METHOD OF QUANTITATIVE HPLC ANALYSIS IN INVESTIGATION OF CYCLOCONDENSATION OF 2-PYRROLIDONE WITH ANTHRANILIC ACID

Kh. R. Nuriddinov, K. Sargazakov, Sh. Abdullaev, and Kh. N. Aripov UDC 547.856:543(062).544

The products of the cyclocondensation of 2-pyrrolidone with anthranilic acid in the presence of condensing agents have been investigated by high-performance liquid chromatography (HPLC). The main product of this reaction is the alkaloid deoxyvasicinone (DOV) [1]. To analyze the starting materials and the reaction products we used reversed-phase HPLC with the UV detection of the separated substances at a wavelength of 254 nm [2]. The HPLC method has not previously been used for studying the products of this reaction.

Chromatography was performed by isocratic elution of the substances with an eluent consisting of methanol or acetonitrile and water in a ratio of 80:20 in a 3.3×150 mm glass column filled with Separon SGXC₁₈ (7 µm) (Czechoslovakia). The ratio of organic solvent and water was selected experimentally. HPP 4001 high-pressure pump; LCD 2563 UV detector; TZ4620 recorder (Czechoslovakia). The methanol and acetonitrile were purified by fractional distillation. Double-distilled water was used. The eluent after preparation was filtered through a filter with 1-4 µm pores and was degassed with a current of helium.

Elution was carried out at a rate of feed of eluent by the pump of 0.2 ml/min. The initial reagents 2-pyrrolidone and anthranilic acid and the products of the cyclocondensation of 2-pyrrolidone with anthranilic acid in the presence of the condensing agents PCl_3 and $SOCl_2$ at various ratios of the reagents were analyzed by this method. In this way, the technical 2-pyrrolidone was shown to contain 3-5% of vinylpyrrolidone. Figures 1a and



Fig. 1. Chromatograms of reaction mixtures: I) anthranilic acid; II) 2-pyrrolidone; III) DOV; IV) unknown substance. Detection in the 0.32 o.u. interval. The DOV peak was recorded in the 3.2 o.u. interval. In b) peak IV was recorded in 1.6 o.u. interval.

Institute of the Chemistry of Plant Substances, Uzbek SSR Academy of Sciences, Tashkent. Translated from Khimiya Prirodnykh Soedinenii, No. 2, pp. 293-294, March-April, 1989. Original article submitted July 8, 1988. b show, respectively, the chromatograms of the HPLC analysis of the reaction mixtures for the cyclocondensation of 2-pyrrolidone with anthranilic acid in the presence of PCl_3 and $SOCl_2$.

For quantitative analysis we used the method of area normalization with a calibration factor. To find the calibration factors we determined the molar extinctions of UV absorption at a wavelength of 254 nm for the standard substances 2-pyrrolidone, anthranilic acid and DOV. In this way the calibration factors for 2-pyrrolidone and anthranilic acid relative to DOV were found: $K_{2-pyrr} = 28.79$ and $K_{anth.ac.} = 0.57$. The calibration factors for the other, unknown, substances, were taken as unity, as for DOV.

As the result of our study of the cyclocondensation of 2-pyrrolidone with anthranilic acid by HPLC methods we have determined the optimum conditions for the reaction and the optimum ratio of the reactants for the highest yield of the desired product -DOV.

Thus, the HPLC method permits an effective and quantitative study of the reactions of organic synthesis and, in particular, the reaction for producing the alkaloid deoxyvasicinone.

LITERATURE CITED

1. S. Yu. Yunusov, Alkaloids [in Russian], Fan, Tashkent (1974).

2. E. L. Styskin, L. B. Itsikson, and E. V. Braude, Practical High-Performance Liquid Chromatography [in Russian], Khimiya, Moscow (1986).