## [E0011]

# Synthesis of 1, 1'-(1, 4- butanediyl)bis(imidazole) under microwave irradiation

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**Abstract:** The compound, 1, 1'-(1,4- butanediyl)bis(imidazole), has been synthesized by the reaction between imidazole, NaOH, and 1,4-dichlorobutane, under a fast and efficient method microwave irradiation. The title compound has been characterized by spectral methods (<sup>1</sup>H-, <sup>13</sup>C NMR and IR spectroscopy).

Keywords: Imidazole, 1, 4-dichlorobutane, Microwave, Spectral methods.

## Introduction

In recent years many rational synthetic strategies have been proposed for the design of metal-based coordination. Most of these structures have relatively large voids and potential interest for host–guest interaction and molecular recognition [1]. Bis(imidazole) ligands bearing alkyl spacers are a good choice of N-donor ligand, and the flexible nature of spacers allows the ligands to bend and rotate when coordinating to metal centers so as to conform to the coordination geometries of metal ions [2-7]. Based on its structure, 1, 1'-(1, 4-butanediyl) bis(imidazole) can be used as a flexible divergent ligand to fabricate coordination polymer materials [8]. The use of microwave irradiation has been employed for a number of organic syntheses to reduce the reaction time, rate enhancement and to increase the selectivity and yields [9-11]. We now to report the synthesis of 1, 1'-(1, 4-butanediyl)bis(imidazole) through the reaction of imidazole, NaOH, and 1,4-dichlorobutane conditions using microwave irradiation.



#### **Results and Discussion**

In the IR spectrum, the characteristic C=N stretching frequency is formed in the region between  $v=1640-1690 \ cm^{-1}$  as a doublet band. The C-N stretching frequency is found in the region between  $v=1100-1230 \ cm^{-1}$ . The absorption bands at  $v=3400 \ cm^{-1}$  are attributed to N-H stretching vibrations. In the <sup>1</sup>H NMR spectrum, there is a doublet peak at  $\delta=1.59 \ ppm$  for CH<sub>2</sub> (5). Also, an observed doublet peak at  $\delta=3.92 \ ppm$  is related to CH<sub>2</sub>(4). A doublet resonance at  $\delta=6-7 \ ppm$  is assigned to the proton of the CH-N(3). Singlet signals can be observed at  $\delta=7.11$  and 7.58 ppm for CH-N(2) and CH-N(1), respectively.

There are five signals in <sup>13</sup>CNMR spectrum. The peak at  $\delta$ =27.66 *ppm* and  $\delta$ =45.28 *ppm* could be assigned to the C<sup>5</sup> and C<sup>4</sup>. The signals of C<sup>3</sup>, C<sup>2</sup>, C<sup>1</sup> appear at  $\delta$ =119.21, 128.39, 137.19 *ppm*, respectively. Based on the presented <sup>1</sup>HNMR, <sup>13</sup>CNMR and IR spectroscopy for this compound, structure can be suggested as shown in scheme 1.



*Scheme1. The structure of 1, 1'-(1, 4- butanediyl)bis(imidazole)* 

### **Experimental Section**

A mixture of imidazole (1.7g, 25mmol), NaOH (1.0g, 25mmol) and 1, 4-dichlorobutane (1.6g, 12.5mmol) in DMSO (5mL) was stirred and then irradiated at 100W for 3 min. After this time, the result white powder was produced (scheme1). mp. 81-83°C. The progress of the reaction was monitored by TLC.

**IR(KBr,** *cm*<sup>-1</sup>): 3441(w), 3124(w), 2940(w), 2983(w), 2357(m), 1671(m), 1651(m), 1378(m), 1281(m), 1108(m), 1083(s), 826(m), 784(s), 731(s), 663(s).

<sup>1</sup>**HNMR (DMSO-d<sub>6</sub>**, *ppm*) δ**H**: 1.59 (d, 4H, CH<sub>2</sub>(5)), 3.92 (d, 4H, CH<sub>2</sub>(4)), 6.86 (d, 1H, CH-N(3)), 7.11 (s, 1H, CH-N(1)), 7.58 (d, 1H, CH-N(2)).

<sup>13</sup>CNMR (DMSO-d<sub>6</sub>, *ppm*) δC: 27.66 (C<sup>5</sup>), 45.28(C<sup>4</sup>), 119.21 (C<sup>3</sup>), 128.39 (C<sup>2</sup>), 137.19 (C<sup>1</sup>).

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#### References

- Ma, J.-F., Yang, J., Zheng, G. L., Li, L., Zhang, Y. M., Li, F. F., Liu, J. F., Polyhedron, 23, 2004, 553.
- 2. Hoskins, B.F., Robson, R., Slizys, D.A., J. Am. Chem. Soc., 119, 1997, 2952.
- 3. Gao, E. Q., Xu, Y. X., Yan, C. H., Cryst. Eng. Comm., 6, 2004, 298.
- 4. Fan, J., Slebodnick, C., Angel, R., Hanson, B.E., Inorg. Chem., 44, 2005, 552.
- Zhang, L., Lü, X. Q., Chen, C. L., Tan, H. Y., Zhang, H. X., Kang, B. S., Cryst. Growth Des., 5, 2005, 283.
- 6. Li, X., Cao, R., Bi, W., Wang, Y., Wang, Y., Li, X., Guo, Z., Cryst. Growth Des., 5, 2005, 1651.
- 7. Cui, G. H., Li, J. R., Tian, J. L., Bu, X. H., Batten, S.R., Cryst. Growth Des., 5, 2005, 1775.
- 8.(a) Ma, J. F., Liu, J. F., Xing, Y., Jia, H. Q., Lin, Y. H., *J. Chem.Soc., Dalton Trans.*, 2000, 2403;
  (b) Ma, J. F., Liu, J. F., Liu, Y. C., Xing, Y., Jia, H. Q., Lin, Y. H., *New J. Chem.*, 2000, 24, 759.
- 9. Singh, V. Singh, J., Kaur, K.P., Kad, G.L., J. Chem. Res., (S), 1997, 58.
- Manhas, M.S., Ganguly, S.N., Mukherjee, S., Jain, A.K., Bose, A.K., *Tetrahedron Lett.*, 47, 2006, 2423.
- 11. Singh, V., Kaur, S., Sapehiyia, V., Singh, J., Kad, G.L., Catal. Commun., 6, 2005, 57.

