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Synthesis and Antimicrobial Evaluation of Substituted (1,1'-Biphenyl)-4-yl(3-methylbenzofuran-2-yl)methanones by Suzuki Cross-Coupling Reaction[1]

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Abstract—A series of new substituted [1,1'-biphenyl]-4-yl(3-methylbenzofuran-2-yl)methanones was prepared by Suzuki cross-coupling from (4-bromophenyl)(3-methylbenzofuran-2-yl)methanone. The obtained compounds were assayed for their in vitro antibacterial activity against different types of bacterial strains; antifungal activity was also examined by inhibitory effect against different fungal strains.

Keywords: benzofuran, Suzuki cross-coupling, antimicrobial activity

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Benzofuran-containing compounds [1] are ubiquitous in nature, particularly among plants. Often such natural products possessing benzofuran core are endowed with useful pharmacological properties. This has generated enormous interest in synthetic products containing benzofuran moiety and has resulted in active development of benzofuran chemistry during last several years. Recent reviews showed that benzofuran [2] is clinically potent moiety possessing diverse biological activities and profound efficacy. Benzofuran derivatives possess a wide range of biological activitiy such as antimicrobial [3–5], antitumor [6, 7], anti-inflammatory [8], antibacterial [9], antifungal [10], antidepressant [11], analgesic [12], and hypoglycemic [13].

Aryl–aryl and aryl–vinyl bonds formation is very important in organic synthesis and has a wide range of applications of industrial interest, including synthesis of pharmaceuticals, polymers, herbicides, and the other materials [14, 15]. Some natural biaryl derivatives can be used as drugs and an example of this is Mastigophorene A [16]. The palladium-catalyzed reaction between organoboron compounds and organic halides that led to the formation of carbon–carbon bond has been first reported by Akira Suzuki and Miyayura and it was a revolution in organic couplings [17]. For the synthesis of biaryl compounds, palladium-catalyzed Suzuki-Miyaura coupling is the most attractive method [18–21]. The most suitable catalyst for Suzuki-Miyaura coupling is $Pd(PPh_3)_4$ because of its tolerance to the functional groups and low sensitivity towards water and air, so that the reactions can take place even in aqueous medium [22].

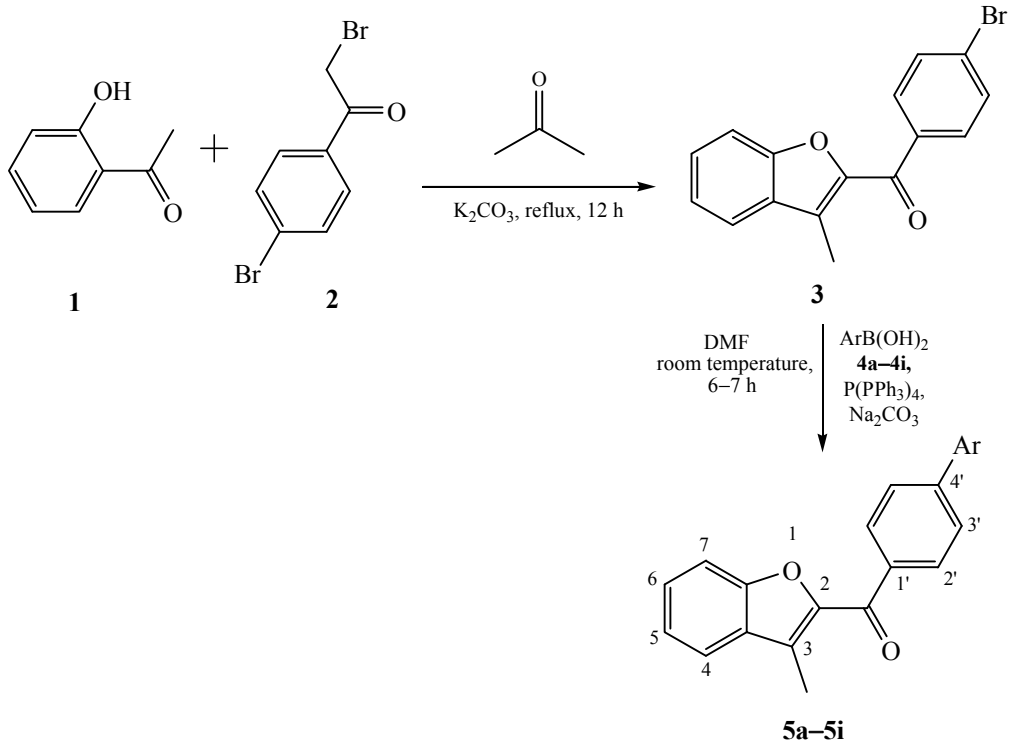
Therefore being inspired with the potential bioactivity of benzofuran and biaryl moieties, we have undertaken the synthesis of some new benzofuran–biaryl derivatives using Suzuki cross-coupling reaction. All the synthesized compounds have been tested in vitro for their antibacterial and antifungal activities.

Synthesis of new benzofuran derivatives **5a–5i** was carried out according to Scheme 1. The carbon–carbon bond was formed by reacting (4-bromophenyl)(3-methylbenzofuran-2-yl)methanone [23] with various arylboronic acids **4a–4i** with the formation of target compounds [1,1'-biphenyl]-4-yl(3-methylbenzofuran-2-yl)methanones **5a–5i** (Table 1).

The proposed structure for compound **5a** was confirmed by the spectral data as [1,1'-biphenyl]-4-yl-(3-methylbenzofuran-2-yl)methanone. In the IR spectrum of **5a**, the peaks at 1077 and 1749 cm^{-1} confirmed the presence of C–O–C motif and carbonyl groups respectively. In ^1H NMR spectrum of **5a** a singlet at δ = 2.68 ppm corresponding to the resonance of CH_3 protons was registered. In ^{13}C NMR spectrum of the discussed compound a signal δ 10.08 ppm corresponding to the methyl carbon resonance and another signal at δ = 185.4 ppm corresponding to resonance of

[1] The text was submitted by the authors in English.

Scheme 1. Synthesis of substituted [1,1'-biphenyl]-4-yl(3-methylbenzofuran-2-yl)methanones.



the conjugated ketone functionality confirmed formation of benzofuran skeleton. Main peak in the mass spectrum of compound **5a** at *m*/*z* 313 corresponding to $[M + H]^+$ ion also confirmed the proposed Suzuki coupled product.

All the compounds were screened for their antibacterial activity [24] against *Staphylococcus aureus*, *Bacillus subtilis*, *Pseudomonas aeruginosa* and *Escherichia coli* using Ampicillin as a reference drug.

Table 1. Synthesis of compounds **5a–5i**

Comp no.	Ar	Time, h	Yield, %
5a	Ph	6	76
5b	4-FC_6H_4	6	72
5c	4-ClC_6H_4	7	74
5d	4-BrC_6H_4	6	70
5e	2,4-$Cl_2C_6H_3$	7	68
5f	4-MeC_6H_4	7	75
5g	4-$MeOC_6H_4$	6	73
5h	4-$CH_3C(O)C_6H_4$	7	65
5i	1-Naphth	6	74

The activity was determined using cup plate agar diffusion method by measuring the zone of inhibition in mm. The compounds were screened at concentration 50 μg/mL in DMSO. From the screening study it was evident that compounds **5a**, **5e**, **5f**, and **5g** showed good antibacterial activity against all the tested organisms (Table 2).

In addition, all the compounds were screened for their antifungal activity [25] against *Aspergillus nigerzeae*, *Penicillium italicum* and *Fusarium oxysporum* using Grieseofulvin as a reference drug. The activity was determined using cup plate agar diffusion method by measuring the zone of inhibition in mm. The compounds were screened at concentration 50 μg/mL in DMSO. The screening revealed that compounds **5a**, **5e**, and **5f** showed good antifungal activity against all the tested organisms (Table 3).

In summary, substituted [1,1'-biphenyl]-4-yl(3-methylbenzofuran-2-yl)methanones were successfully synthesized by Suzuki cross-coupling reaction. Title compounds were prepared in good yields with $Pd(PPh_3)$ in DMF in the presence of Na_2CO_3. Antibacterial and antifungal activity studies revealed that some of them showed good antibacterial and antifungal activity against all the tested organisms.

Table 2. Antibacterial activity of compounds **5a–5i**

Compound	Zone of inhibition, mm			
	gram positive bacteria		gram negative bacteria	
	Staphylococcus aureus	*Bacillus subtilis*	*Pseudomonas aeruginosa*	*Escherichia coli*
5a	29	11	10	27
5b	20	7	5	22
5c	18	10	6	18
5d	15	9	8	21
5e	28	11	9	28
5f	28	11	8	26
5g	27	10	8	28
5h	18	10	4	20
5i	20	8	9	24
Ampicillin	30	12	10	30

EXPERIMENTAL

Stuart SMP30 apparatus was used for determination of melting points. Purity of the compounds was checked by TLC on silica gel 60 F_{254} (Merck). ^{1}H and ^{13}C NMR spectra were recorded on a Bruker Avance II 400 spectrometer using TMS as an internal reference. IR spectra were recorded in KBr on a Shimadzu FTIR 8400S spectrophotometer. Mass spectra were recorded on a Shimadzu GCMS-QP 1000 mass spectrometer. Elemental analysis was carried out on a Vario-11CHN analyzer.

General procedure for the synthesis of [1,1'-biphenyl]-4-yl(3-methylbenzofuran-2-yl)methanones (5a–5i). $Pd(PPh_3)_4$ (0.10 mmol), arylboronic acid (1.30 mmol) **4a–4i** and Na_2CO_3 (3.00 mmol) were added sequentially to a solution of 4-(bromophenyl)(3-methylbenzofuran-2-yl)methanone **3** (1 mmol) in DMF (5 mL). The round-bottom flask with the reaction mixture was filled with nitrogen for 15 min. Then, the reaction mixture was stirred at 80°C for 6–7 h under nitrogen. The mixture was cooled to room temperature, diluted with EtOAc (15 mL), and washed with H_2O and brine. The organic layer was dried over anhydrous magnesium sulfate. The solvent was evaporated and the residue was purified by the means of silica gel column chromatography eluting with ethylacetate–hexane (1 : 3) to afford pure product **5a–5i**.

[1,1'-Biphenyl]-4-yl(3-methylbenzofuran-2-yl)-methanone (5a). White solid, mp 152–154°C. IR spectrum, ν, cm^{-1}: 1077 (C–O–C), 1749 (C=O). ^{1}H NMR spectrum (400 MHz, DMSO-d_6), δ, ppm: 2.68 s (3H, CH_3), 7.33–7.52 m (5H, Ar–H), 7.66–7.73 m (3H, Ar–H), 7.76 d (2H, H$^{3'}$, J = 8.53 Hz), 7.99 d (1H, H$^{3'}$, J = 8.78 Hz), 8.19 d (2H, H$^{2'}$, J = 8.53 Hz). ^{13}C NMR spectrum (101 MHz, DMSO-d_6), δ_C, ppm: 10.0, 112.5, 121.4, 123.3, 123.4, 127.0, 127.3, 128.1, 128.2, 128.9, 130.4, 131.3, 131.6, 136.5, 140.0, 145.3, 148.4, 154.3, 185.4. Found, %: C 84.62; H 5.20; O 10.27. $C_{22}H_{16}O_2$. Calculated, %: C 84.59; H 5.16; O 10.24. M 313 $[M + H]^+$.

Table 3. Antifungal activity of compounds **5a–5i**

Compound	Zone of inhibition, mm		
	Aspergillus nigerzeae	*Penicillium italicum*	*Fusarium oxysporum*
5a	15	25	28
5b	7	15	18
5c	6	12	14
5d	8	10	10
5e	14	21	23
5f	14	21	24
5g	8	14	19
5h	10	18	15
5i	9	14	20
Griseofulvin	12	20	25

(4'-Fluoro-[1,1'-biphenyl]-4-yl)(3-methylbenzofuran-2-yl)methanone (5b). White solid, mp 150–152°C. IR spectrum, ν, cm^{-1}: 1077 (C–O–C), 1749 (C=O). ^1H NMR (400 MHz, DMSO-d_6), δ, ppm: 2.68 s (3H, CH_3), 7.17–7.34 m (4H, Ar–H), 7.51–7.58 m (3H, Ar–H), 7.71–7.40 m (3H, Ar–H, $H^{3'}$), 8.19 d (2H, $H^{2'}$, J = 8.53 Hz). ^{13}C NMR spectrum (101 MHz, DMSO-d_6), δ_C, ppm: 10.0, 112.2, 116.2, 121.4, 123.3, 124.5, 124.6, 127.0, 128.2, 128.9, 129.0, 129.2, 129.7, 129.8, 129.9, 130.6, 130.7, 136.8, 140.1, 154.3, 185.4. Found, %: C 80.03; H 4.60; O 9.72. $C_{22}H_{15}FO_2$ Calculated, %: C 79.99; H 4.58; O 9.69. M 331 $[M + H]^+$.

(4'-Chloro-[1,1'-biphenyl]-4-yl)(3-methylbenzofuran-2-yl)methanone (5c). White solid, mp 161–163°C. IR spectrum, ν, cm^{-1}: 1078 (C–O–C), 1749 (C=O). ^1H NMR spectrum (400 MHz, DMSO-d_6), δ, ppm: 2.68 s (3H, CH_3), 7.15–7.34 m (4H, Ar–H), 7.49–7.58 m (3H, Ar–H), 7.71–7.40 m (3H, Ar–H, $H^{3'}$), 8.19 d (2H, $H^{2'}$, J = 8.53 Hz). ^{13}C NMR spectrum (101 MHz, DMSO-d_6), δ_C, ppm: 10.0, 111.2, 116.4, 121.4, 123.7, 124.5, 124.6, 127.0, 128.2, 128.9, 129.0, 129.1, 129.7, 129.7, 129.9, 130.6, 130.7, 137.8, 140.1, 154.3, 185.4. Found, %: C 76.21; H 4.39; O 9.26. $C_{22}H_{15}ClO_2$. Calculated, %: C 76.19; H 4.36; O 9.23. M 347 $[M + H]^+$.

(4'-Bromo-[1,1'-biphenyl]-4-yl)(3-methylbenzofuran-2-yl)methanone (5d). White solid, mp 154–156°C. IR spectrum, ν, cm^{-1}: 1074 (C–O–C), 1749 (C=O). ^1H NMR spectrum (400 MHz, DMSO-d_6), δ, ppm: 2.67 s (3H, CH_3), 7.18–7.37 m (4H, Ar–H), 7.47–7.58 m (3H, Ar–H), 7.71–7.40 m (3H, Ar–H, $H^{3'}$), 8.23 d (2H, $H^{2'}$, J = 8.53 Hz). ^{13}C NMR spectrum (101 MHz, DMSO-d_6), δ_C, ppm: 10.0, 111.2, 116.4, 121.4, 123.7, 124.5, 124.6, 127.0, 128.2, 128.9, 129.0, 129.1, 129.7, 129.7, 129.9, 130.6, 130.7, 137.8, 140.1, 154.3, 185.4. Found, %: C 67.56; H 3.90; O 8.20. $C_{22}H_{15}BrO_2$. Calculated, %: C 67.53; H 3.86; O 8.18. M 3911 $[M + H]^+$.

(2',4'-Dichloro-[1,1'-biphenyl]-4-yl)(3-methylbenzofuran-2-yl)methanone (5e). White solid, mp 148–150°C. IR spectrum, ν, cm^{-1}: 1077 (C–O–C), 1749 (C=O). ^1H NMR spectrum (400 MHz, DMSO-d_6), δ, ppm: 2.69 s (3H, CH_3), 7.20–7.37 m (3H, Ar–H), 7.47–7.58 m (3H, Ar–H), 7.71–7.38 m (3H, Ar–H, $H^{3'}$), 8.20 d (2H, $H^{2'}$, J = 8.53 Hz). ^{13}C NMR spectrum (101 MHz, DMSO-d_6), δ_C, ppm: 10.0, 111.2, 116.4, 121.4, 123.7, 124.5, 124.6, 127.0, 128.2, 128.9, 129.0, 129.1, 129.7, 129.7, 129.9, 130.6, 130.7, 137.8, 140.1, 154.3, 185.4. Found, %: C 69.33; H 3.75; O 8.43. $C_{22}H_{14}Cl_2O_2$. Calculated, %: C 69.31; H 3.70; O 8.39. M 381 $[M + H]^+$.

(4'-Methyl-[1,1'-biphenyl]-4-yl)(3-methylbenzofuran-2-yl)methanone (5f). White solid, mp 142–144°C. IR spectrum, ν, cm^{-1}: 1075 (C–O–C), 1747 (C=O). ^1H NMR spectrum (400 MHz, DMSO-d_6), δ, ppm: 2.41 s (3H, CH_3), 2.67 s (3H, CH_3), 7.33–7.48 m (5H, Ar–H), 7.64–7.71 m (3H, Ar–H), 7.76 d (2H, $H^{3'}$, J = 8.53 Hz), 7.99 d (1H, $H^{3'}$, J = 8.78 Hz), 8.21 d (2H, $H^{2'}$, J = 8.53 Hz). ^{13}C NMR spectrum (101 MHz, DMSO-d_6), δ_C, ppm: 10.4, 22.4, 111.5, 122.2, 123.5, 124.8, 127.4, 127.4, 128.1, 128.9, 130.7, 131.6, 136.5, 140.0, 148.4, 154.3, 184.5. Found, %: C 84.67; H 5.58; O 9.84. $C_{23}H_{18}O_2$. Calculated, %: C 84.64; H 5.56; O 9.80. M 327 $[M + H]^+$.

(4'-Methoxy-[1,1'-biphenyl]-4-yl)(3-methylbenzofuran-2-yl)methanone (5g). White solid, mp 168–170°C. IR spectrum, ν, cm^{-1}: 1079 (C–O–C), 1750 (C=O). ^1H NMR spectrum (400 MHz, DMSO-d_6), δ, ppm: 2.71 s (3H, CH_3), 3.94 s (3H, OCH_3), 7.34–7.54 m (4H, Ar–H), 7.63–7.78 m (3H, Ar–H), 7.81 d (2H, $H^{3'}$, J = 8.53 Hz), 7.87 d (1H, $H^{3'}$, J = 8.78 Hz), 8.09 d (2H, $H^{2'}$, J = 8.53 Hz). ^{13}C NMR spectrum (101 MHz, DMSO-d_6), δ_C, ppm: 10.4, 54.5, 112.5, 121.2, 123.3, 126.8, 127.4, 128.4, 128.1, 128.9, 130.1, 130.7, 131.6, 136.5, 140.0, 145.3, 148.4, 154.3, 184.5. Found, %: C 80.72; H 5.33; O 14.06 $C_{23}H_{18}O_3$. Calculated, %: C 80.68; H 5.30; O 14.02. M 343 $[M + H]^+$.

1-(4'-(3-Methylbenzofuran-2-carbonyl)-[1,1'-biphenyl]-4-yl)ethanone (5h). White solid, mp 165–167°C. IR spectrum, ν, cm^{-1}: 1072 (C–O–C), 1744 (C=O). ^1H NMR spectrum (400 MHz, DMSO-d_6) δ, ppm: 2.65 s (3H, CH_3), 2.72 s (3H, CO–CH_3), 7.20–7.37 m (5H, Ar–H), 7.47–7.58 m (3H, Ar–H), 7.72 d (2H, $H^{3'}$, J = 8.53 Hz), 8.20 d (2H, $H^{2'}$, J = 8.53 Hz). ^{13}C NMR spectrum (101 MHz, DMSO-d_6), δ_C, ppm: 10.0, 111.2, 115.4, 119.4, 123.7, 124.2, 124.6, 127.0, 128.2, 128.9, 129.0, 129.1, 129.7, 129.7, 129.9, 130.6, 130.7, 137.8, 140.1, 154.3, 185.4. Found, %: C 81.37; H 5.16; O 13.59. $C_{24}H_{18}O_3$. Calculated, %: C 81.34; H 5.12; O 13.54. M 355 $[M + H]^+$.

(3-Methylbenzofuran-2-yl)[4-(naphth-1-yl)phenyl]-methanone (5i). White solid, mp 140–142°C. IR spectrum, ν, cm^{-1}: 1075 (C–O–C), 1748 (C=O). ^1H NMR spectrum (400 MHz, DMSO-d_6), δ, ppm: 2.71 s (3H, CH_3), 7.34–7.75 m (10H, Ar–H), 7.90–7.95 m (3H, Ar–H), 8.25 d (2H, $H^{2'}$, J = 8.53 Hz). ^{13}C NMR spectrum (101 MHz, DMSO-d_6), δ_C, ppm: 10.1, 112.3, 121.4, 123.3, 125.3, 125.7, 126.0, 126.3, 127.0, 128.2, 128.3, 128.4, 129.2, 129.8, 130.1, 131.2, 133.8, 136.6, 139.2, 145.3, 148.4, 154.3, 185.5. Found, %: C 86.19;

H 5.06; O 8.87. $C_{28}H_{18}O_2$. Calculated, %: C 86.16; H 5.01; O 8.83. *M* 363 $[M + H]^+$.

Biological assay. All synthesized compounds were screened for their antibacterial activity against different types of bacterial strains, namely gram negative bacterial strains of *Pseudomonas aeruginosa and Escherichia coli*, and gram positive bacterial strains of *Bacillus subtilis and Staphylococcus aeureus*.

The cultures were diluted with 5% saline and autoclaved; the final volume was of approximate concentration 10^5–10^6 CFU/mL. Benzofuran derivatives **5a–5i** were dissolved in dimethyl sulfoxide to achieve concentration 50 μg/mL for antibacterial activity measurement. For agar disc diffusion method, liquid form of the test compound was soaked on the disc and then allowed to dry in air, such that the disc got completely saturated with the test compound. The saturated discs were introduced onto the upper layer of the medium evenly floated with the bacterial strain. Composition of the nutrient agar medium for the bacteria was agar (15 g), peptone (5 g), yeast extract (3 g), NaCl (5 g), and distilled water (1000 mL).

The discs were dipped in different samples with the investigated compounds, then were placed over the evenly spread bacterial nutrient media and incubated at 37°C for 24 to 48 h to achieve the best results. Afterwards zones of inhibition were measured. All the experiments were carried out three times and the results were expressed as zone of inhibition in mm (Table 2). These zones of inhibition for the synthesized compounds were compared with the zone of inhibition for the referent antibiotic Amoxicillin used at concentration of 50 μg/mL which was also tested under the same conditions against these bacterial species.

Antifungal activity of the synthesized compounds was tested against three pathogenic fungi, namely *Aspergillus nigerzeae*, *Penicillium italicum*, and *Fusarium oxysporum* by the poison plate technique.

The fungi were incubated in PDA at 25±1°C for 5 days to get new mycelium for antifungal assay; then a mycelia as disks of approximately 0.45 cm diameter cut from the culture medium were picked up with a sterilized inoculation needle and inoculated in the center of PDA plate. The test compounds were dissolved in dimethyl sulfoxide, then added to the ptato dextrose agar medium (PDA). Final concentration of the compounds in the medium was adjusted to 50 μg/mL. The inoculated plates were incubated at 25±1°C for 5 days. Grieseofulvin used as fungicide reference drug at concentration 50 μg/mL was also tested under the same conditions against the discussed fungi. Each treatment was done three times. The radial growth of the fungal colonies was measured on the sixth day. In vitro inhibition effect of the test compounds towards the fungi was calculated in terms of inhibition zone in mm (Table 3). The composition of potato dextrose agar medium for fungi was: potatoes (200 g), dextrose (20 g), agar (20 g), and distilled water (1000 mL).

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