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SHORT COMMUNICATIONS

Recyclization of Pyrroloquinoxalinetrione by the Action of *o*-Aminobenzenethiol

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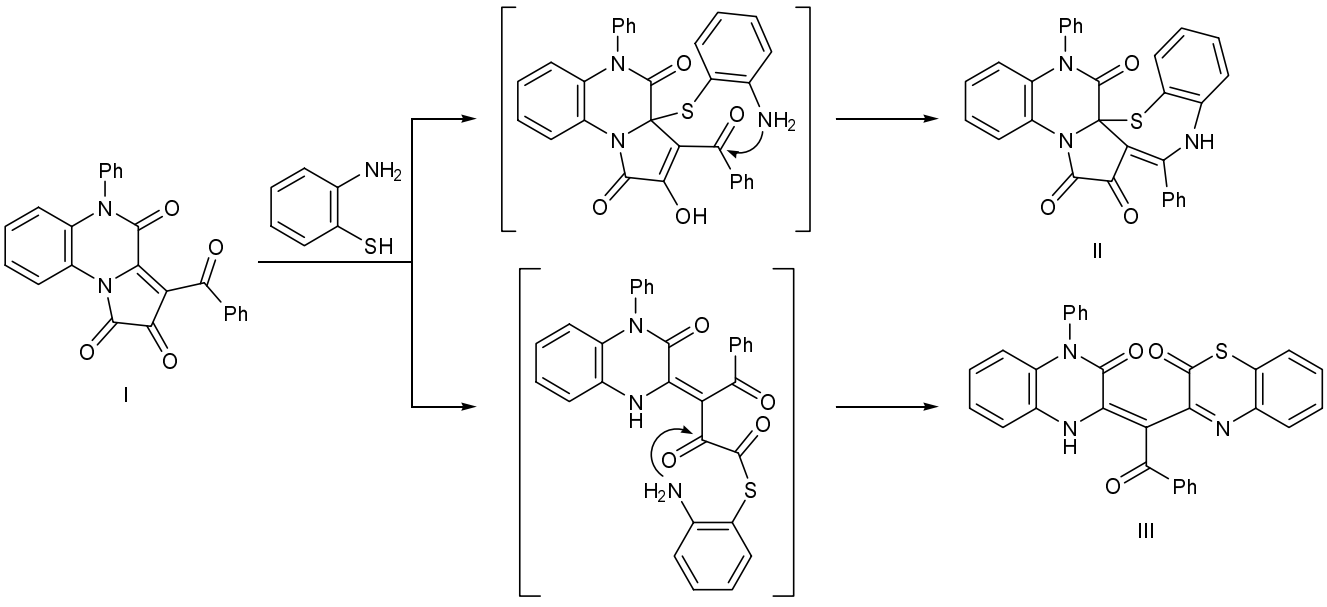
We previously showed that the reaction of 3-aroyl-5-phenylpyrrolo[1,2-*a*]quinoxaline-1,2,4(5*H*)-triones with *o*-aminobenzenethiol involves successive attack by the sulfanyl and amino groups of the latter on the carbon atom in position *3a* and aroyl carbonyl carbon atom, respectively, of the former to give heterocyclization products, 8-aryl-16-phenyl-6*H*-quinoxalino-[1',2':1,2]pyrrolo[2,3-*b*][1,5]benzothiazepine-6,7,-15(9*H*,16*H*)-triones [1].

In the reaction of 3-benzoyl-5-phenylpyrrolo[1,2-*a*]-quinoxaline-1,2,4(5*H*)-trione (**I**) with *o*-aminobenzenethiol in boiling benzene (reaction time 10 min), we isolated the expected product, 8,16-diphenyl-6*H*-quinoxalino[1',2':1,2]pyrrolo[2,3-*b*][1,5]benzothiazepine-6,7,15(9*H*,16*H*)-trione (**II**), and 3-[(1*Z*)-2-oxo-1-(3-oxo-4-phenyl-3,4-dihydroquinoxalin-2(1*H*)-ylidene)-2-phenylethyl]-1,4-benzothiazin-2(3*H*)-one (**III**).

Presumably, benzothiazinone **III** is formed as a result of attack by the sulfanyl group of *o*-aminobenzenethiol on the C^1 carbon atom of **I** with opening of the pyrroledione ring at the C^1–N^{10} bond and subsequent intramolecular cyclization involving the amino group of aminobenzenethiol and carbonyl group in the β-position with respect to the sulfur atom. Among several possible tautomeric forms of benzothiazinone **III**, that giving rise to the strongest intramolecular hydrogen bond is observed.

8,16-Diphenyl-6*H*-quinoxalino[1',2' : 1,2]pyrrolo-[2,3-*b*][1,5]benzothiazepine-6,7,15(9*H*,16*H*)-trione (II) and 3-{(1*Z*)-2-oxo-1-[3-oxo-4-phenyl-3,4-dihydroquinoxalin-2(1*H*)-ylidene]-2-phenylethyl}-1,4-benzotiazin-2(3*H*)-one (III). A solution of 1 mmol of compound **I** and 1 mmol of *o*-aminobenzenethiol in 10 ml of anhydrous benzene was heated for 10 min



under reflux. The mixture was cooled, and the light yellow precipitate of compound **III** was filtered off. Yield 25%, mp 284–286°C (decomp., from benzene). IR spectrum, ν, cm^{-1}: 3030 br (NH), 1728 (SC=O), 1703 (NC=O), 1630 (PhCO). ^1H NMR spectrum, δ, ppm: 6.57 d (1H, 5-H, quinoxaline, J = 7.5 Hz), 7.15–7.98 m (17H; H_{arom}; 6-H, 7-H, and 8-H in quinoxaline, 5-H, 6-H, 7-H, and 8-H in benzothiazine), 13.77 s (1H, NH). ^{13}C NMR spectrum (DMSO-d_6), δ_C, ppm: 115.97–140.04 (C_{arom}), 147.17 (NC=O), 156.15 (C^3, benzothiazine), 164.39 (SC=O), 188.68 (PhCO). Mass spectrum, m/z (I_{rel}, %): 501 (73) $[M]^+$, 445 (9) $[M - 2CO]^+$, 424 (100) $[M - Ph]^+$, 396 (88) $[M - Ph - CO]^+$, 368 (35) $[M - Ph - 2CO]^+$, 323 (21), 262 (87), 234 (27), 212 (34), 205 (38), 109 (25) $[PhS]^+$, 77 (28) $[Ph]^+$. Found, %: C 71.86; H 3.85; N 8.36; S 6.37. $C_{30}H_{19}N_3O_3S$. Calculated, %: C 71.84; H 3.82; N 8.38; S 6.39.

The mother liquor was evaporated to dryness. The residue was dark yellow compound **II**. Yield 64%, mp 239–241°C (decomp., from toluene). IR spectrum, ν, cm^{-1}: 3210 br (NH), 1732 (C^6=O), 1685 (C^7=O), 1670 (C^{15}=O). ^1H NMR spectrum, δ, ppm: 6.41 d (1H, 1-H, J = 7.5 Hz), 7.16–7.56 m (16H, H_{arom}, 2-H, 3-H, 10-H, 11-H, 12-H, 13-H), 7.91 d (1H, 4-H, J = 7.7 Hz), 10.40 s (1H, NH). ^{13}C NMR spectrum (DMSO-d_6), δ, ppm: 67.98 (C^{14a}), 105.82 (C^{7a}), 116.40–137.32 (C_{arom}), 143.64 (C^6), 156.60 (C^{15}), 159.49 (C^8), 174.23 (C^7). Mass spectrum, m/z (I_{rel}, %): 501 (23) $[M]^+$, 445 (55) $[M - 2CO]^+$, 424 (11) $[M - Ph]^+$, 396 (21) $[M - Ph - CO]^+$, 368 (6) $[M - Ph - 2CO]^+$, 262 (100), 234 (23), 223 (11), 212 (46), 109 (28), 77 (20) $[Ph]^+$. Found, %: C 71.88; H 3.80; N 8.34; S 6.40. $C_{30}H_{19}N_3O_3S$. Calculated, %: C 71.84; H 3.82; N 8.38; S 6.39.

The IR spectra were recorded on a UR-20 spectrometer from samples dispersed in mineral oil. The ^1H and ^{13}C NMR spectra were measured on a Bruker WP-400 instrument from solutions in DMSO-d_6 using tetramethylsilane as internal reference. The purity of the products was checked by thin-layer chromatography on Silufol plates using ethyl acetate as eluent; spots were visualized by treatment with iodine vapor.

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