



Synthesis, characterization and biological active 2-(dimethylamino)methylene)cyclohexane-1,3-dione

Veerappan Jeyachandran¹ Madasamy Kumar²

Abstract

In the present work, the precursors 2-((dimethylamino)methylene)cyclohexane-1,3-dione **4** and 2-((dimethylamino)methylene)-5,5-dimethylcyclohexane-1,3-dione **5** were synthesized from the reaction of 1,3-cyclohexanedione **1** or 5,5-dimethyl-1,3-cyclohexanedione **2** with DMF-DMA **3**, respectively following literature procedure.

Keywords

2-((dimethylamino)methylene)cyclohexane-1, 2-((dimethylamino)methylene)-5, 5-dimethyl-1,3-cyclohexanedione

^{1,2}Department of Chemistry, Bharath Institute of Higher Education and Research, Selaiyur, Chennai-600073, Tamil Nadu, India.

Article History: Received 01 October 2020; Accepted 10 December 2020

©2020 MJM.

Contents

1	Introduction	2114
2	General procedure for the synthesis of 4 or 5 ...	2114
	References	2115

1. Introduction

Pyrazoles are five member ring heterocyclic compounds, consisting of a doubly unsaturated five membered ring with two adjacent nitrogen atoms and are also called as azoles. These are aromatic molecules due to their planar conjugated ring structures with six delocalized π -electrons. [1] The term pyrazole was given by Ludwig Knorr in 1883. Being so composed and having pharmacological effects on humans, they are classified as alkaloids, although they are rare in nature. In 1959, the first natural pyrazole, β -[1-pyrazoly]alanine was isolated from the seeds of water melons [Citrullus lanatus]. [2] Literature survey has revealed that till 1930s very little had been done for the synthesis of steroidal pyrazole derivatives. Several pyrazole derivatives have been found to possess significant activities such as 5- α -reductase inhibitor, [3] antiproliferative, [4] antiparasitic [5] and herbicides [6]. pyrazolo[1,5-*a*]quinoline derivatives have been developed for dopamine D4 antagonist agents, [7] GPR109a agonist agents [8] and organic light-emitting devices. [9] However, besides these examples, the pyrazolo[1,5-*a*]quinoline subunit has not been applied to seek further biologically and materially active [10] compounds, which might be due to the lack of general methods for the synthesis of pyrazolo[1,5-*a*]quinoline derivatives [11].

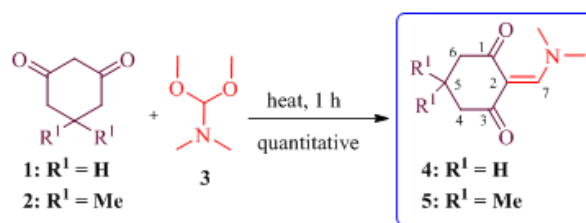
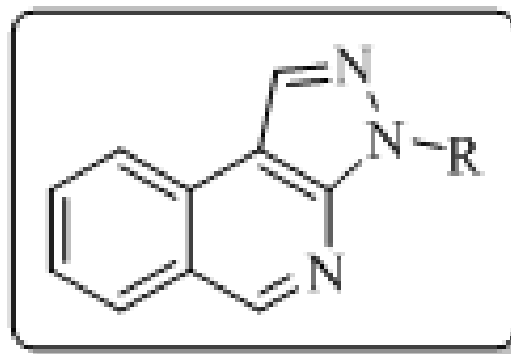


Figure 1. Synthesis of precursors 4, 5



2. General procedure for the synthesis of 4 or 5

A mixture of cyclic 1,3-diketone **1** or **2** (1 mmol) and N,N-dimethylformamide dimethyl acetal **3** (1.5 mmol) was heated to reflux. After 1 hour of continuous reflux the solvent was removed under reduced pressure. The reaction mass upon

cooling to room temperature afforded 4 or 5 as brown crystals, which was used for next step without further purification.

2-((Dimethylamino)methylene)cyclohexane-1,3-dione Brown crystals; Yield 99%; m.p. 85–86°C; ^1H NMR (300 MHz, CDCl_3) δ_{H} : 1.95 (qui, $J = 6.4$ Hz, 2H), 2.47 (t, $J = 6.5$ Hz, 4H), 3.18 (s, 3H), 3.40 (s, 3H), 8.06 (s, 1H) ppm; ^{13}C NMR (75 MHz, CDCl_3) δ_{C} : 19.3, 37.9, 44.4, 48.3, 109.1, 162.0, 195.9 ppm.

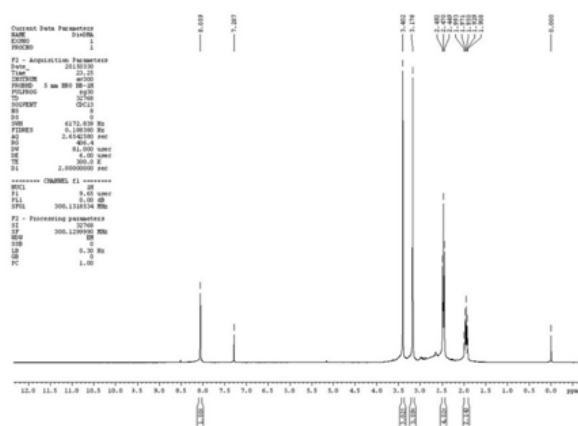


Figure 2. ^1H NMR spectrum of 4

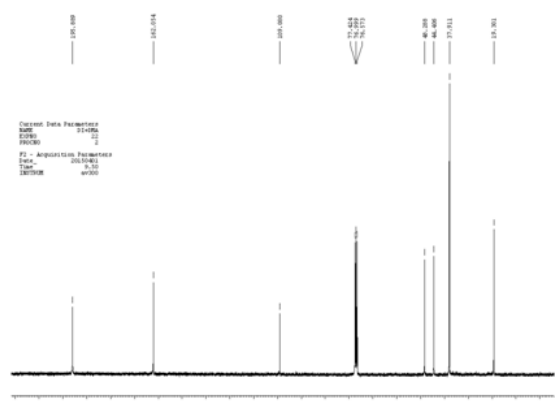


Figure 3. ^{13}C NMR spectrum of 4

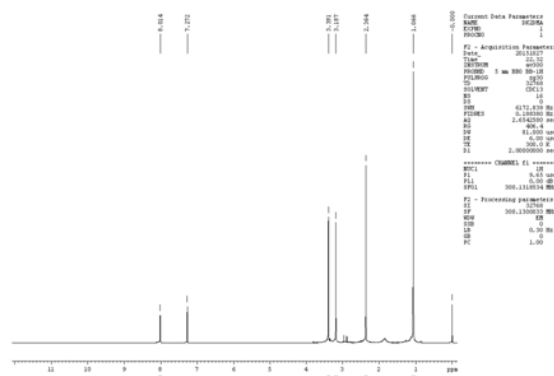


Figure 4. ^1H NMR spectrum of 5

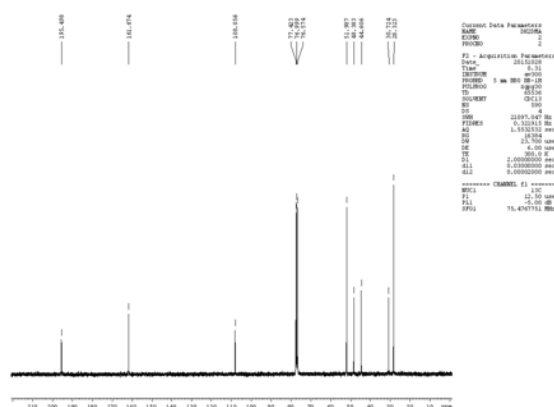


Figure 5. ^{13}C NMR spectrum of 5

- [6] T.A. Henry, *The Plant Alkaloids, Anmol Publication Pvt. Ltd.*, (1999)
- [7] I. B. Dzvinchuk, N. A. Tolmachova, A. N. Chernega, M. O. Lozinskii, *Chem. Heterocycl. Compd.*, 45 (2009) 194.
- [8] R. B. Toche, R. B Bhavsar, M. A. Kazi, S. M. Bagul, M. N. Jachak, *J. Heterocycl. Chem.*, 47 (2010) 287
- [9] Wu. Liqiang, L. Yang, F. Yan, C. Yang, L.Fang, *Bull. Korean Chem. Soc.*, 31 (2010) 1051.
- [10] Wu. Liqiang, Ma. Suying, F.Yan, C. Yang, *Monatsh. Chem.*, 141 (2010) 565.
- [11] B. Jiang, G.Zhang, Ma. Ning, F. Shi, S. J. Tu, P. Kaur, G. Li, *Org. Biomol. Chem.*, 9 (2011) 3834

References

- [1] T. Eicher, S. Hauptmann, *Edition IPnd Wiley-VCH*, ISBN 3527307206 (2003).
- [2] S. Nimbalkar, S. V. Hote, *Int. J. Rec. Innovation Trends in Comp. Comm.*, 3 (2015) 61.
- [3] A. G. Amr, N. A. Abdel-Latif, M. M. Abdlla, *Acta Pharm.*, 56 (2006) 1203.
- [4] S. Chimichi, M. Boccacini, M.M.M. Hassan, G. Viola, F. D. Acqua, M. Curini, *Tetrahedron*, 62 (2006) 90.
- [5] Kobayashi, Hisafumi, Kato, Motto, Nitani, Fumio, *Chem.Abstr.*, 106 (1989) 297008/g.

 ISSN(P):2319 – 3786
 Malaya Journal of Matematik
 ISSN(O):2321 – 5666

