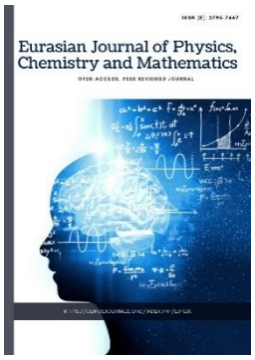

Synthesis Technology and Physicochemical Properties of Bis-Carbamate Mee-1

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ABSTRACT

In this research work, a new bis-carbamate was synthesized on the basis of cresol, a reaction mechanism was proposed, which is produced with raw materials, energy-efficient, environmentally friendly, waste-free and with high efficiency, and theoretical and practical elemental analyzes were conducted. According to the results, it was proved that the elements in the bis-carbamate molecule are actually present in the molecule and the theoretically calculated atomic shares are close to the practically determined results.

Keywords: Bis-carbamate, cresol, dimethylformamide, hexamethylene diisocyanate, synthesis, reaction, mechanism, molecule, elements, microscope.

Introduction. Currently, bis-carbamates are of great interest to scientists as substances with various biological activities. They are used as anesthetics, anticholinesterase agents, preservatives and insecticides, selective herbicides, plant growth regulators, lubricants, semi-products in the synthesis of oligomers, and various polyurethane coatings [1]. In addition, one of the methods of separation of carbon isotopes is the carbamate process. This method is based on the formation of amine carbamate and proceeds with the formation of intermediate products such as carbamic acids as a result of the interaction of carbon dioxide with a primary or secondary amine [2]. You can see how important it is to synthesize bis-carbamates and obtain new derivatives based on the above principles. In particular, the authors obtained various bis-carbamates and their derivatives based on hexamethylenediisocyanate and used them as biostimulants for plant growth [3-5]. Several bis-carbamates were synthesized by the authors based on hexamethylenediisocyanate and cresols [6]. Several spectral analyzes of these substances have been conducted [7]. Their acute toxicity has been studied [8]. In general, the aim of the work is to develop a soda method for the synthesis of N,N′-hexamethylene bis-[(ortho-cresolyl)-carbamate], i.e., MEE-1 bis-carbamate, to make a laboratory device, and to perform elemental analysis as the first analysis, consists of transferring.

Materials and Methods. 10 ml of triethylamine (TEA), 35 ml of dimethylformamide (DMF) are added to 10.8 g (0.1 mol) of ortho-cresol, mixed, dissolved in 20 ml of DMF at room temperature 8.4 g (0.05 mol) of hexamethylene diisocyanate (HMDI) is added. After time, the reaction mixture is stirred at 35-45 ºC for 3.0-4.0 hours,

the contents of the flask are transferred to a glass, and water is added. The resulting precipitate is checked by thin layer chromatography (TLC). After drying, a white powder is obtained; the yield of the product is 18.74 g (97.6% of theory).

Results and Discussions. The technology for the synthesis of MEE-1 in laboratory conditions was developed and assembled by the authors in the laboratory of the "Chemical Technology of Petroleum Refining" department (Fig. 1).

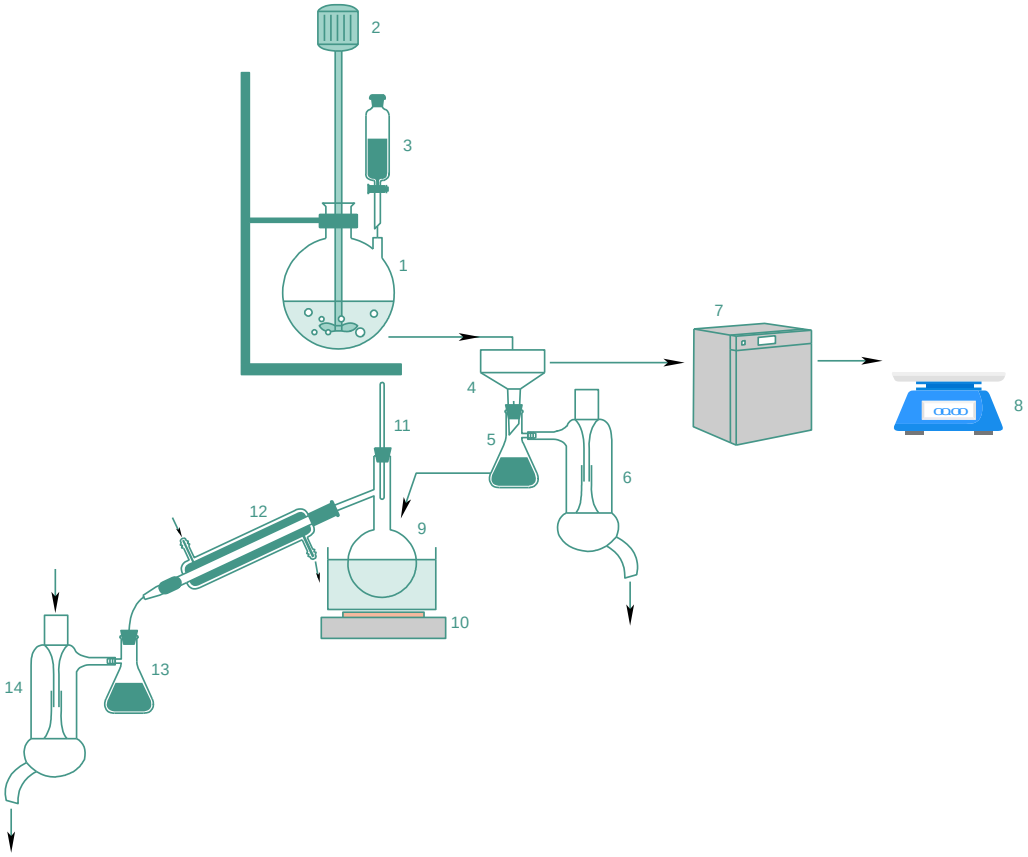


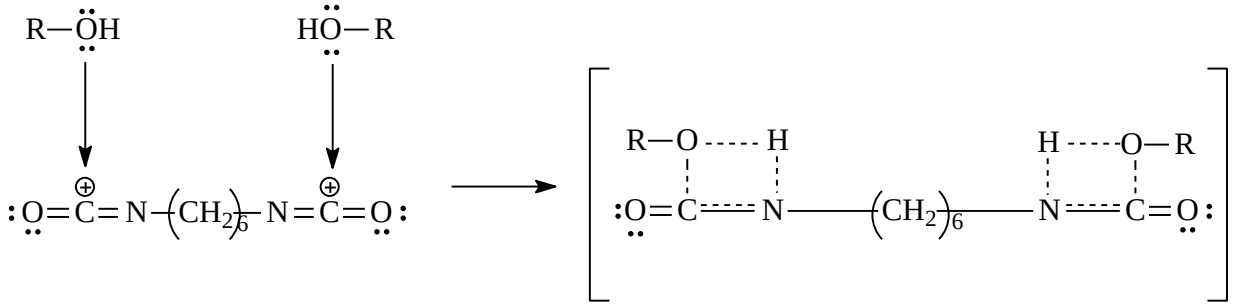
Figure 1. A device for synthesizing MEE-1 in a laboratory

Note 4.: 1- Reactor; 2- electric mixer; 3- separating funnel; 4- Buchner's funnel; 5.13- Bunsen flask; 6.14- vacuum pump; 7- drying cabinet; 8- scales; 9-Wurtz flask; 10- electric plate; 11- thermometer; 12- cooler.

The synthesis process is carried out as follows: 10.8 g (0.1 mol) of ortho-cresol, 10 ml of triethylamine (TEA) catalyst, 35 ml of dimethylformamide (DMF) solvent are loaded into reactor 1 and stirred in mixer 2, and 8.4 dissolved in 20 ml of DMF are passed through separatory funnel 3 g (0.05mol) of hexamethylene diisocyanate (HMDI) was added dropwise and stirred at 35-40 ºC for 4.0 hours. After the mixing is stopped, the mixture is cooled and precipitated. We filter the resulting sediment in a notch filter equipped with 4-Buchner funnel and 5-Bunsen flask for receiving filtrate. To do this, soak the filter paper in methanol alcohol and place it in the funnel. To create a vacuum during the filtration process, we connect the Bunsen flask to the 6th water flow pump. The liquid part of the filtrate is collected in the 5th Bunsen flask. We wash the precipitate remaining in the filter 3-4 times with methanol. The washed wet precipitate is dried in the 7th drying cabinet at a temperature 5-10 ºC higher than room temperature until the moisture content of the precipitate does not exceed 2%. The liquid part of the filtrate from the 5th Bunsen flask is loaded into the 9th Wurtz flask. The Wurtz flask is heated in a water bath to a temperature of 100-150 ºC on a 10-electric plate. In this case, after the TEA catalyst with a boiling point of 89.5 ºC is distilled, a vacuum is

created through the 14th water flow pump. 35 mm s.u. at residual pressure and temperature, the DMF solvent is distilled using condenser 12 at 150 ºC. At the end of the work, the finished product in the form of a white powder was measured on the 8th scale, in which 18.74 g (97.6% of the theoretical) was obtained.

Based on all studies, in our reactions, the hydroxyl group of cresol, which has kept oxygen, attacks the electrophilic center of hexamethylene diisocyanate and forms a transition state, as it has a free pair of electrons, and then regroups into the final product of the reaction. The possible mechanism of the reaction of cresols with hexamethylene diisocyanates can be imagined as follows:



N,N'- geksametilen bis - [(orto - krezolilo) – karbamat]

After that, some physicochemical parameters of the resulting N,N′-hexamethylene bis-[(ortho-cresolyl)-carbamate] i.e. MEE-1 bis-carbamate were studied (Table 1) and elemental analysis was carried out (Tables 2, 3).

Table 1
Properties of MEE-1 bis-carbamate

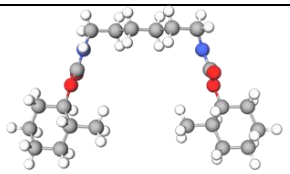
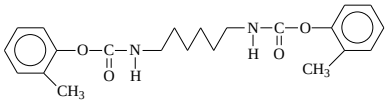
Systematic name	N,N′-hexamethylene bis-[(ortho-cresolyl)-carbamate]	 3D structure of MEE-1
Brutto formula	$C_{22}H_{28}N_2O_4$	
Classification	Organic matter	
Molar mass M_m	384,47	 Structure of MEE-1
Liquefaction temperature ºC	127-129	
R_f value	0,75	

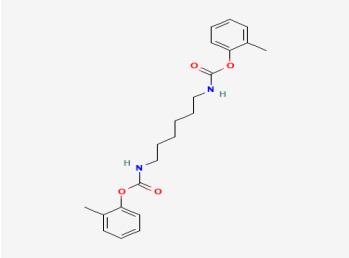
Table 2
Theoretical and practical elemental analysis of MEE-1 bis-carbamate

Element analysis,%					
Calculated		Found		SEM analysis	
C	68,75	C	68,63	C	74,15
H	7,29	H	7,17	O	17,98
N	7,29	N	7,19	N	7,87

An EVO MA 10 scanning electron microscope (SEM) was used to determine the elemental composition of MEE-1 bis-carbamate under vacuum conditions. When comparing the theoretical, practical and calculated results using computer programs and the SEM results, you can see that the results are close to each other (Tables 2, 3).

Table 3

Computer program calculations of MEE-1 bis-carbamate



Compound MEE-1

Formula			$C_{22}H_{28}N_2O_4$
Molecular mass			384.5 u
The number of donors of the hydrogen bond			2
The number of hydrogen bond acceptors			4
Percentage composition			
C	12.0107 u × 22		68.727 %
H	1.00794 u × 28		7.3406 %
N	14.0067 u × 2		7.2863 %
O	15.9994 u × 4		16.646 %

Conclusion. The bis-carbamate synthesis method presented in the scientific work is included in the isocyanate extraction method. It was found that this method is resource- and energy-efficient, environmentally friendly, waste-free, and produces products with high productivity. The elemental analysis of the synthesized MEE-1 bis-carbamate showed that the elements in the bis-carbamate molecule are actually present in the molecule and that other elements are not mixed and obtained in a pure state

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