

Short Note

7-Hydroxy-8-acetylcoumarin *N*-1-(Carboxymethyl)pyridinium Chloride Hydrazone

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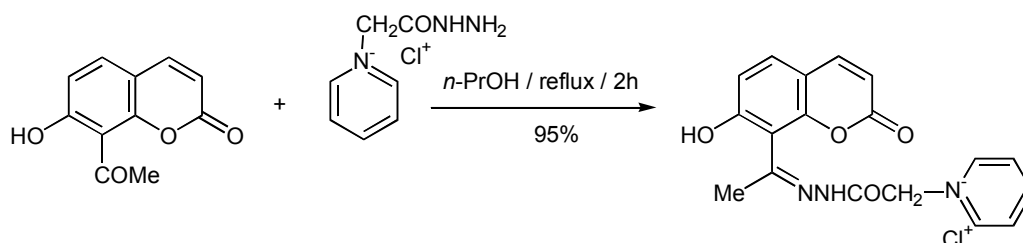
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Received: 24 April 2007; in revised form: 8 August 2008 / Accepted: 18 August 2008 / Published: 24 August 2008

Keywords: 7-hydroxy-8-acetylcoumarin, carbonylhydrazones.

As part of a research programme targeting novel molecules with possible biological activity, derived from *o*-hydroxyaryl ketone hydrazones [1] we attempted to synthesize a water soluble hydrazone of 7-hydroxy-8-acetylcoumarin. Thus, we used 1-(carboxymethyl)pyridinium chloride hydrazide (Girard's Reagent P), a well known reagent that produces water soluble ketone derivatives.



7-Hydroxy-8-acetylcoumarin was prepared according to the literature method [2] whereas, commercially available Girard's Reagent P was supplied by Aldrich. Girard's Reagent P (0.92 g, 4.9 mmol) was added to a solution of 7-hydroxy-8-acetylcoumarin (1 g, 4.9 mmol) in 1-propanol (25 mL). The reaction mixture was refluxed for 2 hours. It was then allowed to cool at room temperature. Subsequently, it was stored in the refrigerator overnight. Filtration of the precipitate, which was formed, afforded of the desired hydrazone as white crystals (1.73 g, 95 %). The product was identified

by its ^1H NMR, ^{13}C NMR and MS without further purification and was found to be a mixture of two isomers as shown by ^1H and ^{13}C NMR data.

M.p. 248-249 °C.

^1H NMR (400 MHz, DMSO- d_6): 2.22 (s, 3H), 2.37 (s, 3H), 5.79 (s, 2H), 5.82 (s, 2H), 6.16-6.22 (m, 2H), 6.98-7.11 (m, 2H), 7.50-7.56 (m, 2H), 7.91-7.97 (m, 2H), 8.13-8.19 (m, 4H), 8.61-8.66 (m, 2H), 9.02-9.11 (m, 4H), 10.60 (s, 1H), 10.70 (s, 1H), 11.18 (s, 1H), 11.46 (s, 1H).

^{13}C NMR (100 MHz, DMSO- d_6): 19.1, 20.0, 62.0, 62.4, 109.7, 111.8, 111.9, 112.0, 112.4, 113.7, 113.8, 113.9, 114.4, 115.5, 128.1, 128.2, 128.3, 129.9, 130.1, 130.3, 145.6, 145.9, 147.0, 147.1, 147.9, 152.9, 153.2, 153.3, 158.8, 159.7, 160.0, 160.8, 161.0, 162.9, 167.1, 167.9.

MS m/z (ESI $^+$) : Calcd. for $\text{C}_{18}\text{H}_{16}\text{N}_3\text{O}_4\text{Cl}$: 711.19647 [$\text{C}_{36}\text{H}_{32}\text{N}_6\text{O}_8\text{Cl}$] $^+$, 675.21979 [$\text{C}_{36}\text{H}_{31}\text{N}_6\text{O}_8$] $^+$, 338.11353 [$\text{C}_{18}\text{H}_{16}\text{N}_3\text{O}_4$] $^+$. Found: 711.19687 [$\text{C}_{36}\text{H}_{32}\text{N}_6\text{O}_8\text{Cl}$] $^+$, 675.22022 [$\text{C}_{36}\text{H}_{31}\text{N}_6\text{O}_8$] $^+$, 338.11320 [$\text{C}_{18}\text{H}_{16}\text{N}_3\text{O}_4$] $^+$.

References

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2. Abramov, M. A.; Dehaen, W. *Synthesis*, **2000**, 11, 1529.

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