A PROJECT REPORT ON

Environmentally Benign Acetylation Reactions

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CERTIFICATE

This is certified that the project report title"Environmentally Benign Acetylation Reactions" completed by B.GOWTHAMI B.AKHIKA A.SOUMYA B.SOUNDARYA…….. under the guidance of Dr. E. Anitha in Chemistry. This has not been submitted to any other institute or University for the award of any degree.

Signature of the Guide

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Environmentally Benign Acetylation Reactions

Aim of the project: To practice the Ecofriendly processes in Organic laboratory.

Objectives: To create the awareness among the students about:

(i) The impotance of Green protocols in organic synthesis.

(ii) The Environmentally benign processes to reduce or eliminate the chemical pollutants during the synthesis.

(iii) To assess the effect of microwave irradiation against thermal heat on the Acetylation reactions.

Keywords:Microwave Radiation, Green Chemistry, Aspirin, Acetanilide.

Introduction:

Microwave-assisted organic synthesis (MAOS) has become an integrant part of environmentally-friendly ("green") chemistry from the last three decades. MAOS was demonstrated for the first time in 1986 by Gedye et al.Synthetic organic reactions performed using Microwave conditions are gaining popularity, primarily to circumvent growing environmental concerns since they are eco-friendly. MAOS eliminates the use of excess solventand moreover offer high yields together with simplicity in processing and handling. Many syntheses take hours under normal heating condition. A microwave emits oscillating magnetic field, cause more interaction between molecules to boost the rate of reaction and allowed to proceed at a fraction of the time withhigh yield.

Now day's this technique is considered as an important approach toward green chemistry, because this technique is more environmentally friendly. Conventional method of organic synthesis usually needslonger heating time, tedious apparatus setup, which result in higher cost of process and the excessive use of solvents/reagents lead to environmental pollution. This growth of green chemistry holds significant potential for a reduction of the by product, a reduction in waste production and a lowering of the energy costs. Acetylation is a process in which an acetyl group is replaced the hydrogen atom during the chemical reaction. The ability of acetyl groups to easily protect a variety of functional groups, such as alcohols, amines, phenols, and thiols, among others, makes acetylation one of the most significant processes in chemical synthesis.

Microwave assisted synthesis of Aspirin

Aim: To prepare the Aspirin by Ecofriendly processes in laboratory.

Objectives:

(i) To reduce the chemical pollutants by avoiding hazardous anhydride.

ii) To reduce the cost of Aspirine by replacing expensive aceticanhydride by acetic acid.

iii) To reduce the reaction time and to boost the better yields.

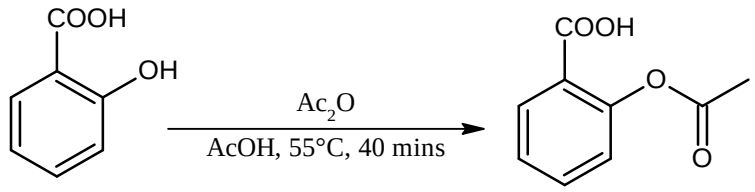
Keywords: Aspirin, Microwaves

Materials: Salicylic acid,glacial acetic acid and acetic anhydride.

Introduction: Aspirin, the Wonder Drug, is an important chemical world-wide, this chemical regulates certain body functions, such as blood vessel elasticity, lowers the risk of forming blood clots in arteries and decreasing the risk of heart attack and also changes the functions of blood platelets. Thus, can aspirin affect blood clotting and ease inflammation and also can be used as a painkiller. As well as being a blood thinner, aspirin is an analgesic that is used to reduce swelling.

Synthesis of Aspirin in Conventional Method:

In conventional method, Aspirin is prepared by acetylation of salicylic acid using acetic anhydride & glacial acetic acid by heating at 55 °C for 40 minutes and the yield of reaction is 70-72%.



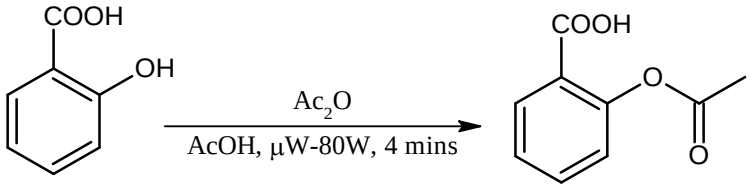
Procedure: Prepare a mixture of 5 ml of each acetic anhydride (0.05 mol)& glacial acetic acid in 100 ml clean and dry beaker. Now add this mixture carefully to 3 gm salicylic acid (0.02 mol) previously weighed &placed in 100 ml round bottom flask & fit the same with a reflux condenser.Boil the reaction mixture at 55 ^{0}C on water bath for duration of 40 min.Pour the hot resulting mixture directly into 100 ml of cold water containing in 500 ml beaker in one lot and stir the content vigorously with a glass rod when the shining tiny crystals of aspirin separate out.Filter off crude &wash the residue with sufficient cold

water, drain well and finally remove the excess water by pressing it between the air to allow it dry completely and recrystallized from ethanol.

The yield of the reaction is 72% (2.82 g of aspirin) and melting point of aspirin is recorded and matched with reported value 136 °C.

Synthesis of Aspirin using Microwave Method:

In Microwave method, Aspirin is prepared by acetylation of salicylic acid using acetic anhydride & glacial acetic acid by irradiating with microwaves at 80 Watts for 3 minutes and the yield of reaction is 88%.



Procedure: Prepare a mixture of 2.5 ml of each acetic anhydride (0.025 mol)& glacial acetic acid in 20 ml clean and dry beaker. Now add this mixture carefully to 3 gm salicylic acid (0.0167 mol) previously weighed &placed in 100 ml flat bottom Borosilflask. Place the flask in domestic Microwave woven, 80W and mixture is irradiated with for 3 mins. Ensure the completion of reaction by TLC. Pour the hot resulting mixture directly into 100 ml of cold water containing in 500 ml beaker in one lot and stir the content vigorously with a glass rod when the shining tiny crystals of aspirin separate out.Filter off crude &wash the residue with sufficient cold water, drain well and finally remove the excess water by pressing it between the air to allow it dry completely and recrystallized from ethanol.

The yield of the reaction is 88% (3.44 g of aspirin) and melting point of aspirin is recorded and matched with reported value 136 °C.

Discussion: Under the conventional condition, salicylic acid and acetic anhydride are taken in 1:2.5 ratio and heated at 55 °C for 40 min. In microwave procedure , salicylic acid and acetic anhydride are taken in 1:1.5 ratio and reaction was completed in 3 min. at 80Watts in the microwave, shorting the reaction time 3 min. Using conventional heating aspirin was isolated with a yield of 72 % while the shorter microwave method resulted in a comparable 88 % yield.

Conclusion: In conclusion Microwave method is an efficient than conventional method in terms of yields and time.