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Synthesis, characterization, and crystal structures of new 3,5-diaryl-1*H*-pyrazoles

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ABSTRACT

Seven new 3,5-diaryl-1*H*-pyrazoles were synthesized by the condensation of 1,3-diketones with hydrazine in ethanol, and characterized. The crystal structures for 3(5)-(4-tert-butylphenyl)-5(3)-(4-fluorphenyl)-1*H*-pyrazole (**5**) and 3-(4-tert-butylphenyl)-5-(6-methoxy-naphthalen-2-yl)-1*H*-pyrazole (**7**) have been determined by X-ray crystal structure analysis. The N–H. . .N intermolecular hydrogen bonds join the molecules into dimers. The NH proton of **5** is disordered, and its dimer is formed by the tautomers **5a5b** or **5b5a**. Compound **7** only exists as a **7a** tautomer, and its dimer consists of the tautomers **7a7a**.

1. Introduction

The pyrazole derivatives are well known for their wide range of biological and pharmacological activities, such as antibacterial, fungicidal, herbicidal, insecticidal, and other biological activities [1–3]. They are also useful intermediates for many industrial products [4,5]. On the other hand, the crystal structures of N-unsubstituted pyrazoles have received much attention due to their variety of packing forms, cyclic dimers, trimers, tetramers and linear chains, all of which involve N–H. . .N hydrogen bonding [6–8]. In this paper, some new 3,5-diaryl-1*H*-pyrazoles were prepared by the reaction of 1,3-diketones with hydrazine monohydrate, and characterized by elemental analysis, FTIR, ^{1}H NMR and ESI-MS. The X-ray diffraction structures of the compounds 3(5)-(4-tert-butylphenyl)-5(3)-(4-fluorphenyl)-1*H*-pyrazole (**5**) and 3-(4-tert-butylphenyl)-5-(6-methoxy-naphthalen-2-yl)-1*H*-pyrazole (**7**) were determined. In the solid state, the molecules are connected by N–H. . .N intermolecular hydrogen bonds to form cyclic dimers.

2. Experimental

2.1. Materials and methods

The required 1,3-diketone compounds were prepared by our group[9]. Other reagents used were of analytical grade and without further purification. Elemental analysis (C, H, N) was performed using a Perkin-Elmer 2400 elemental analyzer. ^{1}H NMR spectra were measured on a Varian Mercury-Plus 400 NMR nuclear magnetic resonance instrument in $CDCl_3$ solution with TMS as internal standard. Infrared spectra (4000–400 cm^{-1}) were recorded on a Nicolet FTIR 5700 spectrophotometer with KBr pellets. Low-resolution electrospray ionization mass spectra (ESI-MS) were obtained on a Finnigan LCQ ADVANTAGE MAX spectrometer. Melting points were determined using X-4 digital melting point apparatus and uncorrected.

2.2. Synthesis

The synthesis of compounds **1**–**7** was conducted as outlined in Scheme 1. Double condensation of 1,3-diketone compounds with hydrazine monohydrate in ethanol afforded 3,5-dissubstituted-1*H*-pyrazoles in moderate to good yields, as shown in Table 1. All products were characterized by element analysis, FTIR, ^{1}H NMR and ESI-MS.

2.2.1. General synthesis for 3,5-diaryl-1H-pyrazoles (**1–7**)

The required 1,3-diketone (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 (detected by thin layer chromatography, TLC), the solvent was removed by evaporation under reduced pressure to obtain the solid mixture, which was recrystallized from ethanol solution to give the corresponding 3,5-disubstituted-1*H*-pyrazole.

2.2.2. 3-(Furan-2-yl)-5-(naphthalen-2-yl)-1H-pyrazole (**1**)

White solid, yield 72%, mp 167–168 °C; IR: (KBr, cm^{-1}) 3132(b, s), 3056(s), 2970(s), 2919(s), 1582(m), 1561(s), 1513(s), 1452(m),

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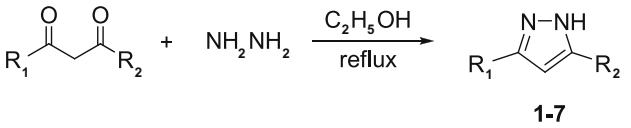


Scheme 1. Reaction of 1,3-diketones with hydrazine.

Table 1
Preparation of 3,5-disubstituted-1*H*-pyrazoles.

1*H*-Pyrazole	R_1	R_2	Mp (°C)	Yield (%)
1	C_4H_3O–	2–$C_{10}H_7$–	167–168	72
2	C_4H_3O–	6–CH_3O–2–$C_{10}H_6$–	188–189	73
3	C_6H_4–	2–$C_{10}H_7$–	119–120	76
4	C_6H_4–	6–CH_3O–2–$C_{10}H_6$–	212–213	71
5	4–$C(CH_3)_3$–C_6H_4–	4–F–C_6H_4–	209–210	56
6	4–$C(CH_3)_3$–C_6H_4–	2–$C_{10}H_7$–	201	70
7	4–$C(CH_3)_3$–C_6H_4–	6–CH_3O–2–$C_{10}H_6$–	244	67

1222(m), 1169(m), 1061(m), 1012(s), 972(s), 896(s), 787(s), 748(s), 727(s); ^{1}H NMR ($CDCl_3$, 400 Hz): δ = 6.47–6.48 (m, 1H, furanyl C–H), 6.68 (bs, 1H, N–H), 6.82(d, 1H, furanyl C–H, J = 2.8 Hz), 6.94(s, 1H, pyrazolyl C–H), 7.42(s, 1H, furanyl C–H), 7.48–7.51(m, 2H, Ar–H), 7.81–7.87(m, 4H, Ar–H), 8.23(s, 1H, Ar–H) ppm; ESI-MS: m/z 261.1 $[M + 1]^+$; Anal. Calcd. for $C_{17}H_{12}N_2O$: C, 78.44; H, 4.65; N, 10.76; Found C, 78.62; H, 4.56; N, 10.64.

*2.2.3. 3-(Furan-2-yl)-5-(6-methoxynaphthalen-2-yl)-1H-pyrazole (**2**)*

Colorless needles, yield 73%, mp 188–189 °C; IR: (KBr, cm^{-1}) 3215(b,s), 3054(m), 2958(m), 2853(m), 1610(m), 1565(s), 1507(m), 1486(m), 1452(s), 1391(s), 1261(s), 1173(s), 1028(s), 973(s), 907(s), 851(s), 793(s), 729(s); ^{1}H NMR ($CDCl_3$, 400 Hz): δ = 3.94(s, 3H, CH_3O), 6.48(s, 1H, furanyl C–H), 6.78(s, 1H, furanyl C–H), 6.88(s, 1H, pyrazolyl C–H), 7.14(d, 1H, Ar–H, J = 3.2 Hz), 7.16(d, 1H, Ar–H, J = 2.4 Hz), 7.43(s, 1H, furanyl C–H), 7.72–7.81(m, 3H, Ar–H), 8.11(s, 1H, Ar–H) ppm, pyrazolyl N–H not found; ESI-MS: m/z 580.8 $[2 M]^+$, 291.2 $[M + 1]^+$; Anal. Calcd. for $C_{18}H_{14}N_2O_2$: C, 74.47; H, 4.86; N, 9.65; Found C, 74.61; H, 4.82; N, 9.55.

*2.2.4. 5-(Naphthalen-2-yl)-3-phenyl-1H-pyrazole (**3**)*

White solid, yield 76%, mp 119–120 °C; IR: (KBr, cm^{-1}) 3228(b,s), 3036(m), 3002(m), 1596(m), 1503(s), 1456(s), 1268(s), 1170(m), 1054(s), 965(s), 830(s), 792(s), 754(s), 695(s); ^{1}H NMR ($CDCl_3$, 400 Hz): δ = 6.35(bs, 1H, N–H), 7.01(s, 1H, pyrazolyl C–H), 7.34–7.49(m, 5H, Ar–H), 7.78–7.84(m, 6H, Ar–H), 8.21(s, 1H, Ar–H) ppm; ESI-MS: m/z 271.2 $[M + 1]^+$; Anal. Calcd. for $C_{19}H_{14}N_2$: C, 84.42; H, 5.22; N, 10.36; Found C,83.92; H, 5.16; N, 10.18.

*2.2.5. 5-(6-Methoxynaphthalen-2-yl)-3-phenyl-1H-pyrazole (**4**)*

White solid, yield 71%, mp 212–213 °C; IR: (KBr, cm^{-1}) 3202(b,s), 3060(m), 3001(m), 2937(m), 1608(m), 1566(s), 1498(s), 1460(s), 1390(s), 1260(s), 1222(s), 1174(s), 1028(s), 963(s), 862(s), 795(s), 769(s), 700(s); ^{1}H NMR ($CDCl_3$, 400 Hz): δ = 3.93(s, 3H, CH_3O), 5.80(bs, 1H, N–H), 6.97(s, 1H, pyrazolyl C–H), 7.01(s, 1H, Ar–H), 7.12–7.14(m, 1H, Ar–H), 7.62(d, 2H, Ar–H, J = 8.8 Hz), 7.70(d, 2H, Ar–H, J = 8.8 Hz), 7.74(d, 2H, Ar–H, J = 8.8 Hz), 7.82(d, 2H, Ar–H, J = 7.2 Hz), 8.10(s, 1H, Ar–H) ppm; ESI-MS: m/z 600.9 $[2 M]^+$, 301.4 $[M + 1]^+$; Anal. Calcd. for $C_{20}H_{16}N_2O$: C, 79.98; H, 5.37; N, 9.33; Found C, 80.13; H, 5.28; N, 9.27.

*2.2.6. 3(5)-(4-Tert-butylphenyl)-5(3)-(4-fluorphenyl)-1H-pyrazole (**5**)*

Colorless needles, yield 56%, mp 209–210 °C; IR: (KBr, cm^{-1}) 3214(b,s), 3055(m), 2964(s), 2868(m), 1608(m), 1502(s), 1447(s), 1364(m), 1263(m), 1217(s), 1158(s), 1049(s), 971(s), 838(s), 794(s), 736(m); ^{1}H NMR ($CDCl_3$, 400 Hz): δ = 1.34(s, 9H, $C(CH_3)_3$), 6.78(s, 1H, pyrazolyl C–H), 7.04–7.09(m, 2H, Ar–H), 7.44(d, 2H, Ar–H, J = 8.4 Hz), 7.64(d, 2H, Ar–H, J = 8.4 Hz), 7.71–7.75(m, 2H, Ar–H) ppm, pyrazolyl N–H not found; ESI-MS: m/z 295.1 $[M + 1]^+$; Anal. Calcd. for $C_{19}H_{19}FN_2$: C, 77.52; H, 6.51; N, 9.52; Found C, 77.46; H, 6.43; N, 9.45.

*2.2.7. 3-(4-Tert-butylphenyl)-5-(naphthalen-2-yl)-1H-pyrazole (**6**)*

White solid, yield 70%, mp 201 °C; IR: (KBr, cm^{-1}) 3229(b,s), 3051(s), 2962(s), 2865(m), 1582(m), 1500(s), 1448(s), 1362(m), 1267(s), 1175(m), 1051(s), 966(s), 835(s), 793(s), 750(s); ^{1}H NMR ($CDCl_3$, 400 Hz): δ = 1.34(s, 9H, $C(CH_3)_3$), 5.20(bs, 1H, N–H), 6.98(s, 1H, pyrazolyl C–H), 7.44–7.78(m, 4H, Ar–H), 7.71(d, 2H, Ar–H, J = 8.4 Hz), 7.70–7.89(m, 4H, Ar–H), 8.21(s, 1H, Ar–H) ppm; ESI-MS: m/z 652.9 $[2 M]^+$, 327.2 $[M + 1]^+$; Anal. Calcd. for $C_{23}H_{22}N_2$: C, 84.63; H, 6.79; N, 8.58; Found C, 84.29; H, 6.70; N, 8.42.

*2.2.8. 3-(4-Tert-butylphenyl)-5-(6-methoxynaphthalen-2-yl)-1H-pyrazole (**7**)*

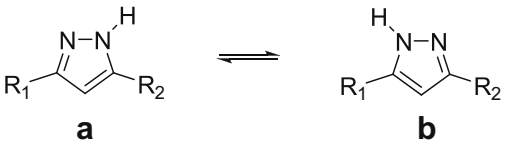
Colorless needles, yield 67%, mp 244 °C; IR: (KBr, cm^{-1}) 3215(b,s), 3056(m), 2963(s), 2868(m), 1614(s), 1585(s), 1507(s), 1445(s), 1393(s), 1261(s), 1168(s), 1029(s), 963(s), 859(s), 794(s); ^{1}H NMR ($CDCl_3$, 400 Hz): δ = 1.34(s, 9H, $C(CH_3)_3$), 3.92(s, 3H, CH_3O), 5.60(bs, 1H, N–H), 6.94(s, 1H, pyrazolyl C–H), 7.00(s, 1H, Ar–H), 7.10–7.13(m, 1H, Ar–H), 7.45(d, 2H, Ar–H, J = 8.4 Hz), 7.60(d, 1H, Ar–H, J = 8.4 Hz), 7.69(d, 2H, Ar–H, J = 8.8 Hz), 7.76(d, 2H, Ar–H, J = 8.4 Hz), 8.10(s, 1H, Ar–H) ppm; ESI-MS: m/z 712.9 $[2 M]^+$, 357.2 $[M + 1]^+$; Anal. Calcd. for $C_{24}H_{24}N_2O$: C, 80.87; H, 6.79; N,7.86; Found C, 80.64; H, 6.65; N, 7.92.

2.3. X-ray crystal structure determination

Suitable crystals of compounds **5** and **7** for X-ray diffraction experiments were grown by slow evaporation from CH_2Cl_2–EtOH

Table 2
Crystal data and structure refinements for **5** and **7**.

Compound	**5**	**7**
Empirical formula	$C_{19}H_{19}FN_2$	$C_{24}H_{24}N_2O$
Formula weight	294.36	356.45
Temperature (K)	298(2)	298(2)
Crystal system	Monoclinic	Monoclinic
Space group	C2/c	C2/c
a (Å)	19.9638(7)	34.9071(10)
b (Å)	17.7081(6)	6.15110(10)
c (Å)	9.7166(3)	21.7645(4)
α (°)	90	90
β (°)	110.4120(10)	123.620(2)
γ (°)	90	90
Volume (Å^3)	3219.33(19)	3891.51(15)
Z	8	4
Density (calc.) (mg/m^3)	1.215	1.217
Absorption coefficient (mm^{-1})	0.080	0.075
F(0 0 0)	1248	1520
Crystal size (mm^3)	0.20 × 0.10 × 0.10	0.13 × 0.10 × 0.10
Theta range for data	1.58 to 28.29	1.40 to 26.00
Collection (°)		
h/k/l	−26.26/−22.23/−12.8	−42.40/−7.7/−26.26
Reflections collected	11163	19456
Independent reflections	3796[R(int) = 0.0858]	3819[R(int) = 0.0932]
Data/restraints/parameters	3796/2/210	3819/0/248
Goodness-of-fit on F^2	0.923	1.028
Final R indices [$I > 2\sigma(I)$]	R^1 = 0.0575	R^1 = 0.0588
	wR^2 = 0.1403	wR^2 = 0.1611
R indices (all data)	R^1 = 0.0985	R^1 = 0.0774
	wR^2 = 0.1606	wR^2 = 0.1713
Largest diff. peak and hole (eÅ^{-3})	0.174 and −0.205	0.308 and −0.239



Scheme 2. Tautomerism of 3,5-disubstituted-1*H*-pyrazole.

(1:2) solutions. Single crystals were selected and mounted on the top of the glass fibres. X-ray single-crystal diffraction measurement was carried out at 298(2) K on a Bruker Smart 1000 CCD area diffractometer equipped with graphite monochromatic MoKα radiation (λ = 0.71073 Å) for data collection. The unit cell dimensions were obtained with the least-squares refinements and the structures were solved by direct methods with SHELXTL-97 program [10]. The final refinement was performed by full-matrix least-squares techniques with anisotropic thermal parameters for the non-hydrogen atoms on F^2. All the H atoms were placed in the calculated positions and constrained to ride on their parent atoms. Multi-scan absorption correction was applied by using the SADABS program [11]. The crystallographic data and refinement information for **5** and **7** are summarized in Table 2.

3. Results and discussion

Compounds **1–7** were synthesized from the corresponding 1,3-diketone compounds according to the published procedures [1,12]. Nucleophilic attack of hydrazine monohydrate with the required 1,3-diketone precursor in hot ethanol in each case led to formation of the required 3,5-disubstituted-1*H*-pyrazole, with isolation of the product involving a solvent evaporation step followed by recrystallization from ethanol solution. The yields were 56–76%.

3.1. Spectral characterization

In IR spectra, compounds **1–7** showed broad bands around 3300–3100 cm^{-1}, strong absorption bands in the regions of 1618–1582 cm^{-1} and 1513–1498 cm^{-1} and Medium bands from 1061 to 1028 cm^{-1} indicating the presence of the characteristic peaks for 1*H*-pyrazole compounds [13,14].

In ^{1}H NMR ($CDCl_3$), the title compounds showed the aromatic protons at δ = 6.47–8.23 ppm and a typical proton chemical shift of 3,5-disubstituted-1*H*-pyrazole (4-H) at δ = 6.78–7.01 ppm. In addition, the N–H resonance of the pyrazole ring of compounds **1**, **3**, **4**, **6**, **7** appeared as a broad and flat signal at δ = 5.20–6.68 ppm, but for compounds **2** and **5** the signal corresponding to the N–H resonance was not observed. Apparently, there is a fast tautomerism of the 1*H*-pyrazoles in $CDCl_3$ solution [15,16] (Scheme 2). This observation is in accordance with the crystal structures of compound **5**.

The mass spectroscopy by ESI-MS showed the molecular ions ($[M + 1]^+$) of all 3,5-disubstituted pyrazoles. However, compounds **2**, **4**, **6**, **7** also displayed their double molecular ions ($2\ M^+$) attributable to the dimers of 3,5-disubstituted-1*H*-pyrazoles. This result is in agreement with the X-ray crystal structures of compound **7**.

3.2. Crystal structures

Compounds **5** and **7** crystallize in the same space group, C2/c (Table 2). Their molecular structures with the atom-numbering scheme and unit cells packing figures are shown in Figs. 1–4, and selected bond lengths and angles are given in Table 3.

The molecules **5** and **7** consist of three ring systems, a pyrazole ring and two aryl rings. The dihedral angles formed by two benzene rings (C1–C6) and (C10–C15) with the pyrazole ring are both

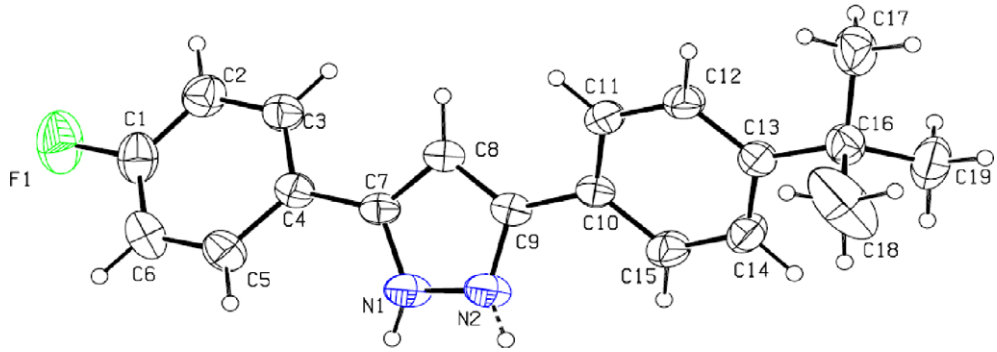


Fig. 1. View of **5** with the atom-labeling scheme and 50% probability displacement ellipsoids.

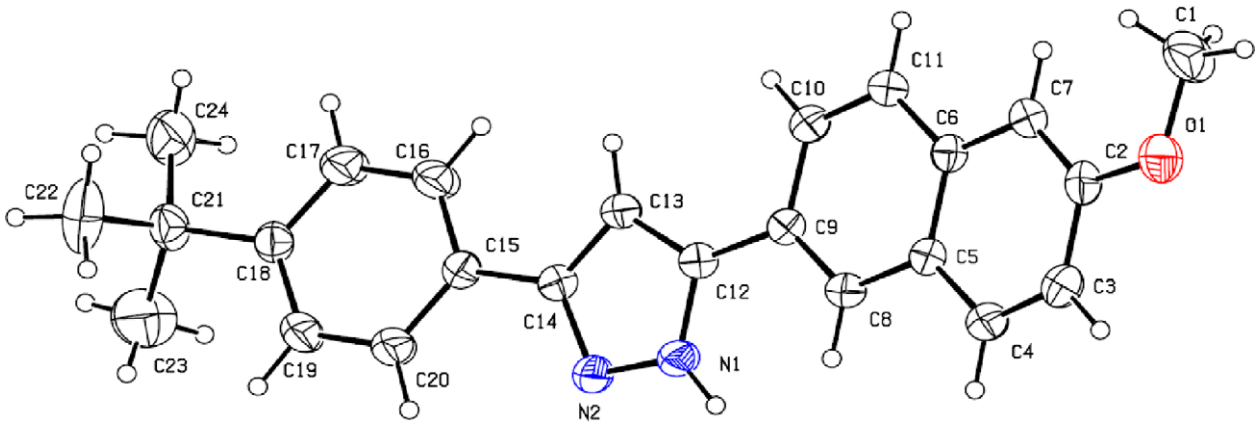


Fig. 2. View of **7** with the atom-labeling scheme and 50% probability displacement ellipsoids.

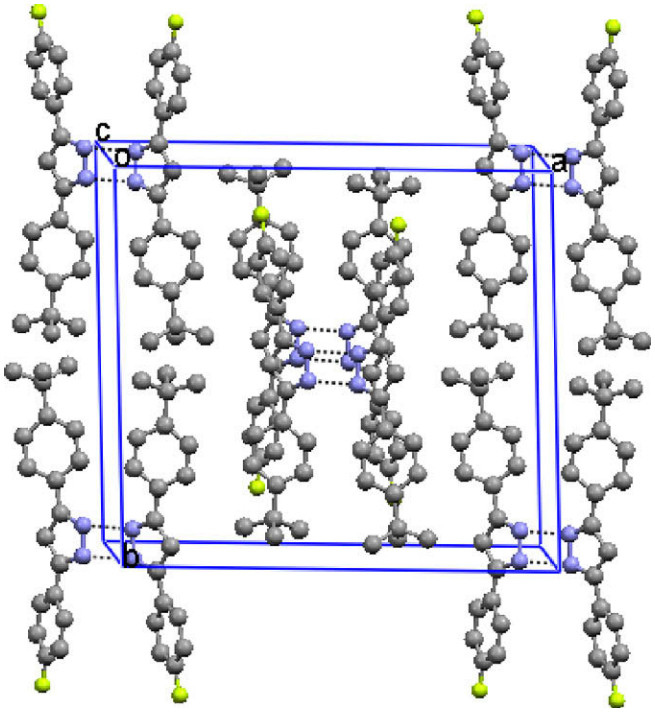


Fig. 3. The crystal structure of **5**, showing the packing mode of the dimers. Hydrogen atoms are omitted for clarity.

25.71° for **5**, but two benzene rings are not planar, those of two aryl rings (C2–C11) and (C15–C20) with the pyrazole ring are 9.07° and 15.01° for **7**, respectively. The molecules are linked together by pairs of intermolecular N–H...N hydrogen bonds to form the dimers [16] (Table 4). The crystals packing are stabilized by van der Waals forces (Figs. 3 and 4).

In compound **5**, the N–H hydrogen linkage of the pyrazole ring is disordered (Figs. 1 and 3), and its dimer is formed by the tautomers **5a5b** or **5b5a**. The measured N1...N1 distance is 2.874(3) Å, showing strong intermolecular N–H...N bonding interactions. The C7–N1 and C9–N2 bond lengths are 1.339(2) Å and 1.3339(19) Å, respectively, displaying double-bond character. This provides additional evidence to the disordered proton of the pyrazole ring.

However, the molecular structure for compound 7 only exists as the **7a** tautomer, and its dimer is formed by the tautomers **7a7a** (Figs. 2 and 4). The N–H bond of the pyrazole system resides at the nitrogen atom N1, adjacent to the naphthyl group. The observed N...N distance of 2.871(2) Å is typical for N–H...N hydrogen bond. The bond lengths of C12–N1 and C14–N2 are 1.3471(19) Å and 1.339(2) Å, respectively.

In conclusion, the analysis of the X-ray crystal structure data of compounds **5** and **7** shows that their molecules form dimmers as a

Table 3
Selected bond lengths (Å) and angles (°) for **5** and **7**.

Compound	Bond lengths	(Å)	Angles	(°)
5	N1–N2	1.351(2)	C7–N1–N2	108.93(14)
	C9–N2	1.3339(19)	C9–N2–N1	109.18(14)
	C8–C9	1.391(2)	N2–C9–C8	107.58(15)
	C7–C8	1.383(2)	C9–C8–C7	106.58(15)
	C7–N1	1.339(2)	C8–C7–N1	107.73(15)
7	N1–N2	1.3549(18)	C12–N1–N2	112.91(13)
	C12–N1	1.3471(19)	C14–N2–N1	104.63(12)
	C13–C12	1.367(2)	N2–C14–C13	110.08(14)
	C14–C13	1.392(2)	C14–C13–C12	107.00(14)
	C14–N2	1.339(2)	C13–C12–N1	105.37(14)

Table 4
Hydrogen-bonding geometry for **5** and **7**.

	D–H...A	D–H (Å)	H...A (Å)	D...A (Å)	D–H...A (°)
5	N1–H1A...N1^{i}	0.85(2)	2.17(2)	2.874(3)	140(3)
7	N1–H1...N2^{ii}	0.86(3)	2.15(2)	2.871(2)	141(2)

Symmetry codes: (i) $1-x, y, -z+3/2$; (ii) $1-x+1/2, -y+1/2, -z+1$.

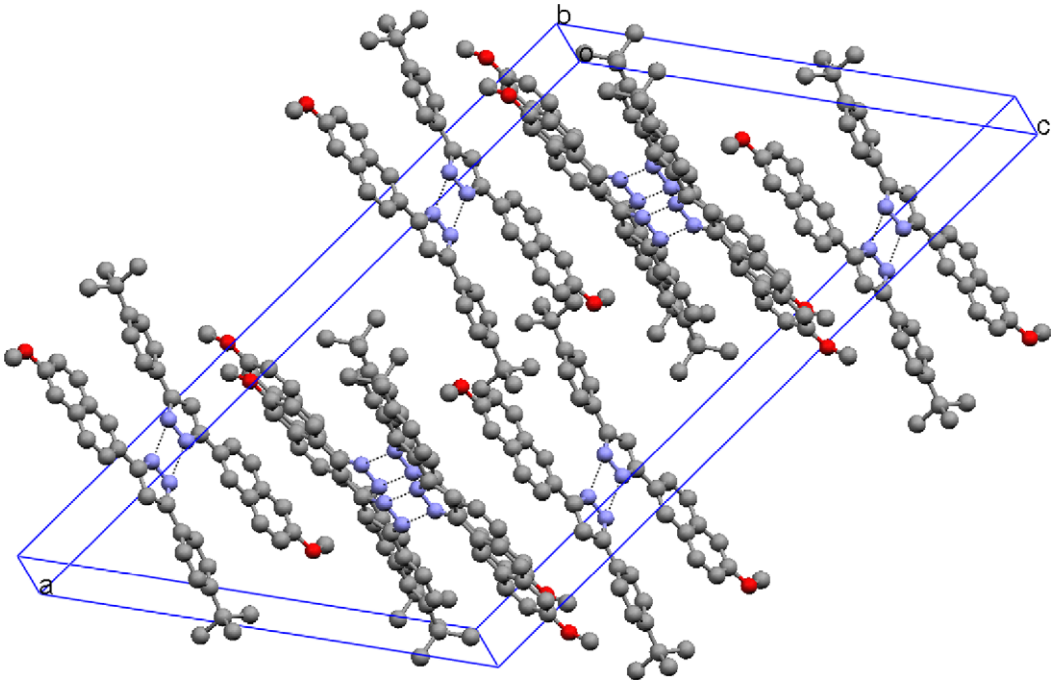


Fig. 4. The crystal structure of **7**, showing the packing mode of the dimers. Hydrogen atoms are omitted for clarity.

result of intermolecular N–H. . .N hydrogen bonding. In compound **5**, the dimer is formed by the tautomers **5a5b** or **5b5a**, and its NH proton is disordered. However, compound **7** is found to only exist as a **7a** tautomer, and its dimer consists of the tautomers **7a7a**. Probably, the different tautomerism in compounds **5** and **7** owe to the influence of fluoride atom on the benzene ring.

4. Supplementary material

Crystallographic data for structures **5** and **7** have been deposited at the Cambridge Crystallographic Data Center, CCDC Nos. 711753, 711755 for compounds **5** and **7**, respectively. Copies of this information may be obtained free of charge from the Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (Fax: +44 1223 336033; email: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

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