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Mild and Solvent-free Synthesis and Antibacterial Evaluation of Novel Sulfonamides Containing Hydroxyl Groups

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A good range of interesting sulfonamides containing hydroxyl functional groups were synthesized under solvent-free conditions. The synthetic route involves the selective reaction of amino alcohols and sulfonating agents. The sulfonamides were obtained by a simple procedure after an easy work-up in high yield and purity. The resulting products were characterized by IR, ^{1}H NMR and ^{13}C NMR analyses. These compounds were tested for antibacterial activity against four bacteria including Escherichia Coli, Staphylococcus Aurous, Bacillus Subtilis and Salmonella Typhi.

Keyword: Sulfonamide, Solvent-free, Antibacterial, Amino alcohol

INTRODUCTION

Sulfonamides have found widespread uses in a number of pharmaceutical applications, such as antiprotozoal [1,2], antifungal [3], anti-inflammatory [4], antimalarial agents [5], carbonic anhydrase inhibitors [6], antiobesity [7], anticancer [8,9] and antialzheimer's [10]. Sulfonamides are the first successfully synthesized antimicrobial drugs [11]. For example, sulfamethoxazole is usually used in the treatment of urinary tract infections [12], bronchitis [13], and prostatitis [14]. Combination of sulfamethoxazole and trimethoprim can be used in the treatment of encephalomyelitis [15], or combination of sulfamethoxazole and azithromycin, in the therapy of ocular toxoplasmosis [16,17].

Commonly, sulfonamides are prepared from sulfonyl chlorides and amines. Aryl sulfonyl chlorides are typically prepared *via* electrophilic aromatic substitution using an excess of chlorosulfonic acid, or oxidative chlorination of sulfur compounds, such as thiols and sulfides, with aqueous chlorine [18] and trichloroisocyanuric acid [19]. Direct synthesis of sulfonamides obtained from sulfonic acids, one-pot synthesis of sulfonamides from Grignard reagents and SO_2, and also from aryl iodide are some other reported methods [20,21].

Amino alcohols are very important and versatile compounds with significant applications in synthetic and medicinal chemistry [22]. In continuation of our interest in synthesis of new sulfonamides [23-26], especially the selective sulfonation of amino alcohols [27], we wish to report here a mild and efficient chemoselective method for the synthesis of sulfonamides derived from amino alcohols, aminophenols and sulfonyl chlorides and their antibacterial effects.

MATERIAL AND METHODS

Apparatus and Analysis

All chemicals were purchased from Merck and Fluka chemical companies and used without further purification. Infrared spectra were recorded on Perkin-Elmer V IR spectrophotometer. ^{1}H NMR and ^{13}C NMR spectra were recorded on Bruker (400 MHz) Avance spectrophotometer in $CDCl_3$ or DMSO-d_6 as the solvent. Thin layer

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chromatography (TLC) was performed on silica gel G (Merck), and spots were isolated by the iodine vapor or by irradiation with UV light (254 nm).

General Procedure for the Synthesis of Sulfonamides

A mixture of amino alcohol (2 mmol), aryl sulfonyl chloride (2 mmol), and anhydrous $NaHCO_3$ (1 g) were stirred at room temperature under solvent-free conditions. The progress of the reaction was monitored by TLC. After completion of the reaction, the product was obtained by one of the two following procedures. Method A: (for the solid product), water was added to the mixture and the solid sulfonamide was collected by filtration and washed with additional water.

Method B: (for the oily product), water was added to the reaction mixture and the product was extracted with EtOAc (20 ml) and dried over $MgSO_4$. The solvent was evaporated and the product was obtained as oil. All of the products were obtained very pure.

Spectral Data of the Synthesized Sulfonamides

***N*-(3-Hydroxypropyl)-4-methylbenzenesulfonamide.** (Table 1, entry 1); oily viscose; R_f = 0.13 (40% ethyl acetate, 60% *n*-hexane); IR (KBr, cm^{-1}) = 3495, 3283 (N-H, O-H), 2949, 2880 (C-H), 1323, 1158 (SO_2), ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) = 1.72 (2H, q, *J* = 12.0 Hz), 2.45 (3H, s), 3.10 (2H, t, *J* = 12.4 Hz), 3.74 (2H, t, *J* = 11.2 Hz), 7.52 (2H, d, *J* = 8.4 Hz), 7.77 (2H, d, *J* = 8.0 Hz); ^{13}C NMR (100 MHz, DMSO-d_6) δ (ppm) = 21.5, 31.4, 40.9, 60.5, 127.0, 129.7, 136.7, 143.

***N*-(2-Hydroxyethyl)-4-methylbenzenesulfonamide.** (Table 1, entry 2); oily viscose; R_f = 0.19 (40% ethyl acetate, 60% *n*-hexane); IR (KBr, cm^{-1}) = 3286 (N-H, O-H), 2932, 2880 (C-H), 1321, 1157 (SO_2), 551 (C-N), ^{1}H NMR (400 MHz, $CDCl_3$) *δ* (ppm) = 2.42 (3H, s), 3.05 (2H, t, *J* = 10.0Hz), 3.6 (2H, t, *J* = 9.6 Hz), 4.10 (2H, s), 7.6 (2H, m), 7.7 (2H, d, *J* = 8.4 Hz); ^{13}C NMR (100 MHz, DMSO-d_6) *δ* (ppm) = 21.5, 45.2, 61.1, 127.0, 129.8, 136.6, 143.5.

***N*,*N*-Bis(2-hydroxyethyl)-4-methylbenzenesulfonamide.** (Table 1, entry 3); oily viscose; R_f = 0.27 (40% ethyl acetate, 60% *n*-hexane); IR (KBr, cm^{-1}) = 3297 (N-H, O-H), 2928, 2879 (C-H), 1336, 1161 (SO_2), 549 (C-N), ^{1}H NMR (400 MHz, $CDCl_3$) *δ* (ppm) = 2.45 (3H, s), 2.86 (1H, br), 3.27 (4H, br), 3.73 (1H, t, *J* = 5.2), 3.88 (4H, br), 7.32 (2H, d, *J* = 7.6 Hz), 7.71 (2H, d, *J* = 8.0 Hz), ^{13}C NMR (100 MHz, DMSO-d_6) *δ* (ppm) = 21.5, 50.87, 53.05, 62.2, 127.3, 129.8, 143.7.

***N*-(4-Hydroxyphenyl)-4-methylbenzenesulfonamide.** (Table 1, entry 4); m.p.: 174 °C; R_f = 0.57 (40% ethyl acetate, 60% *n*-hexane); IR (KBr, cm^{-1}) = 3452, 3241 (N-H, O-H), 1317, 1156 (SO_2), 567 (C-N),), ^{1}H NMR (400 MHz, $CDCl_3$) *δ* (ppm) = 1.61 (1H, s), 2.31 (3H, s), 6.24 (1H, s), 6.72 (2H, d, *J* = 8.4 Hz), 6.93 (2H,d, *J* = 8.8 Hz), 7.24 (2H, d, *J* = 8.0 Hz), 7.59 (2H, d, *J* = 8.0 Hz), ^{13}C NMR (100 MHz, DMSO-d_6) *δ* (ppm) = 21.5, 115.9, 123.5, 125.9, 127.3, 128.9, 129.0, 129.5, 143.5, 152.0.

***N*-Benzyl-*N*-(2-hydroxyethyl)-4-methylbenzenesulfonamide.** (Table 1, entry 5); m.p.: 182 °C; R_f = 0.4 (40% ethyl acetate, 60% *n*-hexane); IR (KBr, cm^{-1}) = 3555 (O-H), 2956, 2931 (C-H) 1315, 1155 (SO_2), 548 (C-N), ^{1}H NMR (400 MHz, $CDCl_3$) *δ* (ppm) = 2.26 (1H, br), 2.46 (3H, s), 3.24 (2H, t, *J* = 10.8 Hz), 3.48 (2H, t, *J* = 11. 2Hz), 4.37 (2H,s), 7.30-7.36 (7H, m), 7.76 (2H, d, *J* = 8.4 Hz), ^{13}C NMR (100 MHz, DMSO-d_6) *δ* (ppm) = 21.5, 50.6, 53.4, 60.9, 127.3, 128.0, 128.3, 128.8, 129.7, 129.9, 136.0, 136.3, 143.7

***N*-(2-(Hydroxymethyl)phenyl)-4-methylbenzenesulfonamide.** (Table 1, entry 6); m.p.: 178 °C; R_f = 0.45 (40% ethyl acetate, 60% *n* -hexane)**;** IR (KBr, cm^{-1}) = 3440 (N-H, O-H), 2922, 2875 (C-H), 1317, 1153 (SO_2), 561 (C-N); ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) = 1.97 (1H, br), 2.41 (3H, s), 4.41 (2H, s), 7.10-7.12, (2H, m), 7.25 (2H, d, *J* = 8.0 Hz), 7.28-7.30 (1H, m), 7.46 (1H, d, *J* = 8.0 Hz), 7.67 (2H, d, *J* = 8.0 Hz), 7.91 ($1H_{N-H}$, s), ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) = 21.5, 63.9, 123.4, 125.3, 127.0, 129.0, 129.3, 129.6, 131.6, 136.4, 136.9, 143.8.

***N*-(2-Hydroxyethyl)-4-methyl-*N*-phenylbenzenesulfonamide.** (Table 1, entry 7); m.p.: 174 °C; R_f = 0.66 (40% ethyl acetate, 60% *n* -hexane), IR (KBr, cm^{-1}) = 3537 (O-H), 2963, 2884 (C-H) 1333, 1164 (SO_2), 584 (C-N). ^{1}H NMR (400 MHz, $CDCl_3$) *δ* (ppm) = 2.44 (3H, s), 3.65 (2H, t, *J* = 10.4 Hz), 3.71 (2H, t, *J* = 6.8 Hz), 7.08-7.10 (2H, m), 7.27 (2H, d, *J* = 8.0 Hz), 7.32-7.33 (3H, m); 7.51 (2H, d, *J* = 8.0), 7.55-7.58 (2H, m), 7.85-7.88 (2H, m), ^{13}C NMR (100 MHz, DMSO-d_6) *δ* (ppm) = 21.6, 53.4, 60.3, 127.7,

128.2, 128.9, 129.2, 120.5, 135.0, 139.4, 143.7.

***N*-(2-Hydroxypropyl)benzenesulfonamide.** (Table 1, entry 8); oily viscose; R_f = 0.23 (40% ethyl acetate, 60% *n* -hexane); IR (KBr, cm^{-1}) = 3501, 3286 (N-H, O-H), 2976, 2929 (C-H), 1327, 1162 (SO_2), 587 (C-N), ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) = 1.11 (3H, d, *J* = 6.4 Hz), 2.78 (1H, dd, *J* = 15.4, *J* = 8.0 Hz), 3.00 (1H, dd, *J* = 15.2, *J* = 2.8 Hz), 3.27 (1H, br), 3.86-3.92 (1H, m), 5.94 (1H, br); 7.48-7.51 (2H, m), 7.55-7.58 (2H, m), 7.85-7.88 (2H, m), ^{13}C NMR (100 MHz, DMSO-d_6) δ (ppm) = 20.41, 50.0, 66.6, 127.0, 129.2, 132.7, 139.6.

***N*-(2-Hydroxyethyl)benzenesulfonamide.** (Table 1, entry 9); oily viscose; R_f = 0.2 (40% ethyl acetate, 60% *n* -hexane)**;** IR (KBr, cm^{-1}) = 3498, 3283 (N-H, O-H), 2939, 2882 (C-H), 1321, 1159 (SO_2), 586 (C-N), ^{1}H NMR (400 MHz, $CDCl_3$) *δ* (ppm) = 3.07 (2H, q, *J* = 4.0 Hz), 3.15 (1H, br), 3.67-3.69 (2H, m), 5.92 (1H, s), 7.49-7.53 (2H, m), 7.56-7.60 (1H, m), 7.87-7.90 (2H, m); ^{13}C NMR (100 MHz, DMSO-d_6) *δ* (ppm) = 45.25, 61.21, 127.0, 129.2, 132.8, 139.5.

***N*-(2-Hydroxyethyl)-*N*-phenylbenzenesulfonamide.** (Table 1, entry 10) m.p.: 164 °C; R_f = 0.46 (40% ethyl acetate, 60% *n*-hexane); IR (KBr, cm^{-1}) = 3530 (O-H), 2936, 2881 (C-H) 1343, 1162 (SO_2), 586 (C-N). ^{1}H NMR (400 MHz, $CDCl_3$) *δ* (ppm) = 2.525 (1H, br), 3.65-3.68 (2H, m), 3.72-3.75 (1H, m), 7.07-7.09 (2H, m), 3.31-3.35 (3H,m), 7.47-7.50 (2H, m); 7.59-7.65 (3H, m), 7.55-7.58 (2H, m), 7.85-7.88 (2H, m), ^{13}C NMR (100 MHz, DMSO-d6) *δ* (ppm) = 46.4, 53.5, 60.2, 61.0, 113.6,127.7, 128.3, 128.9, 129.3, 132.9, 138.0, 139.2.

***N*-Benzyl-*N*-(2-hydroxyethyl)benzenesulfonamide.** (Table 1, entry 11); m.p.: 172 °C; R_f = 0.38 (40% ethyl acetate, 60% *n* -hexane); IR (KBr, cm^{-1}) = 3581 (N-H, O-H) 1332, 1164 (SO_2), 554 (C-N), ^{1}H NMR (400 MHz, $CDCl_3$) *δ* (ppm) = 2.00 (1H, br), 3.28 (2H, t, *J* = 10.8Hz), 3.52 (2H, t, *J* = 10.4 Hz), 4.41 (2H, s), 7.37 (5H,m), 7.58 (2H, t, *J* = 15.2 Hz), 7.65 (1H, t, *J* = 14.8 Hz), 7.90 (2H, d, *J* = 7.2), ^{13}C NMR (100 MHz, DMSO-d6) *δ* (ppm) = 50.7, 53.5, 60.9, 127.2, 128.1,128.3, 128.8, 129.3, 132.8, 136.1, 139.0.

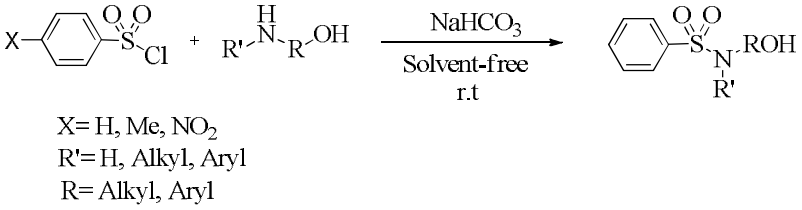
***N*-(2-(Hydroxymethyl)phenyl)benzenesulfonamide.** (Table 1, entry 12); m.p.: 168 °C; R_f = 0.4 (% 40 ethyl acetate, % 60 *n* -hexane); IR (KBr, cm^{-1}) = 3444 (N-H, O-H), 1313, 1156 (SO_2), 550 (C-N); ^{1}H NMR (400 MHz, $CDCl_3$) *δ* (ppm) = 2.13 (1H_{O-H}, br), 4.39 (2H, s), 7.12 (2H, d, *J* = 12.4 Hz), 7.27-7.31 (1H, m), 7.51 (3H, t, *J* = 15.6 Hz), 7.66 (1H, t, *J* = 14.4 Hz), 7.79 (2H, d, *J* = 7.6 Hz), 7.97 (1H, s), ^{13}C NMR (100 MHz, $CDCl_3$) *δ* (ppm) = 63.9, 123.5, 125.5, 127.0, 129.0, 129.0, 129.3, 131.6, 133.0, 136.2, 139.8.

***N*-(2-Hydroxypropyl)-4-nitrobenzenesulfonamide.** (Table 1, entry 13); oily viscose; R_f = 0.23 (40% ethyl acetate, 60% *n* -hexane); IR (KBr, cm^{-1}) = 3579 (N-H, O-H), 2946, 2878 (C-H), 1320, 1167 (SO_2), 561 (C-N), ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) = 1.20 (3H, d, *J* = 6.4 Hz), 1.92 (1H, br), 2.87 (1H, m), 3.173 (1H, m), 3.96 (1H, m), 5.37 (1H, s); 8.40 (2H, d, *J* = 8.8 Hz), 8.09 (2H, d, *J* = 8.8 Hz) ^{13}C NMR (100 MHz, DMSO-d_6) δ (ppm) = 20.8, 49.8, 66.6, 124.5, 128.3, 145.7,150.1.

***N*-(3-Hydroxypropyl)-4-nitrobenzenesulfonamide.** (Table 1, entry 14); oily viscose; R_f = 0.13 (40% ethyl acetate, 60% *n*-hexane), IR (KBr, cm^{-1}) = 3524, 3109 (N-H, O-H), 2961, 2899 (C-H), 1341, 1160 (SO_2), 551 (C-N); ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) = 1.76 (1H, 2HCH_2, m, *J* = 6.0 Hz), 3.22 (2H, q, *J* = 6.0 Hz) , 3.80 (2H, t, *J* = 11.2 Hz), 5.47 (br, 1H), 8.09 (2H, d, *J* = 8.4 Hz), 8.40 (2H, d, *J* = 8.8 Hz); ^{13}C NMR (100 MHz, DMSO-d6) *δ* (ppm) = 31.0, 41.9, 61.3, 124.4, 128.3, 145.9.

***N*-(3-Hydroxyphenyl)-4-nitrobenzenesulfonamide.** (Table 1, entry 15); m.p.: 175 °C; R_f = 0.48 (40% ethyl acetate, 60% *n*-hexane); IR (KBr, cm^{-1}) = 3440 (N-H, O-H), 2922, 2806 (C-H) 1317, 1153 (SO_2), 586 (C-N), ^{1}H NMR (400 MHz, $CDCl_3$) *δ* (ppm) = 4.42 (2H), 7.11-7.18 (2H, m), 7.34 (1H, t, *J* = 14.4 Hz), 7.54 (1H, d, *J* = 8.0 Hz), 7.99 (2H, d, *J* = 8.4 Hz), 8.27 (1H, d, *J* = 8.8 Hz), 8.51 (2H, d, *J* = 8.4 Hz), ^{13}C NMR (100 MHz, DMSO-d6) *δ* (ppm) = 107.9, 111.1, 114.0, 124.1, 128.4, 129.9, 138.9, 144.6, 149.4, 156.4.

***N*-(4-Hydroxyphenyl)-4-nitrobenzenesulfonamide.** (Table 1, entry 16); m.p.: 196 °C; R_f = 0.52 (40% ethyl acetate, 60% *n*-hexane); IR (KBr, cm^{-1}) = 3406, 3222 (N-H, O-H), 1325, 1164 (SO_2), 548 (C-N), ^{1}H NMR (400 MHz, $CDCl_3$) *δ* (ppm) = 6.65 (2H, d, *J* = 8.8 Hz), 6.70 (2H, d, *J* = 8.8 Hz), 7.89 (2H, d, *J* = 11.2 Hz), 8.32 (2H, d, *J* = 11.2 Hz), 9.42 (1H, s), 10.0 (1H, br), ^{13}C NMR (100 MHz, DMSO-d6) *δ* (ppm) = 116.1, 124.9, 125.1, 128.0, 128.7, 145.4, 150.1, 155.8.



Scheme 1

Table 1. Synthesis of Sulfonamides Containing the Hydroxyl Group and their Antibacterial Results[a]

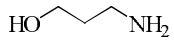
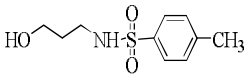
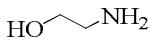
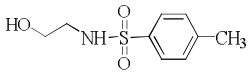
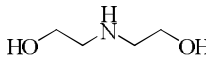
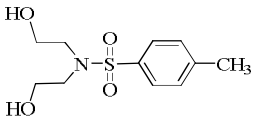
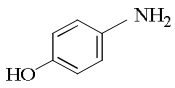
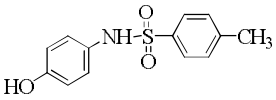
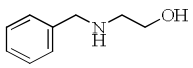
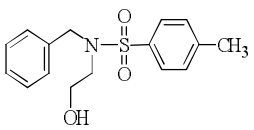
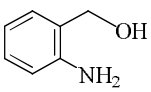
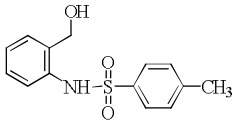
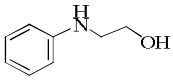
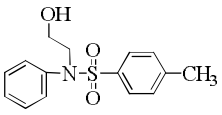
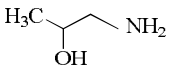
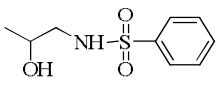
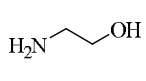
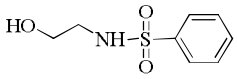
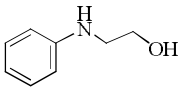
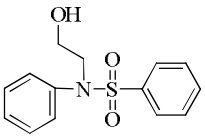
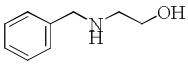
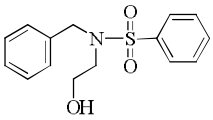
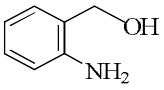
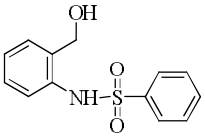
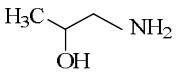
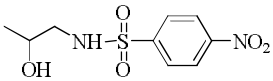
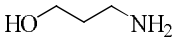
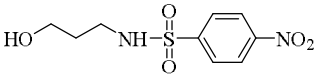
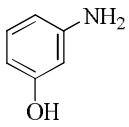
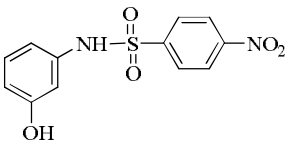
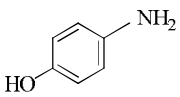
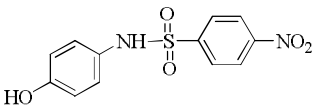
Entry	Amino alcohol	Sulfonamide	Time (min)	Yield (%)[b]	E. coli	S. aureus	Salmonella	Baciluse
1			40	65	-	715	715	-
2			25	55	-	715	357	-
3			65	50	715	178	178	178
4			200	92	715	178	178	90
5			100	85	715	178	178	178
6			180	80	-	-	-	178
7			90	85	715	178	715	-
8			35	65	-	-	357	-

Table 1. Continued

9			30	60	-	715	715	-
10			80	92	>715	715	715	-
11			90	91	715	178	178	90
12			150	79	-	-	-	357
13			25	65	-	715	357	-
14			20	75	-	90	-	-
15			130	86	357	45	-	-
16			120	85	715	357	357	45
17	Sulfa-methoxazole		-	-	24	12	24	12

[a]MICs, μg ml^{-1}. [b]Isolated yield.

RESULTS AND DISCUSSION

A series of sulfonamides containing a hydroxyl group were synthesized under solvent-free conditions (Table 1). The selective nucleophilic substitution reaction of the aliphatic and aromatic amino alcohols and also aminophenols with *p*-toluenesulfonyl chloride, benzenesulfonyl chloride and 4-nitro benzenesulfonyl chloride was carried out at room temperature under solvent-

free conditions. Anhydrous $NaHCO_3$ was used as an inorganic green solid base. Using K_2CO_3 as the base gives rise to di-sulfonylated byproducts in most cases. Sulfonations of aliphatic amino alcohols in the presence of $NaHCO_3$ were carried out in 20-65 min and the products were obtained in 50-75% yields in high purity. On the other hand, the aromatic amino alcohols and amino phenols were sulfonated in 90-200 min and the corresponding sulfonamides were obtained in 79-92% yields in high purity. In all cases the reactions were carried out selectively and the sulfonamides were obtained as the only product.

The synthesized sulfonamides were screened for their preliminary antibacterial activity using the macro broth dilution method against two Gram-positive bacteria, *S. aureus ATCC 25953 and Bacillus Subtilis ATCC 6633*, and two Gram-negative bacteria, *Esherchia coli ATCC 11230 and Salmonella typhi*, to determine the minimum inhibitory concentration (MIC). The MICs values were determined by comparison of them with sulfamethoxazole as the reference drug, the solvent DMSO as the control, and the results are presented in Table 1. The evaluated compounds showed moderate activities against the tested bacteria especially Gram-positive and lower antibacterial activity in comparison to those of sulfamethoxazole. Among them, *N*-(3-hydroxyphenyl)-4-nitrobenzenesulfonamide (Table 1, entry 15) and *N*-(4-hydroxyphenyl)-4-nitrobenzenesulfonamide (Table 1, entry 16) exhibited the most potent antibacterial activity against *S. aureus* and *Bacillus* as Gram-positive bacteria, respectively. The presence of nitro group may be the reason for this activity.

CONCLUSIONS

In conclusion, we synthesized several aryl and alkylsulfonamides containing hydroxy groups as antibacterial agents under solvent-free conditions. Some of the synthesized sulfonamides showed antibacterial activity against Gram-positive and Gram-negative bacterial strains.

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