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Microwave assisted synthesis: A green chemistry approach

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Abstract*:Green Chemistry with its twelve principles would like to see changes in the conventional chemical synthesis and the use of less toxic starting materials. Green Chemistry would like to increases the efficiency of synthetic methods, to use less toxic solvents, reduce the stages of the synthetic routes and minimize waste as far as practically possible. Green chemistry applies across the life cycle of a chemical product, including its design, manufacture, and use. Green chemistry technologies provide a number of benefits, including reduced use of energy and resources, reduced waste, eliminating costly end-of-the-pipe treatments, safer products improved competitiveness of chemical manufacturers and their customers. Looking at the definition of green chemistry, the first thing one sees is the concept of invention and design. In many ways, this is a wonderful time in history to be a chemist or chemical engineer. The challenges that confront the planet are nothing short of the most consequential that humanity has ever faced. Simply put, if we do not meet the challenges of maintaining the viability of the biosphere, humanity will not survive to see any future challenges. Eco-friendly technology is used to produce environment friendly products by using renewable resources. This article is an attempt to provide the basic concept of eco-friendly technology. In this way, chemical synthesis will be part of the effort for sustainable development. Microwave assisted synthesis has revolutionized chemical synthesis. Small molecules can be built in a fraction of the time required by conventional methods. Microwave-assisted synthesis is rapidly becoming the method of choice in modern chemical synthesis and drug discovery. The present article will highlight the applications of microwave-assisted synthesis in organic molecules involved in microwave heating.*

Key words:*sustainable chemistry, microwave chemistry.*

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Introduction

The last decade has seen a huge interest in sustainable, green and organic chemistry[1], particularly as chemical educators look to "green" their undergraduate curricula. In detailing published laboratory experiments and proven case studies, this book discusses concrete examples of green organic chemistry teaching approaches from both lecture/seminar and practical perspectives. The experienced contributors address such topics as the elimination of solvents in the organic laboratory, organic reactions under aqueous conditions, organic reactions in non-aqueous media, greener organic reagents, waste management/recycling strategies, and microwave technology as a greener heating tool. This reference allows instructors to directly incorporate material presented in the text into their courses.

The principles of Green Chemistrycan be particularly useful in addressing these major challenges through the design of next-generation products and processes. Monowave by CEM Company is a high performance microwave reactor (**figure 1& 2**) specially designed for small scale microwave synthesis applications in research and development laboratories. Microwave irradiation nowadays is not only successfully employed for organic synthesis - inorganic synthesis, material science, polymer chemistry, and other disciplines can also be performed successfully. The MAS 24 Auto sampler option allows for unattended sequential processing of 24 experiments.

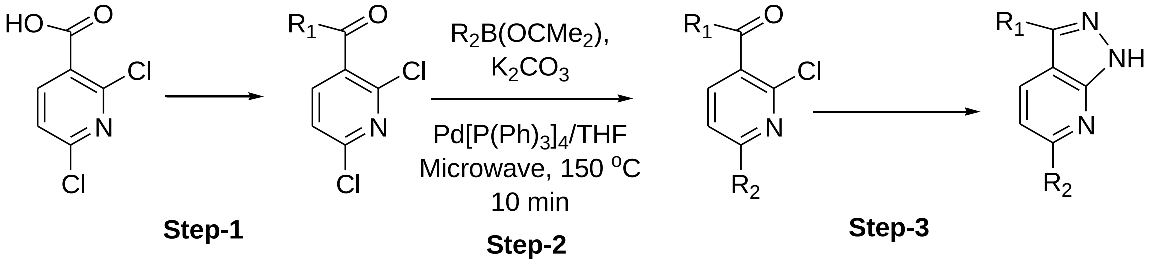
General benefits of microwave synthesis

- New reaction pathways
- Significant rate enhancement
- Reduced overall process time
- Increased yield
- Improved product purity, less by-products
- High reproducibility
- Software-controlled processing

Green chemistry is a welcome step towards protecting the mother earth. The green chemistry revolution provides an enormous number of opportunities to discover and apply new synthetic approaches using alternatives feedstocks, eco-friendly reaction conditions, energy minimizations and the design of less toxic and inherently safer chemicals. The origin and basis of green chemistry for achieving environmental and economic prosperity is inherent in sustainable world. Green chemistry is not different from traditional chemistry in as much as it embraces the same creativity and innovation that has always been central to classical chemistry. However, there lies a difference in that historically synthetic chemists have not been seen to rank the environment consciousness throughout the world there is a change for chemists to develop new products, processes and services that achieve necessary social, economical and environmental objectives. Dr. B. C. Ranu[2] and his group have been working on green chemistry using microwave process. The use of microwave irradiation in organic synthesis has become an increasingly popular topic within the pharmaceutical and academic research arenas. It has proven to be a safe and clean means of performing reactions very rapidly, however, has not been fully explored within a teaching environment. Herein, we describe the use of microwave energy to accelerate reactions typically encountered in an undergraduate teaching laboratory, as well as a few that present more interesting challenges and educational opportunities for the undergraduate student. Each of the reactions has proven to be faster, cleaner, and very interesting to students, serving not only to challenge, but also to more fully engage students in the laboratory. The use of emerging microwave (MW)-assisted chemistry techniques is dramatically reducing chemical waste and reaction times in several organic syntheses and chemical transformations (**figure 3**). According to the work carried out by William G Bornmann[3]and his co-worker including myself have made a series of c-Kit compounds using microwave technology as a greener heating tool. Structural or chemical Diversity was first introduced at the beginning of the synthesis with the condensation of 2,6-dichloro nicotinic acid chloride with various zinc halides (e.g., bromide or iodide) in presence oftetrakis(triphenylphosphine)palladium in THF to give first series of starting materials. The second round of structural or chemical diversity was introduced by the condensation of first series compounds with various boronate esters by means of a Suzuki coupling using microwave conditions at 150^{o}C for 10 min produced the next series of compound at a time 24 compound by the help of CEM microwave reactor. The whole Microwave process takes 5 min per each reaction and yield is more than 25 percentages while in normal process the reaction takes three days, with more solvents and yields are less than 15 percentages.

General Synthetic Methods For Preparing Compounds

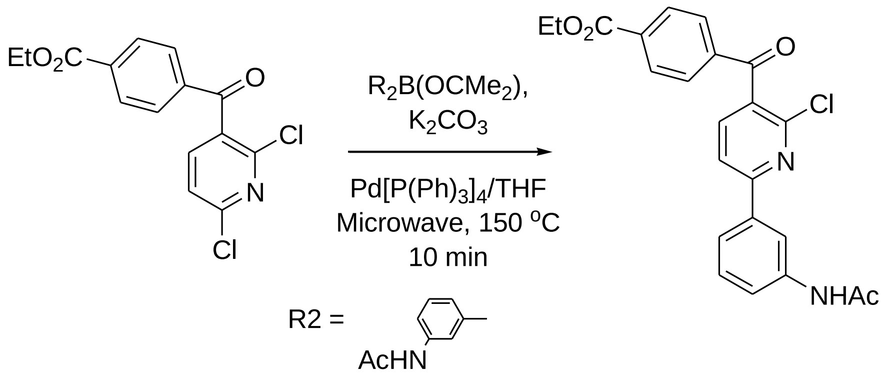
The following scheme can be used to practice the library of compounds using R_1 and R_2 as diverse aryl groups



R_1 = Different substiruted aryl groups
R_2 = Different substiruted aryl groups

Figure 3 GENERAL SYNTHETIC METHODS FOR PREPARING PYRAZOLO COMPOUNDS

Illustrative of Scheme 1 is the synthesis of 3-[6-chloro-5-{4-ethyoxycarbonyl)carbonyl}-2-pyridinyl]acetanilide.



A mixture of (2,6-dichloro-3-pyridinyl)(4-ethyloxycarbonyll)methanone (300 mg, 0.93mmol), 3'-(4,4,5,5-tetramethyl-1,3-2-dioxaborolan-2-yl)acetanilide, potassium carbonate (200 mg), tetrakis(triphenylphosphine)palladium (80 mg) in THF (4 mL) was finished with argon and then subjected to microwave at 155 oC for 10 min. The whole product was adsorbed onto silica, then purified by flash column chromatography over silica gel, using polarity gradient 5-50% EtOAc in hexane to yield acetanilide derivative (200 mg, 49%) as a yellow oil; ^{1}H NMR (600 MHz DMSO-d_6) δ 10.17 (s, 1H), 8.32 (s, 1H), 7.81(m, 4H), 7.47 (t, 1H, J = 7.8 Hz), 7.10 (d, 2H, J = 9.0 Hz), 4.36(q, 2H, J =7.2 Hz), 2.08 (s, 3H), 1.36 (t, 3H, J = 7.2 Hz)^{13}C NMR δ 191.3, 168.5, 164.2, 157.4, 145.9, 140.1, 139.5, 136.8, 133.1, 132.3, 129.5, 128.4, 121.5, 120.8, 119.2, 117.3, 114.5,60.8, 55.7, 14.2; MS ($C_{23}H_{19}ClN_2O_4$) calcd. 422.103 found MH^+423.2.

Microwave synthesis is an important technique for facilitating drug discovery, polymer science, chemical and materials research. It reduces reaction times from days to minutes, increases yields, and in many cases, produces cleaner reactions. It allows access to new reaction schemes, which are not possible using conventional heating.

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