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NOTE

Synthesis and Characterization of 3-(Pyridin-2-ylmethyl)pentane-2,4-dione

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A new compound, 3-(pyridin-2-ylmethyl)-pentane-2,4-dione was synthesized successfully and characterized by ^{1}H NMR, IR, MS and elemental analysis.

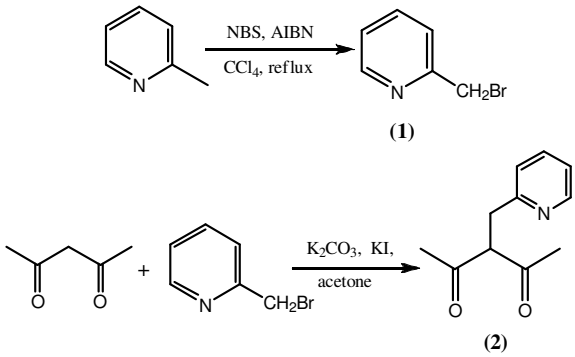
Key Words: 3-(Pyridin-2-ylmethyl)pentane-2,4-dione, 2,4-pentanedione, *N*-bromosuccinimide.

2,4-Pentanedione, also named acetylacetone, is an important chemical intermediate and used widely for the synthesis of pharmaceuticals, feed additives and catalysts *etc.*[1]. Further more, 2,4-pentanedione and its derivatives can coordinate with many metal ions to obtain metal chelates, which can catalyze a number of reactions[2,3]. For example, 2,4-pentanedione chelated nickel complex can catalyze the oligomerization or polymerization of ethylene, propylene, styrene, diene, cyclo-olefins, MMA *etc.*[4-6].

Recently, we synthesized a new compound 3-(pyridin-2-ylmethyl)pentane-2,4-dione, which is derived from 2,4-pentanedione by introducing pyridinylmethyl group into 3-position carbon. This tridentate ligand can also coordinate with metal compounds and the catalytic performance of the obtained metal complexes may be improved due to the introduced pyridinyl group. The synthetic route of 3-(pyridin-2-ylmethyl)-pentane-2,4-dione was shown in **Scheme-I.**

The title compound was characterized by ^{1}H NMR, IR, MS and element analysis and the structure was confirmed. From ^{1}H NMR, it was found that this compound may own two isomers and the molar ratio of two isomers is 2:1 by calculating the integral areas, which is shown in Fig-1.

Preparation of 2-(bromomethyl)pyridine (1)[7-9]: *N*-bromosuccinimide (8.9 g, 0.05 mol), 2-methylpyridine (4.7 g, 0.05 mol) and AIBN (0.3 g) were dissolved in CCl_4 (80 mL) and refluxed for 8 h. After filtration of the suspension, the filtrate was neutralized by Na_2CO_3 solution and washed twice with 100 mL H_2O, then dried by $MgSO_4$. The solution was concentrated under reduced pressure and the residue was purified by column chromatography on silica gel (eluent: petroleum ether : ethyl acetate = 2: 1) to give 2.67 g (31 %) of **1**; ^{1}H NMR (400 MHz, $CDCl_3$): δ 4.53 (s, 2H,-CH_2), 7.18 (m, 1H, Py-H), 7.41 (m, 1H, Py-H), 7.66 (m, 1H, Py-H), 8.55 (m, 1H, Py-H); m.p. 117 ºC. Anal. calcd. for C_6H_6NBr: C, 41.88; H, 3.52; N, 8.13. Found: C, 41.91; H, 3.55; N, 8.11.



Scheme-I Synthesis of 3-(pyridin-2-ylmethyl)pentane-2,4-dione

Preparation of 3-(pyridin-2-ylmethyl)pentane-2,4-dione (2): A mixture of 2,4-pentanedione (2 g, 0.02 mol), sodium carbonate (4.15 g, 0.03 mol) and acetone (60 mL)

were refluxed for 5 h. Then 2-(bromomethyl)pyridine (3.5 g, 0.02 mol) and KI (1.5 g) were added and refluxed again for 8 h. The mixture was filtered and the solvent was removed *in vacuo*. After adding water, the mixture was extracted three times with Et_2O and filtrated after being dried by $MgSO_4$. Then the solvent was removed to leave light red liquid 3.25 g (85 %). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.59 (m, 1H, Py-H), 7.24-7.32 (m, 3H, Py-H), 3.94 (s, 1H, -CH), 3.47 (s, 2H, $-CH_2$), 2.37 (m, 6H, $-CH_3$); IR (cm^{-1}): 3055, 2986, 2925, 1727, 1589, 1473, 1430, 1359, 1241, 1151, 1097, 1044, 996, 943, 847, 755. MS(EI)(m/z): 191 (M^+). Anal. calcd. for $C_{11}H_{13}NO_2$: C, 69.08; H, 6.85; N, 7.33. Found: C, 69.05; H, 6.89; N, 7.31.

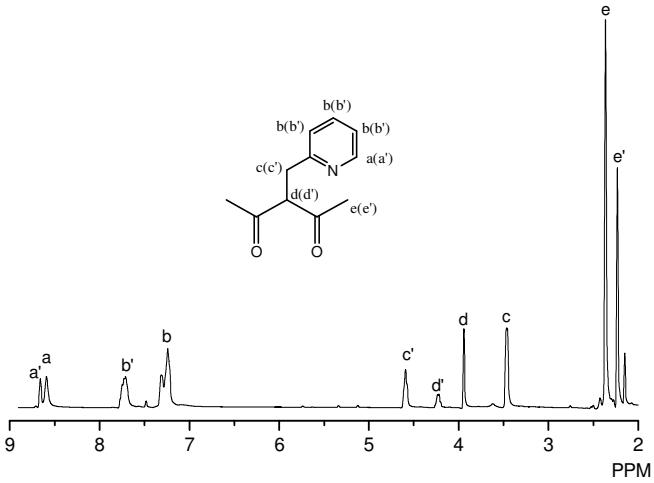


Fig. 1. ^{1}H NMR spectrum of the title compound **2**

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