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SYNTHESIS, CHARACTERIZATION, AND ANTIMICROBIAL STUDIES OF BISSYDNONES BASED ON 4,4′-DIAMINODIPHENYL METHANE

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The aim of this investigation is to present the synthesis and characterization of some 3,3′-(methylenedi-1,4-phenylene)bis(4-substituted aminosulfonyl)sydnone (7a–j) and to evaluate their antimicrobial activity. All newly synthesized compounds were characterized by spectral data as well as by elemental analysis. Some compounds showed excellent activity against Gram-positive and Gram-negative bacteria.

Keywords: Bissydnone; cyclization; 4,4′-diaminodiphenyl methane; mesoionic; microbial

INTRODUCTION

Mesoionic compounds have attracted widespread interest for a long time because of their unusual structure,[1] chemical properties,[2] and synthetic utility.[3] Sydnones are typical mesoionic compounds.[4] Several synthetic methodologies for sydnones have been developed in the past years. A hydrogen atom at the fourth position of the sydnone ring allows substitution with a wide variety of electrophiles, with retention of the ring, typical of aromatic substrates.[5] Since their discovery, sydnones have shown a variety of biological activities[6] carried out from a variety of standpoints.

Sydnones have played a crucial role in the development of theory in heterocyclic chemistry and occupy a unique place in organic chemistry. These observations encouraged us to continue our current work to synthesize new heterocyclic-containing bissydnone sulfonamides based on 4,4′-diamino diphenyl methane.

RESULTS AND DISCUSSION

Herein we describe the synthesis, characterization, and biological evaluation of novel 3,3′-(methylenedi-1,4-phenylene)bis(4-substituted amino sulfonyl)sydnone (**7a–j**). The synthesis of 3,3′-(4,4′-diphenyl)-bissydnonyl methane (**5**) was accomplished by a four-step procedure. Compound **5** was reacted with chlorosulfonic acid and then

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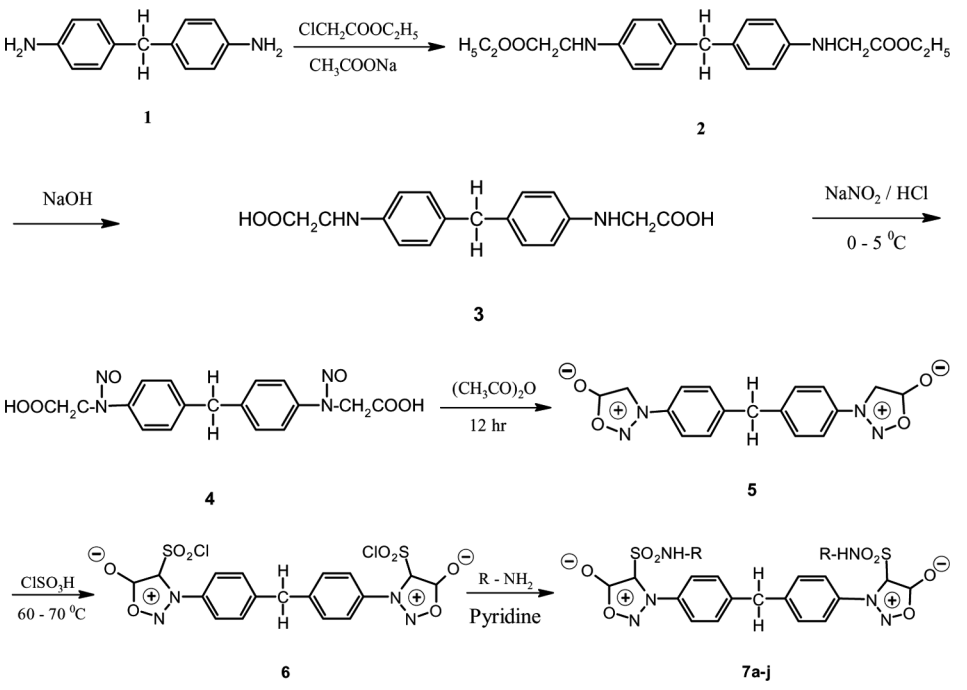
condensed with different amines to give 3,3′-(methylenedi-1,4-phenylene) bis(4-substituted aminosulfonyl) sydnone (**7a–j**), which is a well-established strategy for the synthesis of sulfonamides. Compounds **7a–j** were synthesized as shown in Scheme 1. The structures of the synthesized compounds were established on the basis of analytical and spectral data. The infrared (IR) spectrum of compound **5** showed absorption bands at 1745 cm^{-1}, characteristic of the C=O group. The ^1H NMR spectra of compound **5** showed a singlet at δ 7.76 ppm, characteristic of the proton at C_4 of the sydnone.

The IR spectrum of the compounds displayed absorption bands between 1719 and 1756 cm^{-1}, which is characteristic of the carbonyl group of sydnone, and also showed 1362–1367, 1157–1165, and 2923–2939 cm^{-1}, characteristic of SO_{2asy}, SO_{2sym}, and CH_2 groups. Furthermore, the ^1H NMR spectra of compounds **7a–j** showed signals in the range of δ 8.86–8.96 ppm and 4.61–5.25 ppm for the SO_2NH group and δ 6.80–8.90 ppm for aromatic protons. Further, the ^{13}C NMR spectra exhibited confirmatory signals for the carbonyl carbon around δ 167.00 ppm and the methylene carbon around δ 39.00 ppm. The test compounds **7a–j** were evaluated for their antibacterial activity on four important bacterial stains (Gram-possitive bacteria *Streptococcus pneumoniae* and *Staphylococcus aureus* and Gram-negative bacteria *Escherichia coli* and *Psudomanas aeruginosa*). From the results obtained, most of the compounds possess antibacterial activity. Compound **7h** was most active against Gram-positive bacterial stains and *P. aeruginosa* whereas compound **7e** was



Scheme 1. R =**7a**, 4-ClC_6H_4; **7b**, CH_3; 7c, C_6H_5; **7d**, 3-ClC_6H_4; **7e**, 2-FC_6H_4; **7f**, $CH_2(CH_2)_2CH_3$; **7g**, 3-$CH_3C_6H_4$; **7h**, CH_2-C_6H_5; **7i**, 2-$CH_3C_6H_4$; and **7j**, 4-$NO_2C_6H_4$.

Table 1. Antimicrobial activity of 3,3′-(methylenedi-1,4-phenylene)bis(4-substituted aminosulfonyl) sydnone (**7a–j**)

	Gram-positive organism				Gram-negative organism			
	S. pneumoniae		*S. aureus*		*E. coli*		*P. aeruginosa*	
Compound	IZ	MIC	IZ	MIC	IZ	MIC	IZ	MIC
7a	14	64	16	32	15	64	13	64
7b	15	64	14	64	10	—	13	64
7c	15	32	17	32	13	64	15	32
7d	10	—	18	32	10	—	12	64
7e	17	16	17	32	16	32	10	—
7f	17	16	15	64	14	64	16	32
7g	15	32	17	32	14	64	14	64
7 h	18	16	19	16	10	—	22	16
7i	14	64	14	64	13	64	12	128
7j	14	64	14	64	10	—	10	—
Streptomycin	40	0.25	40	0.125	28	1.0	34	0.5
Penicillin-G	35	0.25	45	0.125	30	0.5	38	0.25

Notes. IZ, inhibition of zone; MIC, minimum inhibitory concentration. Zone diameter of growth inhibition (mm) after 24 h.

most active against *E. coli*. However, compound **7d** was inactive against *S. pneumoniae*, compounds **7b, 7h,** and **7j** were inactive against *E. coli*, and compound **7e** and **7j** were inactive against *P. aeruginosa*. The rest of the compounds were moderate to less active against these species.

Biological Activity

One of the purposes of the present work was to synthesize new heterocyclic compounds that might be of biological interest. The synthesized compounds were tested for their antibacterial activity against Gram-positive and Gram-negative bacteria. Preliminary antimicrobial testing was carried out using the Kirby–Bauer technique.[8] The reference drugs used were streptomycin and penicillin-G, respectively. The activity of the samples and the reference drugs was assayed under identical conditions at 200 μg/mL concentration in N,N-dimethylformamide (DMF), and the zone of inhibition was measured in millimeters. The investigation results are listed in Table 1. In general, all the synthesized 3,3′-(methylenedi-1,4-phenylene)bis(4-substituted aminosulfonyl)sydnones (**7a–j**) exerted a wide range of modest antibacterial activity in vitro activity against the tested organisms.

EXPERIMENTAL

General Procedures

Melting points were determined by the open capillary method and are uncorrected. All compounds were analyzed satisfactorily for C, H, O, N, and S. IR spectra (KBr) were recorded on a Shimadzu (Japan) Fourier transform (FT)–IR spectrophotometer. ^{1}H NMR spectra were recorded on a Bruker Advance II 400-MHz

NMR spectrometer, and ^{13}C NMR spectra were recorded on Bruker Advance II 400-MHz NMR spectrometer using dimethylsulfoxide (DMSO-d_6) as a solvent and tetramethylsilane (TMS) as an internal standard. Elemental analysis was carried out on a Vairo-EL (Elementa) model. Purity of the compounds was checked by thin-layer chromatography (TLC) on silica-gel plates.

Synthesis of 3,3′-(Methylenedi-1,4-phenylene)bis(4-substituted amino sulfonyl) Sydnone Based on 4,4′-Diaminodiphenyl Methane

2,2′-[Methylenebis(1,4-phenyleneimino)]diethyl Acetate (2). 4,4′-Diaminodiphenyl methane (1.98 g, 0.01 mol), ethylchloroacetate (2.13 mL, 0.02 mol) in dry ethanol (10 mL), and anhydrous sodium acetate (3.28 g, 0.04 mol) were refluxed for 5 h. The mixture was diluted with water (10 mL). After standing overnight in the refrigerator, crystalline ester was obtained. The crude solid was purified by recrystallization from ethanol. Yield: 3.01 g, 80%, Mp 110–112 °C. IR (KBr): 2959, 2928, 2875, 2854, 1756, 1521, 1307 cm^{-1}. ^1H NMR (400 MHz, DMSO-d_6): δ 1.34 (t, 6H, CH_3), 3.95 (s, 2H, NH), 4.05 (s, 6H, CH_2), 4.35 (q, 4H, OCH_2), 6.50–7.00 (m, 8H, Ar-H). ^{13}C NMR (40 MHz, DMSO-d_6): δ 14.64, 40.50, 44.26, 61.24, 112.65, 128.95, 129.01, 145.87, 170.97. Anal. calcd. (%) for $C_{21}H_{26}N_2O_4$: C, 68.09; H, 7.07; O, 17.28; N, 7.56. Found: C, 68.13; H, 7.11; O, 17.25; N, 7.58.

2,2′-[Methylenebis(1,4-phenyleneimino)]diacetic Acid (3). Compound **2** (3.70 g, 0.01 mol) and sodium hydroxide (1.2 g, 0.03 mol) were dissolved in a solution of distilled water and ethanol (36 mL/4 mL). The mixture was stirred at reflux temperature for 30 min. The resultant mixture was cooled and acidified with hydrochloric acid. White crystalline product was obtained and recrystallized from ethanol. Yield: 2.23 g, 70%, Mp 130–135 °C. IR (KBr): 2925, 2856, 2570–3209, 1719, 1518, 1297 cm^{-1}. ^1H NMR (400 MHz, DMSO-d_6): δ 4.05 (s, 2H, CH_2), 4.10 (s, 4H, CH_2), 6.35 (s, 2H, NH), 6.40 (s, 2H, OH), 6.50–7.10 (m, 8H, Ar-H). ^{13}C NMR (40 MHz, DMSO-d_6): δ 40.82, 44.75, 112.28, 129.22, 129.47, 145.63, 171.54. Anal. calcd. (%) for $C_{17}H_{18}N_2O_4$: C, 64.96; H, 5.77; O, 20.36; N, 8.91. Found: C, 64.94; H, 5.79; O, 20.32; N, 8.96.

2,2′-{Methylenebis[4,1-phenylene (nitrosoimino)]}diacetic Acid (4)[7]. A freshly prepared sodium nitrate solution (3.32 g, 0.049 mol) was added dropwise to an ice-cold and well-stirred solution of compound **3** (5.02 g, 0.016 mol) in water (40 mL) over a period of 40 min. Concentrated hydrochloric acid was added until complete precipitation occured and was stirred in cold solution for several minutes. The solid nitroso compound was filtered off, washed with cold water, and dried. Yield: 3.80 g, 64%. Mp 130–135 °C. IR (KBr): 2925, 2853, 2570–3200, 1719, 1554, 1325 cm^{-1}. ^1H NMR (400 MHz, DMSO-d_6): δ 4.12 (s, 2H, CH_2), 4.84 (s, 2H, N-CH_2), 7.25–7.54 (m, 8H, Ar-H), 11.36 (s, 2H, COOH). ^{13}C NMR (40 MHz, DMSO-d_6): δ 40.61, 48.94, 122.88, 129.54, 137.20, 138.63, 167.61. Anal. calcd. (%) for $C_{17}H_{16}N_4O_6$: C, 54.84; H, 4.33; O, 25.78; N, 15.05. Found: C, 54.81; H, 4.37; O, 25.81; N, 14.98.

3,3′-(4,4′-Diphenyl)bissydnonyl Methane (5). The dried compound **4** (4.00 g, 0.0107 mol) was stirred for 12 h in 40 mL acetic anhydride. The solution was poured slowly into cold water, which was very well stirred. The pH of the

content was adjusted to 7.0 with 10% sodium bicarbonate solution. The solid crude product was washed well with water and dried. The crude sydnone was recrystallized from benzene–petroleum ether. The product obtained was an orange solid. Yield: 2.52 g, 70%. Mp 120–123 °C. IR (KBr): 3157, 2925, 2853 1745 cm^{-1}. ^1H NMR (400 MHz, DMSO-d_6): δ 4.26 (s, 2H, CH_2), 7.76 (s, 2H, sydnone), 7.14–8.13 (m, 8H, Ar-H). ^{13}C NMR (40 MHz, DMSO-d_6): δ 39.43, 121.89, 128.26, 129.20, 139.36, 141.15, 168.20. Anal. calcd. (%) for $C_{17}H_{12}N_4O_4$: C, 60.71; H, 3.60; O, 19.03; N, 16.66. Found: C, 60.68; H, 3.65; O, 19.98; N, 16.63.

3,3′-(Methylenedi-1,4-phenylene)bis(4-chlorosulfonyl) Sydnone (6). Chlorosulfonic acid (2.32 mL, 0.02 mol) was added dropwise into the mixture of compound **5** (3.36 g, 0.01 mol) and a catalytic amount of P_2O_5 over 30 min with constant stirring at 0–5 °C. The temperature of the well-stirred mixture did not rise above 5 °C. When all the chlorosulphonic acid was added, the mixture was refluxed at about 60 °C for about 1 h. The solution was then poured into a mixture of crushed ice and water with vigorous stirring. Precipitation was collected by filtration, washed three times with water, and dried. Yield: 3.94 g, 74%. Mp 239–241 °C. IR (KBr): 2928, 1745, 1395, 1180 cm^{-1}. ^1H NMR (400 MHz, DMSO-d_6): δ 4.25 (s, 2H, CH_2), 7.29–8.12 (m, 8H, Ar-H). ^{13}C NMR (40 MHz, DMSO-d_6): δ 39.65, 121.76, 122.97, 128.58, 138.86, 140.85, 169.64. Anal. calcd. (%) for $C_{17}H_{12}Cl_2N_4O_8S_2$: C, 38.14; H, 2.26; O, 23.91; N, 10.47; S, 11.98. Found: C, 38.12; H, 2.31; O, 23.95; N, 10.43; S, 12.03.

General Procedure for the Synthesis of 3,3′-(Methylenedi-1,4-phenylene)bis(4-primary substituted amino sulfonyl) Sydnone (7a–j)

3,3′-(Methylenedi-1,4-phenylene)bis(4-chlorosulfonyl)sydnone **6** (5.88 g, 0.011 mol) was dissolved in acetone at room temperature. A solution of appropriate primary amine (0.022 mol) in acetone was added dropwise into compound **6** solution over a period of 5 h with constant stirring; 1.0 mL of pyridine was added to the well-stirred solution after 1 and 2 h during the reaction. The solution was poured onto ice with stirring. The precipitate was collected by filtration, washed with water, and dried. Recrystallization of the crude product was performed from benzene.

3,3′-(Methylenedi-1,4-phenylene)bis(4-{[(4-chlorophenyl)amino]sulfonyl} Sydnone (7a). Yield: 4.33 g, 55%. Mp 130–132 °C; IR (KBr): 2927, 1739, 1365, 1159, 1076 cm^{-1}. ^1H NMR (400 MHz, DMSO-d_6): δ 4.28 (s, 2H, CH_2), 7.20–8.30 (m, 16H, Ar-H), 8.86 (s, 2H, SO_2NH); ^{13}C NMR (40 MHz, DMSO-d_6): δ 39.86, 121.03, 122.60, 129.52, 129.56, 130.92, 131.15, 135.50, 137.30, 141.12, 167.30. Anal. calcd. (%) for $C_{29}H_{20}Cl_2N_6O_8S_2$: C, 48.68; H, 2.82; O, 17.89; N, 11.74; S, 8.96. Found: C, 48.65; H, 2.76; O, 17.93; N, 11.78; S, 8.99.

3,3′-(Methylenedi-1,4-phenylene)bis{4-[(methylamino)sulfonyl] Sydnone (7b). Yield: 3.05 g, 53%. Mp 145–148 °C; IR (KBr): 2965, 2923, 2868, 1736, 1367, 1157 cm^{-1}. ^1H NMR (400 MHz, DMSO-d_6): δ 3.10 (s, 6H, CH_3), 4.29 (s, 2H, CH_2), 4.61 (s, 2H, SO_2NH), 7.3–8.4 (m, 8H, Ar-H); ^{13}C NMR (40 MHz, DMSO-d_6): δ 31.50, 39.89, 122.12, 129.45, 129.90, 137.56, 141.62, 169.30. Anal. calcd. (%) for $C_{19}H_{18}N_6O_8S_2$: C, 43.67; H, 3.47; O, 24.50; N, 16.08; S, 12.27. Found: C, 43.63; H, 3.43; O, 24.43; N, 15.98; S, 12.33.

3,3′-(Methylenedi-1,4-phenylene)bis[4-(anilinosulfonyl)] Sydnone (7c). Yield 4.84 g, 65%. Mp 154–156 °C; IR (KBr): 2927, 1743, 1362, 1164 cm^{-1}. ^1H NMR (400 MHz, DMSO-d_6): δ 4.25 (s, 2H, CH_2), 6.94–8.33 (m, 18H, Ar-H), 8.94 (s, 2H, SO_2NH); ^{13}C NMR (40 MHz, DMSO-d_6): δ 39.46, 121.50, 122.8, 125.20, 129.15, 129.54, 130.6, 136.45, 137.28, 140.56, 168.57. Anal. calcd. (%) for $C_{29}H_{22}N_6O_8S_2$: C, 53.86; H, 3.43; O, 19.79; N, 13.00; S, 9.92. Found: C, 53.83; H, 3.50; O, 19.76; N, 13.04; S, 9.85.

3,3′-(Methylenedi-1,4-phenylene)bis(4-{[(3-chlorophenyl)amino]sulfonyl} Sydnone (7d). Yield 4.57 g, 58%. Mp 129–131 °C; IR (KBr): 2932, 1735, 1365, 1157, 1096 cm^{-1}. ^1H NMR (400 MHz, DMSO-d_6): δ 4.24 (s, 2H, CH_2), 7.15–8.42 (m, 16H, Ar-H), 8.88 (s, 2H, SO_2NH); ^{13}C NMR (40 MHz, DMSO-d_6): δ 39.24, 119.10, 121.08, 121.89, 125.05, 129.34, 130.01, 131.13, 135.75, 138.32, 139.15, 141.6, 168.33. Anal. calcd. (%) for $C_{29}H_{20}Cl_2N_6O_8S_2$: C, 48.68; H, 2.82; O, 17.89; N, 11.74; S, 8.96. Found: C, 48.65; H, 2.85; O, 17.93; N, 11.78; S, 8.94.

3,3′-(Methylenedi-1,4-phenylene)bis(4-{[(2-fluorophenyl)amino]sulfonyl} Sydnone (7e). Yield: 4.21 g, 56%. Mp 134–136 °C; IR (KBr): 2933, 1737, 1366, 1233, 1159 cm^{-1}. ^1H NMR (400 MHz, DMSO-d_6): δ 4.29 (s, 2H, CH_2), 6.80–8.90 (m, 16H, Ar-H), 8.90 (s, 2H, SO_2NH); ^{13}C NMR (40 MHz, DMSO-d_6): δ 39.18, 116.19, 120.89, 122.93, 124.65, 126.65, 127.08, 129.21, 129.84, 139.15, 141.75, 159.76, 168.33. Anal. calcd. (%) for $C_{29}H_{20}F_2N_6O_8S_2$: C, 51.02; H, 2.95; O, 18.75; N, 12.31; S, 9.39. Found: C, 51.08; H, 2.93; O, 18.74; N, 12.36; S, 9.42.

3,3′-(Methylenedi-1,4-phenylene)bis{4-[(butylamino)sulfonyl] Sydnone (7f). Yield: 4.21 g, 63%. Mp 137–139 °C; IR (KBr): 2967, 2929, 2869, 2855, 1734, 1363, 1161 cm^{-1}. ^1H NMR (400 MHz, DMSO-d_6): δ 0.92 (t, 6H, CH_3), 1.72 (m, 4H, CH_2), 2.90 (t, 4H, $NHCH_2$), 4.24 (s, 2H, CH_2), 4.75 (s, 2H, SO_2NH), 7.23–8.30 (m, 8H, Ar-H); ^{13}C NMR (40 MHz, DMSO-d_6): δ 13.42, 21.05, 29.50, 39.31, 42.41, 121.82, 129.11, 129.54, 139.21, 141.12, 168.64. Anal. calcd. (%) for $C_{25}H_{30}N_6O_8S_2$: C, 49.49; H, 4.98; O, 21.10; N, 13.85; S, 10.57. Found: C, 49.53; H, 4.94; O, 21.16; N, 13.83; S, 10.51.

3,3′-(Methylenedi-1,4-phenylene)bis(4-{[(3-methylphenyl)amino]sulfonyl} Sydnone (7g). Yield: 4.53 g, 61%. Mp 149–151 °C; IR (KBr): 2950, 2939, 2879, 1739, 1366, 1165 cm^{-1}. ^1H NMR (400 MHz, DMSO-d_6): δ 2.25 (s, 6H, CH_3), 4.27 (s, 2H, CH_2), 6.9–8.31 (m, 16H, Ar-H), 8.96 (s, 2H, SO_2NH); ^{13}C NMR (40 MHz, DMSO-d_6): δ 21.75, 39.32, 118.32, 121.45, 121.97, 126.28, 128.76, 129.74, 129.76, 136.59, 139.78, 140.27, 141.54, 168.83. Anal. calcd. (%) for $C_{31}H_{26}O_8N_6S_2$: C, 55.18; H, 3.88; O, 18.97; N, 12.46; S, 9.50. Found: C, 55.15; H, 3.93; O, 18.93; N, 12.53; S, 9.55.

3,3′-(Methylenedi-1,4-phenylene)bis{4-[(benzylamino)sulfonyl]} Sydnone (7h). Yield: 4.38 g, 59%. Mp 180–182 °C; IR (KBr): 2939, 1742, 1365, 1162 cm^{-1}. ^1H NMR (400 MHz, DMSO-d_6): δ 4.26 (s, 2H, CH_2), 4.37 (s, 4H, CH_2), 5.25 (s, 2H, SO_2NH), 7.27–8.25 (m, 18H, Ar-H); ^{13}C NMR (40 MHz, DMSO-d_6): δ 39.74, 45.25, 121.50, 127.53, 128.96, 129.35, 129.50, 129.91, 138.74, 141.63, 142.23, 168.95. Anal. calcd. (%) for $C_{31}H_{26}N_6O_8S_2$: C, 55.18; H, 3.88; O, 18.97; N,12.46; S, 9.50. Found: C, 55.09; H, 3.84; O, 18.92; N, 12.55; S, 9.56.

3,3′-(Methylenedi-1,4-phenylene)bis(4-{[(2-methylphenyl)amino]sulfonyl} Sydnone (7i). Yield: 4.01 g, 54%. Mp 169–171 °C. IR (KBr): 2950, 2935, 2883, 1739, 1366, 1165 cm^{-1}. ^1H NMR (400 MHz, DMSO-d_6): δ 2.26 (s, 6H, CH_3), 4.30 (s, 2H, CH_2), 7.26–8.33 (m, 16H, Ar-H), 8.86 (s, 2H, SO_2NH); ^{13}C NMR (40 MHz, DMSO-d_6): δ 18.09, 39.43, 121.74, 123.96, 125.87, 127.81, 129.42, 129.45, 131.20, 132.04, 134.97, 138.26, 141.88, 168.84. Anal. calcd. (%) for $C_{31}H_{26}O_8N_6S_2$: C, 55.18; H, 3.88; O, 18.97; N, 12.46; S, 9.50. Found: C, 55.15; H, 3.83; O, 18.91; N, 12.52; S, 9.42.

3,3′-(Methylenedi-1,4-phenylene)bis(4-{[(4-nitrophenyl)amino]sulfonyl} Sydnone (7j). Yield 4.38 g, 54%. Mp 174–177 °C; IR (KBr): 2936, 1527, 1734, 1367, 1351, 1162 cm^{-1}. ^1H NMR (400 MHz, DMSO-d_6): δ 4.23 (s, 2H, CH_2), 7.33–8.36 (m, 16H, Ar-H), 8.96 (s, 2H, SO_2NH); ^{13}C NMR (40 MHz, DMSO-d_6): δ 39.38, 119.24, 122.13, 126.85, 129.24, 129.95, 138.54, 141.68, 143.03, 145.21, 168.53. Anal. calcd. (%) for $C_{29}H_{20}N_8O_{12}S_2$: C, 47.28; H, 2.74; O, 26.06; N, 15.21; S, 8.71. Found: C, 47.23; H, 2.78; O, 26.11; N, 15.24; S, 8.78.

CONCLUSION

The new sulfonamide derivatives of sydnone were synthesized and evaluated for their antibacterial activity. The activity varies with the different substituents on sulfonamide linkage. The potential of the compounds increases and minimum inhibitory concentration decreases.

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