

CONTINUOUS PROCESS FOR THE PREPARATION OF ETHYL FORMATE

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Ethyl formate is used as an intermediate product in the synthesis of vitamin B₁ [1], as well as in the production of several other organic compounds.

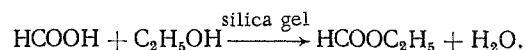
Several methods for the preparation of ethyl formate are known, including the esterification of ethyl alcohol and formic acid in the presence of sulfuric acid [2].

Disadvantages of this method include the use of sulfuric acid, which causes severe corrosion of equipment of utilities, and also the impossibility of regenerating this acid from still residues.

A method has been elaborated at the All-Union Vitamins Scientific-Research Institute, and the Belgorod Vitamins Combine for the preparation of ethyl formate by a continuous process, where a type KSK silica gel with a bulk density of 0.55-0.6 g/cm³ is used as catalyst. The production of ethyl formate by this method was carried out in an industrial plant of the Belgorod Vitamins Combine.

The process proceeded according to the flow diagram shown below (Fig. 1).

A mixture of formic acid and ethyl alcohol prepared in the ratio of 1:1.48 is continuously fed from the storage tank (1) through a pump with an attached flow meter (2) at the rate of 64 liters/h into the reactor (3) filled with silica gel, where the esterification takes place at a temperature of 68-70°:



From the reactor (3), the mixture of vapors of ethyl formate, ethyl alcohol, water, and formic acid is passed to a column for a rectification (4).

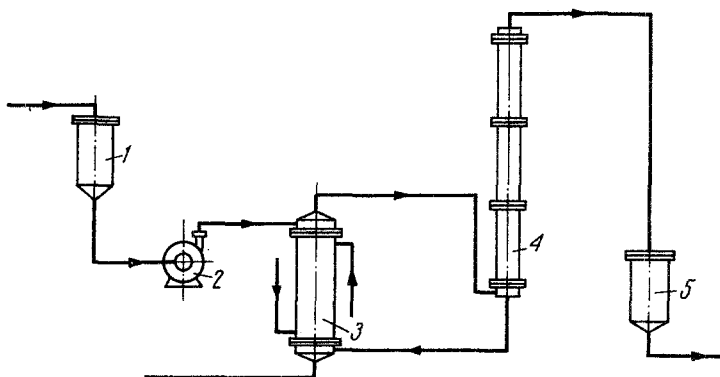


Fig. 1. Flow diagram of a continuous process for the preparation of ethyl formate: 1) Storage tank with a mixture of C₂H₅OH and HCOOH; 2) a pump with a flow meter attached; 3) reactor; 4) rectification column; 5) receiving tank for ethyl formate.

The silica gel used in the esterification process is regenerated by a thermal treatment at a temperature of 100–105° until a bulk density of 0.55–0.6 g/cm³ is attained, and is returned to the production cycle.

The ethyl formate obtained conforms to the requirements of the synthesis of vitamin B₁. Its yield is 90–94%, the content of the main substance being 90–95%.

LITERATURE CITED

1. V. M. Berezovskii, Vitamin Chemistry [in Russian], Moscow (1959).
2. V. M. Tursin, and N. D. Kolotilova, Vitamins [in Russian], Moscow (1959), p. 31.