

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

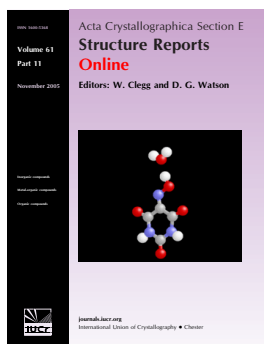
Editors: W.T.A. Harrison, J. Simpson and
M. Weil

N,N'-Bis[(*E*)-quinoxalin-2-ylmethylidene]ethane-1,2-diamine

Digna Varghese, V. Arun, Manju Sebastian, P. Leeju, G. Varsha and K. K. M. Yusuff

Acta Cryst. (2009). E65, o435

This open-access article is distributed under the terms of the Creative Commons Attribution Licence <http://creativecommons.org/licenses/by/2.0/uk/legalcode>, which permits unrestricted use, distribution, and reproduction in any medium, provided the original authors and source are cited.



Acta Crystallographica Section E: Structure Reports Online is the IUCr's highly popular open-access structural journal. It provides a simple and easily accessible publication mechanism for the growing number of inorganic, metal-organic and organic crystal structure determinations. The electronic submission, validation, refereeing and publication facilities of the journal ensure very rapid and high-quality publication, whilst key indicators and validation reports provide measures of structural reliability. In 2007, the journal published over 5000 structures. The average publication time is less than one month.

Crystallography Journals **Online** is available from journals.iucr.org

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

***N,N'*-Bis[(*E*)-quinoxalin-2-ylmethylidene]ethane-1,2-diamine**

Digna Varghese, V. Arun, Manju Sebastian, P. Leeju, G. Varsha and K. K. M. Yusuff*

Department of Applied Chemistry, Cochin University of Science and Technology, Cochin 682 022, Kerala, India

Correspondence e-mail: yusuff@cusat.ac.in

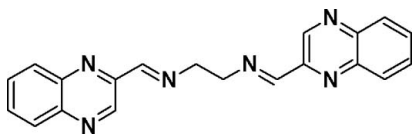
Received 16 December 2008; accepted 23 January 2009

Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.071; wR factor = 0.164; data-to-parameter ratio = 12.4.

In the molecule of the title compound, $\text{C}_{20}\text{H}_{16}\text{N}_6$, the central C—C bond lies on a crystallographic inversion centre. The quinoxalidine ring is nearly planar, with a maximum deviation of 0.021 (2) Å from the mean plane. The crystal structure is stabilized by intermolecular C—H...N interactions, leading to the formation of a layer-like structure, which extends along the a axis.

Related literature

For the synthesis of the Schiff base, see: Zolezzi *et al.* (1999). For the properties of Schiff base ligands, see: Gupta & Sutar (2008); Harmenberg *et al.* (1991); Mayadevi *et al.* (2003); Miller *et al.* (1999); Naylor *et al.* (1993); Sreekala & Yusuff (1994); Xavier *et al.* (2004); Yusuff & Sreekala (1991). For related structures, see: Habibi *et al.* (2006); Taylor & Kennard (1982).



Experimental

Crystal data

$\text{C}_{20}\text{H}_{16}\text{N}_6$
 $M_r = 340.39$
 Triclinic, $P\bar{1}$
 $a = 6.888$ (2) Å
 $b = 7.381$ (3) Å
 $c = 9.638$ (4) Å
 $\alpha = 101.674$ (6)°
 $\beta = 96.233$ (6)°

$\gamma = 116.046$ (5)°
 $V = 420.1$ (3) Å³
 $Z = 1$
 Mo $K\alpha$ radiation
 $\mu = 0.09$ mm⁻¹
 $T = 298$ (2) K
 $0.40 \times 0.24 \times 0.18$ mm

Data collection

Bruker SMART CCD area-detector diffractometer

Absorption correction: multi-scan (SADABS; Sheldrick, 2001)
 $T_{\min} = 0.967$, $T_{\max} = 0.995$

3956 measured reflections
 1465 independent reflections

1239 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.025$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.071$
 $wR(F^2) = 0.164$
 $S = 1.27$
 1465 reflections

118 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.13$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.21$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C1}-\text{H1}\cdots\text{N2}^{\text{i}}$	0.93	2.73	3.647 (4)	168
$\text{C9}-\text{H9}\cdots\text{N1}^{\text{ii}}$	0.93	2.67	3.593 (3)	169

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINTE* (Bruker, 2000); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008) and/or *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

The authors thank Professor M. V. Rajasekharan, School of Chemistry, University of Hyderabad, for kind help and useful discussions. The X-ray data were collected on the diffractometer facilities at the University of Hyderabad provided by the Department of Science and Technology. DV gratefully acknowledges financial support from the Council of Scientific and Industrial Research (CSIR), India. MS thanks KSCSTE, Trivandrum, Kerala, for financial assistance.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FJ2185).

References

- Bruker (2000). *SMART* and *SAINTE*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Gupta, K. C. & Sutar, A. K. (2008). *Coord. Chem. Rev.* **252**, 1420–1450.
- Habibi, M. H., Montazerzohori, M., Lalegani, A., Harrington, R. W. & Clegg, W. (2006). *J. Fluorine Chem.* **127**, 769–773.
- Harmenberg, J., Akesson-Johansson, A., Graslund, A., Malmfors, T., Bergman, J., Wahren, B., Akerfeldt, S., Lundblad, L. & Cox, S. (1991). *Antiviral Res.* **15**, 193–204.
- Mayadevi, S., Prasad, P. G. & Yusuff, K. K. M. (2003). *Synth. React. Inorg. Met. Org. Chem.* **33**, 481–496.
- Miller, J. K., Baag, J. H. & Abu-Omar, M. M. (1999). *Inorg. Chem.* **38**, 4510–4514.
- Naylor, M. A., Stephen, M. A., Nolan, J., Sutton, B., Tocher, J. H., Fielden, E. M., Adams, G. E. & Strafford, I. J. (1993). *Anticancer Drug. Des.* **8**, 439–461.
- Sheldrick, G. M. (2001). *SADABS*. University of Göttingen, Germany.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
- Sreekala, R. & Yusuff, K. K. M. (1994). *Synth. React. Inorg. Met. Org. Chem.* **24**, 1773–1788.
- Taylor, R. & Kennard, O. (1982). *J. Am. Chem. Soc.* **104**, 5063–5070.
- Xavier, K. O., Chacko, J. & Yusuff, K. K. M. (2004). *Appl. Catal. A Gen.* **258**, 251–259.
- Yusuff, K. K. M. & Sreekala, R. (1991). *Synth. React. Inorg. Met. Org. Chem.* **21**, 553–568.
- Zolezzi, S., Decinti, A. & Spodine, E. (1999). *Polyhedron*, **18**, 897–904.

supplementary materials

Acta Cryst. (2009). E65, o435 [doi:10.1107/S1600536809003006]

N,N'-Bis[(*E*)-quinoxalin-2-ylmethylidene]ethane-1,2-diamine

D. Varghese, V. Arun, M. Sebastian, P. Leeju, G. Varsha and K. K. M. Yusuff

Comment

Schiff bases derived from aldehydes and diamines constitute one of the most relevant synthetic ligand systems. They find application in a broad range of transition metal catalyzed reactions including lactide polymerization, epoxidation of olefins, hydroxylation and asymmetric ring opening of epoxides (Gupta & Sutar, 2008). Many drug candidates bearing quinoxaline core structures are in clinical trials in antiviral (Harmenberg *et al.*, 1991), anticancer and central nervous system therapeutic areas (Naylor *et al.*, 1993). Catalytic and antibacterial activities have been observed for the Schiff base complexes derived from Quinoxaline-2-carboxaldehyde (Yusuff & Sreekala, 1991; Sreekala & Yusuff, 1994; Mayadevi *et al.*, 2003). Ethylenediamine groups appear to be of importance for various transition metal catalysis (Miller *et al.*, 1999; Xavier *et al.*, 2004). We have recently prepared the title compound (1), and report here its structure.

The single-crystal X-ray structure determination of (1) was carried out at 298 (2) K. The structure analysis showed that the compound to form in triclinic space group P-1 with $a = 6.888$ (2) Å, $b = 7.381$ (3) Å, $c = 9.638$ (4) Å and $\alpha = 101.674$ (6)°, $\beta = 96.233$ (6)°, $\gamma = 116.046$ (5)° with $z = 1$. A perspective drawing is depicted in figure 1 with the atomic numbering scheme. The C10—N3—C9, N3—C9—C8 angles are 117.9 (2)° and 121.5 (2)° respectively. The N3—C10 and N3—C9 bond lengths are 1.455 (3) Å and 1.260 (3) Å respectively. In this compound (1), the short (C—)H···N contacts are responsible for the stability of layer structure (figure 3) which extends along the a axis (Taylor & Kennard, 1982). The (C—)H···N distances and C—H—N angles are given in table 1.

Experimental

A hot solution of ethylenediamine (1 mmol) in methanol (25 ml) was slowly added over a hot solution of quinoxaline-2-carboxaldehyde (2 mmol) in the same solvent (50 ml). The resulting mixture on cooling yielded the crude product. The precipitated diimine was filtered off and washed with cold methanol. Light yellow single crystals of (1) were obtained from a solution of dichloromethane by slow evaporation.

Refinement

H atoms were positioned geometrically with, C—H = 0.93 Å and refined in riding mode with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Figures

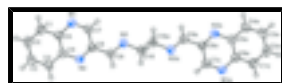


Fig. 1. An ORTEP-3 (Farrugia, 1997) plot of the (I) compound, with the atomic labelling scheme. The shapes of the ellipsoids correspond to 50% probability contours of atomic displacement.

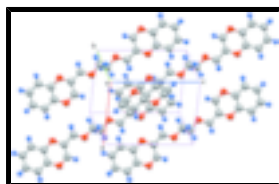


Fig. 2. Unit cell packing diagram of *N,N*-bis[(*E*)-quinoxalin-2-ylmethylidene]ethane-1,2-diamine.

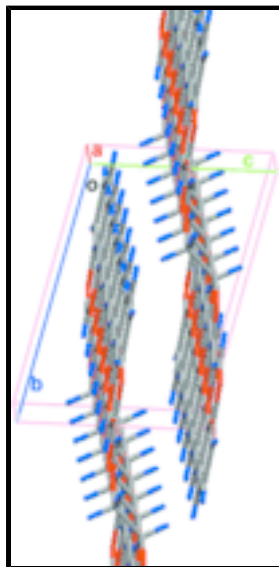


Fig. 3. Pairs of (C–)H⋯N interactions lead to a layer structure along the *a* axis.

N,N'-Bis[(*E*)-quinoxalin-2-ylmethylidene]ethane-1,2-diamine

Crystal data

$C_{20}H_{16}N_6$

$M_r = 340.39$

Triclinic, $P\bar{1}$

Hall symbol: $-P\ 1$

$a = 6.888\ (2)\ \text{\AA}$

$b = 7.381\ (3)\ \text{\AA}$

$c = 9.638\ (4)\ \text{\AA}$

$\alpha = 101.674\ (6)^\circ$

$\beta = 96.233\ (6)^\circ$

$\gamma = 116.046\ (5)^\circ$

$V = 420.1\ (3)\ \text{\AA}^3$

$Z = 1$

$F_{000} = 178$

$D_x = 1.345\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71073\ \text{\AA}$

Cell parameters from 1465 reflections

$\theta = 2.3\text{--}25.0^\circ$

$\mu = 0.09\ \text{mm}^{-1}$

$T = 298\ (2)\ \text{K}$

Plate, yellow

$0.40 \times 0.24 \times 0.18\ \text{mm}$

Data collection

Bruker SMART CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 298\ (2)\ \text{K}$

φ and ω scans

1465 independent reflections

1239 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.025$

$\theta_{\text{max}} = 25.0^\circ$

$\theta_{\text{min}} = 2.2^\circ$

Absorption correction: multi-scan
(SADABS; Sheldrick, 2001) $h = -8 \rightarrow 8$
 $T_{\min} = 0.967, T_{\max} = 0.995$ $k = -8 \rightarrow 8$
 3956 measured reflections $l = -11 \rightarrow 11$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.071$	H-atom parameters constrained
$wR(F^2) = 0.164$	$w = 1/[\sigma^2(F_o^2) + (0.0542P)^2 + 0.1572P]$
$S = 1.27$	where $P = (F_o^2 + 2F_c^2)/3$
1465 reflections	$(\Delta/\sigma)_{\max} < 0.001$
118 parameters	$\Delta\rho_{\max} = 0.13 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\min} = -0.21 \text{ e } \text{\AA}^{-3}$
	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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	0.4604 (3)	0.2446 (3)	0.5782 (2)	0.0439 (6)
N2	0.0673 (3)	0.2571 (3)	0.4735 (2)	0.0405 (6)
N3	0.0679 (3)	0.3587 (3)	0.8476 (2)	0.0425 (6)
C1	0.5203 (4)	0.1943 (4)	0.3355 (3)	0.0482 (7)
H1	0.6488	0.1881	0.3682	0.058*
C2	0.4561 (5)	0.1772 (4)	0.1921 (3)	0.0518 (8)
H2	0.5425	0.1608	0.1280	0.062*
C3	0.2624 (5)	0.1841 (4)	0.1406 (3)	0.0522 (8)
H3	0.2216	0.1735	0.0429	0.063*
C4	0.1338 (4)	0.2062 (4)	0.2329 (3)	0.0473 (7)
H4	0.0032	0.2070	0.1975	0.057*
C5	0.1963 (4)	0.2279 (4)	0.3820 (3)	0.0374 (6)
C6	0.3918 (4)	0.2210 (4)	0.4334 (3)	0.0381 (6)
C7	0.3349 (4)	0.2733 (4)	0.6617 (3)	0.0429 (7)

supplementary materials

H7	0.3764	0.2897	0.7604	0.051*
C8	0.1382 (4)	0.2813 (4)	0.6116 (3)	0.0371 (6)
C9	0.0064 (4)	0.3217 (4)	0.7124 (3)	0.0419 (6)
H9	-0.1251	0.3198	0.6754	0.050*
C10	-0.0707 (4)	0.3998 (4)	0.9395 (3)	0.0438 (7)
H10A	-0.1415	0.2835	0.9804	0.053*
H10B	-0.1857	0.4133	0.8820	0.053*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0419 (12)	0.0551 (14)	0.0405 (12)	0.0272 (11)	0.0095 (10)	0.0150 (10)
N2	0.0409 (12)	0.0465 (13)	0.0350 (12)	0.0231 (11)	0.0064 (9)	0.0084 (10)
N3	0.0463 (13)	0.0504 (13)	0.0342 (12)	0.0257 (11)	0.0108 (9)	0.0113 (10)
C1	0.0440 (15)	0.0514 (17)	0.0495 (16)	0.0233 (14)	0.0136 (13)	0.0113 (13)
C2	0.0565 (18)	0.0512 (17)	0.0433 (16)	0.0210 (14)	0.0221 (13)	0.0093 (13)
C3	0.0596 (18)	0.0568 (18)	0.0332 (14)	0.0228 (15)	0.0095 (13)	0.0101 (13)
C4	0.0497 (16)	0.0554 (18)	0.0345 (14)	0.0243 (14)	0.0039 (12)	0.0122 (12)
C5	0.0375 (14)	0.0327 (13)	0.0385 (14)	0.0138 (11)	0.0077 (11)	0.0098 (11)
C6	0.0380 (13)	0.0377 (14)	0.0372 (13)	0.0173 (12)	0.0074 (11)	0.0096 (11)
C7	0.0448 (15)	0.0524 (16)	0.0313 (13)	0.0233 (13)	0.0052 (11)	0.0127 (12)
C8	0.0368 (13)	0.0367 (14)	0.0357 (14)	0.0173 (11)	0.0061 (11)	0.0071 (11)
C9	0.0414 (15)	0.0457 (16)	0.0406 (15)	0.0229 (13)	0.0076 (12)	0.0113 (12)
C10	0.0443 (15)	0.0528 (17)	0.0368 (14)	0.0235 (13)	0.0139 (11)	0.0139 (12)

Geometric parameters (\AA , $^\circ$)

N1—C7	1.298 (3)	C3—H3	0.9300
N1—C6	1.373 (3)	C4—C5	1.410 (3)
N2—C8	1.315 (3)	C4—H4	0.9300
N2—C5	1.369 (3)	C5—C6	1.409 (3)
N3—C9	1.260 (3)	C7—C8	1.418 (3)
N3—C10	1.455 (3)	C7—H7	0.9300
C1—C2	1.367 (4)	C8—C9	1.472 (3)
C1—C6	1.404 (3)	C9—H9	0.9300
C1—H1	0.9300	C10—C10 ⁱ	1.512 (5)
C2—C3	1.398 (4)	C10—H10A	0.9700
C2—H2	0.9300	C10—H10B	0.9700
C3—C4	1.357 (4)		
C7—N1—C6	115.8 (2)	N1—C6—C1	119.7 (2)
C8—N2—C5	116.2 (2)	N1—C6—C5	120.9 (2)
C9—N3—C10	117.9 (2)	C1—C6—C5	119.4 (2)
C2—C1—C6	119.9 (3)	N1—C7—C8	124.0 (2)
C2—C1—H1	120.1	N1—C7—H7	118.0
C6—C1—H1	120.1	C8—C7—H7	118.0
C1—C2—C3	121.0 (3)	N2—C8—C7	121.6 (2)
C1—C2—H2	119.5	N2—C8—C9	116.9 (2)
C3—C2—H2	119.5	C7—C8—C9	121.5 (2)

C4—C3—C2	120.1 (3)	N3—C9—C8	121.5 (2)
C4—C3—H3	120.0	N3—C9—H9	119.3
C2—C3—H3	120.0	C8—C9—H9	119.3
C3—C4—C5	120.7 (3)	N3—C10—C10	109.5 (3)
C3—C4—H4	119.7	N3—C10—H10A	109.8
C5—C4—H4	119.7	C10—C10—H10A	109.8
N2—C5—C6	121.6 (2)	N3—C10—H10B	109.8
N2—C5—C4	119.4 (2)	C10—C10—H10B	109.8
C6—C5—C4	119.0 (2)	H10A—C10—H10B	108.2

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

Hydrogen-bond geometry ($\text{\AA}, ^\circ$)

<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>
C1—H1 \cdots N2 ⁱⁱ	0.93	2.73	3.647 (4)	168
C9—H9 \cdots N1 ⁱⁱⁱ	0.93	2.67	3.593 (3)	169

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

Fig. 1

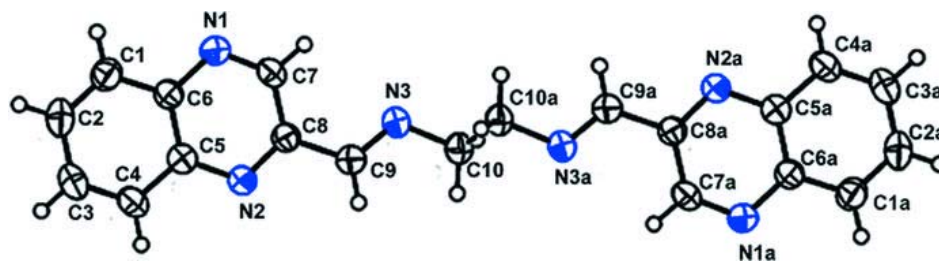


Fig. 2

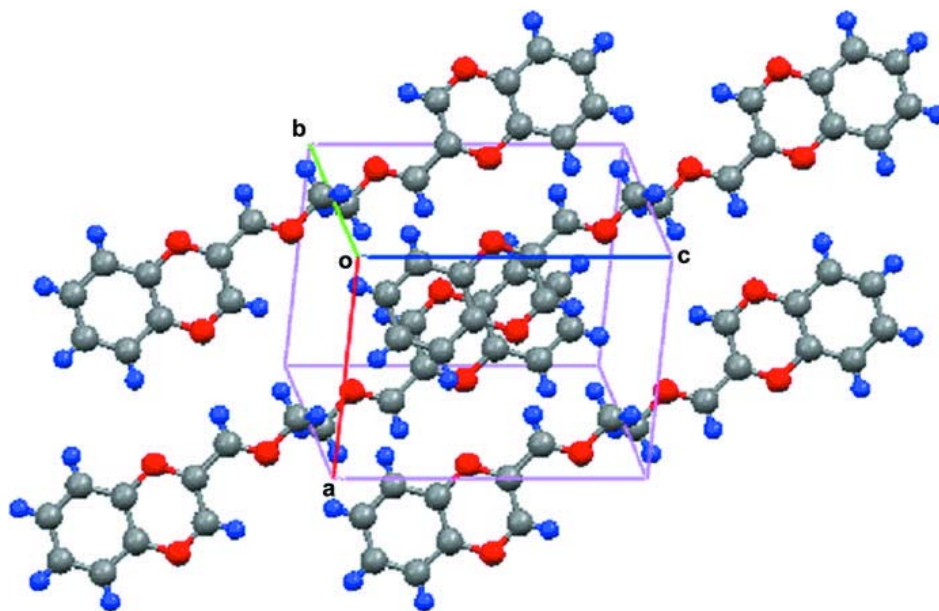


Fig. 3

