Syntheses, Crystal Structures, and Spectral Characterization of Three New Asymmetrical Substituted Triaryltriazoles

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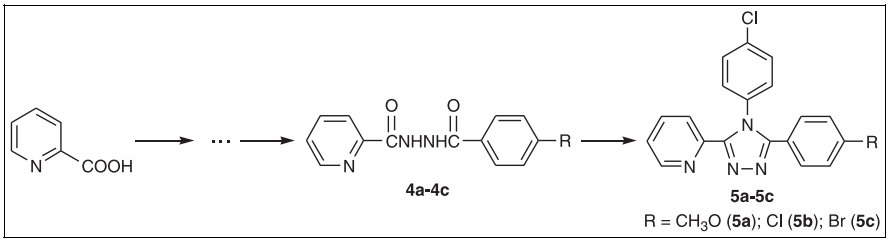

Triaryltriazoles are of interest in iron(II) complexes designed to study spin-crossover properties. In this study, three new asymmetrical substituted triaryltriazoles, 3-(*p*-R-phenyl)-4-(*p*-chlorophenyl)-5-(2-pyridyl)-1,2,4-triazoles (R = OCH_3, **5a**; Cl, **5b**; Br, **5c**), were successfully synthesized from 2-picolinic acid by a three-step reaction through an intermediate *N*-(*p*-R-phenylcarbonyl)-*N*′-(2-pyridylcarbonyl)hydrazine (**4a–4c**). Yield of **5a–5c** is in the range from 74 to 87%. The compounds **5a–5c** were characterized by UV, FTIR, ^{1}H-NMR, electrospray ionization mass spectrum spectra, and elemental analysis. Additionally, the absolute configurations of **5a–5c** were determined by single crystal X-ray crystallography.

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INTRODUCTION

Over the past few decades, 1,2,4-triazoles and their derivatives, a kind of interesting heterocyclic compounds, were found to possess important pharmacological activities, and have been widespread used as antifungus, antitumor, fungicide, weedicide, and so on [1]. Moreover, substituted 1,2,4-triazoles also have attracted considerable attention in coordination chemistry due to their rich and versatile coordination modes [2–4]. Especially, some iron(II) complexes with substituted 1,2,4-triazoles show fascinating spin-crossover properties which can be used in molecular electronics, as information storage and switching materials [5–10].

Recently, some 4-substituted 3,5-di(2-pyridyl)-1,2,4-triazoles and their metal complexes have been prepared successfully by our group [11–19] and others [2, 3]. However, the asymmetrical 3,5-disubstituted triaryltriazoles have been little reported so far [20–23]. As a continuation of our investigation on the asymmetrical 3,5-disubstituted 1,2,4-triazoles and their complexes [24, 25], in this article, we present the syntheses of a series of new asymmetrical substituted triaryltriazoles, 3-(*p*-R-phenyl)-4-(*p*-chlorophenyl)-5-(2-pyridyl)-1,2,4-triazoles (R = OCH_3, **5a**; Cl, **5b**; Br, **5c**) from 2-picolinic acid (Scheme 1). The products **5a–5c** were characterized by UV, FTIR, ^{1}H-NMR, ESI-MS spectra, and elemental analysis. Their single crystal structures have systematically been studied. Structural studies, together with spin-crossover properties on the iron(II) complexes of these ligands, are currently under investigation.

RESULTS AND DISCUSSION

In general, there are three kinds of methods reported for the preparation of 3,4,5-trisubstituted 1,2,4-triazoles (Scheme 2) [26–28]. However, there is a relatively complicated route and the lower yield of the triazole in methods (b) and (c) compared with method (a). Especially, method (a) is considered to be more powerful and practical route for syntheses of 3,4,5-triaryltriazoles. Hence, we choose method (a) for the synthesis of our target compounds—asymmetrical substituted triaryltriazoles **5a–5c**.

The readily available 2-picolinic acid reacting with anhydrous ethanol under the existence of 98% concentrated sulfuric acid gave ethyl pyridine-2-carboxylate (**2**) [29]. Brief treatment of **2** with 80% hydrazine hydrate afforded pyridine-2-carbonylhydrazine (**3**) in 74.5% yield. Then *N*-(*p*-R-phenylcarbonyl)-*N*′-(2-pyridylcarbonyl)hydrazine (**4a–4c**) [30] was obtained in a yield of 78–82% by stirring **3** with the corresponding *p*-R-benzoyl chloride (R = OCH_3, **4a**; Cl, **4b**; Br, **4c**) in anhydrous pyridine at ambient temperature. Finally, under argon atmosphere, the cyclization reaction of

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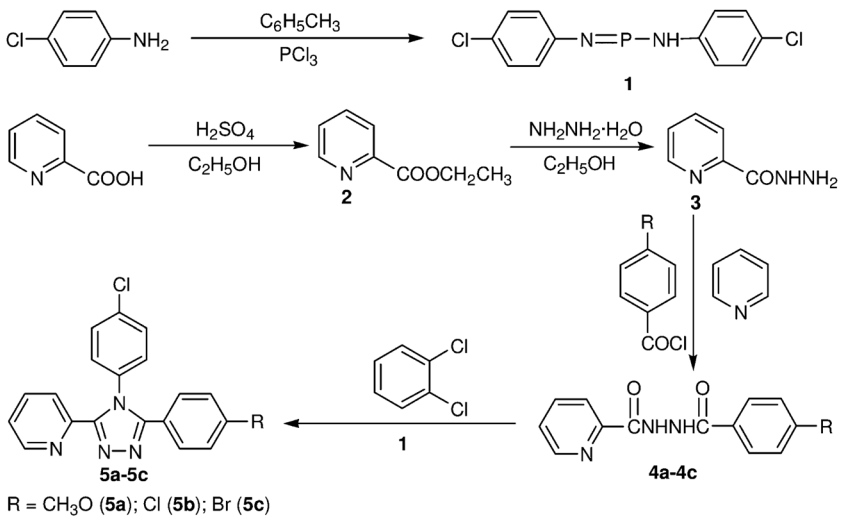
Scheme 1

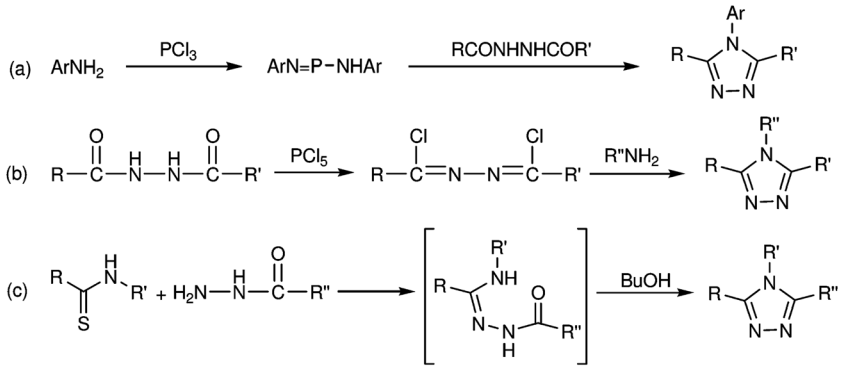


4a–**4c** with 4,4′-dichlorophenylphosphazoanilide (**1**) produced the corresponding 1,2,4-triazoles **5a**–**5c** (yield: **5a**, 86.8%; **5b**, 78.3%; **5c**, 73.8%) in *o*-dichlorobenzene at 190°C. It is worthwhile to note that more reactive phosphazo compounds **1** can be obtained in a yield of 50% by reaction of *p*-chloroaniline with phosphorus trichloride in a dried toluene solution at 125–130°C [31] and should be stored in an inert atmosphere.

The asymmetrical 3,4,5-triaryltriazoles **5a**–**5c** were fully characterized by UV, FTIR, ^{1}H-NMR, ESI-MS spectra, and elemental analysis. The molecular structures of **5a**–**5c** were also confirmed by X-ray crystallography (Table 1). Single crystals of **5a–5c** suitable for X-ray diffraction study were obtained upon slow evaporation from respective ethanol solution at room temperature. The structural features of **5a–5c** with atom-labeling scheme are shown in Figure 1 and selected bond lengths and bond angles are listed in Table 2. The X-ray structure analysis indicate that **5a–5c** consist of three aryl rings and one central 1,2,4-triazole ring. These rings do not share a common plane. The substituted phenyl rings and pyridyl ring lie in a propeller arrangement around the central 1,2,4-triazole ring. Some relevant dihedral angles among the substituted phenyl rings, pyridyl ring, and the 1,2,4-triazole ring are listed in Table 3. In addition, the *N* atom of the pyridyl group is oriented toward the Cl1-phenyl ring.

Although the bond lengths and bond angles in the structures of **5a–5c** are similar and can be comparable with the related compounds [11–13, 20], the stacking modes of **5a**–**5c** are quite different. There is only one kind of π–π interaction between two parallel triazole planes in the crystal of **5a**, whereas there are two kinds of π–π interactions between two pairs of parallel triazole rings in the crystals of **5b** and **5c**. These π–π interactions are summarized in Table 4. In addition, the molecule of **5b** is further stabilized by three kinds of weak C H···π interactions involving (i) C9-H9A and the *p*-Cl1-phenyl ring [C9···π = 3.663(4) Å and C9-H9A···π = 139.6(3)°], (ii) C4-H4A and

Scheme 2



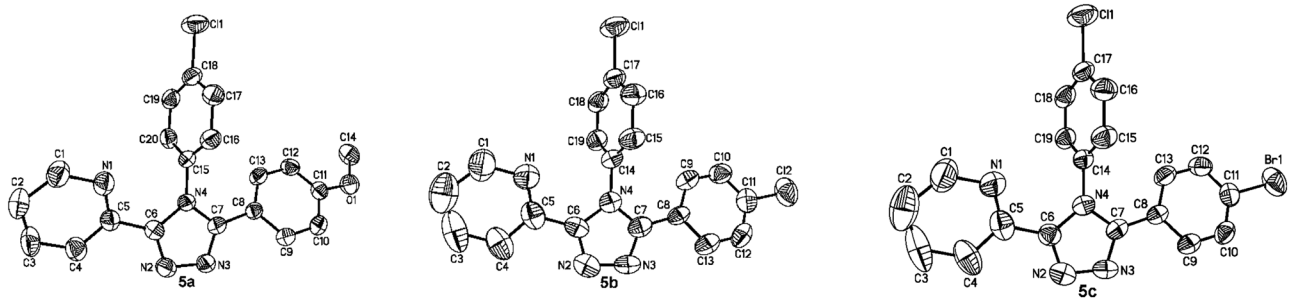


Figure 1. Views of **5a–5c** showing the atom-numbering scheme. Thermal ellipsoids are shown at the 50% probability level. Hydrogen atoms for **5a–5c** are omitted for clarity.

the *p*-Cl2-phenyl ring at (1 − *x*, 1 − *y*, −*z*) [C4···π = 3.366 (4) Å and C4-H4A···π = 116.6(3)°], and (iii) C12-H12A and the pyridyl ring at (1 − *x*, −*y*, −*z*) [C12···π = 3.427(4) Å and C12-H12A···π = 113.8(3)°], however, in the crystals of **5a** and **5c**, there are only two kinds of weak C-H···π interactions involving (i) C4-H4A and the *p*-methoxyphenyl ring at (2 − *x*, 1 − *y*, 1 − *z*) [C4···π = 3.474(3) Å and C4-H4A···π = 131.6(2)°] and (ii) C13-H13A and the *p*-chlorophenyl ring [C13···π = 3.583(3) Å and C13-H13A···π = 133.3(2)°] in **5a**, and (i) C10-H10A and the pyridyl ring at (1 − *x*, −*y*, 2 − *z*) [C10···π = 3.406(5) Å and C10-H10A···π = 111.9(3)°] and (ii) C13-H13A and the *p*-chlorophenyl ring [C13···π = 3.638(5) Å and C13-H13A···π = 141.6(3)°] in **5c**, respectively [25]. The crystal packing diagrams of **5a–5c** showing π–π and C H···π stacking interactions are presented in Figure 2.

EXPERIMENTAL

Melting points were determined with an X-4 digital microscope melting-point apparatus (Beijing) and are uncorrected. UV–vis spectra were recorded on a PerkinElmer Lambda 35 spectrophotometer at room temperature in methanol solution. FTIR spectra were recorded on a Nicolet 380 FTIR instrument using KBr disks in the range 4000–400 cm^{-1}. ^{1}H-NMR spectra were measured on a Bruker AM 500 MHz spectrometer at ambient temperature in $CDCl_3$ or DMSO-d_6. Chemical shifts are reported in parts per million (ppm) downfield from tetramethylsilane (TMS). Electrospray ionization mass spectrum (ESI-MS) was recorded with an LCQ ADVANTAGE MAX mass spectrometer, with MeOH on the mobile phase; the flow rate of the mobile phase was 0.2 $cm^3 \cdot min^{-1}$. The spray voltage, the capillary voltage, and the capillary temperature were 4 kV, 40 V, and 260°C, respectively. Elemental analyses (C, H, N) were carried out with a Thermo Finnigan Flash 1112A elemental analyzer. All

Table 1

Crystallographic data for the compounds **5a**–**5c**.

Compounds	**5a**	**5b**	**5c**
Empirical formula	$C_{20}H_{15}ClN_4O$	$C_{19}H_{12}Cl_2N_4$	$C_{19}H_{12}BrClN_4$
Formula weight	362.81	367.23	411.69
Crystal system	Monoclinic	Monoclinic	Monoclinic
Space group	$P2_1/n$	$P2_1/n$	$P2_1/n$
a (Å)	13.532 (6)	13.596 (3)	13.84 (3)
b (Å)	7.319 (3)	7.1736 (14)	7.169 (14)
c (Å)	17.602 (8)	17.390 (3)	17.62 (3)
β (°)	90.947 (7)	93.274 (3)	92.41 (3)
V (Å^3)	1743.0 (13)	1693.3 (6)	1747 (6)
Z	4	4	4
D_c (g cm^{-3})	1.383	1.440	1.566
μ (mm^{-1})	0.236	0.392	2.515
F (000)	752	752	824
Crystal size (mm)	0.26 × 0.14 × 0.12	0.24 × 0.12 × 0.10	0.24 × 0.16 × 0.10
θ Range	1.91–25.50	1.96–25.50	1.83–25.50
Reflections collected	9202	11,768	9736
Independent reflections	3229 [R_{int} = 0.0598]	3145 [R_{int} = 0.0539]	3219 [R_{int} = 0.0505]
Reflections observed [$I > 2\sigma(I)$]	1770	1614	1768
Data/restraints/parameters	3229/0/235	3145/0/226	3219/0/226
Goodness-of-fit on F^2	0.911	0.967	0.926
R/*w*R [$I > 2\sigma(I)$]	0.0479/0.1083	0.0553/0.1329	0.0474/0.1188
R/*w*R (all data)	0.1020/0.1285	0.1253/0.1546	0.1030/0.1369
Max., Min. Δρ (e·Å^{-3})	0.214, −0.269	0.358, −0.254	0.559, −0.361

Table 2

Selected bond lengths (Å) and angles (°) for **5a**–**5c**.

5a		5b		5c	
N1-C5	1.331 (3)	N1-C5	1.337 (5)	N1-C5	1.347 (6)
N2-N3	1.390 (3)	N2-N3	1.390 (4)	N2-N3	1.390 (5)
N4-C15	1.436 (3)	N4-C14	1.450 (4)	N4-C14	1.450 (5)
Cl1-C18	1.732 (3)	Cl1-C17	1.735 (3)	Cl1-C17	1.736 (5)
O1-C11	1.369 (3)	Cl2-C11	1.746 (4)	Br1-C11	1.902 (5)
C6-N2-N3	107.7 (2)	C6-N2-N3	107.8 (3)	C6-N2-N3	107.5 (3)
C6-C5-N1	117.6 (2)	C6-C5-N1	117.4 (3)	C6-C5-N1	117.5 (4)
C7-N4-C6	105.0 (2)	C7-N4-C6	105.9 (3)	C7-N4-C6	105.7 (3)
C12-C11-O1	124.6 (2)	C12-C11-Cl2	119.9 (3)	C12-C11-Br1	118.6 (4)
C19-C18-Cl1	119.4 (2)	C16-C17-Cl1	118.7 (3)	C16-C17-Cl1	118.7 (3)

chemicals used were of analytical grade and without further purification unless otherwise stated. Toluene was freshly distilled from Na/benzophenone, while pyridine was distilled over NaOH.

4,4′-Dichlorophenylphosphazoanilide (1). A solution of phosphorus trichloride (1.5 mL, 0.017 mol) in dry toluene (20 mL) was added dropwise to a stirring solution of *p*-chloroaniline (10.97 g, 0.086 mol) in 130 mL of anhydrous toluene at room temperature. The suspension was refluxed for 1 h at 125–130°C. Then the hot mixture was immediately filtered and washed with hot toluene. Then the filtrate was evaporated to dryness at reduced pressure and washed thoroughly with petroleum ether and ethanol to obtain **1** as a pure white solid (2.29 g, 50%). FTIR (v, cm^{-1}): 3340, 3165, 3030, 2942, 1596, 1491, 1384, 1093, 972.

Ethyl pyridine–2-carboxylate (2). Ninety-eight percent concentrated sulfuric acid (25 mL) was added dropwise to a solution of 2-picolinic acid (24.6 g, 0.2 mol) in anhydrous ethanol (120 mL) surrounded by a ice-water bath. The mixture was refluxed for 24 h. Upon cooling to ambient temperature, the product was poured into 100 mL ice-water. The resulting solution was neutralized to pH = 7–8 with a solution of potassium carbonate. The precipitate was filtered and the filtrate was extracted with ether (4 × 100 mL). After drying over magnesium sulfate, the organic phases were evaporated to dryness under reduced pressure. The unpurified ester can be used for next hydrazinolysis.

Pyridine–2-carbonylhydrazine (3). A mixture of above ester **2**, 80% hydrazine hydrate (19 mL, 0.4 mol) and ethanol (50 mL) was refluxed for 8 h. Then the solution was evaporated to dryness, and the resulting white solid was recrystallized from anhydrous ethanol to give **3** as colorless needles (20.4 g, 74.5% for a two-step reaction), mp 97–98°C (lit. mp 100–101°C [29]); FTIR (v, cm^{-1}): 3310, 3213, 3051, 1676, 1652, 1594, 1570, 1521, 1473, 1070, 998.

General procedure. ***Preparation of the N-(p-R-phenylcarbonyl)-N′-(2-pyridylcarbonyl)hydrazine (4a–4c).*** A suspension of the corresponding *p*-substituted-benzoyl chloride (0.012 mol) and dry pyridine (10 mol) was added dropwise to a stirring solution of **3** (0.01 mol) in 30 mL of pyridine at 0°C. The mixture was stirred at 0°C for 1 h, then at room temperature for 8 h. Then the mixture was poured into 50 mL ice-water, the precipitate was filtered, dried *in vacuo*, and recrystallized from anhydrous ethanol to give **4a**–**4c** as white solids.

N-(p-Methoxyphenylcarbonyl)-N′-(2-pyridylcarbonyl)hydrazine (4a). This compound was obtained as a white solid 2.13 g (78.5%), mp 153–155°C; FTIR (v, cm^{-1}): 3285, 3021, 2964, 1703, 1642, 1610, 1566, 1507, 1488, 1302, 1259, 1026, 998.4. ^{1}H-NMR (DMSO-d_6, ppm): δ 3.88 (s, 3H, CH_3), 7.18–7.20 (d, 2H, ArH), 7.63–7.66 (t, 1H, PyH), 8.05–8.09 (m, 3H, PyH, ArH), 8.24–8.26 (d, 1H, PyH), 8.80–8.81 (d, 1H, PyH), 10.51–10.53 (d, 2H, NH).

N-(p-Chlorophenylcarbonyl)-N′-(2-pyridylcarbonyl)hydrazine (4b). This compound was obtained as a colorless needle crystal 2.26 g (82.2%), mp 168–170°C; FTIR (v, cm^{-1}): 3179, 3010, 1685, 1641, 1597, 1570, 1513, 1488, 1334, 1095, 1015, 844. ^{1}H-NMR (DMSO-d_6, ppm): δ 7.60–7.63 (d, 2H, ArH), 7.66–7.69 (t, 1H, PyH), 7.93–7.95 (d, 2H, ArH), 8.03–8.07 (m, 2H, PyH), 8.71–8.72 (d, 1H, PyH), 10.63–10.67 (d, 2H, NH).

N-(p-Bromophenylcarbonyl)-N′-(2-pyridylcarbonyl)hydrazine (4c). This compound was obtained as a white solid 2.50 g (78.2%), mp 167–168°C; FTIR (v, cm^{-1}): 3173, 3010, 1690, 1637, 1591, 1508, 1484, 1399, 1072, 1010, 840. ^{1}H-NMR (DMSO-d_6, ppm): δ 7.62–7.65 (t, 1H, PyH), 7.71–7.73 (d, 2H, ArH), 7.84–7.85 (d, 2H, ArH), 7.99–8.05 (m, 2H, PyH), 8.68–8.69 (d, 1H, PyH), 10.58–10.61 (d, 2H, NH).

General procedure. ***Preparation of the 3-(p-R-phenyl)-4-(p-chlorophenyl)-5-(2-pyridyl)-1,2,4-triazole (5a–5c).*** To a solution of the corresponding *N*-(*p*-R-phenylcarbonyl)-*N′*-(2-pyridylcarbonyl)hydrazine (**4a**–**4c**) (4 mmol) in *o*-dichlorobenzene

Table 3

The dihedral angles (°) for **5a**–**5c**.

Compounds	Py/Trz	Cl1-Ph/Trz	CH_3O-Ph/Trz	Cl2-Ph/Trz	Br1-Ph/Trz
5a	23.7 (2)	73.5 (2)	30.4 (2)		
5b	18.5 (3)	75.6 (3)		24.5 (3)	
5c	16.3 (3)	74.9 (3)			22.6 (3)

Table 4

The π–π stacking interactions for **5a**–**5c**.

Compounds	π···π interaction	cent···cent	π···π	Dihedral angle
5a	π (trz)···π (trz)[a]	3.575	3.381	0.0
5b	π (trz)[b]···π (trz)[c]	3.514	3.394	0.0
	π (trz)···π (trz)[d]	3.956	3.738	0.0
5c	π (trz)···π (trz)[e]	3.521	3.396	0.0
	π (trz)···π (trz)[f]	3.979	3.731	0.0

Symmetry codes: (a) 1 – *x*, 2 – *y*, – *z;* (b) 1/2 + *x*, 1/2 – *y*, 1/2 + *z*; (c) 1/2 – *x*, 1/2 + *y*, 3/2 – *z*; (d) – *x*, 1 – *y*, 1 – *z;* (e) 1 – *x*, 1 – *y*, – *z;* (f) –1/2 + *x*, 3/2 – *y*, 1/2 + *z*

(25 mL) was added 4,4′-dichlorophenylphosphazoanilide (**1**) (4.8 mmol) with stirring. The mixture was heated to 190°C for 5 h. After all starting material had been consumed, the solvent was removed under reduced pressure, and the residue was treated with dilute hydrochloric acid (20 mL). The yellow solid was filtered off, and the filtrate was neutralized with a 20% potassium carbonate solution to pH = 9–10. The precipitate was filtered off, washed with ether, and recrystallized from anhydrous ethanol to give **5a**–**5c** as colorless block crystals.

3-(p-Methoxyphenyl)-4-(p-chlorophenyl)-5-(2-pyridyl)-1,2,4-triazole (5a). This compound was obtained as colorless block crystals 1.25 g (86.8%), mp 200–202°C; UV (MeOH, nm): 223 (2.23), 278 (1.97). FTIR (v, cm^{-1}): 3046, 2983, 2830, 1613, 1586, 1561, 1496, 1466, 1254, 1084, 1028, 845, 742. ^{1}H-NMR ($CDCl_3$, ppm): δ 3.80 (s, 3H, CH_3), 6.82–6.84 (d, 2H, ArH), 7.16–7.17 (d, 2H, ArH), 7.20–7.23 (m, 1H, PyH), 7.34–7.38 (t, 4H, ArH), 7.74–7.78 (t, 1H, PyH), 8.16–8.17 (d, 1H, PyH), 8.31–8.32 (d, 1H, PyH). ESI-MS: *m/z* 363 (M^+). *Anal*. Calcd. for $C_{20}H_{15}ClN_4O$: C, 66.21; H, 4.17; N, 15.44. Found: C, 66.08; H, 4.12; N, 15.52.

3,4-Di(p-chlorophenyl)-5-(2-pyridyl)-1,2,4-triazole (5b). This compound was obtained as colorless block crystals 1.15 g (78.3%), mp 213–215°C; UV (MeOH, nm): 224 (2.48), 272 (2.28). FTIR (v, cm^{-1}): 3050, 2979, 1587, 1573, 1493, 1461, 1086, 1015, 999, 843, 753. ^{1}H-NMR ($CDCl_3$, ppm): δ 7.15–7.19 (d, 2H, ArH), 7.22–7.24 (t, 1H, PyH), 7.28–7.31 (d, 2H, ArH), 7.35–7.40 (m, 4H, ArH), 7.75–7.81 (t, 1H, PyH), 8.17–8.20 (d, 1H, PyH), 8.32–8.33 (d, 1H, PyH). ESI-MS: *m/z* 367 (M^+). *Anal*. Calcd. for $C_{19}H_{12}Cl_2N_4$: C, 62.14; H, 3.29; N, 15.26. Found: C, 61.79; H, 3.21; N, 15.38.

3-(p-Bromophenyl)-4-(p-chlorophenyl)-5-(2-pyridyl)-1,2,4-triazole (5c). This compound was obtained as colorless block crystals 1.20 g (73.8%), mp 221–223°C; UV (MeOH, nm): 225 (1.76), 275 (1.65). FTIR (v, cm^{-1}): 3046, 2921, 1587, 1561, 1494, 1460, 1085, 1076, 1000, 843, 740. ^{1}H-NMR ($CDCl_3$, ppm): δ 7.15–7.18 (d, 2H, ArH), 7.22–7.24 (t, 1H, PyH), 7.30–7.33 (d, 2H, ArH), 7.36–7.39 (d, 2H, ArH), 7.45–7.47 (d, 2H, ArH), 7.75–7.81 (t, 1H, PyH), 8.19–8.20 (d, 1H, PyH), 8.32–8.33 (d, 1H, PyH). ESI-MS: *m/z* 413 ($M+H^+$). *Anal*. Calcd. for $C_{19}H_{12}BrClN_4$: C, 55.43; H, 2.94; N, 13.61. Found: C, 55.29; H, 2.86; N, 13.85.

Single-crystal X-ray diffraction analysis of 5a–5c. The well-shaped single crystals of **5a–5c** were selected for lattice parameter determination and collection of intensity data at 296 K on a Bruker SMART CCD diffractometer with a detector distance of 5 cm and frame exposure time of 10 s using a graphite-monochromated Mo K_α (λ = 0.71073 Å) radiation. The structures were all solved by direct methods and refined on F^2 by full-matrix least squares procedures using SHELXTL

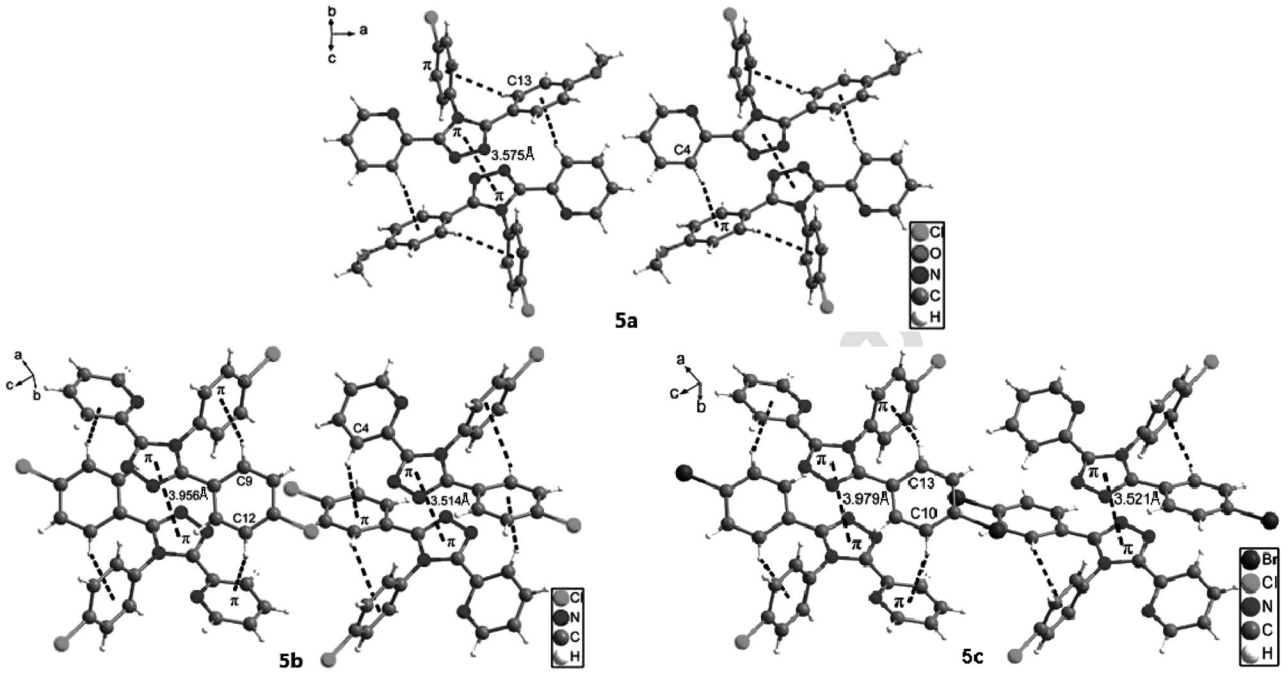


Figure 2. Views of the crystal packing for **5a–5c** showing π–π stacking interactions.

software [32]. All nonhydrogen atoms were anisotropically refined. All H atoms were located from a difference map and refined isotropically. Details on crystal data of **5a–5c** are summarized in Table 1.

CCDC 810287 (**5a**), 810288 (**5b**), and 810289 (**5c**) contain the supplementary crystallographic data for this article. These data can be obtained free of charge from the Cambridge Crystallographic Data Centre *via* www.ccdc.cam.ac.uk/data_request/cif.

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