RESEARCH PAPER | Chemistry | Volume : 5 | Issue : 4 | April 2015 | ISSN - 2249-555X

Green Synthesis of Benzoin Derivatives Using Coenzyme Catalyzed Benzion Condensation Reaction.

KEYWORDS: Green Chemistry, Benzoin condensation

*** Anandrao Kale**

Post Graduate Department of Chemistry, S.M.Joshi College, Hadapsar. Dist-Pune. * Corresponding author

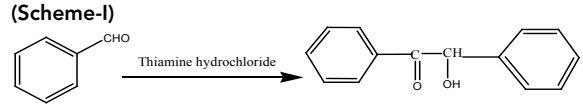
ABSTRACT *The new trends of Green Chemistry is an invention, design, development and application of chemical products and processes to reduce or to eliminate the use and generation of substances hazardous to human health and environment. The Benzoin and its derivatives are synthesized by conventional method by use of sodium cyanide and ethanol with strong heating for the longer time which is found to hazardous and slower conversion An alternative facile route of green approach, eco-friendly and solvent-free reaction procedure with very simple workup conditions is needed for organic synthesis. Present work is carried out for the synthesis of Benzoin and its derivatives by coenzyme catalyzed benzion condensation reaction. The synthesis of ligands benzoin and its substituted derivatives includes three component coupling reaction of benzaldehyde, ethanol and sodium hydroxide. The ice cold clear solution of thiamine hydrochloride in aqueous ethanol is taken in a 50 ml round bottom flask. To this ice cooled freshly prepared (15 ml) sodium hydroxide solution is added. Then Fresh bidistilled benzaldehyde (10 ml) or substituted benzaldehyde was added to the reaction mixture. The mixture was heated gently on a water bath for about 60 min. The mixture was cooled to room temperature and then in ice bath to induce crystallization of the benzoin. The yield is 45% and melting point is found to be 1350C. The ligands obtained is recrystallized; and further characterized by modern spectral methods. The systematic path of the reaction and mechanism is shown in the Scheme-I. The peculiarity of the reaction is rapid conversion, solvent free; no use of catalyst and hazardous chemicals in the synthesis hence, it is eco-friendly green chemistry synthesis.*

Introduction:

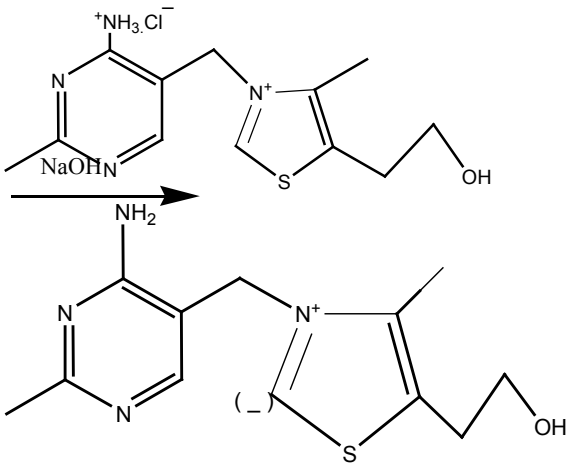
The Green Chemistry approaches is an eco-friendly green synthesis, design, development and application of chemical products and processes modifications to minimize the use and hazardous solvent, reagents and catalysts causing environmental pollutions. It is better to prevent waste than to treat or clean up waste after it is formed. Synthetic methods should be designed to maximize the incorporation of all materials used in the process into the final product. Wherever practicable, synthetic methodologies should be designed to use and generate a substance that poses little or no toxicity to human health and the environment. Chemical products should be designed to preserve efficacy of function while reducing toxicity. The use of auxiliary substances (e.g. solvents, separation agents etc.) should be made unnecessary wherever possible and, innocuous when used. Energy requirements should be recognized for their environmental and economic impacts and should be minimized. Synthetic methods should be conducted at ambient temperature and pressure. A raw material feedstock should be renewable rather than depleting whenever technically and economically practical. Unnecessary derivatization (blocking group, protection/deprotection, and temporary modification of physical/chemical processes) should be avoided whenever possible. Catalytic reagent are superior to stoichiometric reagents. Chemical products should be designed so that at the end of their function they do not persist in the environment and break down into innocuous degradation products. Analytical methodologies need to be further developed to allow for real-time in-process monitoring and control prior to the formation of hazardous substances. Substances and the forms of the substance used in chemical reaction should be chosen so as to minimize the potential of chemical accidents, including releases, explosions, and fires. Experiments should involve the use of alternative reagents which are not only eco-friendly but also be easily available anywhere in the country in bulk quantities at very cheap price. They should not preferably involve the use of organic solvents (like ether, petroleum ether or ethyl acetate); ethanol and methanol are mostly preferred. Modified Experiments, if possible should not involve sophisticated instrumentation techniques like high-pressure system, evacuated system, inert atmosphere using argon, etc. Experiments should avoid tedious experimental procedure like longer reaction time, reaction at high temperature etc. All organic chemistry experiments (preparation, separation of mixture of compounds, identification of functional groups etc.) should preferably be conducted in semi-micro or micro-scale.Present work is carried out for the green synthesis of Benzoin its derivatives Benzoin condensation reactions.

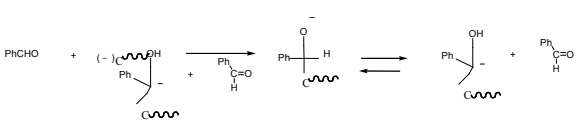
Experimental: The extra pure thiamine hydrochloride (1.75 g) was dissolved in water (about 5 ml) in a 50 ml round bottom flask. Ethanol (95%, 15 ml) was added and the solution was cooled by swirling the flask in an ice water bath. Sodium hydroxide ice cooled solution (5 ml) was added drop wise to the thiamine solution. Fresh bidistilled benzaldehyde (10 ml) was added to the reaction mixture. The mixture was heated gently on a water bath for about 60 min until it was become homogeneous. The mixture was cooled to room temperature and then in ice bath to induce crystallization of the benzoin. The yield is 45% and melting point is found to be 1350C . The ligands obtained is recrystallized; and further characterized by modern IR, NMR, CMR, etc spectral methods.

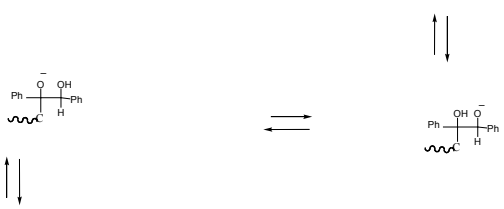
(Scheme-I)

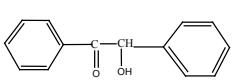


Volume : 5 | Issue : 4 | April 2015 | ISSN - 2249-555X



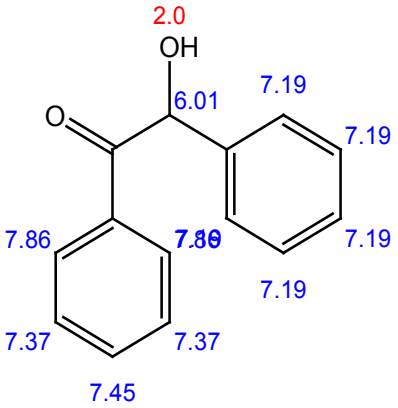






1HNMR

13C NMR



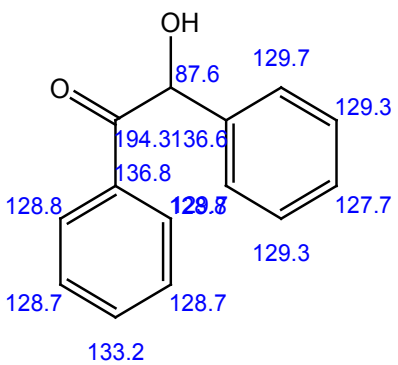


Fig-1

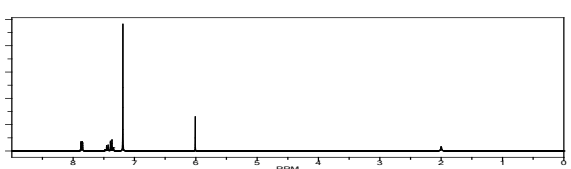
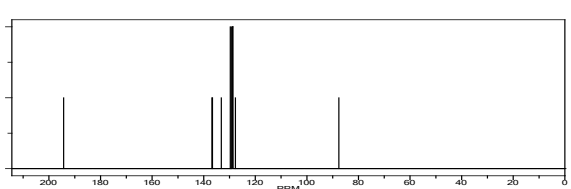


Fig-2



Node	Chemical shift δ in ppm	Node	Chemical shift δ in ppm
CH	6.01	CH	87.6
OH	2.0	C	194.3
CH	7.19	C	136.6
CH	7.19	CH	129.7
CH	7.19	CH	129.3
CH	7.19	CH	127.7
CH	7.81	CH	129.3
CH	7.86	CH	129.7
CH	7.36	C	136.8
CH	7.45	CH	128.8
CH	7.37	CH	128.7
CH	7.86	CH	133.2
CH		CH	128.7
CH		CH	128.8

Volume : 5 | Issue : 4 | April 2015 | ISSN - 2249-555X

Conclusions:

The green procedure indicated in the (Scheme –I) is very good low cost an alternative eco-friendly method. This includes the no use of non green component highly poisonous Sodium cyanide, no use of hazardous organic solvents, no requirement of costly catalyst and the faster conversion with precisely good yield of reaction than the conventional method. The caution of the reaction is that benzaldehyde used in the experiment should be free of benzoic acid Thiamine hydrochloride should be kept in refrigerator when it is not in use. Green chemistry experiments are introduced not to drastically replace the conventional ones rather, they are considered complementary to the existing protocols. This not only provides a wider view of various techniques but also imbibes inquest in innovative minds for future development and growth of the subject in general with due emphasis to green chemistry context. The systematic path of reaction and the method of the green synthesis of Benzoin and its derivatives by Benzoin condensation is shown (Scheme-I). The yield of reactions 45% and the melting point of the product is 1350C The Peculiarity of the reaction is Hazardous and poisonous cyanide ion is replaced by thiamine hydrochloride. Reaction is effected at a lower temperature. rapid conversion, solvent free; no use of catalyst and hazardous chemicals in the synthesis follows eco-friendly green chemistry path of synthesis. The product obtained and the substances used in chemical reaction should minimize the potential of chemical accidents, including releases, fires, and explosions.

REFERENCE P., D. L. Lampman and G.M.Chriz, Introduction to Organic Lab Technique; College Publishing, New York, 1982, 47 | K. Pitchumani, C. Venkatachalapathi and S. Sivasubramanian, Indian J. Chem., 1997, 36B, 187. | B. C. Ranu, A. Hajra and S. S. Dey, Org. Proc. Res. Dev. 2002, 6, 817 | K. Pitchumani, C. Venkatachalapathi and S. Sivasubramanian, Indian J. Chem., 1997, 36B, 187. | S. M. Reed and J. E. Hutchison, Journal of Chemical Education, 2000, 77, 1627. | P. Anastas and J. C. Warner, Green Chemistry: Theory and Practice; Oxford Science Publications, Oxford, 1998 | S. Bhagat, R. Sharma, and A.K. Chakraborti, J. Mol. Cat. A: Chemical 2006, 260, 235-240. | 14 | A.K. Bose,* S. N. Ganguly, M. S. Manhas, S. Rao, J. Speck,U. Pekelny and E. Pombo-Villars, Tetrahedron Lett. , 2006, 47, 1885. | W. E. Bachmann, J. Am. Chem. Soc. 1927,49,246 Org. Synth., 1943, Coll. Vol 2, p. 73 |