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SYNTHESIS OF NEW SUBSTITUTED DIHYDROPYRIDONES

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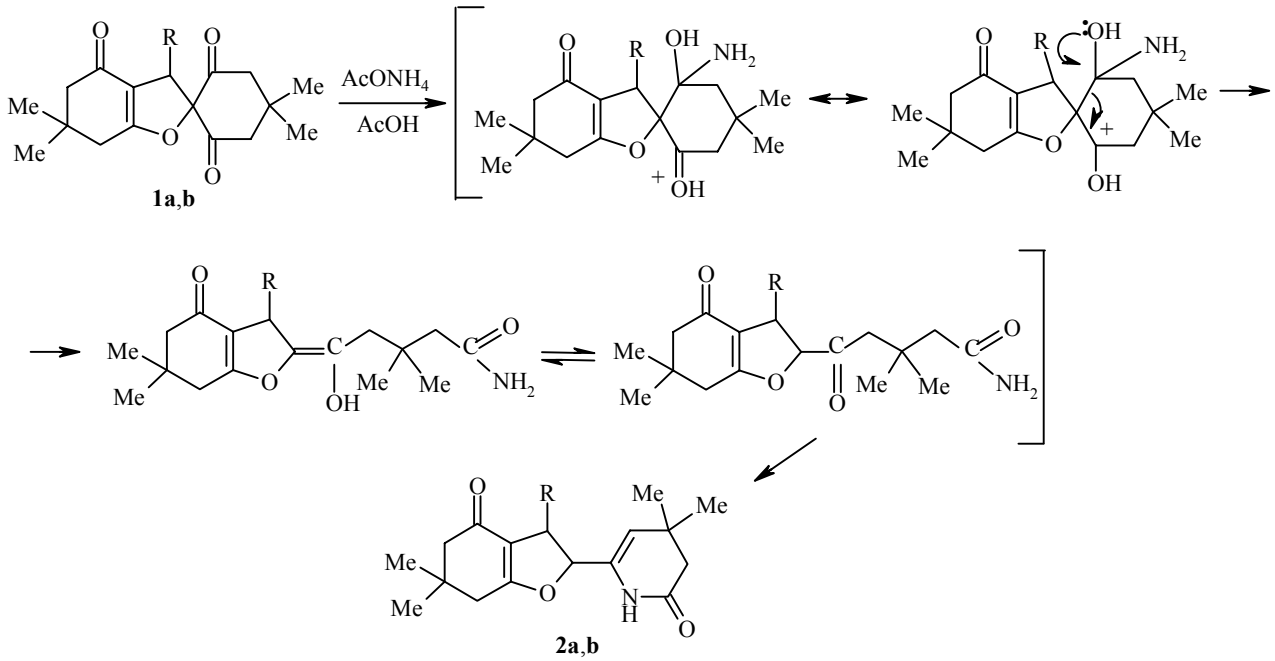
Despite the availability of 2-spiro(3-R-6,6-dimethyl-4-oxo-2,3,4,5,6,7-hexahydrobenzofuran)-2'-(5',5'-dimethyl-1',3'-cyclohexanediones) (**1a,b**), the chemistry of these compounds has hardly been studied [1-4]. Greenberg [2] has reported splitting of the dimedone fragment of spiran **1b** in aqueous dioxane by the action of sodium hydroxide and, then, hydrochloric acid to give the corresponding δ-oxo acid.

We have found that carbonyl-containing condensed spirohydrofurans **1a** and **1b** under conditions of the Chichibabin reaction with $CH_3CO_2NH_4$ and acetic acid are converted to 6-(3-R-6,6-dimethyl-4-oxo-2,3,4,5,6,7-hexahydro-2-benzofuranyl)-4,4-dimethyl-3,4-dihydro-2-pyridones (**2a,b**) in yields of 40 and 78%, respectively. This reaction probably involves formation of δ-oxo acid amides with their subsequent N-heterocyclization.



1, 2 a R = Ph, **b** R = H

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6-(6,6-Dimethyl-4-oxo-3-phenyl-2,3,4,5,6,7-hexahydro-2-benzofuranyl)-4,4-dimethyl-3,4-dihydro-2-pyridone (2a). A mixture of **1a** (1.5 g, 4.1 mmol) and ammonium acetate (1.7 g, 20 mmol) in acetic acid (20 ml) were heated at reflux for 60 h. The reaction mixture was washed with saturated aq. sodium carbonate and extracted with diisopropyl ether. The ethereal extracts were dried over $MgSO_4$ and evaporated. The oily residue was triturated in acetone and crystallized from 2-propanol to give 0.6 g (40%) of **2a**; mp 260-262°C. IR spectrum (neat), ν, cm^{-1}: 1644, 1656 (C=O), 3212-32185 (NH). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 0.69 (3H, s, CH_3); 0.90 (3H, s, CH_3); 1.13 (6H, d, $2CH_3$); 1.60 (1H, s, NH); 1.76 (1H, d, C(3)H); 1.94 (1H, d, C(2)H). 2.21 (4H, s, $2CH_2$); 2.49 (2H, s, CH_2); 4.88 (1H, s, C(5)H); 7.35 (5H, d, Ar). Found, %: C 74.84; H 7.12; N 4.62. $C_{23}H_{27}NO_3$. Calculated, %: C 75.61; H 7.39; N 3.84.

6-(6,6-Dimethyl-4-oxo-2,3,4,5,6,7-hexahydro-2-benzofuranyl)-4,4-dimethyl-3,4-dihydro-2-pyridone (2b) was obtained analogously from spiran **1b** in 78% yield; mp 228-230°C (2-propanol). IR spectrum (neat), ν, cm^{-1}: 1690, 1666 (C=O), 3220-3205 (NH). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 0.90 (12H, d, $4CH_3$); 2.32 (6H, s, $3CH_2$); 2.69 (3H, s, $C(3)H_2$, C(2)H); 5.42 (1H, s, C(5')H); 7.87 (1H, br. s, NH). Found, %: C 70.22; H 7.67; N 5.16. $C_{17}H_{23}NO_3$. Calculated, %: C 70.56; H 8.01; N 4.84.

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