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A SIMPLE METHOD FOR OBTAINING 3-(2,3-DIHYDROXY-4-METHYL-5-OXO-1,3-CYCLOPENTADIENYL)-2(1H)-QUINOXALINONE

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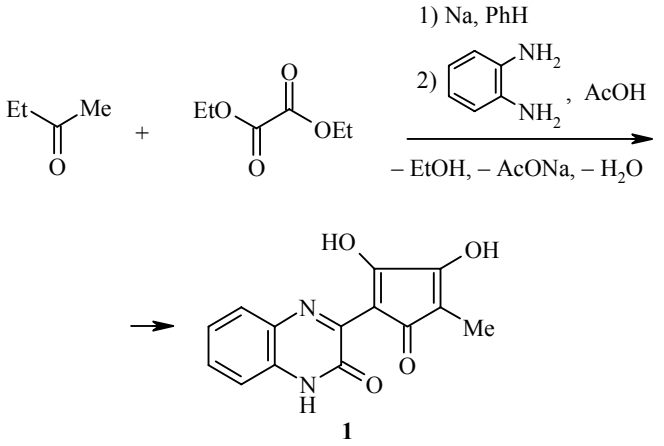
3-Acylmethyl-2(1H)-quinoxalinones are widely used in organic synthesis and are biologically active compounds [1-4]. We recently proposed a convenient one-step method for obtaining 3-(2-oxo-2(R)-ethylidene) derivatives of 2H-1,4-benzoxazin-2-ones and 2(1H)-quinoxalinones by condensation of acetone, pinacoline, or aryl methyl ketones with diethyl oxalate in the presence of base, followed by neutralization and treatment with *o*-aminophenol or *o*-phenylenediamine [5]. The method was limited to the indicated monocyclic structures; bicyclic compounds have not been previously obtained by this method. In continuing the studies, we have developed a very simple preparative method for synthesis of the previously unknown 3-(2,3-dihydroxy-4-methyl-5-oxo-1,3-cyclopentadienyl)-2(1H)-quinoxalinone (**1**) by reaction of 2-butanone with diethyl oxalate in the presence of sodium while boiling the mixture in benzene, followed by treatment with acetic acid and *o*-phenylenediamine.



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The ^{1}H NMR spectra were taken on a Bruker DRX-500 (500 MHz) in DMSO-d_6, internal standard TMS.

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3-(2,3-Dihydroxy-4-methyl-5-oxo-1,3-cyclopentadienyl)-2(1H)-quinoxalinone (1). Sodium (0.46 g, 0.02 mol) was added in small pieces with stirring and cooling to a mixture of 2-butanol (0.9 ml, 0.01 mol), diethyl oxalate (2.8 ml, 0.02 mol), and benzene (20 ml); then the mixture was boiled for 3 h. The solvent was evaporated, the residue was carefully ground with cold water (20 ml), and then AcOH (10 ml) and *o*-phenylenediamine (1.08 g, 0.01 mol) were added with stirring. After 3 h, the precipitate of compound **1** was filtered out and crystallized from DMF. Yield 1.95 g (72%). Decomposition temperature 298-300°C. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.87 (3H, s, CH_3); 7.14 (1H, t, *J* = 8.0, H-6); 7.32 (1H, t, *J* = 8.2, H-7); 7.40 (1H, d, *J* = 8.8, H-8); 7.95 (1H, s, 3'-OH); 8.05 (1H, d, *J* = 9.0, H-5); 11.68 (1H, br. s, $N_{(1)}H$), 14.10 (1H, br. s, 2'-OH). Mass spectrum (Finnigan MAT INCOS 50), *m*/*z* (I_{rel}, %): 270 $[M]^+$ (22), 226 (9), 225 (11), 224 $[M–CO–H_2O]^+$ (22), 223 (9), 198 (10), 197 (12), 169 (22), 168 $[M–C_6H_4CN]^+$ (31), 167 (5), 153 (5), 140 (5), 129 (5), 114 (5), 112 (7), 103 (5), 102 $[C_6H_4CN]^+$ (8), 84 (12), 77 $[C_6H_5]^+$ (15), 76 (10), 75 (5), 73 (32), 70 (7), 63 (8), 52 (7), 51 (11), 50 (11), 45 (8), 44 (10), 42 (22), 41 (13), 40 (10), 39 (18), 38 (5). Found, %: C 61.97; H 3.50; N 10.56. $C_{14}H_{10}N_2O_4$. Calculated, %: C 62.22; H 3.73; N 10.37.

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