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Solvent and Catalyst Free Acylation of Anilines with Acetic Acid

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Abstract: An efficient and green microwave assisted protocol to prepare amide fromamine via acylation without using catalyst provides quantitative yields in short reaction times. Synthesis of Acetanilide under microwave irradiation was described, in which Aniline was directly reacted with glacial acetic acid without any catalytic agents. The reaction took place in 40−50 min in the frequency range of 160 MHz, with almost quantitative yields. By applying green synthesis method, we avoid use of any hazardous or toxic materials. Quantitative yields are produced in a short amount of time by an effective and environmentally friendly microwave assisted approach that produces amide from amine via acylation without the need for a catalyst. Aniline and glacial acetic acid were directly combined during the synthesis of acetanilide under microwave irradiation, without the use of anycatalytic agent.The reaction occurs in the frequencybond of 160 MHz in 40 - 50 min. with almost quantitative yields. We don't employ any harmful or toxic components by using the Green synthesis techniques.

Keywords: Acylation,microwaveirradiation,amide,amine,aniline,acetanilide,greensynthesis.

I. INTRODUCTION

Green Chemistry is the new branch of chemistry which involves pulling together tools, techniques and technologies. It is helpful to chemists and chemical engineers in research, development and production for development of more eco-friendly and efficient products, which may also have significant financial benefits. It is now going to become an essential tool in the field of synthetic chemistry. [3,8] The development of Green Chemistry redefines the role of a solvent. "An ideal solvent facilitates the mass transfer but does not dissolve". In addition, a desirable Green Solvent should be natural, nontoxic, cheap and readily available with additional benefits of aiding the reaction, separation or catalyst recycling. Of various principles of Green Chemistry, the important one is maximizing the Atom Economy which evaluates the efficiency of chemical transformation [9].Amide compounds have attracted much attention because of their extensive application in the pharmacological industry. They have been generally used as protective routes for synthetic routes. An Amide functional group is contained in many bioactive compounds or drugs, an Amide modification is an important optimization methodology for new chemical entities in medicinal Chemistry. Furthermore, peptide bonds, a special kind of Amide bond in life sciences, constitute the back bone of proteins. In the chemical industry, the structure of Amides is involved in many important materials [10]

The formation of Amide bond is generally developed in these ways: the traditional synthetic procedures of the acetamido group compounds are carried out by primary amino compounds with acyl halides, acid anhydrides, esters or acids in different conditions. However all these procedures have usage limitations: the acyl halides and acid anhydrides were hygroscopic and many active acylation agents would be exothermic and produce unwanted acid during the reaction process, so they should be treated carefully under well-cooled and anhydrous conditions in the presence of acid-tricing agents [2]. The esters and acids were low chemically active acylation agents, so they would take longer time for the reaction process with low yields. Some chemical active agents such as N,N'- dicyclohexylcarbodiimide (DCC), N-(3-dimethylaminopropyl)-N-ethylcarbodiimide (EDC), and these should be added to catalyze the reactions because they are harmful and hazardous to body and environment. [13]

Amide bond formations are one of the most important transformations carried out in pharmaceutical synthesis, accounting for 65% of all preliminary screening reactions in industrial medicinal chemistry laboratories as recently reported. Amides also represent very important family of intermediates widely employed in the preparation of fine

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chemicals, cosmetics and food additives.[14] However, the majority of the current employed protocols to form Amides involve the use of stoichiometric activated, toxic and corrosive agents such as acid anhydrides or acyl chlorides with poor atom economy that generate considerable waste. Furthermore, an excess of these reagents is normally needed to achieve optimum amide yields and the reaction is water sensitive with the efficient removal of water being a critical factor in the systems [1].

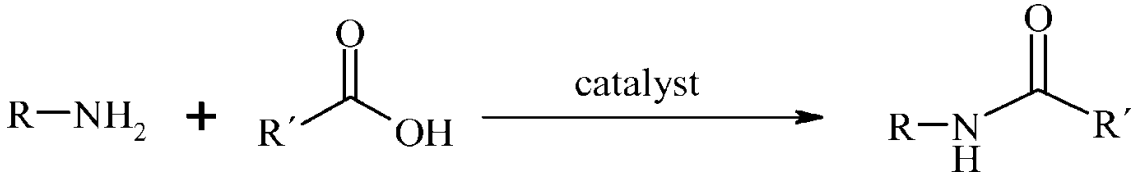
The development of cleaner syntheses is key to reducing the environmental impact of amide formations. In this regard, the direct reaction of Amines with carboxylic acids remains the most attractive approach. These types of reactions are best carried out without catalysis where possible, however, reaction conditions remain harsh in order to accomplish direct Amide formation [1,7]

The acylation of amines is one of the most frequently used transformations in organic synthesis as it provides an efficient and inexpensive means for protecting hydroxyl and amino groups in a multistep synthesis process. For the amide formation mostly acetyl chloride and acetic anhydride are routinely used in the presence of an acidic or base catalyst. However, both of these reagents are corrosive and are not always ideal to use. Moreover, the acidic condition in Lewis acid acylation of amines leads to the cleavage of sensitive functional groups such as acetals and TBDMS ethers. Sc(OTf)3, TMSOTf and clays such as K-10, KSF were reported for acylation of amines with acetic anhydride. Tetrabutylammonium salt, cyanide anion and Cp2Sm(thf)2 are also used in the acylation of amines with an esters. [18]The protection of amino groups by the formation of amides, especially acetamides, is one of the most fundamental and widely used procedures in organic syntheses.[5]

During recent years, microwave irradiations have been widely used in organic syntheses because of its reduction in reaction time and its good yields. Its application in N-acylation amides were also developed, but anhydrides were used to form the amide bonds in anhydrous conditions, and the diversity of amines was not sufficient to elucidate the influential factor of the reaction.[1] Microwave irradiation of organic reactions has rapidly gained in popularity as it accelerates a variety of synthetic transformations via time and energy saving protocol. Microwave assisted organic syntheses are suited to the increased demands in industry, particularly because of short reaction time, selectivity, solvent free technique and also for the expanded reaction range. Acetic acid can alone act as an acetylating agent under microwave irradiation, but this method is applicable with only primary amines. [1,2]

A range of greener catalytic methodologies have been reported for the preparation of amides. These include the N-acylation of amines with organic acids, the use of solid supported reagents (e.g. polymer-bound acylating agents), arylboronic or boronic acid derivatives and the use of microwave irradiation. Many different catalysts have been reported for the acylation of amines including transition metal salts, immobilised ionic liquids on mesoporous materials, and solid acid catalysts[1].

There are many reactions reported using different catalysts. One of the reactions of amides via N-acylation of primary and secondary amines under microwave irradiation was reported using starbon acid catalyst and acetic acid as the acetylating agent. In this reaction typical N-acylation of amine was carried out by adding 2mmol of aniline with 2mmol acetic acid and 0.1g of catalyst in a tube using microwave irradiation at 300 watt for 1-10 minutes at 120-130℃. The yield obtained was quantitative (>98%) with the starbon acid catalyst, which was not seen with any other acid catalyst despite of increasing the time and frequency of the reaction[1,3].



There is also an article on formation of an amide bond using acid without other solvents under microwave irradiation. In this reaction, firstly the benzyl amine and acetic acid were put together in a microwave reaction chamber, and an N-acetylated compound was obtained. In this reaction, acetic acid was used as acylation agent and solvent. Compared with the traditional amide synthetic procedure, this method was mild and convenient with a short time (a few minutes instead of several hours) and high yields (more than 90%). Most of the primary amines react with acetic acid and gave higher

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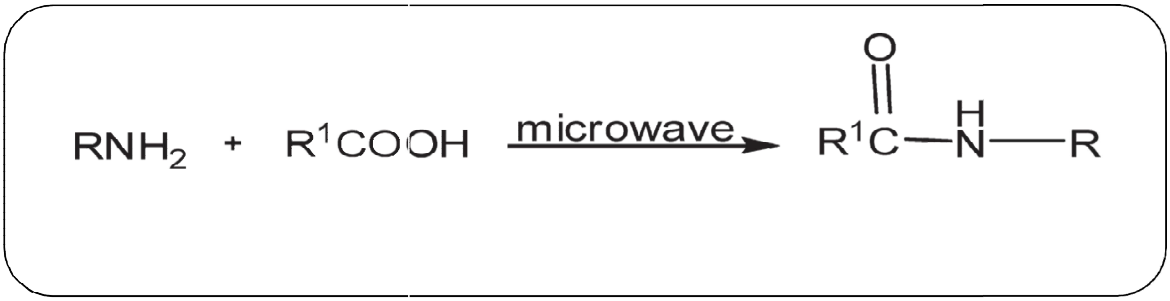
yields, between 90 and 95%. Instead of acetic acid, benzoic acid was used as an acylation reagent and also gave similarly high yields. In this reaction 200mg of aniline was added to 2ml of acetic acid or benzoic acid 800mg under microwave irradiation at 200 watt, 50℃ for 6-8 minutes. The reaction mixture was poured into a mixture of acetic ether and cyclohexane (1:50), then stored for some time in a refrigerator. The white crystal was obtained which was washed and dried out. The yield obtained was 98%. [2]



Acylation of amines with acetic acid using LaY and HY as catalysts are also reported. Recently, Gd(OTf)3 and iodine, ZnO, bromodimethyl- sulfoniumbromide, lanthanide, tosylates, CoCl2.6H2O, ZrOCl2.8H2O have been reported to promote the acylation of amines with acetic anhydride or acetyl chloride.[16] Acylation with acetic anhydride without catalyst has also been reported. The use of acetic acid rather than acetic anhydride or acetyl chloride is both economically and environmentally advantageous, because substances used in chemical synthesis may either be incorporated into final product or into by product as a waste material. Acylation reaction of amines was also carried out by using acid catalyst known as Zeolites. In this reaction 2mmol of aniline was mixed with 2ml of acetic acid and 200 mg of zeolite catalyst was used under microwave irradiation at 600 watts for 30-40 minutes at 117℃. The yield obtained was 73% [5].

$$R\text{-}NH_2 + CH_3COOH \xrightarrow[mi\ rowave\ i\ radiation]{zeolite} R\text{-}NHCOCH_3$$

Acylation reaction of amine is also carried out via catalyst using Zn dust. In this reaction 3.3g of aniline was mixed with 10ml of acetic acid with a0.16g of catalyst. The reaction was heated on a burner for 45 minutes and the yield obtained was 70-80%. In this reaction metal Zn is used as a catalyst which is toxic to the health and environment. [3,19]

$$R\text{-}NH_2 + CH_3CHOOH \xrightarrow{Zn\ dust} R\text{-}NHCOCH_3$$

Although a variety of methods are available for this purpose, acetylation of amines is routinely carried out with acetic anhydride or acetyl chloride in presence of base catalysts. The reaction is generally carried out in the presence of tertiary amines such as triethylamine or pyridine. The use of catalysts such as LiCl, I2, montmorillonite-K 10 or KSF, KF-Al2O3 and AlPO4 have also been reported. Pyridine(tetrahydroborate)zinc complex has also been used for acylation of amines with ethyl acetate. In this reaction 0.093g of aniline in THF and ethyl acetate was prepared which was mixed with the zinc complex of 0.697g and was refluxed for 2.5 hours, the yield obtained was 97%.[4]

$$RR'NH \xrightarrow[THF,reflux]{(Py)Zn(BH4)2} RR'NCOCH_3$$

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II. LITERATURE REVIEW

Acetanilide, derived from aniline through an acetyla-tion reaction, is a white, odorless, crystalline solid with a characteristic amide functional group; its key chemical proper-ties include: de H_2C-COOH/Zn

Structure and Formation:

* Chemical Formula: C6H5NHC(O)CH3
* Formation: Aniline reacts with acetic anhydride (or glacial acetic acid) in the presence of a catalyst like zinc dust to produce acetanilide.
* Functional Group: Amide group (C(O)NH)

Physical Properties:

* Appearance: White, flaky crystals
* Melting Point: Approximately 113-115°C
* Solubility: Slightly soluble in water, more soluble in organic solvents like ethanol

Chemical Properties:

* Weak Base: Due to the resonance within the amide group, acetanilide exhibits weak basic properties
•Acylation: Can undergo further acylation reactions to form diacetanilide
* Halogenation: Can be halogenated on the aromatic ring under appropriate conditions
* Nitration: Can be nitrated on the aromatic ring to produce nitro-substituted acetanilide derivatives

Analysis Methods:

Melting Point Determination:

A simple method to confirm the purity of acetanilide, as a pure sample will melt at a specific temperature range Thin Layer Chromatography (TLC):

Used to identify and compare the presence of acetanilide in a mixture by analyzing its retention factor (Rf) value[24] Infrared Spectroscopy (IR):

Characteristic amide bond stretching vibrations can be observed in the IR spectrum, confirming the presence of the amide group Nuclear Magnetic Resonance (NMR) Spectroscopy:

Provides detailed information about the structure of acetanilide by analyzing the chemical shifts of different hydrogen atoms

Historical Use:

Acetanilide was once used as a pain reliever (under the trade name "Antifebrin") but was later found to be toxic and largely replaced by safer alternatives like acetaminophen.

Synthetic Intermediate:

Today, acetanilide is primarily used as a starting material for synthesizing other organic compounds, including dyes and pharmaceuticals.

Key chemical properties of acetanilide:

* Appearance: White, crystalline powder or flakes
* Molecular formula: C6H5NHCOCH3
* Solubility: Slightly soluble in water, readily soluble in organic solvents like ethanol, ether, chloroform

Pharmacological properties

Acetanilide (N-phenylacetamide) has analgesic and antipyretic properties, and is metabolized into paracetamol (acetaminophen) in the body. It was used as a pain reliever and fever reducer in the late 19th century, but was eventually replaced by acetaminophen because of its toxic side effects.[17]

* Analgesic: Acetanilide relieves mild to moderate pain
* Antipyretic: Acetanilide reduces fever
* Anti-inflammatory: Acetanilide has a limited anti-inflammatory effect

Mechanism of action

* Acetanilide is metabolized in the liver to form paracetamol

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• Paracetamol inhibits the production of prostaglandins, which are lipid compounds that cause inflammation and pain

Side effects

•Acetanilide can cause cyanosis, liver damage, and kidney damage

* Acetanilide can interfere with hemoglobin's ability to carry oxygen
* Acetanilide was introduced in 1886 as an antipyretic and analgesic drug
* Acetanilide was a popular alternative to opium and morphine
* Acetanilide was removed from the market in the 1980s

Applications

Acetanilide has many applic-ations, including as a phar-maceutical, dye, and stabilizer. HN-C-CH Acetanilide Pharmaceutical Application:

* Analgesic: Acetanilide reduces pain and fever
* Antipyretic: Acetanilide reduces fever
* Anti-inflammatory: Acetanilide can be used as an inflammatory agent
* Antimicrobial: Acetanilide derivatives have antimicrobial properties
* Antibiotic: Acetanilide is a building block of penicillin, an antibiotic that treats gram-positive bacteria
* Sulfa drug: Acetanilide is used to synthesize sulfa drugs, which treat bacterial infections.

Dye

* Fabric dye: Acetanilide is used to dye cellulose fabrics
* Textile dye: Acetanilide is used to manufacture colored dyes for textiles Stabilizer
* Hydrogen peroxide stabilizer: Acetanilide prevents hydrogen peroxide from decomposing
* Cellulose ester varnish stabilizer: Acetanilide stabilizes the structure of cellulose ester varnishes .
* Rubber accelerator: Acetanilide is used to speed up the synthesis of rubber
* Plasticizer: Acetanilide is used as a plasticizer in the polymer industry
* Photographic developer: Acetanilide was used as an experimental photographic developer in the 19th century
* Fragrance: Acetanilide can be used in fragrances

(released at high initial velocity into zone of very high rapid air mation). 2.5-10 m/s (500-2000 f/min.) Within each range the appropriate value depends on Lower end of Future Scope

Acetanilide (not acetimide) is the typical derivative obtained from aniline. Here are some potential scopes for acetanilide:

Industrial Applications

1. Dyes and Pigments: Acetanilide is used as an intermediate in the production of dyes, pigments, and pharmaceuticals.
2. Pharmaceuticals: Acetanilide is a precursor to paracetamol (acetaminophen), a widely used analgesic and antipyretic.
3. Agricultural Chemicals: Acetanilide is used in the synthesis of pesticides, herbicides, and fungicides.

Research and Development

1. Organic Synthesis: Acetanilide can be used as a starting material for the synthesis of complex organic compounds, such as heterocycles and natural products.
2. Materials Science: Acetanilide can be used to synthesize polymers, such as polyamides and polyimides, with potential applications in textiles, adhesives, and composites.
3. Biotechnology: Acetanilide can be used as a building block for the synthesis of biomolecules, such as peptides and nucleotides.

Emerging Trends

1. Green Chemistry: Acetanilide can be synthesized using environmentally friendly methods, such as enzymatic catalysis and solvent- free reactions.[21]
2. Nanotechnology: Acetanilide can be used to synthesize nanoparticles with potential applications in medicine, electronics, and energy storage.
3. Sustainable Energy: Acetanilide can be used as a precursor to synthesize materials for energy storage and conversion, such as batteries and fuel cells.[23]

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Pain Management

1. Analgesic properties: Acetanilide has been shown to exhibit analgesic properties, making it a potential candidate for pain management.[22]
2. Development of new analgesics: Researchers may explore modifying acetanilide's structure to create new, more effective analgesics.

Anti-Inflammatory and Antioxidant Properties

1. Inflammation reduction: Acetanilide hasbeen found to possess anti-inflammatory properties, which could be beneficial in treating conditions like arthritis.
2. Antioxidant activity: Its antioxidantproperties may help protect against oxidative stress-related diseases, such as cancer and neurodegenerative disorders.

Antimicrobial and Antibacterial Applications

1. Antibacterial agents: Acetanilide hasdemonstrated antibacterial activity, making it a potential candidate for developing new antibacterial agents.
2. Antimicrobial coatings: Researchers mayexplore using acetanilide-based coatings to prevent microbial growth on medical devices.

Neuroprotective and Neuro regenerative Properties

1. Neuroprotection: Acetanilide may have neuroprotective effects, which could be beneficial in treating neurodegenerative diseases like Alzheimer's and Parkinson's.
2. Neuroregeneration: Its potential neuroregenerative properties may aid in the development of new treatments for nerve damage and neurodegenerative disorders.

Cancer Research

1. Anticancer properties: Acetanilide has been found to exhibit anticancer properties, making it a potential candidate for cancer treatment.
2. Cancer prevention: Researchers may explore using acetanilide as a preventive measure against certain types of cancer.

Future Research Directions

1. Clinical trials: Conducting clinical trials to evaluate the efficacy and safety of acetanilide-based treatments.
2. Mechanism of action: Elucidating the mechanisms by which acetanilide exerts its biological effects.
3. Structure-activity relationships: Investigating the relationships between acetanilide's structure and its biological activity to design more effective analogs.

III. CONCLUSION

The use of acetic acid in the acylation reaction instead of corrosive acetyl chloride or acetic anhydride provides many advantages from both economic and environmental In conclusion, our efficient, atom economic and an environmentally friendly protocol allowed the preparation of a wide range of intermediates for pharmaceuticals (acetanilide). We have developed an efficient, rapid, safe, high yielding and eco- friendly method for Acylation of amines without using catalyst under microwave irradiation. The present method is applicable to primary amines and anilines. Magnesium sulphate heptahydrate-glacial acetic acid system has proved to be a mild, cheap, simple, and benign catalyst for the synthesis of acetanilide from aniline. Lewis acid catalysis nucleophilic acyl substitution reaction by increasing the electrophilicity of the carbonyl group. The method may serve as a sustainable, inexpensive and green route for the acetylation of the primary amines. The method of acetylation reported above is well suited for stu-dents at the undergraduate level since it avoids the use of any catalyst or reducing agent. Since the reaction is carried out in water, it avoids the use of acetic acid as a solvent. The product is obtained immediately on the addition of acetic anhydride and thus eliminates the unnecessary step of refluxing for long peri-ods under anhydrous conditions. This procedure also avoids the use of corrosive chemicals like acetyl chloride. In short, it is a very safe and convenient green procedure for the acetylation of aniline, which gives acetanilide in excellent yields and is easy for students to carry out.

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