

P₂O₅/SiO₂ as an Efficient and Recyclable Catalyst for *N*-Acylation of Sulfonamides under Heterogeneous and Solvent-free Conditions

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A convenient synthetic method for *N*-acylation of sulfonamides in the presence of P₂O₅/SiO₂ is described. Carboxylic acid anhydrides and carboxylic acid chlorides were used as acylating agents and the reactions were carried out in CH₂Cl₂ or solvent-free conditions. The catalyst can be recovered by simple filtration and can be used in the subsequent reactions.

Keywords: *N*-Acylsulfonamide, Solvent-free, Diphosphorous pentoxide, Heterogeneous condition, *N*-Acylation

INTRODUCTION

The *N*-acylsulfonamides constitute an important class of drugs for Alzheimer's disease [1], antibacterial inhibitors of tRNA synthetases [2], antagonists for AngiotensinII [3], and Leukotriene D₄-receptors [4]. The most practical methods for the *N*-acylation of sulfonamides are the reaction of parent sulfonamide with acyl chlorides or anhydrides using trialkylamines and pyridine [5] or alternatively with alkali hydroxides [6]. Another approach utilizes carboxylic acids along with condensing agents such as carbodiimides (EDC, DCC) and *N,N'*-carbonyl diimidazole [2,7]. Recently, Katrizky *et al.* reported *N*-acylation of sulfonamides using *N*-acyl benzotriazole as the acylating agent [8]. The few reports on this transformation under acidic conditions do not systematically examine the scope and limitations of the reactions. Acylation of sulfonamides with concentrated H₂SO₄ in the carboxylic acid anhydride as a solvent [9], or in acetonitrile [10] are among these methods. However, most of these methods have some disadvantages including occurrence

of side reactions especially formation of biacylated by-products [5,] vigorous reaction conditions and tedious work-up [10], use of expensive or unavailable reagents and solvents [7,8], long reaction times and low yield of products [7]. Thus, there is still demand for developing new and mild methods for *N*-acylation of sulfonamides in the presence of inexpensive and bench top reagents.

Solid acids have many advantages such as simplicity of handling, decreasing reactor and plant corrosion problems, and environmentally safe disposal [11]. A literature survey shows that phosphorus pentoxide can be used in several reactions such as formation of amides from ketones [12] and oximes [13], phenolic esters from carboxylic acids [14], acylals from aldehydes [15], sulfonylation of aromatic rings [16], acetalization of carbonyl compounds [17] and synthesis of nitriles from aldehydes [18]. In continuation of our research on synthesis of sulfonamides and *N*-acylsulfonamides [19] we have employed silica supported P₂O₅ in *N*-acylation of sulfonamides.

EXPERIMENTATION

General

All chemicals were purchased from Merck and Fluka

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Chemical Companies. The products were characterized by comparison of their spectral and physical data with those of authentic samples. IR spectra were recorded on Nicolet (impact 400D model) FTIR Spectrometer. ^1H NMR and ^{13}C NMR spectra were recorded on Bruker DRX 300 Avance Spectrometer. Column chromatography was performed using silica gel 60 (230-400 mesh, EtOAc-petroleum ether as eluents). All yields refer to isolated ones.

Preparation of $\text{P}_2\text{O}_5/\text{SiO}_2$

A mixture of phosphorous pentoxide (3 g) and chromatography silica gel (230-400 mesh, 4 g) were placed in a flask and stirred for 4 h. The homogeneous, free flowing and white powder was obtained that is sensitive toward moisture and should be stored in a desiccator.

Preparation of *N*-Butanoyl-4-methyl Benzenesulfonamide: A Typical Procedure for *N*-Acylation of Sulfonamides under Solvent-free Conditions

To a vigorously stirred mixture of 4-methyl benzenesulfonamide (0.342 g, 2 mmol) and $\text{P}_2\text{O}_5/\text{SiO}_2$ [(w/w 75%), 0.35 g], butanoic anhydride (0.632 g, 4 mmol) was added in two portions at 80 °C. The progress of the reaction was monitored by TLC. After the completion of the reaction, ethyl acetate (20 ml) was added and the solid materials were removed by filtration. The filtrate was washed with water (15 ml) and dried over MgSO_4 . After evaporation of the solvent, the crude product was purified by recrystallization from toluene or ethyl acetate/*n*-hexane mixed solvent to afford *N*-butanoyl-4-methyl benzenesulfonamide in 80% yield (0.386 g), m.p.: 82-83 °C, (Lit [20], 82-83 °C) IR ν (cm^{-1}) 3090, 1680, 1601, 1349, 1158, ^1H NMR (DMSO- d_6) δ (ppm) 0.74 (t, $J = 7.3$ Hz, 3H), 1.40 (sext, $J = 7.3$ Hz, 2H), 2.15 (t, $J = 7.3$ Hz, 2H), 2.38 (s, 3H), 7.4 (d, $J = 8.1$ Hz, 2H), 7.79 (d, $J = 8.1$ Hz, 2H), 11.99 (s, 1H); ^{13}C NMR (DMSO- d_6) δ (ppm) 13.6, 17.95, 21.49, 37.58, 127.95, 129.93, 137.08, 144.54, 171.85.

Preparation of *N*-Propanoyl Benzenesulfonamide: A Typical Procedure for *N*-Acylation of Sulfonamides in CH_2Cl_2

A mixture of benzenesulfonamide (0.314 g, 2 mmol), propionyl chloride (0.184 g, 2 mmol) and $\text{P}_2\text{O}_5/\text{SiO}_2$ [(w/w

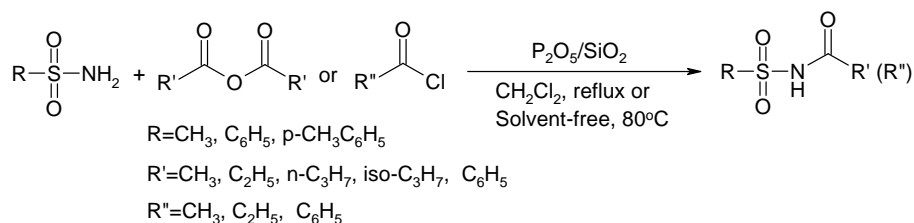
75%), 0.35 g] was refluxed in CH_2Cl_2 (5 ml) for 60 min. The completion of the reaction was monitored by TLC. At this stage, the catalyst was separated by filtration and was washed with CH_2Cl_2 (2×5 ml). The combined organic layers were washed with water (15 ml) and dried over MgSO_4 . After evaporation of the solvent, the crude product was purified by recrystallization from toluene or ethyl acetate/*n*-hexane mixed solvent to afford *N*-propanoyl benzenesulfonamide in 85% yield (0.362 g) m.p.: 75-76 °C, (Lit [21], 74-75) IR ν (cm^{-1}) 3246, 1710, 1341, 1156; ^1H NMR (DMSO- d_6) δ (ppm) 0.88 (t, $J = 7.5$ Hz, 3H), 2.16 (q, $J = 7.5$ Hz, 2H), 7.59-7.94 (m, 5H), 12.06 (s, 1H); ^{13}C NMR (DMSO- d_6) δ (ppm) 8.72, 29.12, 126.02, 127.86, 129.38, 129.59, 172.57. The recovered catalyst was washed with diethyl ether (15 ml) and activated at 70 °C for 2 h prior to reuse.

RESULTS AND DISCUSSION

We report herein the results of $\text{P}_2\text{O}_5/\text{SiO}_2$ catalyzed *N*-acylation of different sulfonamides with some carboxylic acid anhydride and chloride in solvent-free and heterogeneous condition (Scheme 1). In a typical procedure, a mixture of sulfonamide (2 mmol), carboxylic acid anhydride (4 mmol) or carboxylic acid chloride (2mmol) and $\text{P}_2\text{O}_5/\text{SiO}_2$ [(w/w 75%), 0.35 g] was vigorously stirred at 80 °C under solvent-free condition or refluxed in CH_2Cl_2 for the appropriate time. The progress of the reactions was monitored by TLC and the products were isolated in good to high yield after an easy work-up.

First; the reaction conditions were optimized with respect to the solvent, the stoichiometry of reactants, amount of the catalyst, temperature and reaction time. In order to find the best solvent, the reaction of benzenesulfonamide with benzoyl chloride and acetic anhydride was carried out in different solvents (Table 1). CH_2Cl_2 afforded minimum amount of side products in high yield. Thus, it was selected as solvent of choice. Next, the *N*-acylation of benzenesulfonamide was examined with varying amounts of $\text{P}_2\text{O}_5/\text{SiO}_2$ [(w/w 75%)] and acylating agent (Table 2). Initially, the reaction was carried out with 0.35 g of the catalyst using 2 mmol of benzenesulfonamide and 4 mmol of acetic anhydride or benzoyl chloride as acylating agents. The reactions were completed in 20-55 min in good to excellent yields (85-98%)

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Scheme 1. *N*-acylation of sulfonamides with carboxylic acid anhydrides or carboxylic acid chlorides

Table 1. The Effect of Solvent on the *N*-Acylation of Benzenesulfonamide with Benzoyl Chloride and Acetic Anhydride

| Solvent | Acylation with benzoyl chloride | | Acylation with acetic anhydride | |
|-------------------|---------------------------------|-----------|---------------------------------|-----------|
| | Time (min) | Yield (%) | Time (min) | Yield (%) |
| Methylen chloride | 60 | 87 | 45 | 85 |
| n-Hexane | 90 | 80 | 240 | 80 |
| Toluene | 120 | 75 | 270 | 80 |
| Acetonitrile | 150 | 70 | 300 | 75 |
| Choloroform | 270 | 80 | 360 | 75 |
| Ethyl acetate | 360 | 85 | 500 | 50 |

Table 2. The Effect of the Amount of the Catalyst on the *N*-Acylation of Benzenesulfonamide (2 mmol) in CH₂Cl₂ and Solvent-free Conditions

| Entry | P ₂ O ₅ /SiO ₂ [(w/w 75%)] (g) | Acyating agent (mmol) | CH ₂ Cl ₂ | | Solvent-free | |
|-------|---|---|---------------------------------|-----------|--------------|-----------|
| | | | Time (min) | Yield (%) | Time (min) | Yield (%) |
| 1 | 0.35 | PhCOCl (4) | 55 | 88 | 25 | 98 |
| 2 | 0.35 | PhCOCl (3) | 60 | 90 | 25 | 98 |
| 3 | 0.35 | PhCOCl (2) | 60 | 87 | 25 | 98 |
| 4 | 0.35 | (CH ₃ CO) ₂ O (4) | 45 | 85 | 20 | 90 |
| 5 | 0.35 | (CH ₃ CO) ₂ O (3) | 60 | 78 | 40 | 75 |
| 6 | 0.35 | (CH ₃ CO) ₂ O (2) | 90 | 65 | 60 | 60 |
| 7 | 0.2 | PhCOCl (2) | 120 | 80 | 45 | 96 |
| 8 | 0.1 | PhCOCl (2) | 150 | 72 | 90 | 94 |
| 9 | 0.2 | (CH ₃ CO) ₂ O (4) | 95 | 82 | 65 | 88 |
| 10 | 0.1 | (CH ₃ CO) ₂ O (4) | 140 | 75 | 120 | 87 |
| 11 | P ₂ O ₅ (0.15) | PhCOCl (2) | 90 | 65 | 70 | 70 |
| 12 | P ₂ O ₅ (0.15) | C ₂ H ₅ COCl (2) | 190 | 65 | 140 | 70 |
| 13 | P ₂ O ₅ (0.15) | (CH ₃ CO) ₂ O (4) | 100 | 60 | 60 | 80 |
| 14 | P ₂ O ₅ (0.15) | (C ₃ H ₇ CO) ₂ O (4) | 75 | 55 | 60 | 60 |
| 13 | SiO ₂ (0.2 g) | PhCOCl (2) | 24(h) | 12 | 24(h) | 15 |
| 14 | SiO ₂ (0.2 g) | (CH ₃ CO) ₂ O (4) | 24(h) | 10 | 24(h) | 15 |
| 15 | No catalyst | PhCOCl (2) | 24(h) | 0 | 24(h) | 0 |
| 16 | No catalyst | (CH ₃ CO) ₂ O (4) | 24(h) | 0 | 24(h) | 0 |

in both CH_2Cl_2 and solvent-free conditions (Table 2, entries 1 and 4). By reducing the amount of benzoyl chloride to 2 mmol there was not any considerable change in the reaction time or the yield in both solvent-free and heterogeneous conditions (Table 2, entries 2 and 3). However, reducing the acetic anhydride to 2 mmol increased the reaction time and decreased the yield of reaction efficiently (Table 2, entries 5 and 6). On the contrary, by reducing the amount of the catalyst to 0.1 g, there was a considerable change in both the yield and the reaction time in CH_2Cl_2 . In solvent-free condition, there was a negligible change in the yield but the reaction time was increased (Table 2, entries 7-10). In addition, at low temperature and long reaction times only small amounts of the desired products were obtained. For example, the reaction of benzenesulfonamide with acetic anhydride was carried out at different temperatures such as room temperature (20%), 40 °C (28%), 60 °C (43%) and 70 °C (55%) in solvent-free condition. However, the best yield of product (90%) was obtained at 80 °C.

To examine the desirability of this catalyst, we tried the *N*-acylation of benzenesulfonamide with different acylating agents using various catalytic conditions (Table 2, entries 11-16). We found that the yields of products by using the combination of $\text{P}_2\text{O}_5/\text{SiO}_2$ were greater than those with P_2O_5 or SiO_2 separately. The effect of SiO_2 may be due to good dispersion of P_2O_5 on the surface of silica leading to significant improvements in reactivity. Similarly, SiO_2 as a supporter may minimize cross contamination between inorganic and organic compounds. Furthermore, this catalyst has the advantage of being easily removed from the reaction mixture by filtration. A further advantage of silica supported P_2O_5 is its improved storage stability in moisture in comparison to P_2O_5 , which is very sensitive to moisture.

In order to test the general applicability of the reaction in question, benzenesulfonamide, 4-methylbenzenesulfonamide, 4-nitro benzenesulfonamide and methanesulfonamide were subjected to *N*-acylation with different carboxylic acid chlorides and carboxylic acid anhydrides in both CH_2Cl_2 and solvent-free conditions. As shown in Table 3, several structurally varied sulfonamides and acylating agents underwent clean and remarkably fast *N*-acylation reaction. Benzenesulfonamide and benzenesulfonamide with electron-donating groups, that is, 4-methyl benzenesulfonamide, were

converted into their corresponding *N*-acyl sulfonamides with high yield after a short reaction time. Compared to that with electron-withdrawing groups, that is, 4-nitro benzenesulfonamide needed a longer reaction time to form the corresponding *N*-acyl sulfonamides (Table 3 entries 25 and 26). Methanesulfonamide as an example of aliphatic sulfonamide was similarly converted into the corresponding *N*-acyl sulfonamides with good to excellent yield. It seems that there was no steric bulk effect from the substituents at anhydride moiety since isobutanoic anhydride could be applied as efficient candidates for *N*-acylation of different sulfonamides to offer the expected *N*-acylsulfonamides in excellent yields in both heterogeneous and solvent-free conditions (Table 3, entries 14, 19 and 24). Carboxylic acid chlorides were found to be more reactive than carboxylic acid anhydrides. The reaction proceeded smoothly with equimolar of sulfonamides and carboxylic acid chlorides (except acetyl chloride), whereas the reaction with carboxylic acid anhydride needed two equivalents of anhydride with respect to sulfonamide. In addition, comparison between the results obtained in solution and those under solvent-free conditions accompanied with higher yields of products.

$\text{P}_2\text{O}_5/\text{SiO}_2$ is immiscible with non-polar organic compounds or solvents. Thus, the recovery of catalyst is convenient when a non-polar solvent is used as a medium. In this protocol, $\text{P}_2\text{O}_5/\text{SiO}_2$ is a heterogeneous acid catalyst because it is insoluble in the reaction medium, (CH_2Cl_2). The catalyst was recovered after reaction by simple filtration. To rule out the possibility of catalyst leaching, the activity of the recovered catalyst in each reaction was investigated carefully. The experimental results revealed that $\text{P}_2\text{O}_5/\text{SiO}_2$ could be recovered in more than 85% of the cases. In *N*-acylation of benzenesulfonamide with acetyl chloride or acetic anhydride, the catalyst could be reused for three times without significant loss of activity.

In conclusion, $\text{P}_2\text{O}_5/\text{SiO}_2$ was found to be a recyclable and heterogeneous catalyst for the *N*-acylation of various sulfonamides with both carboxylic acid anhydrides and carboxylic acid chlorides in solvent-free and heterogeneous conditions. In addition, the method has advantages in terms of low costs and availability of the chemicals, high yields and short reaction times, operational simplicity and easy work-up.

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Table 3. N-Acylation of Sulfonamides in CH₂Cl₂ or Solvent-free Condition

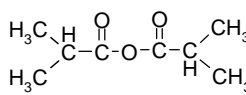
| Entry | Sulfonamide | Acyating agent | Product | CH ₂ Cl ₂ ^a | | Solvent-free ^b | |
|-------|---|---|--|--|----------|---------------------------|----------|
| | | | | Time(min) | Yield(%) | Time(min) | Yield(%) |
| 1 | Ph-SO ₂ NH ₂ | Ph-C(=O)-Cl | Ph-SO ₂ NH-C(=O)-Ph | 60 | 87 | 25 | 98 |
| 2 | Ph-SO ₂ NH ₂ | H ₃ C-C(=O)-Cl | Ph-SO ₂ NH-C(=O)-CH ₃ | 90 | 75 | 50 | 87 |
| 3 | Ph-SO ₂ NH ₂ | C ₂ H ₅ -C(=O)-Cl | Ph-SO ₂ NH-C(=O)-C ₂ H ₅ | 120 | 85 | 60 | 85 |
| 4 | H ₃ C-C ₆ H ₄ -SO ₂ NH ₂ | Ph-C(=O)-Cl | H ₃ C-C ₆ H ₄ -SO ₂ NH-C(=O)-Ph | 90 | 82 | 70 | 94 |
| 5 | H ₃ C-C ₆ H ₄ -SO ₂ NH ₂ | H ₃ C-C(=O)-Cl | H ₃ C-C ₆ H ₄ -SO ₂ NH-C(=O)-CH ₃ | 120 | 78 | 30 | 80 |
| 6 | H ₃ C-C ₆ H ₄ -SO ₂ NH ₂ | C ₂ H ₅ -C(=O)-Cl | H ₃ C-C ₆ H ₄ -SO ₂ NH-C(=O)-C ₂ H ₅ | 240 | 70 | 60 | 85 |
| 7 | H ₃ C-SO ₂ NH ₂ | Ph-C(=O)-Cl | H ₃ C-SO ₂ NH-C(=O)-Ph | 45 | 85 | 10 | 95 |
| 8 | H ₃ C-SO ₂ NH ₂ | H ₃ C-C(=O)-Cl | H ₃ C-SO ₂ NH-C(=O)-CH ₃ | 60 | 80 | 40 | 92 |
| 9 | H ₃ C-SO ₂ NH ₂ | C ₂ H ₅ -C(=O)-Cl | H ₃ C-SO ₂ NH-C(=O)-C ₂ H ₅ | 120 | 70 | 55 | 90 |
| 10 | Ph-SO ₂ NH ₂ | Ph-C(=O)-O-C(=O)-Ph | Ph-SO ₂ NH-C(=O)-Ph | 105 | 90 | 95 | 90 |
| 11 | Ph-SO ₂ NH ₂ | H ₃ C-C(=O)-O-C(=O)-CH ₃ | Ph-SO ₂ NH-C(=O)-CH ₃ | 45 | 85 | 20 | 90 |
| 12 | Ph-SO ₂ NH ₂ | H ₅ C ₂ -C(=O)-O-C(=O)-C ₂ H ₅ | Ph-SO ₂ NH-C(=O)-C ₂ H ₅ | 25 | 85 | 15 | 80 |
| 13 | Ph-SO ₂ NH ₂ | H ₇ C ₃ -C(=O)-O-C(=O)-C ₃ H ₇ | Ph-SO ₂ NH-C(=O)-C ₃ H ₇ | 30 | 80 | 25 | 85 |
| 14 | Ph-SO ₂ NH ₂ |  | Ph-SO ₂ NH-C(=O)-CH(CH ₃) ₂ | 35 | 95 | 30 | 98 |
| 15 | H ₃ C-SO ₂ NH ₂ | Ph-C(=O)-O-C(=O)-Ph | H ₃ C-SO ₂ NH-C(=O)-Ph | 180 | 82 | 150 | 85 |
| 16 | H ₃ C-SO ₂ NH ₂ | H ₃ C-C(=O)-O-C(=O)-CH ₃ | H ₃ C-SO ₂ NH-C(=O)-CH ₃ | 40 | 85 | 15 | 80 |
| 17 | H ₃ C-SO ₂ NH ₂ | H ₅ C ₂ -C(=O)-O-C(=O)-C ₂ H ₅ | H ₃ C-SO ₂ NH-C(=O)-C ₂ H ₅ | 15 | 80 | 5 | 83 |

Table 3. Continued

| Entry | Sulfonamide | Acylying agent | Product | CH ₂ Cl ₂ ^a | | Solvent-free ^b | |
|-------|--|----------------|---------|--|----------|---------------------------|----------|
| | | | | Time(min) | Yield(%) | Time(min) | Yield(%) |
| 18 | H ₃ C-SO ₂ NH ₂ | | | 25 | 75 | 20 | 87 |
| 19 | H ₃ C-SO ₂ NH ₂ | | | 25 | 96 | 15 | 97 |
| 20 | | | | 160 | 80 | 120 | 90 |
| 21 | | | | 60 | 75 | 25 | 87 |
| 22 | | | | 45 | 83 | 20 | 85 |
| 23 | | | | 35 | 78 | 30 | 80 |
| 24 | | | | 30 | 97 | 25 | 98 |
| 25 | | | | 80 | 78 | 60 | 85 |
| 26 | | | | 90 | 75 | 75 | 80 |

^aReflux. ^b80 °C

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