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3(2,4-Dinitrobenzyl)-2,4-pentanedione

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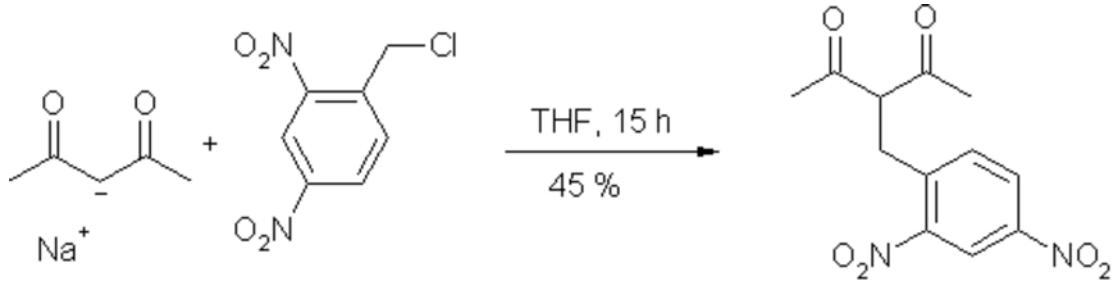

1 **2** **3**

Substituted pentanediones are widely used as ligands for metal complexes.[1] We report the synthesis and characterization of a 3-substituted pentanedione with an electron poor dinitrobenzyl substituent. The compound is sensitive and decomposes readily in the presence of ammonia.

To a solution of sodium acetyl acetonate (1.22 g, 10 mmol) (**1**) in THF (200 ml) were added 2,4-dinitrobenzylchloride (2.16 g, 10 mmol) (**2**) in 25 ml of THF at 0 °C. The reaction mixture was heated to reflux 1.5 h and kept at room temperature overnight. The solution was diluted with Et_2O (100 ml), acidified with aqueous HCl (2 M), washed three times with H_2O, dried over $MgSO_4$ and the solvent was removed in vacuo. The crude product was purified by column chromatography on silica gel [petrol ether (60/70)/Et_2O 3:1] to yield 1.28 g (45 %) of **3** (R_f = 0.35); yellow-orange solid, mp. 118 - 120 °C.

IR (KBr): 3117 cm^{-1}, 1700, 1604, 1539, 1350, 1174, 734.

UV/Vis (CH_3CN): lmax (log e) = 192 nm (4.248), 200 (4.153), 250 (4.177).

^1H NMR (400 MHz, $CDCl_3$): Enol form: d = 2.04 (s, 6H, CH_3), 4.08 (s, 2H, benzyl-CH_2), 7.54 (d, 3J = 8.7 Hz, 1H, arene-H), 8.42 (dd, 3J = 8.7 Hz, 4J = 2.4 Hz, 1H, arene-H), 8.84 (m, 1H, arene-H), 16.96 (s, 1H, enol-H). Keto form: d = 2.27 (s, 6H, CH_3), 3.49 (d, 3J = 6.7 Hz, 2H, benzyl-CH_2), 4.20 (t, 3J = 6.7 Hz, 1H, CH), 7.74 (d, 3J = 8.5 Hz, 1H, arene-H), 8.36 (dd, 3J = 8.5 Hz, 4J = 2.4 Hz, 1H, arene-H), 8.84 (m, 1H, arene-H).

^{13}C NMR (100 MHz, $CDCl_3$): d = 23.16 (+), 30.04 (+), 30.17 (-), 30.89 (-), 67.14 (+), 105.00 (C_{quat}), 120.51 (+), 120.57 (+), 127.22 (+), 127.40 (+), 130.30 (+), 135.32 (+), 140.60 (C_{quat}), 141.66 (C_{quat}), 192.31 (C_{quat}), 201.84 (C_{quat}).

MS (70 eV), m/z (%): 237 (2) [M+-CO-CH_3], 43 (100) [CO-CH_3].

EA Calculated (%): C 51.43 H 4.32 N 10.00
found (%): C 51.26 H 4.31 N 9.96.

References and Notes

1. Murakami, Y.; Nakamura, K.; Uchida, H.; Kanaoka, Y. *Inorg. Chim. Acta*, **1968**, *2*(2), 133 - 8.

Sample availability: available form the authors.