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Easy and Efficient Microwave-Assisted Synthesis of 1,2,3-Triazolyl-Tethered 2-Pyridinylbenzimidiazoles and Their Antimicrobial Activity

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Abstract—A series of novel 1,2,3-triazole-tethered 2-pyridinylbenzimidazole derivatives have been synthesized form 2-(pyridine-3-yl)benzimidazole under microwave irradiation. The microwave-assisted method proved to be simple and easy-to-perform and provided high yields within a short time. The structure of the synthesized compounds was confirmed by different spectroscopic techniques such as IR, ^{1}H and ^{13}C NMR, and mass spectrometry. The synthesized compounds were screened for their antimicrobial activity against four bacterial strains and two fungal strains.

Keywords: 1,2,3-triazole, 2-pyridylbenzimidazole, microwave irradiation, click reaction, antimicrobial activity

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INTRODUCTION

Benzimidazole core is present in some marketed drugs. Examples are Albendazole (used to treat infections caused by dog and pork tapeworms), Veliparib (a potential anticancer drug acting as a PARP inhibitor), Nocodazole (antineoplastic agent), Adibenden (potential phosphodiesterase inhibitor), and Lerisetron (5-HT3 antagonist) (Fig. 1). In addition, various benzimidazole derivatives were reported to exhibit a wide variety of biological activities, including antimicrobial [1], anticancer [2], anti-inflammatory [3], antioxidant [4], antidiabetic [5], anti-HIV [6], and anticonvulsant [7] activities. Moreover, 1,2,3-triazole and its derivatives have attracted considerable attention over the past few decades due to their chemotherapeutical value as

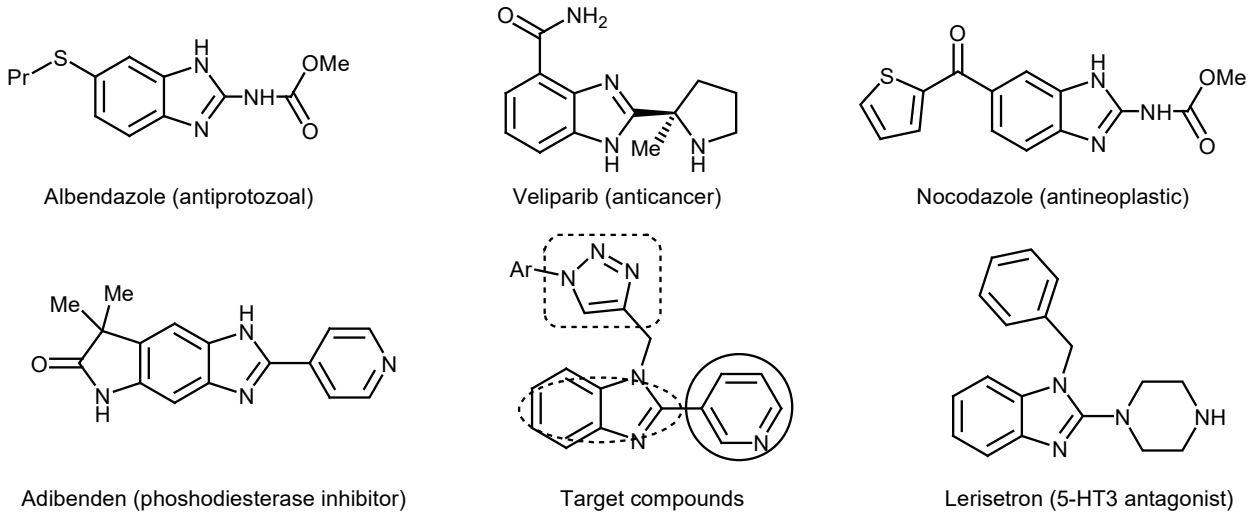
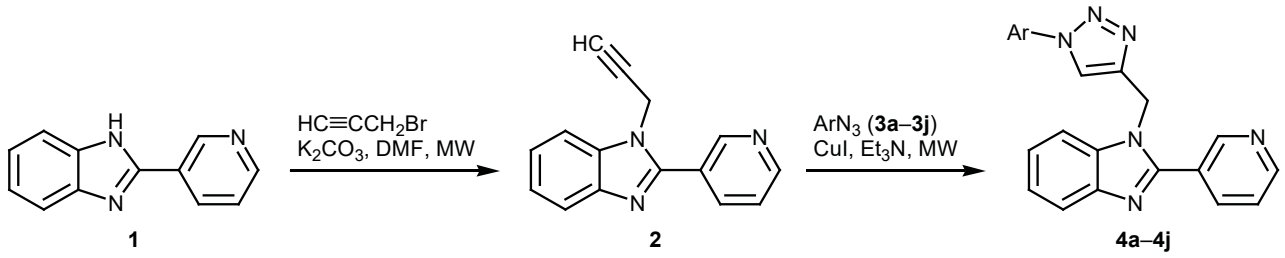


Fig. 1. Representative marketed drugs containing a benzimidazole nucleus.

Scheme 1.



Ar = Ph (**a**), 4-MeC_6H_4 (**b**), 4-$MeOC_6H_4$ (**c**), 2-$MeOC_6H_4$ (**d**), 4-ClC_6H_4 (**e**), 4-$O_2NC_6H_4$ (**f**), 2-$O_2NC_6H_4$ (**g**), 2-Cl-4-$O_2NC_6H_3$ (**h**), 4-FC_6H_4 (**i**), 4-BrC_6H_4 (**j**).

antimicrobial [8], anti-inflammatory [9], anticonvulsant [10], anticancer [11], anti-HIV [12], and anti-tubercular agents [13].

On the other hand, recent green chemistry methods have gained popularity as a nonconventional technique for rapid organic synthesis. There is increasing interest in the use of microwave irradiation in organic synthesis due to several advantages like mild conditions, clean reactions, convenient operation, high selectivity, simple workup, spontaneity of the reaction process, and environmental friendliness. As part of our ongoing research program, the above listed facts encouraged us to design and synthesize a novel series of benzimidazole derivatives, 1-[(1-aryl-1*H*-1,2,3-triazol-4-yl)-methyl]-2-(pyridin-3-yl)-1*H*-benzimidazoles, under microwave irradiation and evaluate them for antimicrobial activity.

RESULTS AND DISCUSSION

The synthetic route to novel 1,2,3-triazole-tethered 2-pyridinylbenzimidazole derivatives is shown in Scheme 1. The key starting material was 2-(pyridine-3-yl)-1*H*-benzimidazole (**1**) which was prepared as per literature procedure by the reaction of *o*-phenylenediamine with pyridine-3-carbaldehyde. Compound **1** was alkylated with propargyl bromide in DMF using K_2CO_3 as a base under microwave irradiation at 180 W for 4 min to afford 1-(prop-2-yn-1-yl)-2-(pyridin-3-yl)-1*H*-benzimidazole (**2**) in 94% yield.

The synthesis of compound **4a** via copper-catalyzed azide–alkyne cycloaddition of **2** and phenyl azide (**3a**) was selected as a model process (Scheme 2). Initially, the reaction of **2** and **3a** was carried out in the presence of copper(I) iodide in four different solvents like triethylamine (TEA), ethyl(diisopropyl)amine (DIPEA), DMF, and DMSO. The maximum yield of **4a** (68%) was obtained in TEA at room temperature. In our effort to increase the yield, the model reaction was carried out under microwave irradiation, other conditions being equal. Surprisingly, a remarkable improvement in the yield from 68% to 94% was observed. Encouraged by this result, we studied the effect of microwave power and performed a series of experiments at 100, 180, 360, and 480 W. As expected, the highest yield within the shortest reaction time was obtained in the reaction performed at an MW power of 360 W (Table 1, entry no. 9).

Next, the scope and generality of the described reaction were examined by reacting 1-(prop-2-yn-1-yl)-2-(pyridin-3-yl)-1*H*-benzimidazole (**2**) with differently substituted aryl azides **3a**–**3j** under the optimized conditions. The results showed that both electron-donating and electron-withdrawing substituents in the aryl azide were well tolerated, and the corresponding products **4a**–**4j** were obtained in good yields.

Antimicrobial activity. Compounds **4a**–**4j** were tested in vitro against four bacterial strains, namely *B. faecalis*, *S. aureus*, *K. pneumoniae*, and *E. coli*, using

Scheme 2.

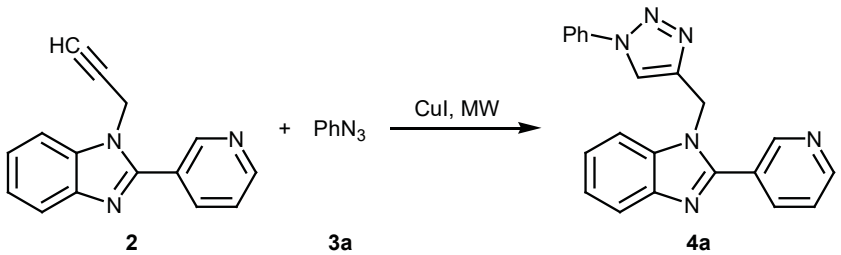


Table 1. Optimization of the conditions for the synthesis of compound **4a**

Entry no.	Solvent	Conditions	Reaction time	Yield,[a] %
1	TEA	Room temperature	8 h	64
2	DIPEA	Room temperature	8 h	58
3	DMF	Room temperature	8 h	55
4	DMSO	Room temperature	8 h	42
5	MeCN	Room temperature	8 h	38
6	EtOH	Room temperature	8 h	47
7	TEA	MW, 100 W	8 h	74
8	TEA	MW, 180 W	6 h	78
9	**TEA**	**MW, 360 W**	**6 min**	**94**
10	TEA	MW, 480 W	6 min	82

ampicillin as a standard antibacterial drug. The antibacterial activity was determined using the cup plate agar diffusion method by measuring the zone of inhibition in mm at a concentration of 100 μg/mL in DMSO. Compounds **4e**–**4h** and **4j** demonstrated good activity, while the others were weakly or moderately active. Compounds **4a**–**4j** were also tested for their in vitro antifungal activity against *A. niger* and *C. metapsilosis* using griseofulvin as a standard drug. The antifungal activity was evaluated by the cup plate agar diffusion method by measuring the zone of inhibition in mm at a concentration of 500 μg/mL in DMSO. Compounds **4e**, **4i**, **4h**, and **4j** demonstrated a good activity against the tested fungi.

EXPERIMENTAL

The melting points were determined in open capillary tubes and are uncorrected. The purity of the isolated compounds was checked by TLC using precoated silica gel 60 UV254 plates (Merck). Microwave-assisted reactions were carried out in a Milestone multi-SYNTH microwave system. The IR spectra were recorded in KBr on a Shimadzu FT-IR-8400s spectrometer. The ^{1}H and ^{13}C NMR spectra were measured on a Bruker Avance II 400 MHz spectrometer using $CDCl_3$ as a solvent and tetramethylsilane as an internal standard. The mass spectra were recorded on a Shimadzu GCMS-QP 1000 EX mass spectrometer.

General procedure for the preparation of 1-[(1-aryl-1*H*-1,2,3-triazol-4-yl)methyl]-2-(pyridin-3-yl)-1*H*-benzimidazoles 4a–4j. A mixture of 1-(prop-2-yn-1-yl)-2-(pyridin-3-yl)-1*H*-benzimidazole (**2**, 1 mmol), aryl azide **3a**–**3j** (1.2 mmol), and copper(I) iodide (2 mmol) in triethylamine (3 ml) was subjected to microwave irradiation at 360 W for 6–8 min. The progress of the reaction was monitored by TLC. After completion of the reaction, the mixture was poured into ice water, and a solid product slowly precipitated. It was filtered off, washed with water, dried, and purified by column chromatography using *n*-hexane–ethyl acetate (8:2) as an eluent.

1-[(1-Phenyl-1*H*-1,2,3-triazol-4-yl)methyl]-2-(pyridin-3-yl)-1*H*-benzimidazole (4a). Yield 94%, off-white solid, mp 129–131°C. IR spectrum, ν, cm^{-1}: 1421, 1589, 1649. ^{1}H NMR spectrum, δ, ppm: 5.65 s (2H, NCH_2), 7.26–7.32 m (2H, H_{arom}), 7.47–7.66 m (4H, H_{arom}), 7.70–7.74 m (2H, H_{arom}), 7.86 d (2H, J = 8.0 Hz, H_{arom}), 8.42–8.45 m (1H, H_{arom}), 8.78 d (1H, J = 2.4 Hz, H_{arom}), 8.93 s (1H, 5-H, triazole), 9.17 s (1H, H_{arom}). ^{13}C NMR spectrum, δ_C, ppm: 39.9, 111.2, 119.3, 120.1, 122.0, 122.4, 123.0, 128.8, 129.8, 135.6, 136.4, 136.8, 142.6, 143.4, 149.8, 150.7. Mass spectrum: *m*/*z* 353 (I_{rel} 100%) [M + H]$^+$. Found, %: C 71.52; H 4.53; N 23.82. $C_{21}H_{16}N_6$. Calculated, %: C 71.57; H 4.58; N 23.85.

1-{[1-(4-Methylphenyl)-1*H*-1,2,3-triazol-4-yl]-methyl}-2-(pyridin-3-yl)-1*H*-benzimidazole (4b). Yield 92%, off-white solid, mp 148–150°C. IR spectrum, ν, cm^{-1}: 1422, 1591, 1652. ^{1}H NMR spectrum, δ, ppm: 2.41 s (3H, CH_3), 5.66 s (2H, NCH_2), 7.22–7.26 m (2H, H_{arom}), 7.48–7.62 m (3H, H_{arom}), 7.69–

7.73 m (2H, H_{arom}), 7.86 d (2H, J = 8.0 Hz, H_{arom}), 8.41–8.44 m (1H, H_{arom}), 8.79 d (1H, J = 2.4 Hz, H_{arom}), 8.91 s (1H, 5-H, triazole), 9.70 s (1H, H_{arom}). ^{13}C NMR spectrum, δ_C, ppm: 25.1, 39.7, 111.2, 118.9, 120.4, 121.9, 122.6, 123.0, 126.7, 128.5, 129.8, 135.6, 136.4, 136.7, 142.6, 143.5, 149.8, 150.6. Mass spectrum: m/z 367 (I_{rel} 100%) $[M + H]^+$. Found, %: C 72.08; H 4.93; N 22.92. $C_{22}H_{18}N_6$. Calculated, %: C 72.11; H 4.95; N 22.94.

1-{[1-(4-Methoxyphenyl)-1*H*-1,2,3-triazol-4-yl]-methyl}-2-(pyridin-3-yl)-1*H*-benzimidazole (4c). Yield 92%, off-white solid, mp 143–145°C. IR spectrum, ν, cm^{-1}: 1423, 1589, 1654. ^1H NMR spectrum, δ, ppm: 3.81 s (3H, OCH_3), 5.63 s (2H, NCH_2), 7.11–7.13 d (2H, J = 8.4 Hz, H_{arom}), 7.29 m (2H, H_{arom}), 7.74–7.77 m (4H, H_{arom}), 7.70–7.74 m (2H, H_{arom}), 8.46 d (1H, J = 9.8 Hz, H_{arom}), 8.82 s (1H, 5-H, triazole), 9.55 br.s (1H, H_{arom}). ^{13}C NMR spectrum, δ_C, ppm: 39.9, 55.5, 111.2, 114.8, 119.3, 121.8, 121.9, 122.4, 122.9, 129.7, 136.8, 143.2, 150.7, 159.3. Mass spectrum: m/z 383 (I_{rel} 100%) $[M + H]^+$. Found, %: C 69.12; H 4.72; N 21.95. $C_{22}H_{18}N_6O$. Calculated, %: C 69.10; H 4.74; N 21.98.

1-{[1-(2-Methoxyphenyl)-1*H*-1,2,3-triazol-4-yl]-methyl}-2-(pyridin-3-yl)-1*H*-benzimidazole (4d). Yield 90%, off-white solid, mp 162–164°C. IR spectrum, ν, cm^{-1}: 1425, 1590, 1648. ^1H NMR spectrum, δ, ppm: 3.83 s (3H, OCH_3), 5.66 s (2H, NCH_2), 7.09–7.13 m (1H, H_{arom}), 7.26–7.33 m (3H, H_{arom}), 7.50–7.79 m (5H, H_{arom}), 8.48–8.51 m (1H, H_{arom}), 8.60 s (1H, 5-H, triazole), 8.78 d (1H, J = 3.6 Hz, H_{arom}), 9.19 br.s (1H, H_{arom}). ^{13}C NMR spectrum, δ_C, ppm: 39.8, 56.1, 111.3, 113.0, 119.3, 120.8, 122.4, 122.9, 123.7, 125.4, 125.6, 125.9, 126.4, 130.8, 135.6, 136.9, 142.0, 142.6, 149.8, 150.4, 150.5, 151.5. Mass spectrum: m/z 383 (I_{rel} 100%) $[M + H]^+$. Found, %: C 69.12; H 4.72; N 21.95. $C_{22}H_{18}N_6O$. Calculated, %: C 69.10; H 4.74; N 21.98.

1-{[1-(4-Chlorophenyl)-1*H*-1,2,3-triazol-4-yl]-methyl}-2-(pyridin-3-yl)-1*H*-benzimidazole (4e). Yield 93%, off-white solid, mp 125–127°C. IR spectrum, ν, cm^{-1}: 1424, 1592, 1654. ^1H NMR spectrum, δ, ppm: 5.66 s (2H, NCH_2), 7.26–7.33 m (2H, H_{arom}), 7.65–7.72 m (5H, H_{arom}), 7.91 d (2H, J = 8.8 Hz, H_{arom}), 8.43 d (1H, J = 8.0 Hz, H_{arom}), 8.80 br.s (1H, H_{arom}), 8.94 s (1H, 5-H, triazole), 9.15 br.s (1H, H_{arom}). ^{13}C NMR spectrum, δ_C, ppm: 39.7, 111.3, 119.3, 121.8, 122.0, 122.4, 123.0, 124.8, 129.0, 129.8, 133.0, 135.1, 136.8, 142.6, 143.6, 149.7, 153.0. Mass spectrum: m/z 387 (I_{rel} 100%) $[M + H]^+$. Found, %: C 65.22; H 3.88; N 21.70. $C_{21}H_{15}ClN_6$. Calculated, %: C 65.20; H 3.91; N 21.73.

1-{[1-(4-Nitrophenyl)-1*H*-1,2,3-triazol-4-yl]-methyl}-2-(pyridin-3-yl)-1*H*-benzimidazole (4f). Yield 89%, off-white solid, mp 152–154°C. IR spectrum, ν, cm^{-1}: 1420, 1588, 1656. ^1H NMR spectrum, δ, ppm: 5.69 s (2H, NCH_2), 7.26–7.33 m (2H, H_{arom}), 7.63–7.73 m (3H, H_{arom}), 8.18 d (2H, J = 8.4 Hz, H_{arom}), 8.39–8.46 m (3H, H_{arom}), 8.78 br.s (1H, H_{arom}), 9.11 s (1H, 5-H, triazole), 9.35 br.s (1H, H_{arom}). ^{13}C NMR spectrum, δ_C, ppm: 39.8, 111.1, 119.3, 120.6, 122.4, 122.4, 123.0, 125.5, 129.5, 129.7, 133.0, 135.6, 136.8, 140.6, 142.6, 144.1, 146.7, 149.7, 150.5. Mass spectrum: m/z 398 (I_{rel} 100%) $[M + H]^+$. Found, %: C 63.42; H 3.83; N 24.69. $C_{21}H_{15}N_7O_2$. Calculated, %: C 63.47; H 3.80; N 24.67.

1-{[1-(2-Nitrophenyl)-1*H*-1,2,3-triazol-4-yl]-methyl}-2-(pyridin-3-yl)-1*H*-benzimidazole (4g). Yield 90%, off-white solid, mp 148–150°C. IR spectrum, ν, cm^{-1}: 1424, 1589, 1653. ^1H NMR spectrum, δ, ppm: 5.69 s (2H, NCH_2), 7.28–7.34 m (2H, H_{arom}), 7.33–7.96 m (6H, H_{arom}), 8.21 d (1H, J = 8.4 Hz, H_{arom}), 8.42 d (1H, J = 6.8 Hz, H_{arom}), 8.80 br.s (1H, H_{arom}), 8.81 s (1H, 5-H, triazole), 9.32 br.s (1H, H_{arom}). ^{13}C NMR spectrum, δ_C, ppm: 39.9, 111.2, 119.4, 120.6, 122.4, 123.0, 125.2, 125.5, 127.7, 128.9, 129.7, 131.3, 132.7, 134.4, 136.5, 140.5, 143.1, 143.9, 146.9, 149.6, 150.7. Mass spectrum: m/z 398 (I_{rel} 100%) $[M + H]^+$. Found, %: C 63.42; H 3.83; N 24.69. $C_{21}H_{15}N_7O_2$. Calculated, %: C 63.47; H 3.80; N 24.67.

1-{[1-(2-Chloro-4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl]methyl}-2-(pyridin-3-yl)-1*H*-benzimidazole (4h). Yield 88%, off-white solid, mp 137–139°C. IR spectrum, ν, cm^{-1}: 1422, 1593, 1655. ^1H NMR spectrum, δ, ppm: 5.73 s (2H, NCH_2), 7.27–7.33 m (2H, H_{arom}), 7.33–7.78 m (3H, H_{arom}), 7.98 d (1H, J = 8.8 Hz, H_{arom}), 8.36–8.45 m (2H, H_{arom}), 8.62 d (1H, J = 2.6 Hz, H_{arom}), 8.78 br.s (1H, H_{arom}), 8.81 s (1H, 5-H, triazole), 9.16 br.s (1H, H_{arom}). ^{13}C NMR spectrum, δ_C, ppm: 40.1, 111.2, 119.3, 122.5, 123.0, 123.5, 125.2, 125.8, 126.1, 129.7, 129.2, 132.1, 135.6, 136.8, 138.8, 142.4, 142.8, 146.5, 148.2, 149.7, 150.5. Mass spectrum: m/z 432 (I_{rel} 100%) $[M + H]^+$. Found, %: C 58.40; H 3.24; N 22.65. $C_{21}H_{14}ClN_7O_2$. Calculated, %: C 58.41; H 3.27; N 22.70.

1-{[1-(4-Fluorophenyl)-1*H*-1,2,3-triazol-4-yl]-methyl}-2-(pyridin-3-yl)-1*H*-benzimidazole (4i). Yield 90%, off-white solid, mp 135–137°C. IR spectrum, ν, cm^{-1}: 1422, 1590, 1652. ^1H NMR spectrum, δ, ppm: 5.67 s (2H, NCH_2), 7.15–7.18 m (2H, H_{arom}),

7.62–7.67 m (5H, H_{arom}), 7.83 d (2H, J = 8.8 Hz, H_{arom}), 8.41 d (1H, J = 8.0 Hz, H_{arom}), 8.78 br.s (1H, H_{arom}), 8.93 s (1H, 5-H, triazole), 9.12 br.s (1H, H_{arom}). ^{13}C NMR spectrum, δ_C, ppm: 39.7, 111.3, 117.8, 119.2, 121.6, 122.1, 122.5, 123.4, 125.2, 128.7, 128.7, 133.0, 135.4, 136.5, 142.2, 143.5, 149.7, 153.1. Mass spectrum: m/z 373 (I_{rel} 100%) $[M + H]^+$. Found, %: C 68.13; H 4.05; N 22.65. $C_{21}H_{15}FN_6$. Calculated, %: C 68.10; H 4.08; N 22.69.

1-{[1-(4-Bromophenyl)-1*H*-1,2,3-triazol-4-yl]-methyl}-2-(pyridin-3-yl)-1*H*-benzimidazole (4j). Yield 89%, off-white solid, mp 126–128°C. IR spectrum, ν, cm^{-1}: 1421, 1589, 1649. ^1H NMR spectrum, δ, ppm: 5.66 s (2H, NCH_2), 7.22–7.27 m (2H, H_{arom}), 7.65–7.72 m (5H, H_{arom}), 7.90 d (2H, J = 8.4 Hz, H_{arom}), 8.41 d (1H, J = 8.0 Hz, H_{arom}), 8.80 br.s (1H, H_{arom}), 8.93 s (1H, 5-H, triazole), 9.15 br.s (1H, H_{arom}). ^{13}C NMR spectrum, δ_C, ppm: 39.7, 111.2, 119.3, 121.9, 122.2, 122.7, 123.2, 125.1, 128.6, 129.8, 132.9, 135.1, 136.8, 142.5, 143.6, 149.8, 153.2; Mass spectrum, m/z 431/433 (I_{rel} 100%) $[M + H]^+$. Found, %: C 58.46; H 3.48; N 19.52. $C_{21}H_{15}BrN_6$. Calculated, %: C 58.48; H 3.51; N 19.49.

CONCLUSIONS

A novel series of 1,2,3-triazole-tethered 2-pyridinyl-benzimidazole derivatives have been synthesized with high efficiency by click reaction under microwave irradiation. The described procedure proved to be simple and easy-to-perform, and it provided high yields within a short time.

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CONFLICT OF INTEREST

No conflict of interest was declared by the authors.

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