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ORIGINAL RESEARCH

Cooperative N–H···N, N–H···O, and O–H···N bonded molecular dimers of two new 3,5-diaryl-1*H*-pyrazoles

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Abstract Two new 3,5-diaryl-1*H*-pyrazoles: 3(5)-(4-tert-butylphenyl)-5(3)-(naphthalene-2-yl)-1*H*-pyrazole (**1**) and 5-(4-(benzyloxy)phenyl)-3-(furan-2-yl)-1*H*-pyrazole (**2**) were synthesized and characterized. Two strong ions peaks $[2M]^+$ and $[2M + Na]^+$ observed in the ESI–MS spectra are attributed to the dimerization process in solution formed by intermolecular N–H···N hydrogen bonds. The crystal structures have been determined by X-ray crystal structure analysis. Compound **1** exists as a pair of tautomers **1a** and **1b**, and its dimer [$R_2^2(6)$ motif] is formed by the tautomers **1a** and **1b**. Compound **2** only exists as a **2a** tautomer, and interesting intermolecular N–H···O and O–H···N hydrogen bonds link two pyrazoles and two methanol molecules, leading to the formation of an $R_4^4(10)$ dimer motif.

Keywords 1*H*-pyrazole · Intermolecular hydrogen bonds · Tautomers · Dimer

Introduction

Pyrazoles and their derivatives represent one of the most active classes of compounds that exhibit important biological activities. Over the past few years, considerable evidences have been accumulated to demonstrate the efficiency of pyrazole derivatives, such as antibacterial, fungicidal, herbicidal, insecticidal, and other biological activities [1–5]. By far, the most prevalent method for preparation of pyrazoles is the double condensation of 1,3-diketones with hydrazine and hydrazine derivative [6–8]. Other methods for the synthesis of pyrazoles have been reported [9–12]. In addition, structural chemistry of N-unsubstituted pyrazoles has received much attention in their solid for several possible N–H···N hydrogen-bonding patterns: monomer, dimer, trimer, tetramer, hexamer, and catemer, all of which involve N–H···N hydrogen interactions using both N atoms of the pyrazoles [13–20]. Further more, pyrazoles dimerization in the gas phase also has been reported [21–23]. On the other hand, the tautomerism and proton transfer in pyrazoles have been communicated [24–27]. Recently, we studied the solid state structures and anti-microbial activity of some new substituted pyrazoles [28, 29]. As a continuation of our study, herein we report the crystal structures of two new 3,5-diaryl-1*H*-pyrazoles, in which intermolecular hydrogen bonds interactions lead to the formation of dimers with $R_2^2(6)$ and $R_4^4(10)$ motifs.

Experimental

Materials and equipments

The required 1,3-diketone compounds were synthesized according to our published procedures [30, 31]. All the commercially available chemicals were of reagent grade and were used without further purification. Infrared spectrum was obtained on a Nicolet 5700 FT-IR instrument with the compound in a KBr disk matrix. The ^{1}H NMR spectrum was obtained with a Varian Mercury-Plus400 instrument. Sample was dissolved in $CDCl_3$ referenced to TMS. The elemental analysis was performed by Perkin Elmer 2400. Electrospray ionization mass spectrum (ESI–MS)

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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 min^{-1}; the spray voltage, the capillary voltage, and the capillary temperature were 4 kV, 40 V, and 220 °C, respectively.

Synthesis of 3(5)-(4-tert-butylphenyl)-5(3)-(naphthalene-2-yl)-1*H*-pyrazole (1)

1-(4-tert-butylphenyl)-3-(naphthalene-2-yl)propane-1,3-dione (0.01 mol) was dissolved in hot ethanol (30 mL). To this solution hydrazine monohydrate (0.01 mol) in ethanol was added, and the mixture was refluxed under stirring for 3 h. After completion of the reaction, the solution was removed by evaporation. The residual solid was recrystallized from dilute ethanol solution to give compound **1**. Colorless needles, yield 79%. Main IR (KBr, cm^{-1}): 3229 (s, N–H), 1630, 1602, 1500 (s, C=C, C=N); ^{1}H NMR ($CDCl_3$, 400 Hz): $\delta = 1.34$ (s, 9H, CH_3), 5.30(bs, 1H, pyrazolyl N–H), 6.98 (s, 1H, pyrazolyl C–H), 7.44–7.48 (m, 4H), 7.71 (d, 2H, $J = 8.4$ Hz), 7.79–7.89 (m, 4H), 8.21 (s, 1H). Anal. Calcd (%) for $C_{23}H_{22}N_2$: C, 84.63; H, 6.79; N, 8.58. Found C, 84.52; H, 6.81; N, 8.52.

Table 1 Crystal data and structure refinements for compounds **1** and **2**

Compounds	1	2
Empirical formula	$C_{23}H_{22}N_2$	$C_{21}H_{20}N_2O_3$
Formula weight	326.43	348.39
Temperature (K)	298(2)	298(2)
Crystal system	Triclinic	Monoclinic
Space group	*P*1	*P*2(1)
a (Å)	10.2430(3)	16.080(3)
b (Å)	13.2720(4)	7.4574(14)
c (Å)	14.1420(4)	16.156(3)
α (°)	89.9760(10)	90
β (°)	77.5260(10)	110.796(4)
γ (°)	77.0560(10)	90
Volume (Å^3)	1827.13(9)	1811.1(6)
Z	4	4
Density (calc.) (Mg m^{-3})	1.187	1.278
Absorption coefficient (mm^{-1})	0.069	0.086
F(000)	696	736
Crystal size (mm^3)	0.16 × 0.04 × 0.02	0.10 × 0.06 × 0.05
Theta range for data (°)	2.09–25.00	2.23–26.00
Index ranges	$-12 \leq 12$, $-15 \leq 15$, $-16 \leq 16$	$-11 \leq 19$, $-9 \leq 5$, $-19 \leq 16$
Reflections collected	15817	7291
Independent reflections	6372 [*R*(int) = 0.0972]	5241 [*R*(int) = 0.0206]
Data/restraints/ parameters	6372/0/457	5241/1/471
Goodness-of-fit on F^2	0.905	1.140
Final *R* indices [$I > 2\sigma(I)$]	$R_1 = 0.0919$, $wR_2 = 0.1757$	$R_1 = 0.0674$, $wR_2 = 0.1377$
R indices (all data)	$R_1 = 0.2446$, $wR_2 = 0.2185$	$R_1 = 0.0824$, $wR_2 = 0.1458$
Largest diff. peak and hole (e°/A^{-3})	0.199 and –0.184	0.170 and –0.223

Synthesis of 5-(4-(benzyloxy)phenyl)-3-(furan-2-yl)-1*H*-pyrazole (2)

Compound **2** was prepared by a similar procedure for compound **1**. Colorless needles, yield 74%. Main IR (KBr, cm^{-1}): 3111 (s, N–H), 1617, 1509, 1454 (s, C=C, C=N); ^{1}H NMR ($CDCl_3$, 400 Hz): $\delta = 5.10$ (s, 2H, CH_2), 6.46–6.48 (m, 1H), 6.71 (s, 1H, pyrazolyl C–H), 6.72 (d, 1H, $J = 3.2$ Hz), 7.02 (d, 2H, $J = 8.4$ Hz), 7.34–7.46 (m, 6H), 7.66 (d, 2H, $J = 8.4$ Hz) ppm, pyrazolyl N–H not found. Anal. Calcd (%) for $C_{20}H_{16}N_2O_2$: C, 75.93; H, 5.10; N, 8.85. Found C, 75.88; H, 5.13; N, 8.83.

Table 2 Selected bond lengths (Å) and angles (°) for compounds **1** and **2**

1			
N1A–N2A	1.334(4)	N1B–N2B	1.344(5)
C11A–C12A	1.365(6)	C11B–C12B	1.394(6)
C12A–C13A	1.391(5)	C12B–C13B	1.381(6)
C13A–N2A	1.347 (5)	C13B–N2B	1.354(5)
C11A–N1A	1.348(5)	C11B–N1B	1.366(5)
C11A–C12A–C13A	107.0(4)	C11B–C12B–C13B	108.4(4)
C12A–C13A–C14A	131.2(5)	C12B–C13B–C14B	134.4(4)
C12–C13A–N2A	108.2(4)	C12–C13B–N2B	104.7(4)
C13A–N2A–N1A	107.1(4)	C13B–N2B–N1B	112.9(4)
N2A–N1A–C11A	111.1(4)	N2B–N1B–C11B	106.0(4)
N1A–C11A–C12A	106.6(4)	N1B–C11B–C12B	107.9(4)
2			
N1A–N2A	1.353(3)	N1B–N2B	1.359(4)
C5A–C6A	1.389(5)	C5B–C6B	1.392(5)
C6A–C7A	1.376(4)	C6B–C7B	1.380(5)
C7A–N2A	1.331(4)	C7B–N2B	1.345(5)
C5A–N1A	1.329(4)	C5B–N1B	1.320(5)
C5A–C6A–C7A	105.6(3)	C5B–C6B–C7B	105.6(4)
C6–C7A–N2A	106.7(3)	C6–C7B–N2B	106.8(3)
C7A–N2A–N1A	111.9(3)	C7B–N2B–N1B	110.9(3)
N2A–N1A–C5A	105.4(3)	N2B–N1B–C5B	106.2(3)
N1A–C5A–C6A	110.4(3)	N1B–C5B–C6B	110.6(3)

X-ray structure determination

Suitable crystals of compounds **1** and **2** for X-ray diffraction experiments were grown by slow evaporation from $CH_3OH–CH_2Cl_2$ (1:1) solutions. Single crystals were selected and mounted on the top of the glass fibers. Crystal of **2** was sealed in glass tube because the crystal was susceptible to solvent loss. X-ray single crystal diffraction measurement was carried out at 298(2) K on a Bruker Smart 1000 CCD area diffractometer using graphite monochromatic Mo Kα radiation ($\lambda = 0.71073$ Å) for data collection. The structure was solved by direct methods and expanded using Fourier difference techniques with SHELXS-97 [32]. Refinements were made by full-matrix least squares on all F^2 data using SHELXL-97 [33]. H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C–H distances of 0.93–0.97 Å, and with $U_{iso}(H) = 1.2U_{eq}(C)$. All the H atoms on O and N atoms were placed in geometrically idealized positions and as riding atoms, with O–H and N–H distances of 0.82 and 0.86 Å, respectively, and $U_{iso}(H) = 1.5U_{eq}(O)$ and $U_{iso}(H) = 1.2U_{eq}(N)$. The crystallographic data and structure refinement information for **1** and **2** are shown in Table 1 and selected bond lengths and angles in Table 2.

Results and discussion

ESI–MS analysis

Electrospray mass spectrometry provided a method of detecting molecular interaction in solution. For compound **1**, two intense peaks with m/z 652.80 and 674.78 corresponded to $[2M]^+$ and $[2M + Na]^+$, the ion at m/z 327.16 was assigned as the $[M + 1]^+$ (Fig. 1). As shown in Fig. 2,

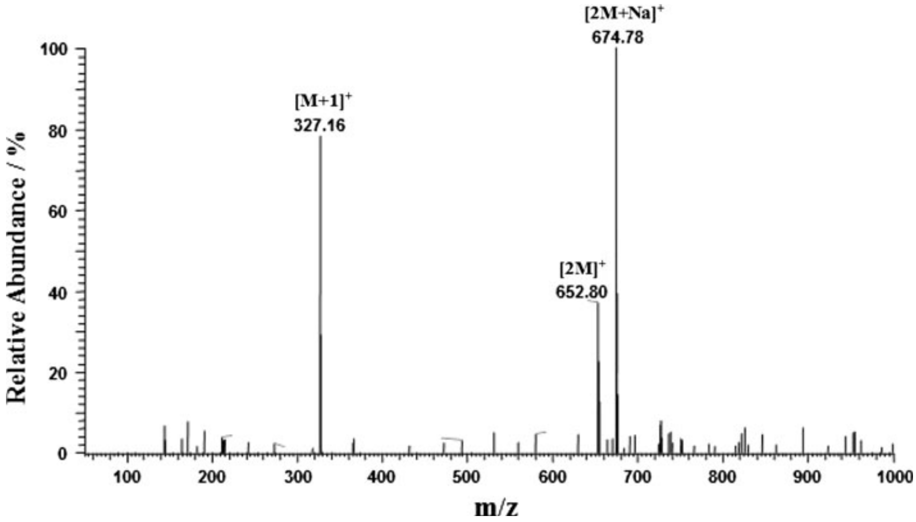


Fig. 1 Positive ion ESI–MS of compound **1** in methane solution

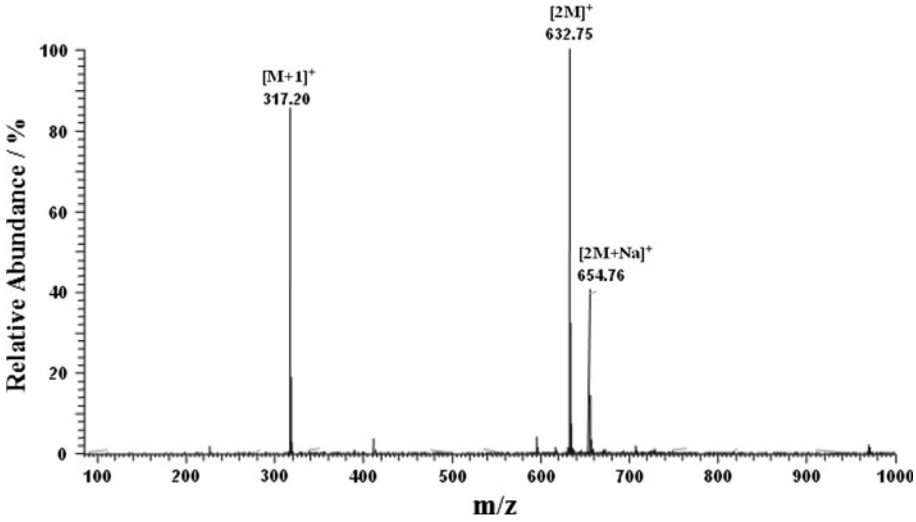
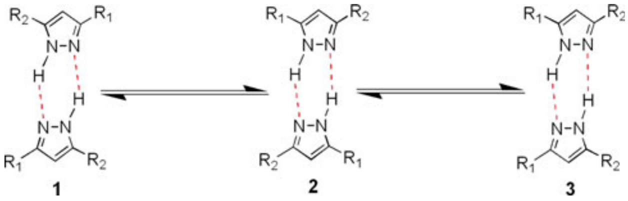


Fig. 2 Positive ion ESI–MS of compound **2** in methane solution



Scheme 1 The dimers connected by N–H···N hydrogen bonds

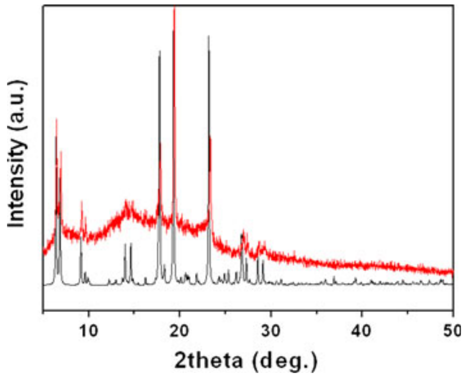


Fig. 3 PXRD patterns for 1 recrystallized from $C_2H_5OH–CH_2Cl_2$ solution (*top*) and simulated from single-crystal data (*below*)

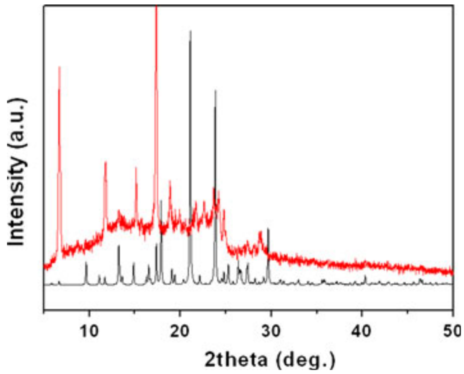


Fig. 4 PXRD patterns for 2 recrystallized from $C_2H_5OH–CH_2Cl_2$ solution (*top*) and simulated from single-crystal data (*below*)

the most intense ion peak for **2** was observed at m/z 632.75 corresponding to $[2M]^+$. The peaks at m/z 654.76 and 317.20 were also assigned as the $[2M + Na]^+$ and $[M + 1]^+$. The ESI–MS spectrum evidently indicates that the compounds **1** and **2** have a dimer formation tendency in solution attributed to the dimerization process by intermolecular N–H···N hydrogen bonds. This result is in agreement with the X-ray crystal structure. The dimers connected by N–H···N hydrogen bonds are shown in Scheme 1.

XRD analysis

Initially, we attempted to obtain single crystals of **1** and **2** from $C_2H_5OH–CH_2Cl_2$, but it was unsuccessful for **2**. Fortunately, we got the crystals of them from $CH_3OH–CH_2Cl_2$ solutions, which indicated that CH_3OH might play an important role in the crystal packing in **2**. Powder X-ray diffraction (PXRD) was used to characterize the phase structures of the products. As shown in Fig. 3, the measured PXRD pattern of **1** closely matched the one simulated from single-crystal diffraction data. However, for pattern of **2** (Fig. 4), the positions of several intensive peaks changed relative to that of the single crystals, indicating that the structure of single crystals from $CH_3OH–CH_2Cl_2$ solutions is a different phase structure of crystals recrystallized from $C_2H_5OH–CH_2Cl_2$.

Crystal structures

It is well known that pyrazole has two resonant isomers, and the hydrogen proton can transfer between two nitrogen atoms. For compound **1**, the molecule contains a pair of independent pyrazole molecules in the asymmetric unit (Fig. 5). Proton of the pyrazole group in one molecule is resided at the nitrogen atom N1A, adjacent to the naphthyl substituent, and proton of the pyrazole group in another molecule is resided at the nitrogen atom N2B, adjacent to the phenyl substituent, and this two independent pyrazole molecules are fitly a pair of tautomers **1a** and **1b**. In two independent pyrazole molecules, all the C–N bond lengths

Fig. 5 View of compound **1**, with 30% probability displacement ellipsoids

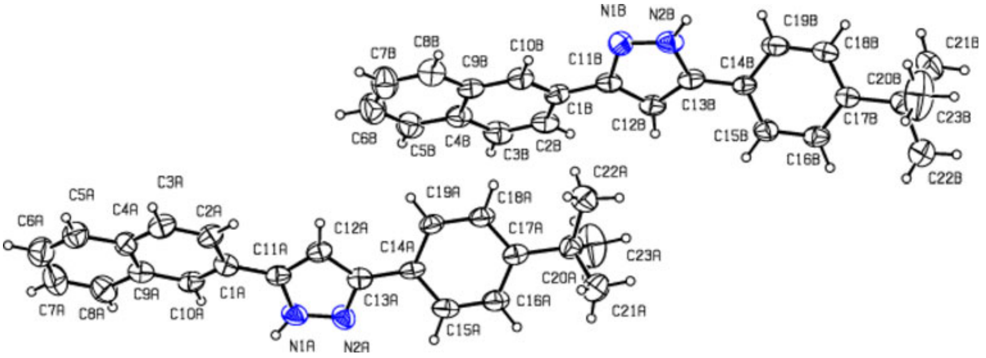


Table 3 Hydrogen-bonding geometry for compounds **1** and **2**

D–H···A	D–H (Å)	H···A (Å)	D···A (Å)	D–H···A (°)	Symmetry codes
1					
N1A–H1A···N1B	0.86	2.28	2.909(5)	130.6	–*x*, –1 + *y*, *z*
N2B–H2C···N2A	0.86	2.12	2.808(5)	136(2)	*x*, 1 + *y*, *z*
C8B–H8B···π ($Cg4^{a}$)	0.93	2.99	3.709(6)	136	2 – *x*, 1 – *y*, 1 – *z*
C19B–H19B···π (*Cg*7)	0.93	3.00	3.776(6)	142	1 – *x*, 2 – *y*, 1 – *z*
2					
O2S–H2S···N1A	0.82	1.95	2.771(4)	178	–*x* + 1, *y* + 1/2, –*z*
N2B–H2B···O2S	0.86	1.96	2.804(4)	169	–*x* + 2, *y* – 1/2, –*z* + 1
N2A–H2A···O1S	0.86	1.96	2.804(4)	165	–*x* + 1, *y* – 1/2, –*z*
O1S–H1S···N1B	0.82	2.00	2.802(4)	163	–*x* + 2, *y* + 1/2, –*z* + 1
C3B–H3B···π (*Cg*8)	0.93	2.72	3.507(4)	143	–*x* + 2, *y* + 1/2, –*z* + 1
C12A–H12A···π ($Cg4^{b}$)	0.93	2.92	3.614(4)	132	–*x* + 1, *y* + 1/2, –*z* + 1
C14A–H14B···π (*Cg*8)	0.93	2.99	3.840(5)	147	*x*, *y* – 1, *z*
C14B–H14D···π ($Cg4^{b}$)	0.93	2.98	3.797(5)	143	*x*, *y*, *z*
C17B–H17B···π (*Cg*5)	0.93	2.90	3.596(5)	133	–*x* + 2, *y* + 1/2, –*z* + 1
C19A–H19A···π (*Cg*3)	0.93	2.92	3.687(5)	141	–*x* + 1, *y* + 1/2, –*z* + 1

$Cg4^{a}$ is the centroid of atoms C14A–C19A, *Cg*7 of atoms C1B–C4B/C9B/C10B, *Cg*8 of atoms C15B–C20B, $Cg4^{b}$ of atoms C15A–C20A, *Cg*5 of atoms O1B/C1B–C4B and *Cg*3 of atoms C8A–C13A

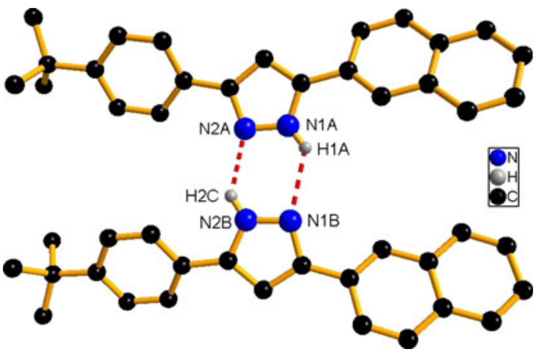
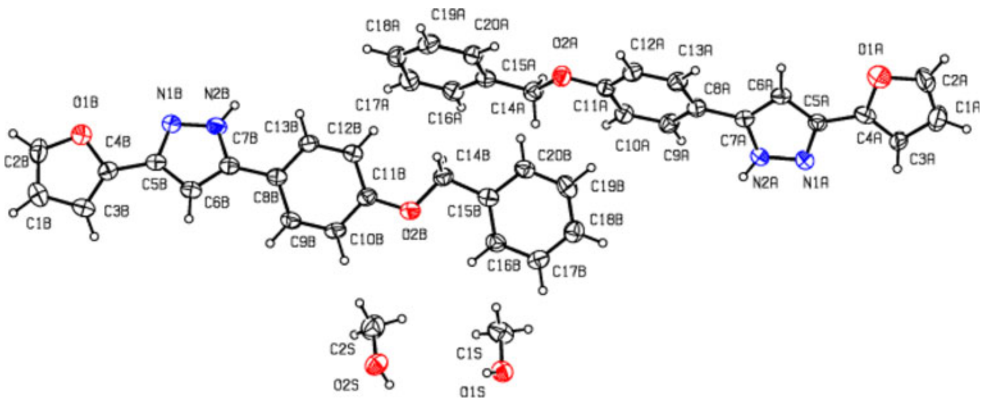


Fig. 6 The hydrogen bond system between compounds **1**, *the dashed lines* represent the intermolecular hydrogen bonds (H atoms on C atoms were omitted for clarity)

[1.347(5)–1.366(5) Å] (Table 3) are almost equal, and are remarkably shorter than the normal C–N bond (1.47–1.50 Å), but close to that of the C=N bond (1.34–1.38 Å), which are the result of the conjugation effect, indicating the significant double bond characteristics as well as the considerable delocalization of the electron density of pyrazole ring. In one molecule, the pyrazole ring (N1A–N2A/C11A–C13A) makes dihedral angles of 24.1(3)° and 23.9(2)° with benzene ring and naphthalene ring, respectively, the corresponding angles in the other molecule are 26.5(3)° and 23.3(2)°. As shown in Fig. 6, a pair of tautomers **1a** and **1b** are linked by intermolecular N–H···N hydrogen bonds to form dimer with an $R_2^2(6)$ motif. The measured N1A···N1B and N2A···N2B distance are 2.909(5), 2.808(5) Å, showing strong intermolecular N–H···N bonding interactions, which is comparable to that of the N–H···N bonding observed in the related pyrazole molecules, 3-methyl-5-phenylpyrazole and 3,5-diphenylpyrazole, which formed the tetrameric structures in the solid state [2.824(5) ~ 2.913(5) Å] [34, 35].

For compound **2**, the molecule also contains two independent pyrazole molecules; in addition, two methanol molecules are in the asymmetric unit (Fig. 7). The proton of the pyrazole group only resides at the nitrogen atom N2, adjacent to the phenyl group, indicating the molecular

Fig. 7 View of compound **2**, with 30% probability displacement ellipsoids



structure exists as a **2a** tautomer. The conformations of the two molecules also are slightly different: in one molecule, the furan ring shows a *trans* geometry, that is, the furan O1A atom is located at the opposite side of the pyrazole N1A atom. However, in another molecule, the furan ring shows a *cis* geometry, and the furan O1B atom is located at the same side of the pyrazole N1B atom. The central benzene ring (C8A–C13A) makes dihedral angles of 4.1(2)° and 32.1(3)° with rings (C15A–C20A) and (O1A/C1A–C4A), respectively, in one molecule; the corresponding angles in the other molecule are 9.0(2)° and 3.2(2)°. The pyrazolyl ring makes dihedral angles of 2.0(3)° and 13.2(2)° with two furan rings, respectively. The crystal structure shows that the acidic proton of one pyrazole molecule is directly linked to the oxygen atom of methanol solvated, from which its proton is also formed due to strong hydrogen bonding with nitrogen atom of another pyrazole molecule. Similar to **1**, compound **2** is also organized as dimer; however, the intermolecular hydrogen bond involves two methanol molecules. As shown in Fig. 8, two pyrazole molecules are linked with two methanol molecules by intermolecular N–H···O and O–H···N hydrogen bonds to form an $R_4^4(10)$ dimer motif.

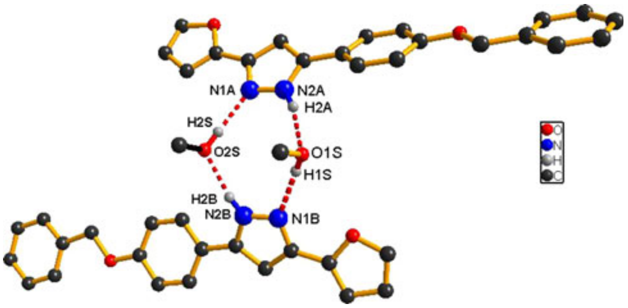


Fig. 8 The hydrogen bond system between compound **2**, *the dashed lines* represent the intermolecular hydrogen bonds (H atoms on C atoms were omitted for clarity)

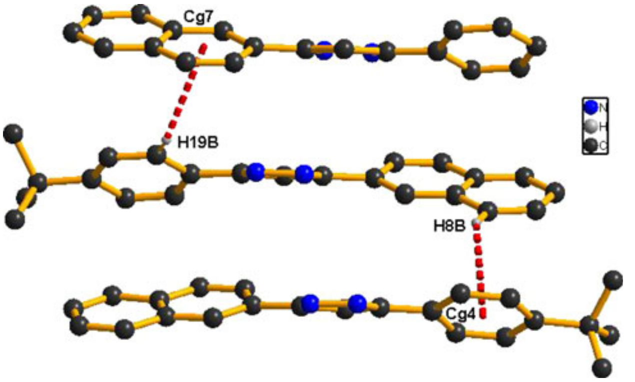


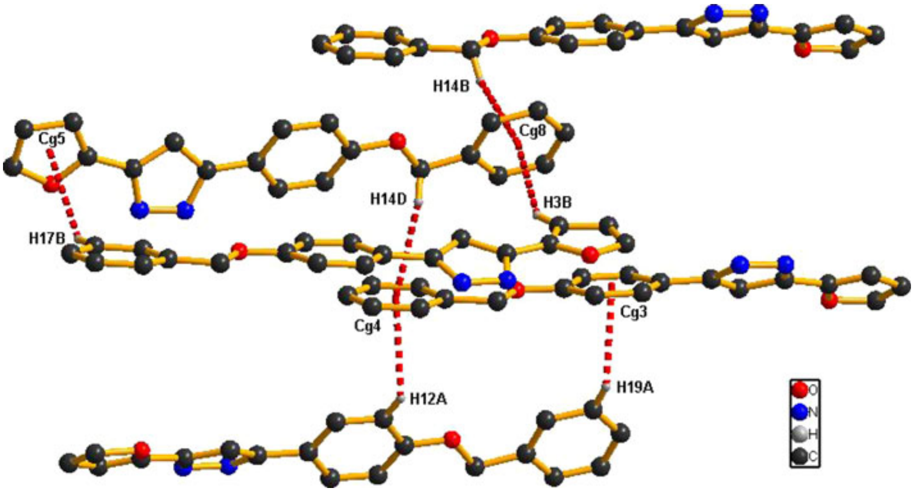
Fig. 9 The C–H···π hydrogen bonds between compound **1**

In addition, the crystal structures of compounds **1** and **2** both exhibit weak C–H···π interactions (Figs. 9, 10), the stability of crystal structure can be accounted by these interactions.

Conclusion

Two new 3,5-diaryl-1*H*-pyrazoles: 3(5)-(4-tert-butylphenyl)-5(3)-(naphthalene-2-yl)-1*H*-pyrazole (**1**) and 5-(4-(benzyloxy)phenyl)-3-(furan-2-yl)-1*H*-pyrazole (**2**) were synthesized and characterized. Two strong ions peaks $[2M]^+$ and $[2M + Na]^+$ observed in the ESI–MS spectra are attributed to the dimerization process in solution formed by intermolecular N–H···N hydrogen bonds. Structure analysis indicates compound **1** exists as a pair of tautomers **1a** and **1b**, and its dimer [$R_2^2(6)$ motif] is formed by the tautomers **1a** and **1b**. Compound **2** only exists as a **2a** tautomer, and intermolecular N–H···O and O–H···N hydrogen bonds link two pyrazole and two methanol molecules, leading to the formation of an $R_4^4(10)$ dimer motif.

Fig. 10 The C–H···π hydrogen bonds between compound **2**



Supplementary material

CCDC 751406 and 749402 for compounds 1 and 2 contain the supplementary crystallographic data for this article. These data can be obtained free of charge from www.ccdc.cam.ac.uk/conts/retrieving.html or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; Fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk.

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