out in 3.5 hours and the yield of 5-chloroisobenzofuran-1,3-dione was determined. The product yield was 12.4% when the reaction was carried out at 30°C. The maximum product yield was 40.3% when the reaction was carried out at 80°C. Product yield decreased when the temperature was raised above 80°C. This situation can be explained by the reduction of chlorine solubility, oxidation of phthalic anhydride or its destructive change.

Based on the data from the research results, a graph of temperature dependence of the yield of 2-5-chloroisobenzofuran-1,3-dione was drawn (Graph 1).





Figure. Temperature dependence of the yield of 5-chloroisobenzofuran-1,3-dione at different concentrations of the catalyst

Conclusion. In conclusion, it can be said that the solvent DMSO, the catalyst AlCl3, the reaction temperature 80°C and the duration of the reaction 3.5 hours were the solvent DMSO and the product yield was 40.3%. Based on the results of the conducted research, it was

determined that the yield of 5-chloroisobenzofuran-1,3-dione has the highest value when the duration of the reaction is 3.5 hours. This phenomenon is explained by the saturation of a certain volume of DMSO solvent in 3.5 hours.

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