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RECYCLIZATION OF PYRROLO[2,1-*c*][1,4]-BENZOXAZINE-1,2,4-TRIONES BY THE ACTION OF AN ACTIVATED ENAMINE

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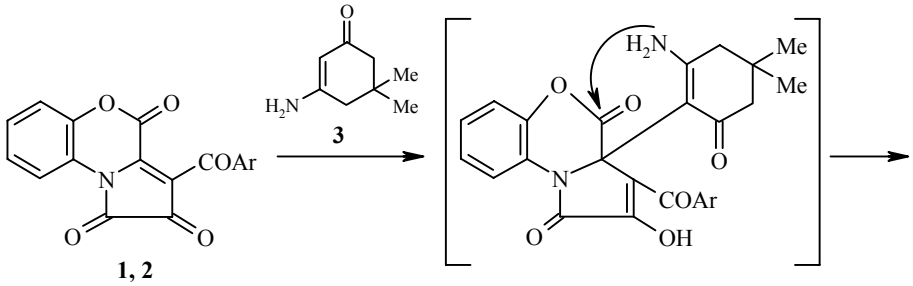
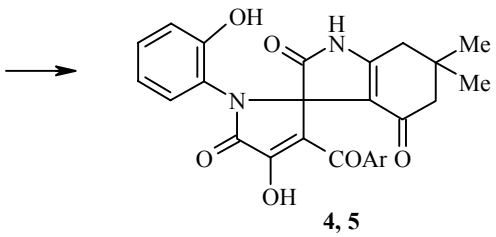
In previous work [1], we have described the recyclization of 3-aroyl-2,4-dihydro-1H-pyrrolo[2,1-*c*]-[1,4]benzoxazine-1,2,4-triones (**1, 2**) by the action of *o*-phenylenediamine, which is an NH-binucleophile [1], with the simultaneous opening of the oxazine ring at the $C_{(4)}$–$O_{(5)}$ and $C_{(3a)}$–$N_{(10)}$ bonds.





1, **4** Ar = *p*-MeC_6H_4, **2**, **5** Ar = α-$C_{10}H_7$

Brief heating of a solutions of **1** or **2** with 3-amino-5,5-dimethyl-2-cyclohexen-1-one (**3**) in acetonitrile gives 6,6-dimethyl-2,4-dioxo-2,3,4,5,6,7-hexahydro-1H-indole-3-spiro-2-(3-aroyl-4-hydroxy-1-*o*-hydroxyphenyl-5-oxo-2,5-dihydropyrroles) (**4, 5**), which were identified by X-ray diffraction structural analysis.

The first step in this reaction probably features the addition of the activated β-CH group of enamine **3** to $C_{(3a)}$ in **1** and **2** as described for *o*-phenylenediamine [1] and other nucleophilic reagents [2, 3] with subsequent closure of the pyrrole ring by intramolecular attack of the free amino group of the lactonic carbonyl group.

6,6-Dimethyl-2,4-dioxo-2,3,4,5,6,7-hexahydro-1H-indole-3-spiro-2-(4-hydroxy-1-*o*-hydroxyphenyl-5-oxo-3-*p*-tolyl-2,5-dihydropyrrole) (4). A solution of **1** (0.001 mol) and a solution of enamine **3** (0.001 mol) in absolute acetonitrile (10 ml) was heated at reflux for 5 min and cooled. The precipitate formed was filtered

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off to give 0.41 g (87%) of compound **4**; mp 228-230°C (acetonitrile). IR spectrum in vaseline mull, ν, cm^{-1}: 3590 (phenol OH), 3400 br (NH, enol OH), 1750 ($C_{(5)}$=O), 1700 ($C_{(2)}$=O), 1650 (COC_6H_4Me-*p*). ^1H NMR spectrum (DMSO-d_6, 400 MHz, HMDS as the standard), δ, ppm: 0.73 (3H, s, Me); 0.85 (3H, s, Me); 1.97 (2H, s, $C_{(7)}H_2$); 2.05 (2H, s, $C_{(5)}H_2$); 6.72-7.60 (8H, m, $2C_6H_4$); 9.24 (1H, s, phenol OH); 10.79 (1H, s, NH); 11.90 (1H, br. s, enol OH). Found, %: C 68.64; H 5.21; N 5.95. $C_{27}H_{24}N_2O_6$. Calculated, %: C 68.63; H 5.12; N 5.93.

6,6-Dimethyl-2,4-dioxo-2,3,4,5,6,7-hexahydro-1H-indole-3-spiro-2-(4-hydroxy-1-*o*-hydroxyphenyl-5-oxo-3-α-naphthoyl-2,5-dihydropyrrole) (5) was obtained in 76% yield (0.39 g); mp 214-215°C (acetonitrile). IR spectrum in vaseline mull, ν, cm^{-1}: 3560 (phenol OH), 3320 br (NH, enol OH), 1730 (C(5)=O), 1690 ($C_{(2)}$=O), 1670 ($COC_{10}H_7$-α). ^1H NMR spectrum (DMSO-d_6, 400 MHz, HMDS as the standard), δ, ppm: 0.75 (3H, s, Me); 0.88 (3H, s, Me); 2.00 (2H, s, $C_{(7)}H_2$); 2.30 (2H, d, $C_{(5)}H_2$); 6.74-8.30 (11H, m, C_6H_4+$C_{10}H_7$); 9.29 (1H, s, phenol OH); 10.85 (1H, s, NH); 12.05 (1H, br. s, enol OH). Found, %: C 70.82; H 4.73; N 5.53. $C_{30}H_{24}N_2O_6$. Calculated, %: C 70.86; H 4.76; N 5.51.

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