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SYNTHESIS OF SUBSTITUTED 4a,10b-DIHYDRO-1H-CHROMENO-[3,4-*c*]-PYRIDINE-2,4,5-TRIONES *via* THE REFORMATSKY REACTION

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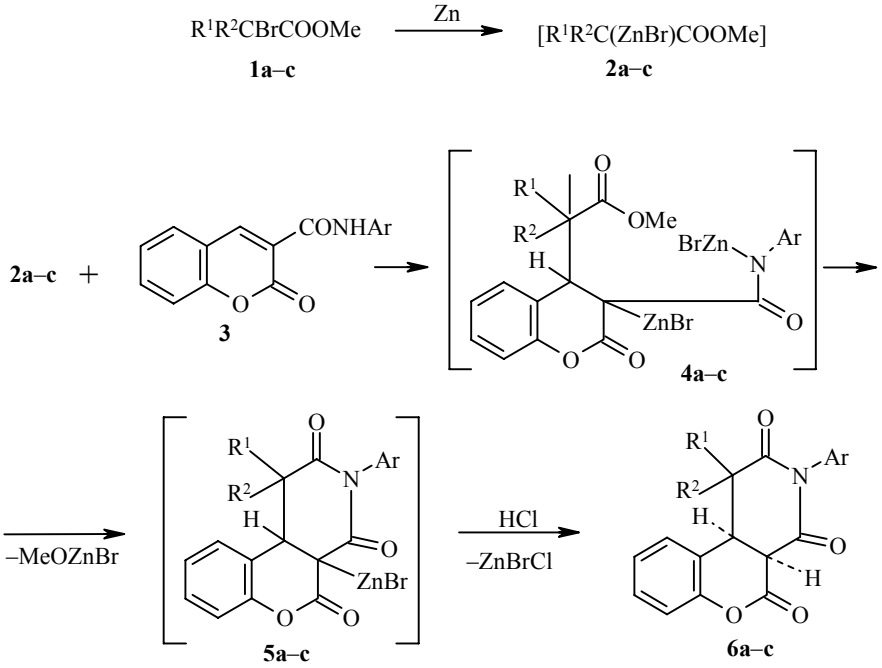
In a continuation of a systematic study of the reaction of zinc intermediates with derivatives of 2-oxochromen-3-carboxylic acids [1], we have observed a new and unexpected method for the heterocyclization of N-arylamides of 2-oxochromen-3-carboxylic acids **3** under the influence of the organozinc reagents **2a-c** prepared from methyl α-bromoacetate, methyl α-bromobutyrate, and methyl α-bromoisobutyrate (**1a-c**). The reactions were carried in a mixture of ether–HMTPA–THF (1:1:1), apparently *via* the intermediate compounds **4a-c** which underwent self-cyclization to the intermediates **5a-c**, which gave the required products, substituted 4a,10b-dihydro-1H-chromeno[3,4-*c*]pyridine-2,4,5-triones, **6a-c**, after hydrolysis.



1–6 Ar = 4-MeC$_6$H$_4$; **a** R^1 = R^2 = H; **b** R^1 = H, R^2 = Et; **c** R^1 = R^2 = Me

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Compounds **6a-c** were formed as a single geometric isomer, most likely with the hydrogens on C(4a) and C(10b) in an eclipsed position.

3-*p*-Tolyl-4a,10b-1H-chromeno[3,4-*c*]pyridine-2,4,5-trione (6a). Yield 72%; mp 215-216°C. IR spectrum (nujol mull), ν, cm^{-1}: 1690, 1770. ^1H NMR spectrum (60 MHz, $CDCl_3$), δ, ppm: 2.27 (3H, s, Me); 2.80-3.30 (2H, m, CH_2); ~3.75, 4.03 (2H, m, d, CH–CH); 6.70-7.40 (8H, m,  , C_6H_4). Found, %: C 71.55; H 3.71. $C_{19}H_{12}NO_4$. Calculated, %: C 71.69; H 3.80.

1-Ethyl-3-*p*-tolyl-4a,10b-1H-chromeno[3,4-*c*]pyridine-2,4,5-trione (6b). Yield 68%; mp 181-182°C. IR spectrum (nujol mull), ν, cm^{-1}: 1690, 1760. ^1H NMR spectrum (60 MHz, $CDCl_3$), δ, ppm: 1.00 (3H, t, $CH_2\underline{CH_3}$); 1.40-2.10 (2H, m, $\underline{CH_2}CH_3$); 2.26 (3H, s, $C_6H_4\underline{CH_3}$); 2.55-2.90 (1H, m, CH); 3.60, 4.06 (2H, m, d, CH–CH); 6.70-7.40 (8H, m,  , C_6H_4). Found, %: C 72.65; H 4.58. $C_{21}H_{16}NO_4$. Calculated, %: C 72.82; H 4.65.

1,1-Dimethyl-3-*p*-tolyl-4a,10b-1H-chromeno[3,4-*c*]pyridine-2,4,5-trione (6c). Yield 81%; mp 231-232°C. IR spectrum (nujol mull), ν, cm^{-1}: 1690, 1770. ^1H NMR spectrum (60 MHz, $CDCl_3$), δ, ppm: 1.10, 1.30 (6H, s, CMe_2); 2.30 (3H, s, $C_6H_4\underline{CH_3}$); 3.82, 4.28 (2H, m, d, CH–CH); 6.70-7.40 (8H, m,  , C_6H_4). Found, %: C 72.82; H 4.65. $C_{21}H_{16}NO_4$. Calculated, %: C 72.71; H 4.69.

REFERENCES

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