



## A Novel and Green Method for *N*-acylation of Amines and Sulfonamides under Ultrasound Irradiation

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### ABSTRACT

A facile and versatile method for acylation of structurally diverse amines and sulfonamides under focused ultrasonic irradiation in catalyst-free and solvent-free conditions is reported. There are several advantages to this approach such as simple and easier workup conditions, small amount of time and high yielding. The acylation reaction was carried out with acetic anhydride. All structures of synthesized products have been identified by NMR and mass spectroscopy.

**Keywords:** *N*-acylation, sulfonamide, ultrasonic irradiation, green chemistry.

### INTRODUCTION

The *N*-acyl sulfonamide moiety is a common feature in several important bio molecules and has emerged as an important class drug synthesis<sup>1-3</sup>. A large number of novel *N*-acylated-sulfonamides-sulfonamides molecules have been investigated for antibacterial activity<sup>4</sup>. They are also known as therapeutic agents for Alzheimer's disease<sup>5</sup> and anti-proliferative agents<sup>6</sup>.

In the literature, several reaction pathways for the preparation of *N*-acyl sulfonamides have been developed, most of them were carried out

by the condensation of the various sulfonamides with acyl chloride or anhydride under basic conditions<sup>7</sup>. Acylation of sulfonamides can also be achieved with concentrated H<sub>2</sub>SO<sub>4</sub><sup>8</sup>, Lewis acids<sup>9</sup> or heterogeneous solid acid<sup>10</sup> in acetonitrile under acidic conditions. Another attractive method based on using of palladium-catalyzed acylation of aryl with sulfonamides under microwave irradiation<sup>11</sup>.

Recently, an efficient method for acylation of sulfonamides in acetonitrile with H<sub>6</sub>P<sub>2</sub>W<sub>18</sub>O<sub>62</sub> as acidic solid catalyst is described by Bougheloum *et al*<sup>12</sup>. Synthetic methods should be developed, to produce molecules that are no toxic to human health

and the environment, because we are all inhabitants on Earth, everyone is a stakeholder, and every person aims contributing to environmental protection locally, nationwide and worldwide.

Chemists have focused on maximizing yield, reducing energy and minimizing the number of steps by application of the green chemistry principle of atom economy to chemical reactions.

Chemical synthesis usually requires an energy source; there are several types of traditional energy such as: light, heat and ionizing radiation. Sonication chemistry can be replaced the traditional energy used for the chemical reaction by irradiation ultrasonic.

In this protocol, ultrasound provides strange mechanism for supplying high energy chemistry, based on acoustic cavitation<sup>13-16</sup>. It was suggested that sonication also assists in the breakdown of intermediates and desorption of the products from the surface, The use of ultrasound in chemical reactions provides specific activation based on a physical-chemical phenomena which are currently a matter of advanced research<sup>17-18</sup>. In recent years, ultrasound irradiation has been considered as a successful method in organic synthesis, because it has several advantages such as; shorter reaction time, higher yield and milder conditions<sup>19-22</sup>.

Herein, we describe a practical procedure for the acylation of various amines and sulfonamides with acetic anhydride at room temperature and without a solvent using ultrasonic irradiation.

## EXPERIMENTAL

### General

The sulfonamide derivatives were prepared according to the literature<sup>23</sup>. All reactions were monitored by thin layer chromatography using Merck TLC Silicagel 60 F254, using the solvents indicated. Silica gel column chromatography was performed over. Sonication was performed in a FUNGILAB ultrasonic bath at a frequency of 40 kHz and nominal power 260 W. Melting points were measured in open capillary tubes on an electro thermal-apparatus.

<sup>1</sup>H-NMR and <sup>13</sup>C-NMR spectra were recorded on Bruker spectrometer (400 and 100MHz)

using (CDCl<sub>3</sub>) as solvent and (TMS) as an internal standard. IR spectra were recorded on a Perkin-Elmer FT-600 spectrophotometer. Mass spectra were performed on a Shimadzu QP operating at an IP of 30 or 70 eV.

Typical experimental procedure for *N*-acylation of amines and sulfonamides

In a glass tube was placed a mixture of amine or sulfonamide derivatives (1 equiv) and acetic anhydride (1equiv) at room temperature. The mixture was immersed in the water of the sonicator bath for appropriate time at room temperature. Reaction was monitored by TLC.

After completion of the reaction, the reaction mixture was diluted with the water, and extracted with ethyl acetate, the organic layer was dried over anhydrous sodium sulfate and the resulting residue was purified by silica gel chromatography using dichloromethane-petroleum ether (9:1), to give the *N*-acylamines or *N*-acylsulfonamides in good yields.

### **N-Phenylacetamide**

Oil; 88 % yield; <sup>1</sup>H NMR (CDCl<sub>3</sub>):7.2-7.5 (m, 5H, Ar), 2.2 (s, 3H, CH<sub>3</sub>); <sup>13</sup>C NMR (CDCl<sub>3</sub>): 168.9, 138.5, 128.9, 128.0, 121.6, 24.0; LCMS (+) *m/z*, 136.07 [M+H]<sup>+</sup>, C<sub>8</sub>H<sub>9</sub>NO.

### **N-(4-Methoxyphenyl)acetamide**

Oil; 89 % yield; <sup>1</sup>H NMR (CDCl<sub>3</sub>): 6.9-7.4 (m, 4H, Ar), 2.2 (s, 3H, CH<sub>3</sub>), 3.6 (s, 3H, CH<sub>3</sub>); <sup>13</sup>C NMR (CDCl<sub>3</sub>):168.9, 158.9, 130.8, 122.6, 114.5, 55.8, 24.2; LCMS (+) *m/z*, 166 [M+H]<sup>+</sup>, C<sub>9</sub>H<sub>11</sub>NO<sub>2</sub>.

### **N-(3,4-Dihydroisoquinolin-2(1H)-yl)acetamide**

Powder; 70 % yield; mp 110-111 °C; IR (KBr): 1660 (CO); <sup>1</sup>H NMR (CDCl<sub>3</sub>):7.2-7.0 (m, 4H, Ar), 4.4 (s, 2H, CH<sub>2</sub>), 3.4 (t, 2H, J5.5 Hz, CH<sub>2</sub>), 2.6 (t, 2H, J5.8 Hz, CH<sub>2</sub>), 2.1 (s, 3H, CH<sub>3</sub>); <sup>13</sup>C NMR (CDCl<sub>3</sub>):169.2, 134.1, 127.7, 127.5, 126.9, 126.3, 49, 47, 28.6, 21.1;LCMS (+) *m/z*, 176.10 [M+H]<sup>+</sup>, C<sub>11</sub>H<sub>13</sub>NO.

### **N-(4-Phenylpiperazin-1-yl)acetamide**

Powder; 74 % yield; mp 113-114 °C; IR (KBr): 1752 (CO);<sup>1</sup>H NMR (CDCl<sub>3</sub>):7.45-6.95 (m, 5H, Ar), 3.62 (t, 4H, J5.2, 4.8 Hz, CH<sub>2</sub>), 3.25

(t, 4H,  $J_{5.2}$ , 4.8 Hz, CH<sub>2</sub>), 2.1 (s, 3H, CH<sub>3</sub>); <sup>13</sup>C NMR (CDCl<sub>3</sub>):168.9, 149.6, 129.6, 121.9, 114.3, 53.3, 46.2, 21.1; LCMS (+) *m/z*, 205.13 [M+H]<sup>+</sup>, C<sub>12</sub>H<sub>16</sub>N<sub>2</sub>O.

#### N-(N-Cyclohexylsulfonyl) acetamide

Powder; 94 % yield; mp 198-199 °C; IR (KBr): 3372 and 3265 (NH), 1730(CO), 1360 and 1153 (SO<sub>2</sub>); <sup>1</sup>H NMR (CDCl<sub>3</sub>):1.6-2.1 (m, 10H, CH<sub>2</sub>), 5.2 (d, 1H,  $J_{7.35}$  Hz, CH), 2.0 (s, 3H, CH<sub>3</sub>); <sup>13</sup>C NMR (CDCl<sub>3</sub>):171.0, 43.9, 32.6, 25.7, 24.8, 21.6; LCMS (+) *m/z*, 222.09 [M+H]<sup>+</sup>, C<sub>8</sub>H<sub>16</sub>N<sub>2</sub>O<sub>3</sub>.

#### N-(N-Propylsulfonyl) acetamide

Powder; 90 % yield; mp 168-169 °C; IR (KBr): 3370 and 3263(NH), 1721(CO), 1380, 1154(SO<sub>2</sub>); <sup>1</sup>H NMR (CDCl<sub>3</sub>):3.2 (m, 2H, CH<sub>2</sub>), 2.1 (s, 3H, CH<sub>3</sub>), 1.65 (m, 2H, CH<sub>2</sub>), 1.01 (t, 3H,  $J_{5.02}$  Hz, CH<sub>3</sub>); <sup>13</sup>C NMR (CDCl<sub>3</sub>):172.1, 42.5, 21.9, 21.6, 11.2; LCMS (+) *m/z*, 181.06 [M+H]<sup>+</sup>, C<sub>5</sub>H<sub>12</sub>N<sub>2</sub>O<sub>3</sub>S.

#### N-(N-(Tert-butyl)sulfonyl) acetamide

Powder; 95 % yield; mp 136-137 °C; IR (KBr): 3374 and 3267 (NH), 1735 (CO), 1362 and 1154 (SO<sub>2</sub>); <sup>1</sup>H NMR (CDCl<sub>3</sub>):1.15 (s, 9H, 3CH<sub>3</sub>), 2.01 (s, 3H, CH<sub>3</sub>); <sup>13</sup>C NMR (DMSO-*d*<sub>6</sub>):19.1, 30.6, 30.6, 30.6, 38.7; MS (+) *m/z*, 212.07 [M+NH<sub>4</sub>]<sup>+</sup>, C<sub>6</sub>H<sub>14</sub>N<sub>2</sub>O<sub>3</sub>S.

#### N-(N-Benzylsulfonyl) acetamide

Powder; 96% yield; mp 121-122 °C; IR (KBr): 3371 and 3268 (NH), 1738 (CO), 1363 and 1156 (SO<sub>2</sub>); <sup>1</sup>H NMR (CDCl<sub>3</sub>):1.90 (s, 3H, CH<sub>3</sub>), 4.06 (s, 2 H, CH<sub>2</sub>), 7.2 (m, 1H, Ar); <sup>13</sup>C NMR

(DMSO-*d*<sub>6</sub>):22.9, 44.2, 125.8, 126.2, 126.7, 127.9, 128.6, 142.3; MS (+) *m/z*, 246.06 [M+NH<sub>4</sub>]<sup>+</sup>, C<sub>9</sub>H<sub>12</sub>N<sub>2</sub>O<sub>3</sub>S.

#### N-(N-(3-Fluorophenyl)sulfonyl) acetamide

Powder; 93 % yield; mp 147-148 °C; IR (KBr): 3260 and 3068 (NH), 1694 (CO), 1368 and 1162 (SO<sub>2</sub>); <sup>1</sup>H NMR (CDCl<sub>3</sub>):1.95 (s, 3 H, CH<sub>3</sub>), 6.9 (m, 1H, Ar), 7.06 (m, 2H, Ar), 7.3 (m, 1H, Ar); <sup>13</sup>C NMR (DMSO-*d*<sub>6</sub>):22.9, 105.6, 110.1, 114.6, 130.7, 139.5, 163.9, 168.6; MS (+) *m/z*, 250.04 [M+NH<sub>4</sub>]<sup>+</sup>, C<sub>8</sub>H<sub>9</sub>N<sub>2</sub>O<sub>3</sub>SF.

#### N-(3,4-Dihydroisoquinolin-2(1H)-yl-sulfonyl) acetamide

Cristal; 90% yield; mp 173-174 °C; IR (KBr): 3261 (NH), 1695 (CO), 1361 and 1155 (SO<sub>2</sub>); <sup>1</sup>H NMR (CDCl<sub>3</sub>):8.1 (s, 1H, NH), 7.2-7.0 (m, 4H, Ar), 4.5 (s, 2H, CH<sub>2</sub>), 3.7 (t, 2H,  $J_{5.7}$  Hz, CH<sub>2</sub>), 2.9 (t, 2H,  $J_{5.8}$  Hz, CH<sub>2</sub>), 2.1 (s, 3H, CH<sub>3</sub>); <sup>13</sup>C NMR (CDCl<sub>3</sub>):173, 134.1, 127.5, 126.9, 126.3, 125.7, 48.1, 47.2, 25.3, 21.6; MS (+) *m/z*, 255.07 [M+NH<sub>4</sub>]<sup>+</sup>, C<sub>11</sub>H<sub>14</sub>N<sub>2</sub>O<sub>3</sub>S.

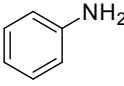
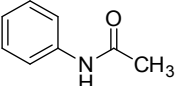
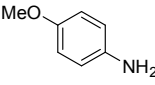
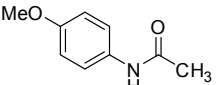
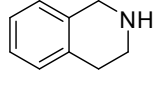
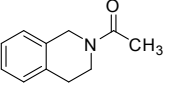
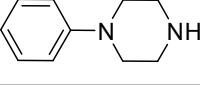
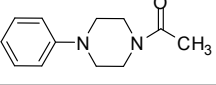
#### N-((4-Phenylpiperazin-1-yl)sulfonyl) acetamide

Cristal; 94 % yield; mp 176-177 °C; IR (KBr): 3260 (NH), 1697(CO), 1361 and 1155 (SO<sub>2</sub>); <sup>1</sup>H NMR (CDCl<sub>3</sub>):7.7-6.9 (m, 5H, Ar), 3.1 (t, 4H,  $J_{5.4}$  Hz, CH<sub>2</sub>), 2.5 (t, 4H,  $J_{5.2}$  Hz, CH<sub>2</sub>), 2.3 (s, 3H, CH<sub>3</sub>); <sup>13</sup>C NMR (CDCl<sub>3</sub>):173, 149.6, 129.6, 121.9, 114.3, 50.3, 43.4, 21.5; LCMS (+) *m/z*, 284.10 [M+H]<sup>+</sup>, C<sub>12</sub>H<sub>17</sub>N<sub>3</sub>O<sub>3</sub>S.

#### N-(N-(4-Methoxyphenyl)sulfonyl) acetamide

Powder; 80% yield; mp 151-152 °C; IR (KBr): 3260 and 3251 (NH), 1690 (CO), 1365 and

Table 1: Synthesis of N-acylamines

Entry	Substrate	Product	Time (min)	Yield (%)
1			15	88
2			15	89
3			30	70
4			30	74

1158 (SO<sub>2</sub>); <sup>1</sup>H NMR (CDCl<sub>3</sub>):7.1-7.5 (m, 4H, Ar), 3.8 (s, 3H, CH<sub>3</sub>), 2.3 (s, 3H, CH<sub>3</sub>); <sup>13</sup>C NMR (CDCl<sub>3</sub>):171, 153.3, 130, 124.5, 115.1, 55.8, 21.6; LCMS (+) *m/z*, 245.05 [M+H]<sup>+</sup>, C<sub>9</sub>H<sub>12</sub>N<sub>2</sub>O<sub>4</sub>.

**(R)-N-(N-(1-Phenylethyl)sulfamoyl) acetamide**

Powder; 85 % yield; mp 171-172 °C; IR (KBr): 3370 and 3265 (NH), 1698 (CO), 1361 and 1152 (SO<sub>2</sub>); <sup>1</sup>H NMR (CDCl<sub>3</sub>):8.1 (s, 1H, NH), 7.5-7.2 (m, 5H, Ar), 5.9 (s, 1H, NH), 5.15 (m, 1H, \*CH),

**Table 2: Synthesis of N-acyl sulfonamides**

Entry	Substrate	Product	Time (min)	Yield (%)
5			30	94
6			15	90
7			15	95
8			30	96
9			30	93
10			40	90
11			60	60
12			35	80
13			20	85
14			30	83

2.00 (s, 3H, CH<sub>3</sub>), 1.50 (d, 3H, *J* 6.9 Hz, CH<sub>3</sub>); <sup>13</sup>C NMR (CDCl<sub>3</sub>): 171.3; 145.1; 128.2; 126.8; 126.4; 47.2; 21.0; 20.6 ppm; LCMS (+) *m/z*, 243.07 [M+H]<sup>+</sup>, C<sub>10</sub>H<sub>14</sub>N<sub>2</sub>O<sub>3</sub>S.

### Ethyl-1-(N-acetylsulfamoyl)pyrrolidine-2-carboxylate

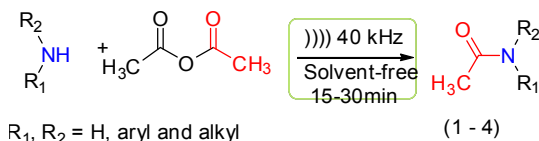
Powder; 83 % yield; mp 199-200°C; IR (KBr): 3254 (NH), 1733 (CO), 1692 (CO), 1369 and 1166 (SO<sub>2</sub>); <sup>1</sup>H NMR (CDCl<sub>3</sub>): 8.7 (s, 1H, NH), 4.7 (m, 1H, \*CH), 4.3 (q, 2 H, *J* 2.4 Hz, CH<sub>2</sub>), 3.4-3.7 (m, 6H, CH<sub>2</sub>), 2.1 (s, 3H, CH<sub>3</sub>), 1.3 (t, 3H, *J* 2.4 Hz, 3H, CH<sub>3</sub>); <sup>13</sup>C NMR (CDCl<sub>3</sub>): 171.1, 171.0, 61.3, 60.6, 55.2, 27.7, 21.4, 21.6, 14.1; LCMS (+) *m/z*, 265.08 [M+H]<sup>+</sup>, C<sub>9</sub>H<sub>16</sub>N<sub>2</sub>O<sub>5</sub>S.

## RESULTS AND DISCUSSION

Following our efforts to develop novel methods for acylation<sup>24-27, 12</sup> and in continuation of our investigation on the use of ultrasound irradiation to accelerate reactions<sup>28-30</sup>, we describe a novel method for acylation the different amines and sulfonamides with acetic anhydride under ultrasonic irradiation, in absence of solvent and catalyst.

The main mechanism of sonochemical was the formation of cavitation bubbles<sup>31</sup>, under certain conditions, some bubbles implode, generating very high temperatures and pressures, these transient, localized hot spots drive high-energy chemical reactions<sup>32-34</sup>.

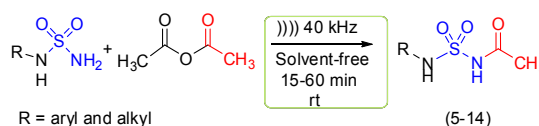
In order to carry out the acylation of the amine function under ultrasonic irradiation, the amine was chosen as a model substrate, the reaction of amine with acetic anhydride using ultrasonic irradiation as shown in scheme 1, the reaction was completed at 15-30 min, the acylated products were isolated in good yields (Scheme 1).



**Scheme 1: Catalyst-free synthesis of N-acylamines under ultrasonic irradiation**

The *N*-acylation of amines was successfully achieved using acetic anhydride under ultrasound irradiation. The reactions proceed smoothly in high yields (70-89 %) and in short reaction time (table 1). The structures of all compounds were unambiguously confirmed by spectroscopic methods. Proton nuclear magnetic resonance spectra showed singlets at 2.20-2.25 ppm corresponding to the proton of CH<sub>3</sub> acetyl. Infrared spectra showed bands at 1650-1700 cm<sup>-1</sup> of functional group C=O.

Encouraged by this result, we extended the reaction to sulfonamides derivatives using ultrasonic irradiation (Scheme 2). The results are reported in (Table 2, entry 5-14). In all cases, we obtained the corresponding products *N*-acyl sulfonamides in good to excellent yields.



**Scheme 2: Catalyst-free synthesis of N-acylsulfonamides under ultrasonic irradiation**

For the resulting products, Infrared spectra showed band at 1650-1700 cm<sup>-1</sup> of functional group (C=O) of acetyl moiety and band at 1170-1395 cm<sup>-1</sup> of sulfonyl group. In Proton nuclear magnetic resonance spectra, the structure of *N*-acyl sulfonamides was confirmed by a singlet at 2.27 ppm corresponding to CH<sub>3</sub> group.

The structures of all molecules synthesized were confirmed by usual spectroscopic methods, IR, <sup>1</sup>H NMR, <sup>13</sup>C NMR and mass spectrometry.

## CONCLUSION

In summary, the acylation of amines or sulfonamides was successfully achieved using acetic anhydride under ultrasound irradiation. This methodology provides a green and much improved approach over the classic methods. Ultrasound can be an effective technology for process intensification in with cavitation collapse can affect chemical transformations. The use of

ultrasound is expected to enhance the rate of reaction by various mechanisms.

The non-conventional method offer advantages over conventional process, short time span to complete reaction, easy work procedure and excellent yields.

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