

IN REACTIONS OF CONDENSED HETEROCYCLIC COMPOUNDS OF ZUCCHINI-FELDS

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ABSTRACT

In an article from 2-aminobenzoic acid and pyrrolidone-2 in the presence of various substances, for example, PCl_5 , POCl_3 , 2,3-trimethylene-3,4-dihydrobenzo[2,3-d]pyrimidin-4-one was synthesized. Its reduction reaction with NaBH_4 was carried out. The resulting 2,3-trimethylene-1,2,3,4-tetrahydrobenzo[2,3-d]pyrimidine-4-one - a three-component combination of carbonyl, amine and hydrophosphoryl compound leads to α -aminophosphonates, phosphoric acid-formaldehyde; Reactions with aldehydes in a three-component system have been studied.

Keywords: 2,3-trimethylene-3,4-dihydrobenzo[2,3-d]pyrimidin-4-one, 2,3-trimethylene-1,2,3,4-tetrahydrobenzo[2,3d]pyrimidin-4-one, 2-aminobenzoic acid, pyrrolidone-2, synthesis reactions.

INTRODUCTION

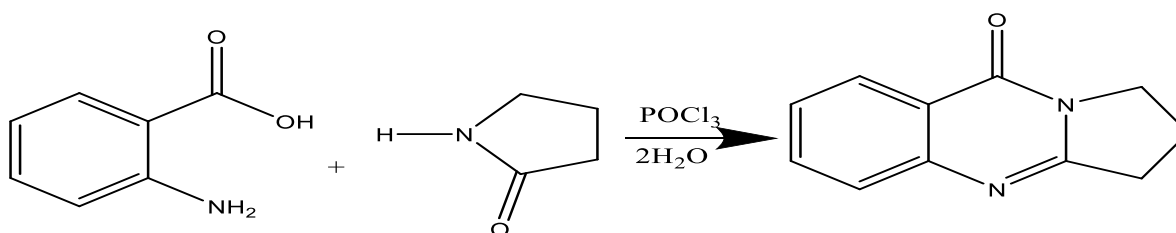
In the world, the synthesis of new physiologically active derivatives of benzo[2,3-d]pyrimidine and the creation of modern drugs on their basis is carried out using high technologies. It is known that the anti-cancer drugs used so far destroy dangerous cancer cells while damaging healthy cells.

Representatives of benzo[2,3-d]pyrimidines, preferred polycyclic anticancer drugs, pyrimidine and pyrimic acids with antibacterial effects, have been developed by world scientists. Medical institutions have a practical interest in the properties of this substance.

Organophosphorus compounds are ubiquitous in nature and are used in agriculture, medicine and industry [1-3]. Some organophosphorus compounds are important pesticides [4], bactericides [5-7] and antibiotics [5]. Phosphorus analogues of α -pyrones act as HIV protease inhibitors [8]. α -Aminophosphate acids are important motifs among organophosphorus compounds in medicinal chemistry due to their clear structural similarity to α -amino acids [9-16].

MATERIALS AND METHODS

Tricyclic 2,3-tremethylenebenzo[2,3-d]pyrimidin-4-one (1) was synthesized from 2-aminobenzoic (anthranilic) acid, which was condensed with pyrrolidone-2. In the presence of POCl_3 .



Aminobenzoic (anthranilic) and 2-aminopyridine-3-carboxylic acids of the lactam series with 2-aminofen-3-carbonic, 2- reduce the yield of the condensation product. So, if 2,3-trimethylenethieno[2,3-d]-pyridine-4-one was obtained with a yield of 81%, and 2,3-polymethylene-quinazolone with a yield of 56-70%, then in 2 cases 2,3-trimethylenebenzo-[2,3-d]pyrimidin-4-one (1) it was 74%. This fact is explained by the fact that the leakage of p-electrons is facilitated by an excess of the thiophene ring, and the p-deficient benzene ring enhances this condensation.

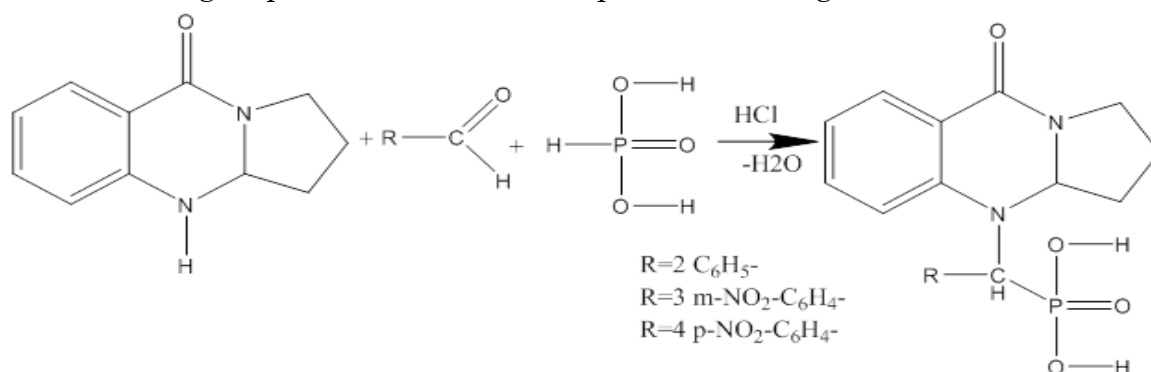
Obviously, the reconnection of N1 = C2 in HN-CH leads to a significant shift in the chemical shifts of the protons of methylene groups towards strong fields. A significant shift in the proton signals of the pyridine ring after recovery was also found. So, at the initial stage, they appear in ppm: 7.29 (1H, dd, J = 4.5, 7.9, C6-H), 8.53 (1H, dd, J = 2.0, 7.9, C5-H), 8.89 (1H, dd, J = 2.0, 4.6, C7-H), respectively; and in the reduction product, the same protons are observed in the following regions: 6.74 (1H, dd, J = 5.0; 7.6, C6-H), 8.06 (1H, dd, J = 1.8; 7.4, C5 - H) and 8.10 (1H, dd, J = 1.8; 5.0, C7-H), respectively; That is, there is a shift in chemical changes in protons towards strong fields. The asymmetric carbon proton C2-H was identified at 5.10.

ANALYSIS OF THE RESULTS

Trehicomponent reaction of 2,3-trimethylene-1,2,3,4 with dihydrobenzo[2,3-d]pyrimidin-4-one, p and m-nitro-benzaldehyde and phosphoric acid in preliminary experiments Optimization of reaction conditions was chosen as a model reaction for The procedures used for the synthesis of α -aminophosphonates in this work are presented in Table 1, records 3-5. The product of α -aminophosphonates was obtained by microwave irradiation of aldehyde, amine and phosphoric acid without solvent for 1 min. Toluene, without catalyst, the resulting product was well formed.

Thin-layer chromatography (TLC) was used to control the reaction and determine the purity of the products. The reaction was carried out with a catalytic amount of HCl in toluene for 30 min. All of these compounds are easily soluble in polar organic solvents.

The IR spectra of the compounds (3-5) showed the presence of the N-CH₂ band at 1550 cm⁻¹. The sharp band observed in the range of 1240-1291 cm⁻¹ is due to P=O, and the stretch band P-C appeared in the range of 740-770 cm⁻¹. All stretch frequencies are summarized in Table 2. The NMR spectra of H compounds (3-5) in the solvent 1DMSO-d₆ were recorded. Aromatic protons of α -aminophosphonic acids appeared as a multiplet in the region d 6.15-8.69. The proton of the P-C-H group resonated as a multiplet in the d region of 3.77-4.86.



EXPERIMENTAL PART

Synthesis of 2,3-trimethylene-3,4-dihydrobenzo[2,3-d]pyrimidine-4-one hydrochloride (1). POCl₃ (90 mL) was added to a mixture of 2-aminobenzoic acid (65.3 g, 0.47 mol) and pyrrolidone-2 (47.94 g, 0.564 mol) and stirred at room temperature. The reaction mixture was heated at 80-90°C for 2-3 hours, cooled to room temperature, and 100 mL of water was added. The aqueous solution was treated with a 5% NaOH solution to pH 7.5-8. Extracted with chloroform (3x100 ml), the organic phase was dried by Na₂SO₄, filtered and the solvent was evaporated. The yield of compound 1 was 30.76 g (71%), mp 139-140°C. R_f=48 (10:1 chloroform-methanol): for a solution of 28.05 g (0.15 mol) of 2,3-trimethylene-3,4-dihydrobenzo[2,3-d]pyrimidine-4. (1) 30 g (0.78 mol) of sodium borohydride was added to 350 mL of alcohol. The reaction mixture was boiled in a water bath for 3 hours and left for twenty-four hours. The solvent was removed, the remaining product was dissolved in water and left for 1 hour. The precipitate was filtered, washed 3 times with water, dried and recrystallized from hexane. The yield of compound 2 was 28.35 g (91%), T_s = 136-139°C. R_f = 0.58 (10:1 chloroform-methanol). Synthesis of compound 3. The mixture of paraform (0.005 mol), 2,3-trimethylene-1,2,3,4-tetrahydrobenzo[2,3d]pyrimidine-4-one (0.005 mol) and phosphidic acid in dry toluene was stirred for 1 hour. The temperature was then raised to 80-95 °C for 3 hours. The reaction was controlled with TLC. After completion of the reaction, toluene was removed by distillation and the residue was purified by column chromatography (5:4, benzene:hexane). The yield of compound 3 was 1.124 g (91%), T_s=167-169°C. R_f = 0.4 (5:4, benzene:hexane). Synthesis of compound 4. A mixture of m-nitrobenzaldehyde (0.005 mol), 2,3-trimethylene-1,2,3,4-tetrahydrobenzo[2,3-d]pyrimidine-4-one (0.005 mol) and phosphidic acid was stirred in dry toluene for 10 minutes at room temperature. The temperature was then raised to reflux for 5 h. The reaction was controlled with TLC. After completion of the reaction, toluene was removed by distillation and the residue was purified by column chromatography (5:4, benzene:hexane). The yield of compound 4 was 1.76 g (89%), T_s = 155-158°C. R_f = 0.52 (5: 4, benzene: hexane). Synthesis of compound 5. A mixture of p-nitrobenzaldehyde (0.01 mol), 2,3-trimethylene-1,2,3,4-tetrahydrobenzo[2,3-d]pyrimidine-4-one (0.01 mol) and phosphidic acid was stirred in dry toluene. . for 10 minutes at room temperature. The temperature was then raised to reflux for 5 h. The reaction was controlled with TLC. After completion of the reaction, toluene was removed by distillation and the residue was purified by column chromatography (5:4, benzene:hexane). The yield of compound 5 was 1.78 g (90%), T_s = 149-152°C. R_f = 0.4 (5:4, benzene:hexane).

INFERENCE

A new method for producing 2,3-trimethylenebenzo[2,3-d]-pyrimidine-4-one by condensation with lactam. Carries out a selective renovation of the 'w gardens.

The synthesis of a new α-aminophosphonic acid was carried out with a high yield using a single-ring tricate.

Component reaction process, Zucchini-Fields reaction. It involves the reaction between 2,3-trimethylene-1,2,3,4-tetrahydrobenzo[2,3-d]pyrimidin-4-one substituted with aromatic

aldehydes and phosphoric acid in dry toluene at the reflux temperature. Their structures were determined by IR elemental analysis, ¹H-NMR and mass spectrum.

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