REACTION OF 2-AMINOBENZOTHIAZOLE WITH FORMAMIDE

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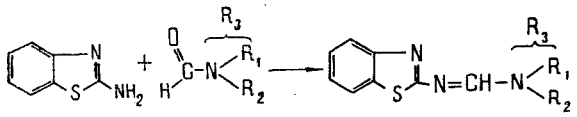
UDC 547.53+547.789+542.91

Many benzothiazole derivatives exhibit a fairly high biological activity, which has permitted their use in practice [1].

The possibility has been shown previously of synthesizing benzimidazolylformamidine from methyl benzimidazol-2-ylcarbamate and dimethylformamide [2]. The required benzimidazolylformamidine has also been obtained by the reaction of 2-aminobenzimidazole with dimethylformamide in the presence of $POCl_3$, $SOCl_2$, PCl_5, and PCl_3.

In order to find a synthesis of potential pesticides, we have studied the reaction of 2-aminobenzothiazole (2-ABT) with some formamides in the presence of phosphorus oxychloride or thionyl chloride in accordance with the scheme given below



The reaction was performed in an excess of formamide at a ratio of 2 ABT to formamide to $POCl_3$ of 1:5:2 for 2 h.

TABLE 1. Benzothiazol-2-ylformamidines

R_1	R_2	Yield, %	mp, °C*	R_f†	Mol. wt. (mass spectrum)
CH_3	CH_3	30	93—94	0,516	205
C_2H_5	C_2H_5	24	115—116	0,481	233
C_4H_9	C_4H_9	13,8	239—240	0,472	289
C_6H_5	C_6H_5	30	70—71	0,507	329
CH_3	C_6H_5	26	137—138	0,504	267
C_2H_5	C_6H_5	15	222—225	0,45	281
R_3					
		14	108—109	0,561	230
		35	63—65	0,623	245
		30	240—241	0,53	247

*All the compounds were recrystallized from dilute ethanol.
†TLC: Silufol UV-254; benzene—acetone (4:3) system; visualizing agent: a mixture of equal volumes of 5% $NaHCO_3$ and 1% $KMnO_4$ solutions.

Institute of the Chemistry of Plant Substances, Academy of Sciences of the Uzbek SSR, Tashkent. Translated from Khimiya Prirodnykh Soedinenii, No. 6, pp. 792-793, November-December, 1982. Original article submitted June 11, 1982.

 0009-3130/82/1806-0762$07.50

The IR spectrum of the compounds obtained showed strong bands in the 3400—3380 cm^{-1} region corresponding to the stretching vibrations of the N=CH group of the side chain, and also absorption bands at 3060—3080 cm^{-1}, corresponding to the stretching vibrations of CH groups. The fragmentation in the mass spectra corresponded to the structures of the compoun obtained. The breakdown of the molecular ions took place similarly to that of the molecular ion of 2-(dimethylaminomethylideneamino)benzimidazole [2]. The elementary analyses of the compounds obtained corresponded to the calculated figures.

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PURIFICATION OF UBIQUINONE

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UDC 5435—48:577.15

The process of obtaining ubiquinone (coenzyme Q_9) consists in saponifying a microbial fat with hot ethanolic alkali, extracting the unsaponifiable lipids, and freeing the extracts from paraffins by column chromatography. At the present time, in purification with the aid of preparative chromatography alumina is used as the adsorbent, but the high activity of this sorbent leads to losses of ubiquinone.

We propose a method for purifying ubiquinone using preparative liquid chromatography in a column filled with silica gel. The absorption of the pure substance is in the 250-280 nm region, which permits it to be revealed with the aid of a UV detector. The column was filled by the suspension method under a pressure of 300 atm with type LG (5/40) silica gel. As the eluting liquid we used a solvent system consisting of hexane and diethyl ether. A product containing 60% of pure ubiquinone was deposited on the column.

In order to search for the optimum conditions of performing the purification process we used the experimental-planning procedure. As the result of a statistical analysis, a system of regression equations adequately describing the process of isolating the pure substance was obtained:

$$K = -32{,}8 - 29{,}3\ V^2 + 62{,}5\ V + 3{,}2\ P - 0{,}0048\ S^2 + 0{,}22\ S - 3{,}4\ VP,\quad \tau = 31{,}8 + 26{,}3\ V^2 - 51{,}6V + 0{,}51\ P^2 - 2{,}3\ P - 0{,}063\ S.$$

where K is the separation factor of the main substance and the impurity; S is the polarity of the solvent; V is the velocity of movement of the eluent; P is the charge on the column; and τ is the time of performing the process.

The equations found showed that the maximum yield of ubiquinone can be obtained at a rate of elution of 1 ml/min in a system consisting of n-hexane and diethyl ether (5:2).

The annual demand for ubiquinone is about 300 kg. On working under the optimum conditions using a separating column with dimensions of 10-30 cm it is possible to isolate 100 kg of product with a purity of more than 97%, which satisfies the requirements of the technical conditions.

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Technological Institute of the Food Industry, Moscow. Translated from Khimiya Prirodnykh Soedineii, No. 6, p. 793, November-December, 1982. Original article submitted June 17, 1982.

0009-3130/82/1806-0763$07.50