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SHORT COMMUNICATIONS

Recyclization of 3-Heteroylpyrrolo[2.1-c][1.4]benzoxazine-1,2,4-triones at Treatment with *o*-Phenylenediamine*

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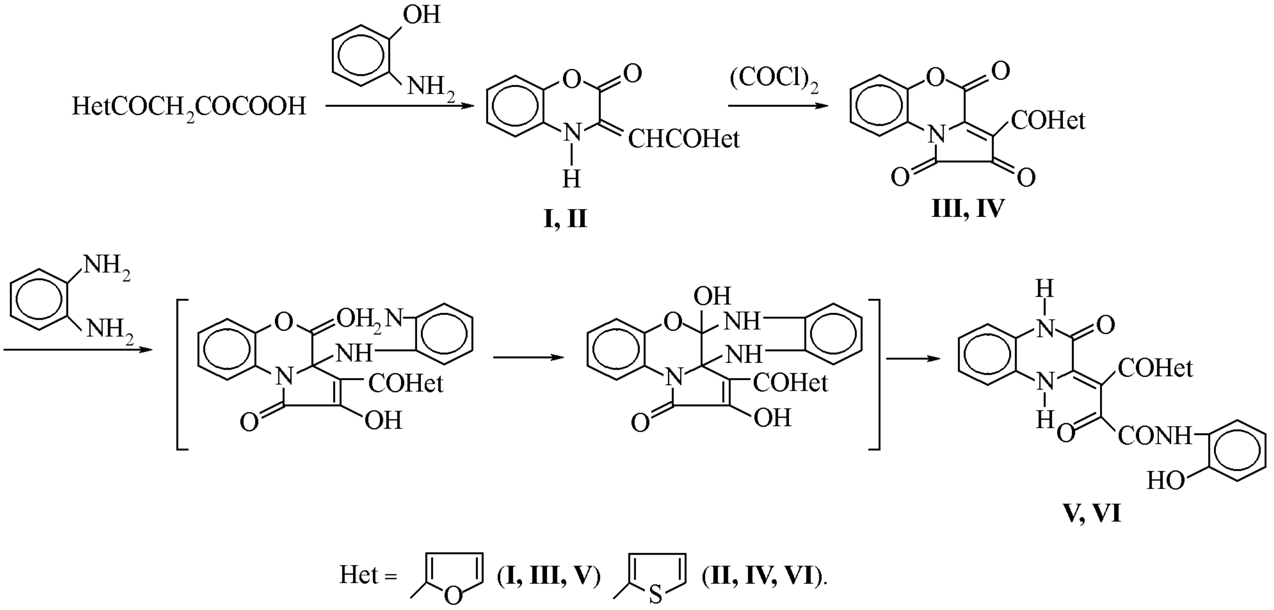
In reaction of 4-heteryl-2,4-dioxabutanoic acids and o-aminophenol we obtained for the first time 3-heteroylmethylene-3,4-dihydro-2*H*-1,4-benzoxazine-2-ones (**I, II**) that by reaction with oxalyl chloride [1] furnished 3-heteroyl-2,4-dihydro-1*H*-pyrrolo[2.1-c][1.4]benzoxazine-1,2,3-triones (**III, IV**). When treated with o-phenylenediamine compounds **III, IV** undergo recyclization with simultaneous opening of the oxazine and dihydropyrroledione rings [2] yielding *N*-*o*-hydroxyphenylamides of (*Z*)-4-heteryl-2,4-dioxo-3-(2-oxo-1,2,3,4-tetrahydro-3-quinoxalinylidene)butanoic acids **V, VI**.

3-Furoylmethylene-3,4-dihydro-2*H*-1,4-benzoxazin-2-one (I). In dioxane solution was boiled for 3–5 min 1.82 g (0.01 mol) of 2,4-dioxo-4-furylbutanoic acid and 1.08 g (0.01 mol) of o-aminophenol. On cooling the separated precipitate was filtered off and recrystallized from ethanol. Yield 1.84 g (72%). yellow crystals, mp 184–185°C (from ethanol). IR spectrum, ν, cm^{-1}: 3150 br. (NH), 1750 (C^2=O), 1640 (CO furoyl). ^{1}H NMR spectrum [$(CD_3)_2CO$], δ, ppm: 6.27 s (1H, CH), 6.29 t (1H, C^4H furyl), 6.75 d (1H, C^3H furyl), 7.03 m (4H, H arom), 7.52 d (1H, C^5H furyl), 12.02 br.s (1H,



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N^4H). Found, %: N 5.63. $C_{14}H_9NO_4$. Calculated, %: N 5.49.

Compound **II** was prepared by similar procedure. Yield 2.14 g (79%), mp 231–232°C (AcOH). IR spectrum ν, cm^{-1}: 3100 br. (NH), 1750 (C^2=O), 1630 (CO thenoyl). ^1H NMR spectrum [$(CD_3)_2CO$], δ, ppm: 6.75 s (1H, CH), 7.14 t (1H, C^4H thienyl), 7.28 d (1H, C^3H thienyl), 7.50 m (4H, H arom), 7.97 d (1H, C^5H thienyl), 12.48 br.s (1H, N^4H). Found, %: N 5.22; S 11.93. $C_{14}H_9NO_3S$. Calculated, %: N 5.16; S 11.82.

3-Furoyl-2,4-dihydro-1*H*-pyrrolo[2.1-c][1.4]-benzoxazine-1,2,3-trione (III). To a solution of 1.28 g (0.005 mol) of compound **I** in 50 ml of anhydrous dichloroethane was added 0.005 mol of oxalyl chloride, and the mixture was heated under reflux; to the outlet of the reflux condenser was fit a drying tube filled with calcium chloride. The reaction mixture was cooled, the separated precipitate was filtered off and washed with anhydrous dichloroethane. Yield 1.22 g (79%0, dark violet crystals, decomp. temp. 194–196°C (from anhydrous dichloroethane). IR spectrum, ν, cm^{-1}: 1780 (C^4=O), 1750 br. (C^1=O, C^2=O), 1640 (CO furoyl). ^1H NMR spectrum [$(CD_3)_2CO$], δ, ppm: 6.71 t (1H, C^4H furyl), 7.21 m (4H, H arom + 2H, C^3H + C^5H furyl). Found, %: N 4.76. $C_{16}H_7O_6N$. Calculated, %: N 4.53.

Compound **IV** was similarly prepared. Yield 1.35 g (89%), dark violet crystals, decomp. temp. 185–187°C (from anhydrous dichloroethane). IR spectrum, ν, cm^{-1}: 1760 (C^4=O), 1740 br. (C^1=O, C^2=O), 1630 (CO thenoyl). ^1H NMR spectrum [$(CD_3)_2CO$], δ, ppm: 7.25 m (4H, H arom + 3H, H thienyl.). Found, %: N 4.59; S 10.24. $C_{16}H_7NO_5S$. Calculated, %: N 4.37; S 10.01.

(*Z*)-2,4-dioxo-3-(2-oxo-1,2,3,4-tetrahydro-3-quinoxalinylidene)-4-furylbutanoic acid *N*-*o*-hydroxyphenylamide (V). To a solution of 0.93 g (0.003 mol) of compound **III** in 30 ml of anhydrous dioxane was added a solution of 0.33 g (0.003 mol) of *o*-phenylenediamine in 20 ml of anhydrous dioxane, the reaction mixture was heated to boiling, cooled, the separated precipitate was filtered off and recrystallized from DMSO. Yield 0.88 g (73%), orange crystals, mp 217–219°C (from DMSO). IR spectrum, ν, cm^{-1}: 3280, 3180 ($CO\underline{NH}$, OH), 3100 br. (N^4H), 1690 ($\underline{CO}NH$), 1660 (CO furoyl), 1610 br (C^2=O), 1530 (amide **II**). ^1H NMR spectrum [$(CD_3)_2CO$], δ, ppm: 7.27 m (8H, H arom + 3H, H furyl), 9.18 br.s (1H, OH), 9.59 s (1H, $CO\underline{NH}$), 10.25 s (1H, $CO\underline{NH}$ quin., 12.22 br.s (1H, N^4H). Found, %: N 10.37. $C_{22}H_{15}N_3O_5$. Calculated, %: N 10.47.

Compound **VI** was obtained similarly. Yield 0.84 g (67%), orange crystals, mp 245–247°C (from DMSO). IR spectrum, ν, cm^{-1}: 3250, 3150 ($CO\underline{NH}$, OH), 3080 br (N^4H), 1680 ($\underline{CO}NH$), 1650 (CO thenoyl), 1620 br (C^2=O), 1530 (amide II). ^1H NMR spectrum [$(CD_3)_2CO$], δ, ppm: 6.66 t (1H, C^4H thienyl), 7.22 m (4H, H arom + 1H, H thienyl), 7.89 d (1H, C^5H thienyl.), 9.30 br.s (1H, OH), 9.57 s (1H, $CO\underline{NH}$), 10.50 s (1H, $CO\underline{NH}$ quin.), 12.57 br.s (1H, N^4H). Found, %: N 10.21; S 7.93. $C_{22}H_{15}N_3O_4S$. Calculated, %: N 10.07; S 7.68.

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