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ORIGINAL PAPER

Synthesis and Crystal Structures of *N*,*N*′-Disubstituted Piperazines

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Abstract The structure of 1,4-diphenylpiperazine (**1**) was determined; it crystallized in the orthorhombic space group *Pbca*, $a = 8.6980(7)$, $b = 8.4287(7)$, $c = 17.6359(15)$, $V = 1292.94(19)$, $Z = 4$. Three novel *N*,*N*′-disubstituted piperazines were synthesized via reductive amination of piperazine or *N*-diphenylmethylpiperazine. The products were characterized by NMR and X-ray crystallography. 1,4-Diphenethylpiperazine (**2**) crystallized in the monoclinic space group *C*2/*c*, $a = 17.9064(13)$, $b = 6.2517(5)$, $c = 14.9869(11)$, $\beta = 90.613(4)$, $V = 1677.6(2)$, $Z = 4$. 1-Benzhydryl-4-benzylpiperazine (**3**) crystallized in the monoclinic space group *Pn*, $a = 5.9450(2)$, $b = 19.0722(4)$, $c = 8.6084(2)$, $\beta = 96.4600(10)$, $V = 98.1790(10)$, $Z = 2$. 1-Benzhydryl-4-(pyridin-2-ylmethyl) piperazine (**4**) crystallized in the monoclinic space group *P*2/*c*, $a = 13.5637(2)$, $b = 5.82170(10)$, $c = 24.0645(4)$, $\beta = 90.613(4)$, $V = 1888.16(5)$, $Z = 4$. Comparison of the structures showed significant sp^2 character for the piperazine nitrogen atoms in **1**. Each structure showed multiple intermolecular non-bonding interactions.

Keywords Piperazine · X-ray · Crystal structure · Resonance · Amination

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Introduction

N,*N*′-Disubstituted piperazines have found application as ligands in metal complexes and form the basis of various natural products that exhibit favorable pharmacological properties. Piperazine is conveniently substituted at the *N* and *N*′ positions via reductive amination and nucleophilic substitution reactions. Synthetic piperazines are important in biomedical applications as ion channel and anticancer agents [1, 2]. The substitutional flexibility of piperazines also makes them tunable ligands. The behavior of piperazine ligands in metal complexes is also of interest because they can act either as chelating or bridging ligands. Although the cyclohexane-type ring is relatively flexible, the chelate bite of the nitrogen atoms requires adoption of a boat conformation leading to significant ring strain. Therefore, the piperazine is at least as likely to engage in bridging behavior between metal centers despite the well-known thermodynamic preference for chelation [3–11]. Many, but not all, chelated piperazine complexes feature ancillary coordination from pendant groups. A variety of bridging piperazine complexes has been reported, including metal dimers and tetramers, polymeric chains, and 2-D and 3-D frameworks [3, 12–22].

The four disubstituted piperazine ligands whose structures are reported herein, and which are shown in Chart 1, were prepared with the intention of producing photoluminescent network complexes with copper(I) salts. Copper(I) halide complexes tend to form oligomeric units or metal organic networks having copper coordination numbers of 2, 3, and/or 4 [20–26]. We have shown that certain low-coordinate copper(I) systems are reactive toward volatile nucleophiles, forming the basis for potential luminescence detectors [23–25]. Therefore, by controlling substituent size at the piperazine *N* and *N*′ positions we

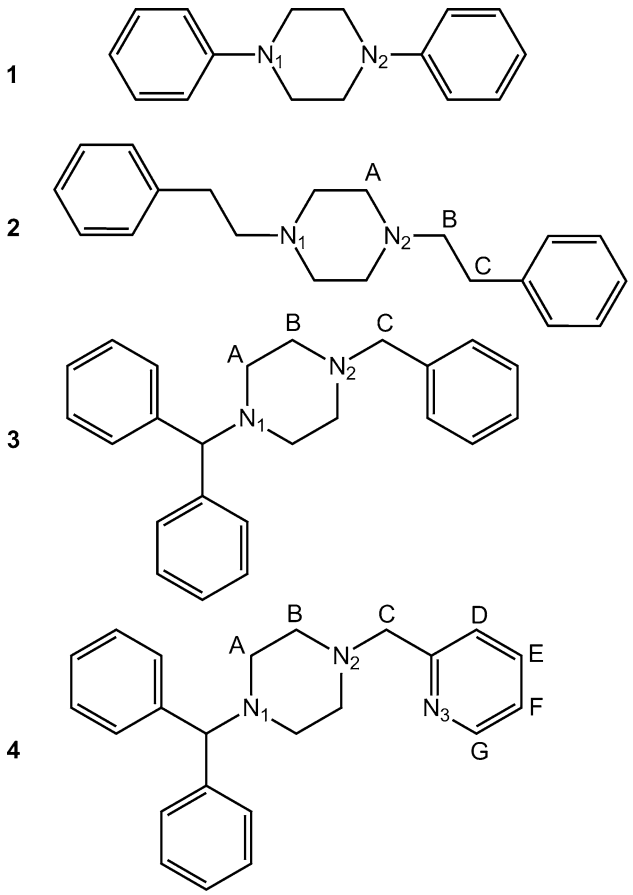


Chart 1 Disubstituted piperazines reported herein

hope to exert control over polymerization and copper(I) coordination number. Here we report the structures of four *N*,*N*′-disubstituted piperazine molecules.

Experimental

General

All reagents were purchased from Aldrich or Acros and were used as received including, piperazine, phenylacetaldehyde, diphenylmethylpiperazine, 2-pyridinecarboxaldehyde, and sodium triacetoxyborohydride ($NaBH(OAc)_3$), except for benzaldehyde, which was distilled prior to use. 1,4-Diphenylpiperazine (**1**) was purchased from MP Biomedical. Piperazines (**2**–**4**) were prepared via reductive amination as described below [20–27]. NMR data were recorded on a Varian Mercury 400 instrument (s = singlet, d = doublet, t = triplet, br = broad, Ph = phenyl, Py = pyridyl). Analyses for C, H, and N were carried out by Atlantic Microlabs, Norcross, GA or using a Thermo Scientific Flash 2000 Organic Elemental Analyzer with a Mettler Toledo XP6 Microbalance.

Synthesis and Crystallization

*1,4-Diphenylpiperazine (**1**)*

Commercial **1** (0.477 g, 2.00 mmol) was heated with CuI (0.190 g, 1 mmol) in 30 mL MeCN solution to 100 °C for 3 days in a sealed tube. No reaction took place, but **1** recrystallized, forming X-ray quality crystals.

*1,4-Diphenethylpiperazine (**2**)*

Piperazine (0.431 g, 5.00 mmol) and phenylacetaldehyde (1.204 g, 10.00 mmol) were dissolved in 30 mL of CH_2Cl_2 and stirred under Ar at room temp. To this solution a few drops of trifluoroacetic acid (0.2 mL) were added, and the mixture was allowed to stir for 30 min, producing a clear yellow solution. Next, $NaBH(OAc)_3$ (2.109 g, 10.00 mmol) dissolved in 25 mL CH_2Cl_2 was added, and the solution allowed to stir overnight under Ar. The resulting solution was washed with 1 M NaOH (aq), then saturated $NaHCO_3$ (aq), and finally deionized water. The organic layer was placed in the freezer and allowed to crystallize. The crystalline precipitate was collected via filtration (1.174 g, 79.2 %). X-ray quality crystals were grown by cooling a 0.15 M solution in CH_2Cl_2 to −5 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.62 (m, 12H, CH_2^A, CH_2^B), 2.81 (dd, J = 11.7, 7.7 Hz, 4H, CH_2^C), 7.20 (m, 6H, $Ph^{o,p}$), 7.27 (t, J = 7.4 Hz, 4H, Ph^m). $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 33.82, 53.38, 60.73, 126.28, 128.62, 128.92, and 140.53. Anal. Calcd. for $C_{20}H_{26}N_2$: C, 81.59; H, 8.89; N, 9.52. Found: C, 81.12; H, 8.73; N, 8.74.

*1-Benzhydryl-4-benzylpiperazine (**3**)*

N-Diphenylmethylpiperazine (3.061 g, 12.00 mmol) and benzaldehyde (1.281 g, 12.00 mmol) were dissolved in 30 mL CH_2Cl_2 and stirred under Ar at room temp. To this solution a few drops of trifluoroacetic acid (0.2 mL) were added, and the mixture was allowed to stir for 30 min, producing a clear solution. Next, $NaBH(OAc)_3$ (2.539 g, 12.00 mmol) dissolved in 25 mL CH_2Cl_2 was added, and the mixture was allowed to stir overnight under Ar. The resulting solution was washed with 1 M NaOH (aq), then saturated $NaHCO_3$ (aq), and finally deionized water. The organic layer was passed through a plug of activated Al_2O_3 and evaporated in vacuo, resulting in a white powder that was dried overnight under vacuum (1.340 g, 59.29 %). X-ray quality crystals were obtained through slow evaporation of a pentane solution. ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.46 (m, 8H, CH_2^A, CH_2^B), 3.51 (s, 2H, CH_2^C), 4.22 (s, 1H, CH), 7.15 (t, J = 7.0 Hz, 2H, $CHPh_2^p$), 7.25 (m, 9H, Ph), 7.39 (d, J = 7.0 Hz, 4H, $CHPh_2^o$). $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 52.14, 53.59, 65.30, 76.44, 127.06,

Table 1 Crystal and structure refinement data

	1,4-Diphenylpiperazine (**1**)	1,4-Diphenethyl-piperazine (**2**)	1-Benzhydryl-4-benzylpiperazine (**3**)	1-Benzhydryl-4-(pyridin-2-ylmethyl)-piperazine (**4**)
CCDC deposit no.	863662	863658	863660	863661
Color and habit	Colorless plate	Colorless prism	Colorless plate	Colorless plate
Size, mm	0.28 × 0.27 × 0.14	0.32 × 0.12 × 0.07	0.44 × 0.16 × 0.06	0.44 × 0.19 × 0.07
Formula	$C_{16}H_2N_{18}$	$C_{20}N_2H_{26}$	$C_{24}N_2H_{26}$	$C_{23}N_3H_{25}$
Formula weight	238.67	296.46	342.48	343.47
Space group	*Pbca*	*C2/c*	*Pn*	*P2/c*
a, Å	8.6980(7)	17.9064(13)	5.9450(2)	13.5637(2)
b, Å	8.4287(7)	6.2517(5)	19.0722(4)	5.82170(10)
c, Å	17.6359(15)	14.9869(11)	8.6084(2)	24.0645(4)
β, °	90	90.613(4)	98.1790(10)	96.4600(10)
Volume, Å^3	1292.94(19)	1677.6(2)	966.13(4)	1888.16(5)
Z	4	4	2	4
ρ_{calc}, g cm^{-3}	1.224	1.174	1.177	1.208
F_{000}	512	648	368	736
μ(Cu Kα), mm^{-1}	0.556	0.516	0.522	0.551
Radiation	Cu Kα (λ = 1.54178 Å)	Cu Kα (λ = 1.54178 Å)	Cu Kα (λ = 1.54178 Å)	Cu Kα (λ = 1.54178 Å)
Temperature, K	100	100	100	100
Residuals:[a] R; R_w	0.0353, 0.0906	0.0374, 0.0943	0.0279, 0.0684	0.0312, 0.0801
Goodness of fit	1.071	1.075	1.055	1.044

[a] $R = R_1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|$ for observed data only. $R_w = wR_2 = \{\Sigma[w(F_o^2 - F_c^2)^2]/\Sigma[w(F_o^2)^2]\}^{1/2}$ for all data

127.18, 128.21, 128.36, 128.62, 129.45, 138.37, 143.04. Anal. Calcd. for $C_{24}H_{26}N_2$: C, 84.17; H, 7.65; N, 8.18. Found: C, 83.89; H, 7.53; N, 7.96.

*1-Benzhydryl-4-(pyridin-2-ylmethyl)piperazine (**4**)*

N-Diphenylmethylpiperazine (2.526 g, 10.00 mmol) and 2-pyridinecarboxaldehyde (1.070 g, 10.00 mmol) were dissolved in 30 mL CH_2Cl_2 and stirred under Ar at room temp. To this solution a few drops of trifluoroacetic acid (0.2 mL) were added and the mixture was allowed to stir for 30 min, producing a clear yellow solution. Next, $NaBH(OAc)_3$ (2.158 g, 10.00 mmol) dissolved in 25 mL CH_2Cl_2 was added, and the mixture was allowed to stir overnight under Ar. The resulting solution was washed with 1 M NaOH (aq), then saturated $NaHCO_3$ (aq), and finally deionized water. The organic layer was passed through a plug of activated Al_2O_3 and evaporated *in vacuo*. The resulting thick yellow oil solidified into a beige powder under vacuum (1.34 g, 59.3 %). X-ray quality crystals were obtained by layering a 20 mM solution in CH_3CN with ether in a 5 mm diameter tube. ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.44 (br s, 4H, CH_2^A), 2.54 (br s, 4H, CH_2^B), 3.66 (s, 2H, CH_2^C), 4.23 (s, 1H, CH), 7.15 (m, 3H, Ph^p, Py^E), 7.25 (t, J = 7.8 Hz, 4H, Ph^m), 7.37 (d, J = 7.8, 1H, Py^D), 7.40 (d, J = 6.3 Hz, 4H, Ph^o), 7.61 (td, J = 7.8, 1.6 Hz, 1H, Py^F), 8.54 (d, J = 5.1 Hz, 1H, Py^G). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 51.81, 53.57, 64.57, 76.17, 121.88, 123.13, 126.82, 127.94, 128.38, 136.21, 142.75, 149.21, 158.64. Anal. Calcd. for $C_{23}H_{25}N_3$: C, 80.43; H, 7.33; N, 12.24. Found: C, 79.81; H, 7.30; N, 11.90.

X-ray Crystallography

Crystals were mounted on glass fibers. All measurements were made using graphite-monochromated Cu Kα radiation on a Bruker-AXS three-circle diffractometer, equipped with a SMART Apex II CCD detector. Initial space group determination was based on a matrix consisting of 120 frames. The data were reduced using SAINT+ [28], and empirical absorption correction applied using SADABS [29].

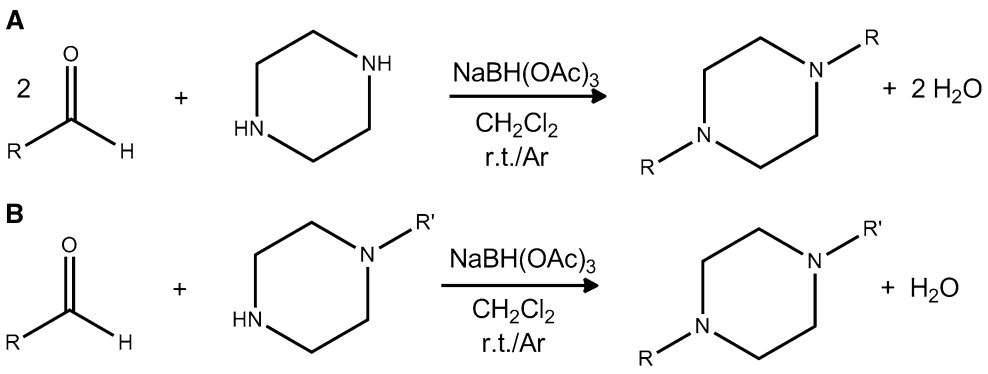
Structures were solved using direct methods. Least-squares refinement for all structures was carried out on F^2. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms in were located in the Fourier difference map and then allowed to refine isotropically. Structure solution, refinement and the calculation of derived results were performed using the SHELXTL package of computer programs [30]. Details of the X-ray experiments and crystal data are summarized in Table 1. Selected bond lengths and bond angles are given in Table 2.

Table 2 Selected bond lengths and angles

1,4-Diphenylpiperazine (**1**)		1,4-Diphenethylpiperazine (**2**)	
Bond lengths			
N(1)–C(1)	1.4589(15)	N–C(1)	1.4589(17)
N(1)–C(2)	1.4695(15)	N–C(2)	1.4617(16)
N(1)–C(3)	1.4157(15)	N–C(3)	1.4611(16)
Bond angles			
C(3)–N(1)–C(1)	116.84(9)	C(1)–N–C(2)	108.67(10)
C(3)–N(1)–C(2)	115.48(9)	C(1)–N–C(3)	110.58(10)
C(1)–N(1)–C(2)	110.18(9)	C(2)–N–C(3)	112.14(10)
1-Benzhydryl-4-benzylpiperazine (**3**)		1-Benzhydryl-4-(pyridin-2-ylmethyl)piperazine (**4**)	
Bond lengths			
N(1)–C(8)	1.459(2)	N(1)–C(4)	1.4583(14)
N(1)–C(10)	1.4600(18)	N(1)–C(1)	1.4585(13)
N(1)–C(7)	1.463(2)	N(1)–C(18)	1.4600(13)
N(2)–C(11)	1.4686(18)	N(2)–C(3)	1.4678(13)
N(2)–C(9)	1.4690(19)	N(2)–C(2)	1.4699(13)
N(2)–C(12)	1.477(2)	N(2)–C(5)	1.4807(13)
C(10)–C(11)	1.514(2)	C(3)–C(4)	1.5161(15)
C(8)–C(9)	1.518(2)	C(1)–C(2)	1.5153(15)
Bond angles			
C(8)–N(1)–C(10)	108.61(11)	C(4)–N(1)–C(1)	108.69(8)
C(8)–N(1)–C(7)	111.86(12)	C(4)–N(1)–C(18)	111.64(8)
C(10)–N(1)–C(7)	110.26(12)	C(1)–N(1)–C(18)	111.32(8)
C(11)–N(2)–C(9)	107.92(11)	C(3)–N(2)–C(2)	108.83(8)
C(11)–N(2)–C(12)	110.04(12)	C(3)–N(2)–C(5)	108.81(8)
C(9)–N(2)–C(12)	110.60(11)	C(2)–N(2)–C(5)	110.93(8)
N(1)–C(8)–C(9)	110.67(12)	N(1)–C(1)–C(2)	110.20(9)
N(2)–C(9)–C(8)	111.23(12)	N(2)–C(2)–C(1)	110.47(9)
N(1)–C(10)–C(11)	110.27(12)	N(2)–C(3)–C(4)	111.99(9)
N(2)–C(11)–C(10)	110.73(13)	N(1)–C(4)–C(3)	109.82(9)

Results and Discussion

Compounds **2**–**4** were prepared through reductive amination by stirring overnight in the presence of reducing agent, as indicated in Scheme 1. For the homo-disubstituted **2**, piperazine was reacted with two equivalents of phenylacetaldehyde. The hetero-disubstituted **3** and **4** were prepared by reacting *N*-diphenylmethylpiperazine with



Scheme 1 Syntheses of disubstituted piperazines

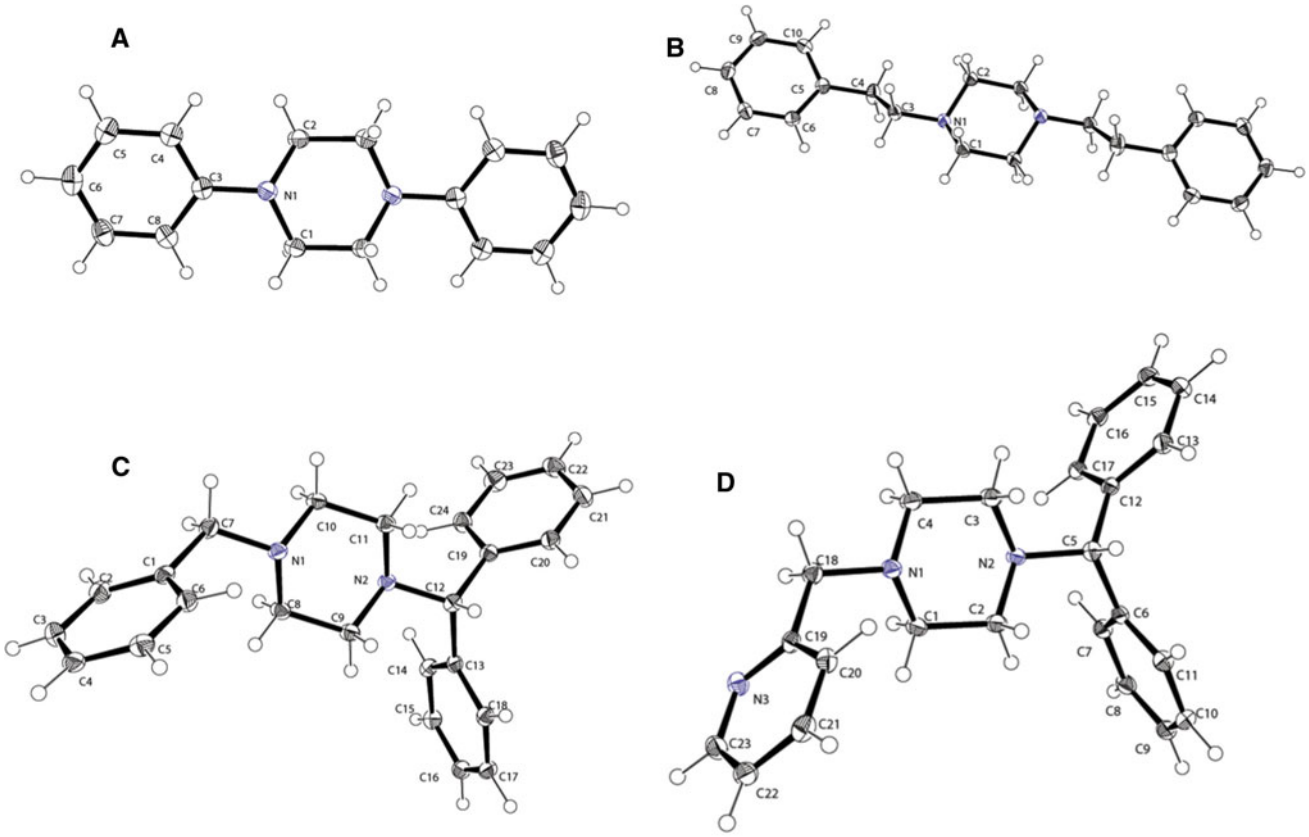
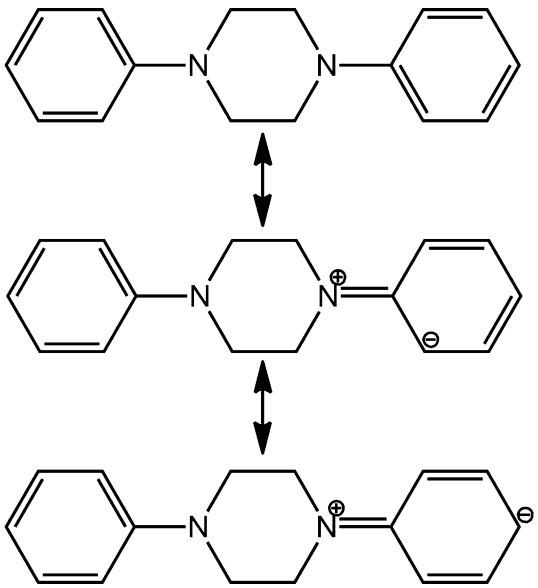


Fig. 1 Thermal ellipsoid (50 % probability) drawings of **1**, **2**, **3**, and **4** (**A**–**D**)



Scheme 2 Resonance in 1,4-diphenylpiperazine

benzaldehyde and 2-pyridinecarboxaldehyde, respectively. The reactions produced beige to brown solids that precipitated out of solution upon cooling or evaporation. The

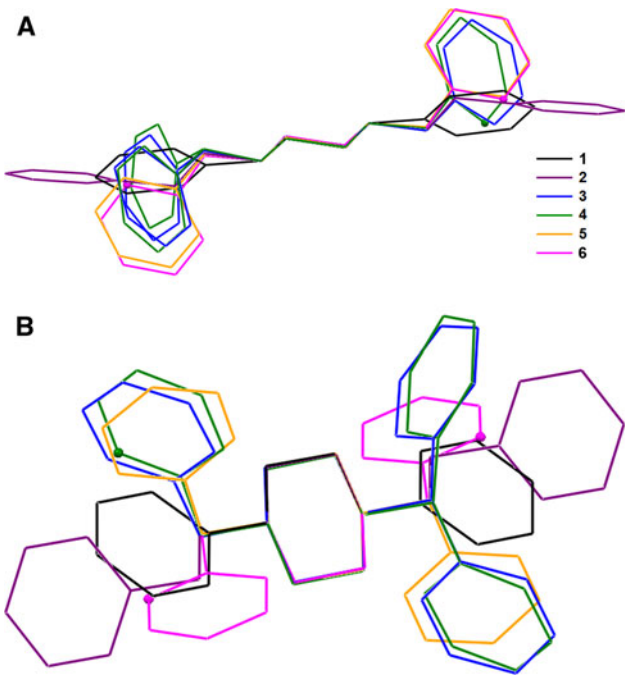


Fig. 2 Wireframe representations of compounds **1**–**6** overlaid using the piperazine rings. Ring nitrogen atoms are shown as spheres

analytically pure compounds were then crystallized for X-ray diffraction. For structural comparison, commercial 1,4-diphenylpiperazine (**1**) was also crystallized for X-ray diffraction.

The novel structures of **1**–**4** are shown in Fig. 1. Crystallographic determination information is given in Table 1 and selected bond lengths and angles in Table 2. For compounds **1** and **2**, the molecules were situated around an inversion center, located at the center of the piperazine ring. For compounds **3** and **4** the entire molecule comprised the asymmetric unit.

Numerous related *N*,*N*′-disubstituted piperazines have been reported, including ring-substituted *N*,*N*′-diphenylpiperazines [31–36], piperazines with the N-$CHPh_2$ substituent [37–42], the simple *N*,*N*′-dibenzylpiperazine (**5**) [43], and 1,4-bis(pyridin-2-ylmethyl)piperazine (**6**) [3, 4]. In the present cases all piperazine carbon–nitrogen and carbon–carbon bond lengths were found to be within the expected range. However, for compound **1** the phenyl groups appear to force a more planar conformation with bond angles about the nitrogen atoms, approaching the 120° sp^2 value. This effect may be attributed to the delocalization of the nitrogen lone pair caused by resonance with the aromatic system, as shown in Scheme 2. In addition, the N(1)–C(3) bond length of 1.4157(15) Å in **1** is significantly shorter than substituent N–C bond lengths (1.4589(17)–1.4807(13) Å) in **2**–**6**, again suggesting a resonance effect in the case of **1**. In structures **2**–**6** the aromatic substituents are all at least one carbon removed from the piperazine ring, allowing for a more tetrahedral conformation and angles much closer to the canonical sp^3 hybrid value of 109.5°. The distinctions between compounds **1** and **2**–**6** are apparent in the structural overlay shown in Fig. 2.

Multiple short, non-bonded intermolecular interactions were observed in all four structures. In each case the relevant C–H···X angle was greater than 140°. Compound **1** showed close intermolecular interactions between H1B and C7 (2.880(14) Å), H2B and C5 (2.826(14) Å), H4 and C6 (2.862(14) Å) and H6 and C4 (2.768(16) Å). Compound **2** demonstrated intermolecular interactions between H6 and a phenyl ring centroid C5–C10 (2.70(3) Å) and between H3A and C7 (2.887(14) Å). Compound **3** revealed a variety of intermolecular interactions: between H9B and C4 (2.687(18) Å), ring centroid C13–C18 and H4 (2.82(3) Å), and bond centroid C4/C5 and H7B (2.74(3) Å). Finally, a very close intermolecular interaction was seen in compound **4**: between the pyridyl nitrogen N3 and phenyl H9 with a distance of 2.557(14) Å. Further intermolecular interactions were found between H1A and C8 (2.883(13) Å), and between H22 and phenyl centroid C6–C11 (2.61(2) Å). In all four compounds, these non-bonded interactions weakly knit the structures into three dimensional networks.

Conclusions

We have synthesized and characterized three *N*,*N*′-disubstituted piperazines in which the substituents are linked to the ring by a saturated carbon. Comparison of these and related structures to the newly presented *N*,*N*′-diphenylpiperazine reveals flattening in the latter, indicative of resonance π-bond character for the N–Ph substituents. Non-bonding interactions are present in all four new structures.

References

1. Choi Y-H, Baek DJ, Seo SH, Lee JK, Pae AN, Cho YS, Min S-J (2011) Bioorg Med Chem Lett 21:215–219
2. Rossi C, Porcelloni M, D'Andrea P, Fincham C, Ettorre A, Mauro S, Squarcia A, Bigioni M, Parlani M, Nardelli F, Binaschi M, Maggi CA, Fattori D (2011) Bioorg Med Chem Lett 21:2305–2308
3. Ratilainen J, Airola K, Fröhlich R, Nieger M, Rissanen K (1999) Polyhedron 18:2265–2273
4. Halfen JA, Uhan JM, Fox DC, Mehn MP, Que L Jr (2000) Inorg Chem 39:4913–4920
5. Ciccarese A, Clemente DA, Fanizzi FP, Marzotto A, Valle G (1998) Inorg Chim Acta 275:410–418
6. Ciccarese A, Clemente DA, Fanizzi FP, Marzotto A, Valle G (1998) Acta Crystallogr C 54:1779–1781
7. Beller M, Trauthwein H, Eichberger M, Breindl C, Muller TE, Zapf A (1998) J Organomet Chem 566:277–285
8. Anderson CE, Apperley DC, Battsanov AS, Dyer PW, Howard JAK (2006) Dalton Trans 4134–4145
9. Fritz T, Steinfeld G, Kersting B (2007) Z Naturforsch B 62:508–518
10. Hollo-Sitkei E, Tarkanyi G, Parkanyi L, Megyes T, Besenyi G (2008) Eur J Inorg Chem 1573–1583
11. Zhang W, Xiong RG, Huang S (2008) J Am Chem Soc 130:10468–10469
12. Obaidi NA, Harnor TA, Jones CJ, McCleverty JA, Paxton K (1987) J Chem Soc Dalton Trans 2653–2660
13. Soma T, Miyamoto TK, Iwamoto T (1997) Chem Lett 319
14. Stocker FB, Staeva TB, Rienstra CM, Britton D (1999) Inorg Chem 38:984–991
15. Yang S, Zou Y, Zhu H-L (2005) Acta Crystallogr E 61:m219–m220
16. Zhang R-F, Zhao B, Wang H-S, Cheng P (2007) Inorg Chem Commun 10:1226–1228
17. Yilmaz VT, Guney S, Kazak C (2008) Polyhedron 27:1381–1386
18. Farnum GA, Knapp WR, LaDuca RL (2009) Polyhedron 28:291–299
19. Braga D, Maini L, Mazzeo PP, Ventura B (2010) Chem Eur J 16:1553–1559
20. Lim MJ, Murray CA, Tronic TA, deKrafft KE, Ley AN, deButts JC, Pike RD, Lu H, Patterson HH (2008) Inorg Chem 47:6931–6947
21. Hou Q, Yu J-H, Xu J-N, Yang Q-F, Xu J-Q (2009) Inorg Chim Acta 362:2802–2806
22. Braga D, Grepioni F, Maini L, Mazzeo PP, Ventura B (2011) New J Chem 35:339–344
23. Ley AN, Dunaway LE, Brewster TP, Dembo MD, Harris TD, Baril-Robert F, Li X, Patterson HH, Pike RD (2010) Chem Commun 46:4565–4567

24. Dembo MD, Dunaway LE, Jones JS, Lepekhina EA, McCullough SM, Ming JL, Li X, Baril-Robert F, Patterson HH, Bayse CA, Pike RD (2010) Inorg Chim Acta 364:102–114
25. Miller KM, McCullough SM, Lepekhina EA, Thibau IJ, Pike RD, Li X, Killarney JP, Patterson HH (2011) Inorg Chem 50:7239–7249
26. Araki H, Tsuge K, Sasaki Y, Ishizaka S, Kitamura N (2005) Inorg Chem 44:9667–9675
27. Denmark SE, Fu J (2002) Org Lett 4:1951–1953
28. SAINT PLUS (2001) Bruker Analytical X-ray Systems, Madison, WI
29. SADABS (2001) Bruker Analytical X-ray Systems, Madison, WI
30. Sheldrick GM (2008) Acta Crystallogr A 64:112–122
31. Owston PG, Peters R, Ramsammy E, Tasker PA, Trotter J (1980) J Chem Soc Chem Commun 1218–1220
32. Gourdon A, Launay J-P, Bujoli-Doeuff M, Heisel F, Miehe J-A, Amouyal E, Boillet M-L (1993) J Photochem Photobiol A 71:13–25
33. Spange S, El-Sayed M, Muller H, Rheinwald G, Lang H, Poppitz W (2002) Eur J Org Chem 4159–4168
34. MacPhee JM, Guzman HL, Almarsson O (2003) J Am Chem Soc 125:8456–8457
35. Lynch DE, McClenaghan I (2004) Acta Crystallogr C 60:o1–o5
36. Cherkasov V, Druzhkov N, Kocherova T, Fukin G, Shavyrin A (2011) Tetrahedron 67:80–84
37. Naveen S, Swamy SN, Basappa B, Rangappa KS (2006) Anal Sci 22:x41–x42
38. Kumar CSA, Prasad SBB, Thimmegowda NR, Rangappa KS, Chandrappa S, Naveen S, Sridhar MA, Prasad JS (2007) Mol Cryst Liq Cryst 469:111–119
39. Vinaya K, Naveen S, Kumar CSA, Benakaprasad SB, Sridhar MA, Prasad JS, Rangappa KS (2008) Struct Chem 19:765–770
40. Amor FB, M'hamed MO, Mrabet H, Driss A, Efrit ML (2008) Acta Crystallogr E 64:o1872
41. Naveen S, Kumar CSA, Prasad SBB, Vinaya K, Prasad JS, Rangappa KS (2009) J Chem Crystallogr 39:395–398
42. Naveen S, Kumar CSA, Manjunath HR, Prasad SBB, Sridhar MA, Rangappa KS, Prasad JS (2009) Mol Cryst Liq Cryst 503:151–158
43. Zhang M, Zhou Y-H, Hou L-H, Yang X-H (2010) Acta Crystallogr E 66:o3336