

Short Note

2,6-Bis(9-ethyl-9H-carbazolymethylene)cyclohexanone

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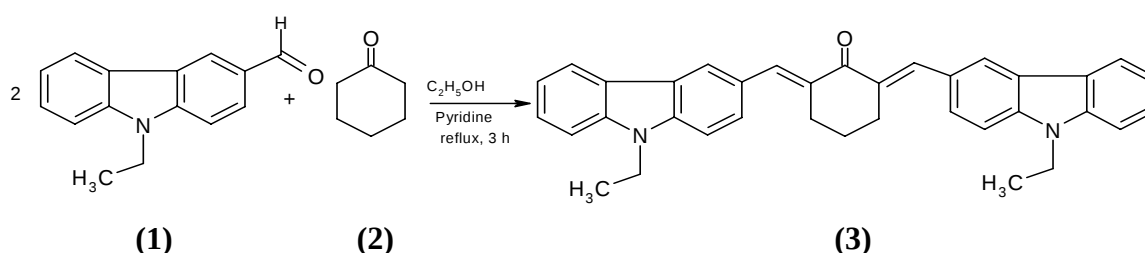
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Abstract: The title compound, 2,6-bis(ethyl-9-ethyl-9H-carbazolymethylene)cyclohexanone has been synthesized by condensation of 9-ethylcarbazole-3-aldehyde and cyclohexanone in ethanol in the presence of pyridine. The structure of this new compound was confirmed by elemental analysis, IR, ^1H NMR, ^{13}C NMR and EI-MS spectral analysis.

Keywords: carbazole aldehyde; cyclohexanone; Knoevenagel condensation

Knoevenagel condensations are among the frequently applied reactions in organic synthesis, providing a good synthetic pathway to form donor-acceptor dienes known as methylene [1]. Compounds with donor-acceptor dienes have become of much interest in recent years in the context of photoelectronics [2], photophotonics [3], photodynamic therapy [4], electrochemical sensing [5], optical limiting [6], langmuir film and photoinitiated polymerization [7]. As evident from the literature, it was noted that a lot of research has been carried out on donor-acceptor dienes, but no work has been done on bis-donor-acceptor dienes. In this paper we report the synthesis of a bis-carbazole from a carbazole aldehyde. With regard to the stereochemistry of the product, we assume the *E,E*-isomer (only one singlet of the olefinic protons was observed in the ^1H NMR spectrum) for steric reasons, but the *Z,Z*-isomer cannot be completely excluded.



A mixture of 9-ethylcarbazole-3-aldehyde (1.0 g, 0.0044 mol), cyclohexanone (0.215 g, 0.0022 mol) and a few drops of pyridine in ethanol (15 mL) was heated for 3 h. The progress of the reaction was monitored by TLC. The solid that separated from the cooled mixture was collected and recrystallized from a methanol-chloroform mixture.

Yield: 20%; m.p. 81 °C.

EI-MS m/z (rel. int.%): 509 (50) $[M+1]^+$.

IR (KBr) $\nu_{\max}$ cm^{-1} : 3053 (C-H_{aromatic}), 1681 (C=O), 1589 (C=C), 1147 (C-N).

^1H NMR (600 MHz, CDCl_3) δ : 8.17 (H-1, d, $J = 7.8$ Hz), 8.02 (H-2, d, $J = 8.4$ Hz), 7.25 (H-3, s), 7.55 (H-4, d, $J = 7.2$ Hz), 7.49 (H-5, dd, $J = 8.4$ Hz, 7.8 Hz), 7.34 (H-6, dd, $J = 7.2$ Hz, 1.2 Hz), 7.55 (H-7, dd, $J = 7.2$ Hz, 7.8 Hz), 8.65 (H-8, s), 4.43 ($\text{CH}_3\text{-CH}_2\text{-N}$, t, $J = 7.2$ Hz), 1.64 ($\text{CH}_3\text{-CH}_2\text{-N}$, q, $J = 7.2$ Hz).

^{13}C NMR (600 MHz, CDCl_3) δ : 201.74, 198.65, 191.89, 145.15, 143.55, 140.64, 140.33, 139.96, 137.96, 129.06, 128.43, 127.20, 126.72, 126.11, 125.88, 125.11, 124.37, 123.03, 122.84, 121.53, 120.98, 120.30, 119.68, 109.31, 108.94, 108.33, 58.49, 40.17, 37.93, 29.24, 27.35, 23.95, 13.84.

Anal. calc. for $\text{C}_{36}\text{H}_{32}\text{ON}_2$: C, 85.03, H, 6.29, N, 5.51. Found: C, 84.98, H, 6.24, N, 5.47.

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References and Notes

1. Asiri, A.M. Synthesis and characterization of dyes exemplified by 2-arylidene-1-dicyanomethyleneindane. *Dyes Pigm.* **1999**, 42, 209–213.
2. Rosso, V.; Loicp, J.; Renotte, Y.; Lion, Y. Optical non-linearity in Disperse Red 1 dye-doped sol gel. *J. Non-Cryst. Solids* **2004**, 342, 140–145.
3. Cotter, D.; Manning, R.J.; Blow, K.J.; Ellis, A.D.; Kelly, A.E.; Nesses, D. Nonlinear optics for high-speed digital information processing. *Science* **1999**, 286, 1523–1528.
4. Gaikwad, P. On the optical limiting and Z-scan of hexamethylin-dotricarbocyanine perchlorate dye. *Opt. Mater.* **2009**, 31, 1559–1563.
5. Tardivo, J.P.; Giglio, A.D.; Oliveir, C.S.; Gabrielli, D.S.; Junqueira, H.C.; Tada, D.B.; Severino, D.; Turchiello, R.F.; Baptist, M.S. Methylene blue in photodynamic therapy: From basic mechanisms to clinical applications. *Photodiagn. Photodyn. Ther.* **2005**, 2, 175–191.

6. Tonle, I.K.; Ngameni, E.; Tcheumi, H.L.; Tchieda, V.; Carteret, C.; Walcarius, A. Sorption of methylene blue on an organoclay bearing thiol groups and application to electrochemical sensing of the dye. *Talanta* **2008**, *74*, 489–497.
7. Prieto, I.; Fernandez, A.L.; Munoz, E.; Martin, M.T.; Camacho, L. Langmuir-Blodgett films containing water-soluble molecules: The methylene blue-dimyristoyl phosphatidic acid system. *Thin Solid Films* **2008**, *284–285*, 162–165.

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