

Acta Crystallographica Section E

## Structure Reports

Online

ISSN 1600-5368

## 4,4'-[Piperazine-1,4-diylbis(propylene-nitrilomethylidene)]diphenol

Ruibo Xu,<sup>a</sup> Xingyou Xu,<sup>b\*</sup> Xujie Yang,<sup>c</sup> Yuping Huang<sup>a</sup> and Chunmei Xu<sup>a</sup>

<sup>a</sup>Department of Chemical Engineering, Huaihai Institute of Technology, Lianyungang 222005, People's Republic of China, <sup>b</sup>Huaiyin Institute of Technology, Huai'an 223003, People's Republic of China, and <sup>c</sup>Materials Chemistry Laboratory, Nanjing University of Science & Technology, Nanjing 210094, People's Republic of China  
Correspondence e-mail: xuruibo9125@163.com

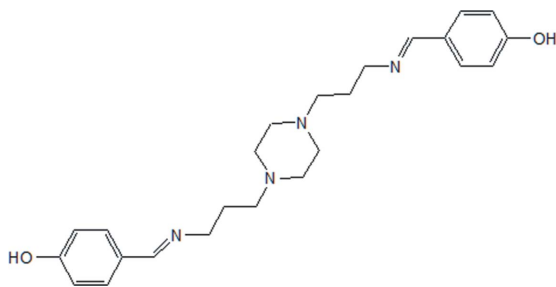
Received 25 October 2009; accepted 30 October 2009

Key indicators: single-crystal X-ray study;  $T = 298$  K; mean  $\sigma(\text{C}-\text{C}) = 0.005$  Å;  $R$  factor = 0.043;  $wR$  factor = 0.103; data-to-parameter ratio = 7.4.

In the title molecule,  $\text{C}_{24}\text{H}_{32}\text{N}_4\text{O}_2$ , the piperazine ring adopts a chair conformation and the dihedral angle between the two benzene rings is  $35.4(1)^\circ$ . In the crystal structure, intermolecular  $\text{O}-\text{H}\cdots\text{N}$  hydrogen bonds link molecules into chains along [001].

## Related literature

For the properties of piperazine derivatives, see: Keypour *et al.* (2008, 2009); Paital *et al.* (2009). For related structures, see: Thirumurugan *et al.* (1998); Yogavel *et al.* (2003).



## Experimental

## Crystal data

$\text{C}_{24}\text{H}_{32}\text{N}_4\text{O}_2$   
 $M_r = 408.54$   
Monoclinic,  $Cc$   
 $a = 5.9701(10)$  Å  
 $b = 30.159(3)$  Å

$c = 12.8348(18)$  Å  
 $\beta = 97.558(2)^\circ$   
 $V = 2290.9(6)$  Å<sup>3</sup>  
 $Z = 4$   
Mo  $K\alpha$  radiation

$\mu = 0.08$  mm<sup>-1</sup>  
 $T = 298$  K

0.17 × 0.15 × 0.11 mm

## Data collection

Siemens SMART CCD diffractometer  
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)  
 $T_{\min} = 0.987$ ,  $T_{\max} = 0.992$

5997 measured reflections  
2016 independent reflections  
1387 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.068$

## Refinement

$R[F^2 > 2\sigma(F^2)] = 0.043$   
 $wR(F^2) = 0.103$   
 $S = 0.96$   
2016 reflections  
271 parameters

2 restraints  
H-atom parameters constrained  
 $\Delta\rho_{\max} = 0.14$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.19$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O2}-\text{H2}\cdots\text{N1}^i$	0.82	1.98	2.762 (4)	159
$\text{O1}-\text{H1}\cdots\text{N2}^{ii}$	0.82	2.00	2.780 (4)	159

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

Data collection: SMART (Siemens, 1996); cell refinement: SAINT (Siemens, 1996); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); software used to prepare material for publication: SHELXTL.

This project was supported by the Key Project for Fundamental Research of the Jiangsu Provincial Educational Committee (07KJA150011), the Opened Funds of Jiangsu Key Laboratory of Marine Biotechnology of Huaihai Institute of Technology (2009HS06) and the QingLan Project of Jiangsu Province (2008).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2937).

## References

- Keypour, H., Rezaeivala, M., Valencia, L. & Pérez-Lourido, P. (2008). *Polyhedron*, **27**, 3172–3176.  
Keypour, H., Rezaeivala, M., Valencia, L., Pérez-Lourido, P. & Mahmoudkhani, A. H. (2009). *Polyhedron*, doi:10.1016/j.poly.2009.07.012.  
Paital, A. R., Mandal, D., Huang, X., Li, J., Aromic, G. & Ray, D. (2009). *Dalton Trans.* pp. 1352–1362.  
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.  
Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.  
Siemens (1996). *SMART* and *SAINTE*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.  
Thirumurugan, R., Shanmuga Sundara Raj, S., Shanmugam, G., Fun, H.-K., Marappan, M. & Kandaswamy, M. (1998). *Acta Cryst.* **C54**, 644–645.  
Yogavel, M., Selvanayagam, S., Velmurugan, D., Shanmuga Sundara Raj, S., Fun, H.-K., Marappan, M. & Kandaswamy, M. (2003). *Acta Cryst.* **E59**, o83–85.

**supplementary materials**

*Acta Cryst.* (2009). E65, o2996 [ doi:10.1107/S1600536809045620 ]

## 4,4'-[Piperazine-1,4-diylbis(propylenenitrilomethylidyne)]diphenol

R. Xu, X. Xu, X. Yang, Y. Huang and C. Xu

### Comment

Piperazine derivatives possess interesting structures and properties (Yogavel *et al.*, 2003; Thirumurugan *et al.*, 1998; Keypour *et al.*, 2008,2009; Paital *et al.*, 2009), therefore, our group have designed and prepared series of Schiff bases and complexes derived from substituted piperazines. As part of our work, the title compound (I) a potential hexadentate Schiff base ligand, was synthesized in our group and herein we report the crystal structure.

The molecular structure of (I) is shown in Fig. 1. In (I), the C8—N3 and C18—N4 double bond lengths are comparable to reported values (Yogavel *et al.*, 2003; Thirumurugan *et al.*, 1998). The dihedral angle between the two benzene rings (C9—C14 and C19—C24) is 35.4 (1)°. In the piperazine ring, atoms C1/C2/C3/C4 are essentially planar (the mean deviation from the plane is 0.0032 Å), and atoms N1 and N2 atom lie 0.6936 and 0.6693 Å either side of this plane. This four atom plane makes a dihedral angle of 50.9 (3)° with the plane of atoms C2/N2/C3 and 52.7 (3)° with atoms C1/N1/C4, respectively, in accordance with the chair conformation of the piperazine ring. In the crystal structure, intermolecular O—H···N hydrogen bonds link molecules into one-dimensional chains along [001](Fig.2).

### Experimental

A solution of *N,N*-bis(*N*-aminopropyl)-piperazine (1.5 mmol in 10 ml anhydrous methanol) was added dropwise with constant stirring to a solution of parahydroxybenzaldehyde (3 mmol in 15 ml anhydrous methanol) at 325 K for 3 h. The resulting mixture was filtered. After cooling, the filtrate was evaporated at ambient environment. Several days later, pink crystals suitable for X-ray analysis were collected and washed with small amount of methanol and dried at room temperature.

### Refinement

H atoms were placed in calculated positions with C—H = 0.97 Å(piperaziny), 0.93 Å(benzene), 0.82 Å(hydroxyl) and 0.97 Å(methylene), and refined in riding mode with  $U_{\text{iso}}(\text{H}) = 1.5 U_{\text{eq}}(\text{O})$  and  $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$ .

### Figures

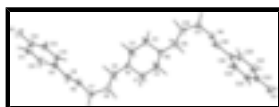


Fig. 1. The molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level.



Fig. 2. One-dimensional chain structure of (I) constructed by O—H···N intermolecular hydrogen bonds (dashed lines) along [001].

## 4,4'-[Piperazine-1,4-diylbis(propylenenitrilomethylidene)]diphenol

### Crystal data

$C_{24}H_{32}N_4O_2$	$F_{000} = 880$
$M_r = 408.54$	$D_x = 1.185 \text{ Mg m}^{-3}$
Monoclinic, $Cc$	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
Hall symbol: C -2yc	Cell parameters from 1760 reflections
$a = 5.9701 (10) \text{ \AA}$	$\theta = 2.7\text{--}24.9^\circ$
$b = 30.159 (3) \text{ \AA}$	$\mu = 0.08 \text{ mm}^{-1}$
$c = 12.8348 (18) \text{ \AA}$	$T = 298 \text{ K}$
$\beta = 97.558 (2)^\circ$	Lamellate, pink
$V = 2290.9 (6) \text{ \AA}^3$	$0.17 \times 0.15 \times 0.11 \text{ mm}$
$Z = 4$	

### Data collection

Siemens SMART CCD diffractometer	2016 independent reflections
Radiation source: fine-focus sealed tube	1387 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.068$
$T = 298 \text{ K}$	$\theta_{\text{max}} = 25.0^\circ$
$\varphi$ and $\omega$ scans	$\theta_{\text{min}} = 1.4^\circ$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -7 \rightarrow 6$
$T_{\text{min}} = 0.987$ , $T_{\text{max}} = 0.992$	$k = -30 \rightarrow 35$
5997 measured reflections	$l = -15 \rightarrow 15$

### Refinement

Refinement on $F^2$	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.043$	H-atom parameters constrained
$wR(F^2) = 0.103$	$w = 1/[\sigma^2(F_o^2) + (0.0507P)^2]$
$S = 0.96$	where $P = (F_o^2 + 2F_c^2)/3$
2016 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
271 parameters	$\Delta\rho_{\text{max}} = 0.14 \text{ e \AA}^{-3}$
2 restraints	$\Delta\rho_{\text{min}} = -0.19 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: none

*Special details*

**Geometry.** All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

**Refinement.** Refinement of  $F^2$  against ALL reflections. The weighted  $R$ -factor  $wR$  and goodness of fit  $S$  are based on  $F^2$ , conventional  $R$ -factors  $R$  are based on  $F$ , with  $F$  set to zero for negative  $F^2$ . The threshold expression of  $F^2 > \sigma(F^2)$  is used only for calculating  $R$ -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement.  $R$ -factors based on  $F^2$  are statistically about twice as large as those based on  $F$ , and  $R$ -factors based on ALL data will be even larger.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	0.6528 (5)	0.39796 (9)	0.5395 (2)	0.0440 (7)
N2	0.6327 (5)	0.34403 (9)	0.7270 (2)	0.0450 (7)
N3	0.6917 (7)	0.47133 (10)	0.2563 (2)	0.0619 (9)
N4	0.5753 (7)	0.26995 (10)	1.0120 (2)	0.0604 (9)
O1	1.0094 (5)	0.38009 (9)	-0.1521 (2)	0.0684 (8)
H1	0.8981	0.3653	-0.1744	0.103*
O2	0.2701 (4)	0.37289 (8)	1.40788 (19)	0.0592 (7)
H2	0.3880	0.3735	1.4486	0.089*
C1	0.5611 (7)	0.41527 (12)	0.6330 (3)	0.0509 (10)
H1A	0.4439	0.4369	0.6113	0.061*
H1B	0.6804	0.4300	0.6789	0.061*
C2	0.4643 (7)	0.37801 (12)	0.6919 (3)	0.0524 (10)
H2A	0.4043	0.3901	0.7526	0.063*
H2B	0.3403	0.3644	0.6467	0.063*
C3	0.7284 (7)	0.32708 (12)	0.6338 (3)	0.0510 (10)
H3A	0.6111	0.3122	0.5871	0.061*
H3B	0.8464	0.3057	0.6560	0.061*
C4	0.8247 (6)	0.36472 (12)	0.5762 (3)	0.0504 (10)
H4A	0.9453	0.3788	0.6226	0.060*
H4B	0.8890	0.3530	0.5163	0.060*
C5	0.7518 (7)	0.43362 (13)	0.4807 (3)	0.0552 (10)
H5A	0.8202	0.4203	0.4237	0.066*
H5B	0.8712	0.4479	0.5273	0.066*
C6	0.5862 (8)	0.46839 (13)	0.4356 (3)	0.0628 (11)
H6A	0.5490	0.4873	0.4920	0.075*
H6B	0.4481	0.4540	0.4044	0.075*
C7	0.6779 (9)	0.49699 (13)	0.3524 (3)	0.0680 (12)
H7A	0.5796	0.5224	0.3361	0.082*
H7B	0.8268	0.5079	0.3798	0.082*
C8	0.8797 (8)	0.46994 (12)	0.2204 (3)	0.0555 (11)
H8	1.0022	0.4850	0.2563	0.067*
C9	0.9117 (7)	0.44553 (11)	0.1246 (3)	0.0479 (9)
C10	1.1116 (7)	0.44905 (13)	0.0817 (3)	0.0608 (10)

## supplementary materials

---

H10	1.2280	0.4664	0.1153	0.073*
C11	1.1427 (7)	0.42709 (14)	-0.0112 (3)	0.0584 (10)
H11	1.2774	0.4304	-0.0395	0.070*
C12	0.9734 (6)	0.40044 (12)	-0.0611 (3)	0.0471 (9)
C13	0.7736 (6)	0.39566 (12)	-0.0171 (3)	0.0501 (10)
H13	0.6592	0.3775	-0.0497	0.060*
C14	0.7435 (6)	0.41775 (12)	0.0751 (3)	0.0472 (9)
H14	0.6100	0.4140	0.1041	0.057*
C15	0.5266 (7)	0.30844 (12)	0.7830 (3)	0.0566 (10)
H15A	0.4159	0.2933	0.7332	0.068*
H15B	0.4465	0.3219	0.8360	0.068*
C16	0.6907 (8)	0.27448 (12)	0.8355 (3)	0.0591 (11)
H16A	0.8220	0.2896	0.8713	0.071*
H16B	0.7411	0.2555	0.7821	0.071*
C17	0.5864 (9)	0.24597 (14)	0.9141 (3)	0.0703 (13)
H17A	0.4353	0.2372	0.8841	0.084*
H17B	0.6759	0.2193	0.9286	0.084*
C18	0.3869 (7)	0.28242 (12)	1.0347 (3)	0.0530 (10)
H18	0.2584	0.2760	0.9879	0.064*
C19	0.3598 (7)	0.30661 (11)	1.1315 (3)	0.0446 (9)
C20	0.5407 (7)	0.31283 (13)	1.2093 (3)	0.0563 (11)
H20	0.6820	0.3019	1.1994	0.068*
C21	0.5156 (7)	0.33506 (13)	1.3016 (3)	0.0566 (10)
H21	0.6392	0.3387	1.3530	0.068*
C22	0.3063 (6)	0.35184 (11)	1.3173 (3)	0.0462 (9)
C23	0.1256 (7)	0.34603 (12)	1.2404 (3)	0.0517 (9)
H23	-0.0150	0.3574	1.2501	0.062*
C24	0.1506 (7)	0.32352 (12)	1.1488 (3)	0.0545 (10)
H24	0.0260	0.3196	1.0980	0.065*

### Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
N1	0.0460 (18)	0.0507 (17)	0.0371 (16)	0.0038 (15)	0.0126 (15)	-0.0067 (14)
N2	0.0486 (18)	0.0493 (17)	0.0378 (16)	0.0054 (15)	0.0086 (14)	-0.0041 (14)
N3	0.086 (3)	0.053 (2)	0.047 (2)	-0.0058 (19)	0.0136 (19)	-0.0030 (17)
N4	0.084 (3)	0.0542 (19)	0.045 (2)	0.0001 (19)	0.0140 (19)	-0.0078 (16)
O1	0.0580 (18)	0.092 (2)	0.0577 (17)	-0.0007 (15)	0.0161 (15)	-0.0221 (16)
O2	0.0517 (17)	0.0707 (18)	0.0567 (17)	0.0012 (14)	0.0131 (14)	-0.0143 (14)
C1	0.060 (2)	0.054 (2)	0.041 (2)	0.0106 (19)	0.0137 (19)	-0.0060 (18)
C2	0.049 (2)	0.067 (2)	0.043 (2)	0.012 (2)	0.0145 (19)	-0.0047 (19)
C3	0.057 (2)	0.054 (2)	0.043 (2)	0.0123 (19)	0.0086 (19)	-0.0054 (18)
C4	0.045 (2)	0.065 (3)	0.042 (2)	0.0102 (18)	0.0110 (18)	-0.0056 (19)
C5	0.063 (3)	0.058 (2)	0.046 (2)	-0.007 (2)	0.014 (2)	-0.0035 (19)
C6	0.083 (3)	0.052 (2)	0.056 (3)	0.003 (2)	0.019 (2)	-0.002 (2)
C7	0.105 (4)	0.049 (2)	0.052 (2)	-0.004 (2)	0.019 (2)	-0.008 (2)
C8	0.070 (3)	0.049 (2)	0.047 (2)	-0.005 (2)	0.004 (2)	-0.0016 (18)
C9	0.055 (2)	0.043 (2)	0.044 (2)	0.0047 (18)	0.0010 (19)	0.0022 (17)

C10	0.054 (3)	0.069 (3)	0.057 (2)	-0.009 (2)	-0.001 (2)	-0.007 (2)
C11	0.046 (2)	0.073 (3)	0.056 (2)	0.001 (2)	0.008 (2)	-0.003 (2)
C12	0.047 (2)	0.051 (2)	0.042 (2)	0.0076 (18)	0.0023 (19)	0.0014 (19)
C13	0.052 (2)	0.049 (2)	0.049 (2)	-0.0023 (18)	0.003 (2)	-0.0043 (18)
C14	0.049 (2)	0.049 (2)	0.045 (2)	0.0014 (18)	0.0081 (18)	0.0016 (18)
C15	0.059 (2)	0.064 (2)	0.048 (2)	-0.008 (2)	0.0096 (19)	-0.005 (2)
C16	0.083 (3)	0.050 (2)	0.045 (2)	0.012 (2)	0.012 (2)	-0.0052 (19)
C17	0.113 (4)	0.052 (2)	0.048 (2)	0.001 (3)	0.018 (3)	-0.005 (2)
C18	0.066 (3)	0.049 (2)	0.042 (2)	-0.011 (2)	0.001 (2)	0.0043 (18)
C19	0.055 (2)	0.045 (2)	0.0339 (19)	-0.0059 (18)	0.0061 (17)	0.0028 (16)
C20	0.052 (2)	0.068 (3)	0.051 (2)	0.000 (2)	0.014 (2)	-0.010 (2)
C21	0.047 (2)	0.077 (3)	0.046 (2)	-0.006 (2)	0.005 (2)	-0.016 (2)
C22	0.050 (2)	0.046 (2)	0.044 (2)	-0.0031 (19)	0.010 (2)	-0.0013 (18)
C23	0.047 (2)	0.055 (2)	0.053 (2)	0.0088 (19)	0.006 (2)	0.0049 (19)
C24	0.054 (2)	0.058 (2)	0.047 (2)	0.001 (2)	-0.0086 (19)	0.0080 (19)

*Geometric parameters (Å, °)*

N1—C4	1.467 (5)	C8—C9	1.466 (5)
N1—C1	1.478 (4)	C8—H8	0.9300
N1—C5	1.481 (4)	C9—C10	1.383 (5)
N2—C2	1.465 (5)	C9—C14	1.395 (5)
N2—C15	1.479 (4)	C10—C11	1.398 (5)
N2—C3	1.483 (4)	C10—H10	0.9300
N3—C8	1.269 (5)	C11—C12	1.381 (6)
N3—C7	1.467 (5)	C11—H11	0.9300
N4—C18	1.256 (5)	C12—C13	1.392 (5)
N4—C17	1.458 (5)	C13—C14	1.390 (5)
O1—C12	1.361 (4)	C13—H13	0.9300
O1—H1	0.8200	C14—H14	0.9300
O2—C22	1.367 (4)	C15—C16	1.513 (6)
O2—H2	0.8200	C15—H15A	0.9700
C1—C2	1.511 (5)	C15—H15B	0.9700
C1—H1A	0.9700	C16—C17	1.520 (5)
C1—H1B	0.9700	C16—H16A	0.9700
C2—H2A	0.9700	C16—H16B	0.9700
C2—H2B	0.9700	C17—H17A	0.9700
C3—C4	1.509 (5)	C17—H17B	0.9700
C3—H3A	0.9700	C18—C19	1.468 (5)
C3—H3B	0.9700	C18—H18	0.9300
C4—H4A	0.9700	C19—C20	1.384 (5)
C4—H4B	0.9700	C19—C24	1.394 (5)
C5—C6	1.504 (6)	C20—C21	1.386 (5)
C5—H5A	0.9700	C20—H20	0.9300
C5—H5B	0.9700	C21—C22	1.387 (5)
C6—C7	1.529 (6)	C21—H21	0.9300
C6—H6A	0.9700	C22—C23	1.374 (5)
C6—H6B	0.9700	C23—C24	1.382 (5)
C7—H7A	0.9700	C23—H23	0.9300

## supplementary materials

---

C7—H7B	0.9700	C24—H24	0.9300
C4—N1—C1	107.4 (3)	C10—C9—C8	120.7 (4)
C4—N1—C5	110.5 (3)	C14—C9—C8	121.2 (4)
C1—N1—C5	111.9 (3)	C9—C10—C11	121.5 (4)
C2—N2—C15	109.7 (3)	C9—C10—H10	119.3
C2—N2—C3	108.3 (3)	C11—C10—H10	119.3
C15—N2—C3	112.1 (3)	C12—C11—C10	120.0 (4)
C8—N3—C7	118.2 (4)	C12—C11—H11	120.0
C18—N4—C17	119.5 (4)	C10—C11—H11	120.0
C12—O1—H1	109.5	O1—C12—C11	118.1 (3)
C22—O2—H2	109.5	O1—C12—C13	122.8 (3)
N1—C1—C2	110.5 (3)	C11—C12—C13	119.1 (3)
N1—C1—H1A	109.5	C14—C13—C12	120.6 (3)
C2—C1—H1A	109.5	C14—C13—H13	119.7
N1—C1—H1B	109.5	C12—C13—H13	119.7
C2—C1—H1B	109.5	C13—C14—C9	120.7 (3)
H1A—C1—H1B	108.1	C13—C14—H14	119.7
N2—C2—C1	112.5 (3)	C9—C14—H14	119.7
N2—C2—H2A	109.1	N2—C15—C16	114.4 (3)
C1—C2—H2A	109.1	N2—C15—H15A	108.7
N2—C2—H2B	109.1	C16—C15—H15A	108.7
C1—C2—H2B	109.1	N2—C15—H15B	108.7
H2A—C2—H2B	107.8	C16—C15—H15B	108.7
N2—C3—C4	110.4 (3)	H15A—C15—H15B	107.6
N2—C3—H3A	109.6	C15—C16—C17	112.4 (4)
C4—C3—H3A	109.6	C15—C16—H16A	109.1
N2—C3—H3B	109.6	C17—C16—H16A	109.1
C4—C3—H3B	109.6	C15—C16—H16B	109.1
H3A—C3—H3B	108.1	C17—C16—H16B	109.1
N1—C4—C3	112.1 (3)	H16A—C16—H16B	107.9
N1—C4—H4A	109.2	N4—C17—C16	111.1 (3)
C3—C4—H4A	109.2	N4—C17—H17A	109.4
N1—C4—H4B	109.2	C16—C17—H17A	109.4
C3—C4—H4B	109.2	N4—C17—H17B	109.4
H4A—C4—H4B	107.9	C16—C17—H17B	109.4
N1—C5—C6	114.6 (3)	H17A—C17—H17B	108.0
N1—C5—H5A	108.6	N4—C18—C19	123.2 (4)
C6—C5—H5A	108.6	N4—C18—H18	118.4
N1—C5—H5B	108.6	C19—C18—H18	118.4
C6—C5—H5B	108.6	C20—C19—C24	117.8 (3)
H5A—C5—H5B	107.6	C20—C19—C18	121.0 (4)
C5—C6—C7	112.6 (4)	C24—C19—C18	121.2 (4)
C5—C6—H6A	109.1	C19—C20—C21	121.3 (4)
C7—C6—H6A	109.1	C19—C20—H20	119.3
C5—C6—H6B	109.1	C21—C20—H20	119.3
C7—C6—H6B	109.1	C20—C21—C22	120.1 (4)
H6A—C6—H6B	107.8	C20—C21—H21	120.0
N3—C7—C6	110.8 (3)	C22—C21—H21	120.0
N3—C7—H7A	109.5	O2—C22—C23	118.2 (3)

C6—C7—H7A	109.5	O2—C22—C21	122.6 (3)
N3—C7—H7B	109.5	C23—C22—C21	119.1 (3)
C6—C7—H7B	109.5	C22—C23—C24	120.7 (3)
H7A—C7—H7B	108.1	C22—C23—H23	119.6
N3—C8—C9	122.7 (4)	C24—C23—H23	119.6
N3—C8—H8	118.6	C23—C24—C19	120.9 (4)
C9—C8—H8	118.6	C23—C24—H24	119.5
C10—C9—C14	118.1 (3)	C19—C24—H24	119.5
C4—N1—C1—C2	-57.8 (4)	O1—C12—C13—C14	-178.9 (3)
C5—N1—C1—C2	-179.3 (3)	C11—C12—C13—C14	0.7 (6)
C15—N2—C2—C1	-179.4 (3)	C12—C13—C14—C9	0.8 (5)
C3—N2—C2—C1	-56.8 (4)	C10—C9—C14—C13	-2.5 (5)
N1—C1—C2—N2	59.2 (4)	C8—C9—C14—C13	178.4 (3)
C2—N2—C3—C4	56.1 (4)	C2—N2—C15—C16	-172.3 (3)
C15—N2—C3—C4	177.3 (3)	C3—N2—C15—C16	67.3 (4)
C1—N1—C4—C3	59.4 (4)	N2—C15—C16—C17	165.2 (3)
C5—N1—C4—C3	-178.3 (3)	C18—N4—C17—C16	109.1 (5)
N2—C3—C4—N1	-60.0 (4)	C15—C16—C17—N4	-77.3 (5)
C4—N1—C5—C6	177.5 (3)	C17—N4—C18—C19	179.8 (3)
C1—N1—C5—C6	-62.9 (4)	N4—C18—C19—C20	-7.1 (5)
N1—C5—C6—C7	-164.6 (3)	N4—C18—C19—C24	173.7 (3)
C8—N3—C7—C6	-125.9 (4)	C24—C19—C20—C21	0.1 (5)
C5—C6—C7—N3	70.6 (5)	C18—C19—C20—C21	-179.0 (3)
C7—N3—C8—C9	-179.6 (3)	C19—C20—C21—C22	-0.4 (6)
N3—C8—C9—C10	172.5 (4)	C20—C21—C22—O2	178.0 (3)
N3—C8—C9—C14	-8.3 (5)	C20—C21—C22—C23	0.1 (5)
C14—C9—C10—C11	2.7 (6)	O2—C22—C23—C24	-177.6 (3)
C8—C9—C10—C11	-178.1 (3)	C21—C22—C23—C24	0.4 (5)
C9—C10—C11—C12	-1.3 (6)	C22—C23—C24—C19	-0.7 (5)
C10—C11—C12—O1	179.2 (3)	C20—C19—C24—C23	0.4 (5)
C10—C11—C12—C13	-0.4 (6)	C18—C19—C24—C23	179.6 (3)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
O2—H2 $\cdots$ N1 <sup>i</sup>	0.82	1.98	2.762 (4)	159
O1—H1 $\cdots$ N2 <sup>ii</sup>	0.82	2.00	2.780 (4)	159

Symmetry codes: (i) *x*, *y*, *z*+1; (ii) *x*, *y*, *z*-1.

Fig. 1

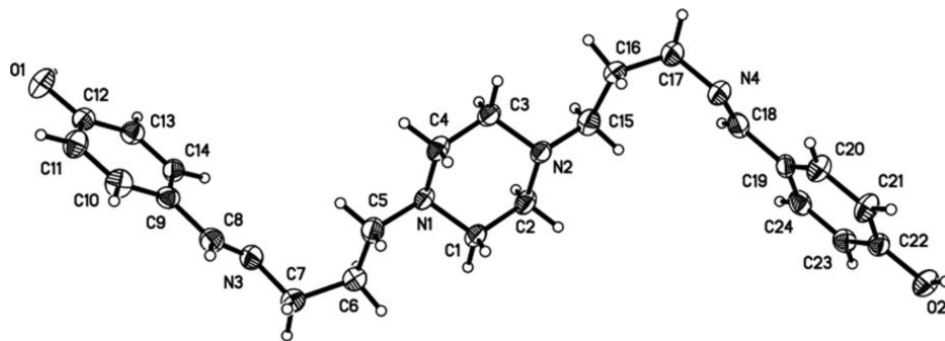


Fig. 2

