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Preparation of isocyanates by carbamates thermolysis on the example of butyl isocyanate

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Abstract

Butyl isocyanate is widely distributed as a precursor for the production of a number of biologically active substances: fungicides, preservatives, insecticides, personal care products, etc. Nowadays, there are a number of methods for the preparation of isocyanates, which can be divided into liquid phase and gas phase. One of the perspective methods for the production of isocyanates is the thermolysis of carbamate and/or the actions of various reaction activating agents, accompanied by the elimination of alcohol, but this process is reversible, which greatly complicates its use in industry. The paper presents the results of studies of noncatalytic thermal decomposition of N-alkylcarbamates with the formation of alkylisocyanates on the example of butylisocyanate in the gas phase, flow reactor in a wide temperature range (200 to 450 °C). In addition, a series of experiments was carried out using a catalyst, dibutyltin dilaurate, in order to reduce the thermolysis temperature and increase the yield of the final product. To implement the isocyanate production process, an experimental laboratory setup, consisting of a gas flow meter (argon) regulator, a packed column (for heating) and a sorption solution tank, was developed and tested. The thermolysis of N-n-butylcarbamate was carried out in two variations: the preparation of an individual *n*-butylisocyanate and the passage of reaction products through a sorption solution linking the *n*-butyl isocyanate to *N*-*n*-butyl-*N* '-(1-phenylethyl)urea, which allows to estimate the yield of the target *n*-butylisocyanate without additional losses. The analysis of the obtained substances was carried out by high performance liquid chromatography with a UV detector (target product) and a mass detector (analysis of by-products). According to the results of research, a modification of the laboratory facility was proposed, as well as *n*-butylisocyanate was obtained with a yield of 49% on the basis of a new technique.

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