

Acta Crystallographica Section E

## Structure Reports

Online

ISSN 1600-5368

11,12-Dichlorodibenzo[*a,c*]phenazine

Arta Imeri, Neil M. Glagovich and Guy Crundwell\*

Department of Chemistry, Central Connecticut State University, New Britain, CT 06053, USA

Correspondence e-mail: crundwellg@mail.ccsu.edu

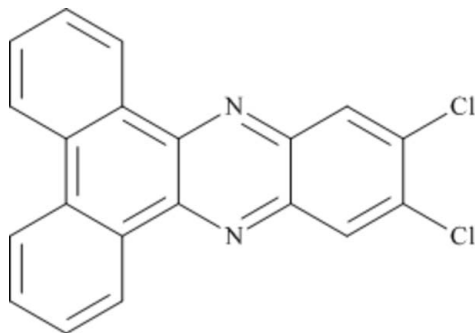
Received 1 November 2012; accepted 4 December 2012

Key indicators: single-crystal X-ray study;  $T = 293$  K; mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å;  $R$  factor = 0.039;  $wR$  factor = 0.104; data-to-parameter ratio = 23.1.

The title compound,  $\text{C}_{20}\text{H}_{10}\text{Cl}_2\text{N}_2$ , has crystallographic twofold rotational symmetry [maximum deviation from the least-squares plane = 0.038 (1) Å]. In the crystal, weak  $\pi$ - $\pi$  ring stacking interactions occur down the  $a$ -axis direction [minimum centroid-centroid separation = 3.7163 (8) Å].

## Related literature

For the synthesis of the title compound, see: Bellizzi *et al.* (2006). For the structures of similar compounds, see: Bellizzi *et al.* (2006); Day *et al.* (2002); Richards *et al.* (2009).



## Experimental

## Crystal data

$\text{C}_{20}\text{H}_{10}\text{Cl}_2\text{N}_2$   
 $M_r = 349.20$   
 Monoclinic,  $C2/c$   
 $a = 3.8583$  (3) Å  
 $b = 26.2739$  (13) Å  
 $c = 15.1147$  (10) Å  
 $\beta = 94.877$  (6)°

$V = 1526.67$  (17) Å<sup>3</sup>  
 $Z = 4$   
 Mo  $K\alpha$  radiation  
 $\mu = 0.43$  mm<sup>-1</sup>  
 $T = 293$  K  
 $0.32 \times 0.14 \times 0.09$  mm

## Data collection

Oxford Diffraction Xcalibur  
 Sapphire3 CCD diffractometer  
 Absorption correction: multi-scan  
 (*CrysAlis PRO*; Oxford Diffraction, 2009)  
 $T_{\min} = 0.915$ ,  $T_{\max} = 1.000$

5082 measured reflections  
 2520 independent reflections  
 1372 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.020$

## Refinement

$R[F^2 > 2\sigma(F^2)] = 0.039$   
 $wR(F^2) = 0.104$   
 $S = 0.83$   
 2520 reflections

109 parameters  
 H-atom parameters constrained  
 $\Delta\rho_{\max} = 0.26$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.25$  e Å<sup>-3</sup>

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2009); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2009); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

This research was funded by a CCSU-AAUP research grant.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZS2242).

## References

- Bellizzi, M., Crundwell, G., Zeller, M., Hunter, A. D. & McBurney, B. (2006). *Acta Cryst.* **E62**, o5249–o5251.  
 Day, M. W., Amashukeli, X. A. & Gray, H. B. (2002). Private communication (refcode MIVRUE). CCDC, Cambridge, England.  
 Oxford Diffraction (2009). *CrysAlis CCD*, *CrysAlis PRO* and *CrysAlis RED*. Oxford Diffraction Ltd, Yarnton, England.  
 Richards, G. J., Hill, J. P., Okamoto, K., Shundo, A., Akada, M., Elsegood, M. R. J., Mori, T. & Ariga, K. (2009). *Langmuir Lett.* **25**, 8408–8413.  
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.  
 Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.

## supporting information

*Acta Cryst.* (2013). E69, o48 [https://doi.org/10.1107/S1600536812049690]

## 11,12-Dichlorodibenzo[a,c]phenazine

Arta Imeri, Neil M. Glagovich and Guy Crundwell

### S1. Comment

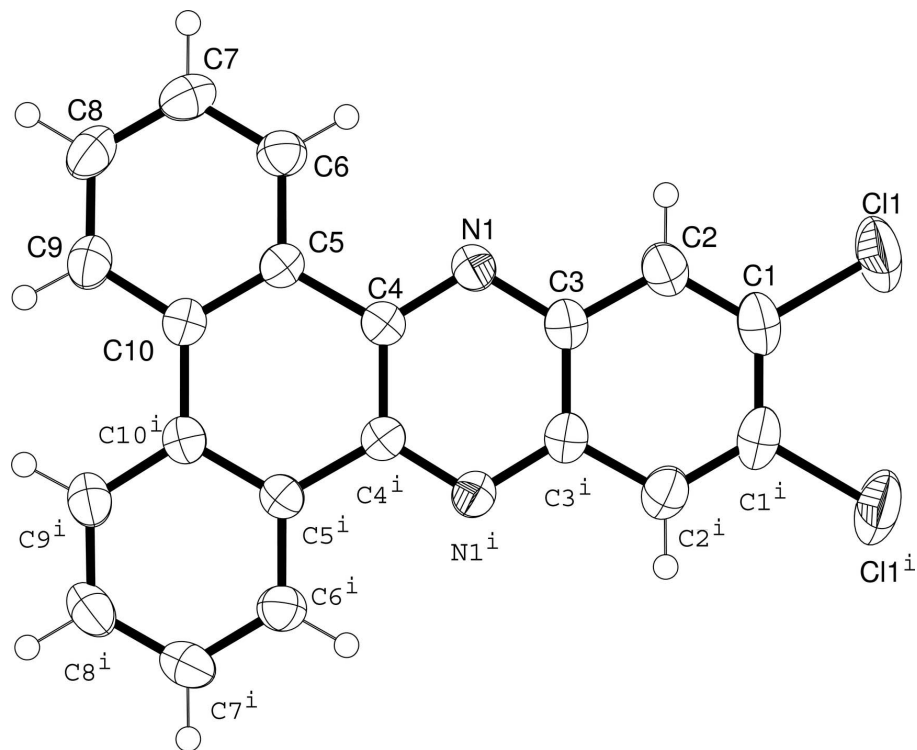
The title compound  $C_{20}H_{10}Cl_2N_2$  is planar [maximum deviation from the l.s. plane over the 24 atoms = 0.038 (1) Å] and lies on a crystallographic twofold axis (Fig. 1). All bond lengths and angles fall within typical ranges found in other dibenzo[a,c]phenazines (Day *et al.*, 2002; Richards *et al.*, 2009). In the crystal, the molecules are involved in weak  $\pi$ - $\pi$ -stacking interactions [minimum ring centroid separation = 3.7163 (8) Å], giving stacks down the *a* axial direction of the unit cell.

### S2. Experimental

To a 25 ml round bottom flask equipped with a reflux condenser was added 0.27 g (1.5 mmol) of 4,5-dichloro-1,2-phenylenediamine, 0.27 g (1.3 mmol) of phenanthraquinone, and 10 ml of glacial acetic acid (Bellizzi *et al.*, 2006) and the mixture was heated with refluxing for 6 h. After this time, the resulting yellow solution was concentrated under reduced pressure, and the product obtained was purified by recrystallization from methanol, producing 0.48 g of the title compound as a yellow solid (m.p. 273 °C; yield: 92%). *R*<sub>f</sub> 0.31 (SiO<sub>2</sub>, 80% hexanes-ethyl acetate); <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ 9.35 (dd, 2H, *J* = 8.0 Hz, *J* = 1.5 Hz), 8.58 (d, 2H, *J* = 8.0 Hz, *J* = 1.2 Hz), 8.47 (s, 2H), 7.86 (dt, 2H, *J* = 7.2 Hz, *J* = 1.5 Hz), 7.774 (dt, 2H, *J* = 7.2 Hz, *J* = 1.2 Hz); <sup>13</sup>C NMR (300 MHz, CDCl<sub>3</sub>) δ 143.30, 140.76, 134.30, 132.32, 130.92, 129.85, 129.79, 128.16, 126.45, 123.03; UV/Vis (CH<sub>2</sub>Cl<sub>2</sub>; λ<sub>max</sub>) 260 nm.

### S3. Refinement

Hydrogen atoms were included in calculated positions with a C—H distance of 0.93 Å in the refinement in a riding motion approximation, with  $U_{iso} = 1.2U_{eq}$  of the carrier atom.



**Figure 1**

A view of the title compound showing atom numbering, with displacement ellipsoids drawn at the 50% probability level. Symmetry code (i)  $(-x, y, -z + 1/2)$ , indicates atoms related by twofold rotational symmetry.

### 11,12-Dichlorodibenzo[a,c]phenazine

#### Crystal data

$C_{20}H_{10}Cl_2N_2$   
 $M_r = 349.20$   
 Monoclinic,  $C2/c$   
 Hall symbol:  $-C 2yc$   
 $a = 3.8583 (3) \text{ \AA}$   
 $b = 26.2739 (13) \text{ \AA}$   
 $c = 15.1147 (10) \text{ \AA}$   
 $\beta = 94.877 (6)^\circ$   
 $V = 1526.67 (17) \text{ \AA}^3$   
 $Z = 4$

$F(000) = 712$   
 $D_x = 1.519 \text{ Mg m}^{-3}$   
 Melting point: 546 K  
 Mo  $K\alpha$  radiation,  $\lambda = 0.71073 \text{ \AA}$   
 Cell parameters from 1714 reflections  
 $\theta = 4.1\text{--}32.5^\circ$   
 $\mu = 0.43 \text{ mm}^{-1}$   
 $T = 293 \text{ K}$   
 Needle, yellow  
 $0.32 \times 0.14 \times 0.09 \text{ mm}$

#### Data collection

Oxford Diffraction Xcalibur Sapphire3 CCD  
 diffractometer  
 Radiation source: Enhance (Mo) X-ray Source  
 Graphite monochromator  
 Detector resolution:  $16.1790 \text{ pixels mm}^{-1}$   
 $\omega$  scans  
 Absorption correction: multi-scan  
 (*CrysAlis PRO*; Oxford Diffraction, 2009)  
 $T_{\min} = 0.915$ ,  $T_{\max} = 1.000$

5082 measured reflections  
 2520 independent reflections  
 1372 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.020$   
 $\theta_{\text{max}} = 32.6^\circ$ ,  $\theta_{\text{min}} = 4.1^\circ$   
 $h = -5 \rightarrow 5$   
 $k = -27 \rightarrow 38$   
 $l = -21 \rightarrow 10$

Refinement

Refinement on $F^2$	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.039$	H-atom parameters constrained
$wR(F^2) = 0.104$	$w = 1/[\sigma^2(F_o^2) + (0.0575P)^2]$
$S = 0.83$	where $P = (F_o^2 + 2F_c^2)/3$
2520 reflections	$(\Delta/\sigma)_{\max} = 0.001$
109 parameters	$\Delta\rho_{\max} = 0.26 \text{ e } \text{\AA}^{-3}$
0 restraints	$\Delta\rho_{\min} = -0.25 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	

Special details

**Experimental.** Hydrogen atoms were included in calculated positions with a C—H distance of 0.93 Å and were included in the refinement in riding motion approximation with  $U_{\text{iso}} = 1.2U_{\text{eq}}$  of the carrier atom.

**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 ( $\text{\AA}^2$ )

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
C11	0.13563 (12)	1.243825 (14)	0.15408 (3)	0.07256 (18)
C1	0.0618 (3)	1.18700 (5)	0.20681 (10)	0.0463 (3)
C2	0.1244 (3)	1.14234 (5)	0.16590 (9)	0.0430 (3)
H2	0.2072	1.1427	0.1099	0.052*
C3	0.0651 (3)	1.09527 (4)	0.20751 (8)	0.0355 (3)
N1	0.1324 (3)	1.05122 (4)	0.16541 (7)	0.0356 (2)
C4	0.0669 (3)	1.00812 (4)	0.20704 (8)	0.0308 (3)
C5	0.1338 (3)	0.95984 (4)	0.16356 (8)	0.0306 (3)
C6	0.2605 (3)	0.95979 (5)	0.07952 (8)	0.0390 (3)
H6	0.3017	0.9906	0.0519	0.047*
C7	0.3246 (4)	0.91492 (5)	0.03731 (9)	0.0447 (3)
H7	0.4078	0.9152	-0.0187	0.054*
C8	0.2642 (4)	0.86907 (5)	0.07896 (9)	0.0472 (4)
H8	0.3089	0.8386	0.0508	0.057*
C9	0.1394 (3)	0.86825 (5)	0.16127 (9)	0.0421 (3)
H9	0.1010	0.8371	0.1880	0.051*
C10	0.0685 (3)	0.91342 (4)	0.20592 (8)	0.0321 (3)

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C11	0.0829 (3)	0.0336 (2)	0.1023 (4)	-0.00414 (17)	0.0140 (3)	0.0163 (2)
C1	0.0413 (8)	0.0305 (6)	0.0665 (9)	-0.0025 (5)	0.0004 (7)	0.0067 (6)

C2	0.0440 (8)	0.0364 (7)	0.0490 (8)	-0.0003 (6)	0.0053 (6)	0.0062 (6)
C3	0.0333 (7)	0.0311 (6)	0.0417 (6)	-0.0006 (5)	0.0010 (5)	0.0014 (5)
N1	0.0380 (6)	0.0323 (5)	0.0366 (5)	0.0000 (4)	0.0047 (5)	0.0029 (4)
C4	0.0281 (7)	0.0325 (6)	0.0315 (6)	0.0005 (4)	0.0014 (5)	0.0018 (5)
C5	0.0272 (6)	0.0327 (6)	0.0315 (6)	0.0025 (5)	0.0006 (5)	-0.0012 (5)
C6	0.0393 (7)	0.0410 (7)	0.0375 (7)	0.0028 (5)	0.0074 (6)	0.0012 (5)
C7	0.0450 (8)	0.0526 (8)	0.0373 (7)	0.0081 (6)	0.0072 (6)	-0.0067 (6)
C8	0.0549 (9)	0.0403 (7)	0.0464 (8)	0.0090 (6)	0.0041 (6)	-0.0115 (6)
C9	0.0476 (8)	0.0317 (6)	0.0467 (8)	0.0028 (5)	0.0018 (6)	-0.0027 (5)
C10	0.0290 (6)	0.0316 (6)	0.0351 (6)	0.0014 (4)	-0.0012 (5)	-0.0011 (5)

*Geometric parameters (Å, °)*

C11—C1	1.7274 (13)	C5—C10	1.4101 (15)
C1—C2	1.3576 (17)	C6—C7	1.3731 (17)
C1—C1 <sup>i</sup>	1.428 (3)	C6—H6	0.9300
C2—C3	1.4147 (16)	C7—C8	1.3879 (18)
C2—H2	0.9300	C7—H7	0.9300
C3—N1	1.3564 (14)	C8—C9	1.3719 (18)
C3—C3 <sup>i</sup>	1.418 (2)	C8—H8	0.9300
N1—C4	1.3301 (14)	C9—C10	1.4037 (15)
C4—C4 <sup>i</sup>	1.438 (2)	C9—H9	0.9300
C4—C5	1.4616 (15)	C10—C10 <sup>i</sup>	1.475 (2)
C5—C6	1.3991 (16)		
C2—C1—C1 <sup>i</sup>	120.17 (8)	C7—C6—C5	120.89 (12)
C2—C1—C11	119.62 (11)	C7—C6—H6	119.6
C1 <sup>i</sup> —C1—C11	120.20 (5)	C5—C6—H6	119.6
C1—C2—C3	120.76 (12)	C6—C7—C8	119.38 (12)
C1—C2—H2	119.6	C6—C7—H7	120.3
C3—C2—H2	119.6	C8—C7—H7	120.3
N1—C3—C2	119.52 (11)	C9—C8—C7	120.69 (12)
N1—C3—C3 <sup>i</sup>	121.43 (6)	C9—C8—H8	119.7
C2—C3—C3 <sup>i</sup>	119.05 (7)	C7—C8—H8	119.7
C4—N1—C3	116.93 (10)	C8—C9—C10	121.36 (12)
N1—C4—C4 <sup>i</sup>	121.64 (6)	C8—C9—H9	119.3
N1—C4—C5	118.57 (10)	C10—C9—H9	119.3
C4 <sup>i</sup> —C4—C5	119.79 (6)	C9—C10—C5	117.61 (11)
C6—C5—C10	120.06 (11)	C9—C10—C10 <sup>i</sup>	122.27 (7)
C6—C5—C4	119.85 (10)	C5—C10—C10 <sup>i</sup>	120.12 (6)
C10—C5—C4	120.08 (10)		
C3—N1—C4—C4 <sup>i</sup>	-0.40 (17)	N1—C4—C4 <sup>i</sup> —N1 <sup>i</sup>	0.24 (19)
C4—N1—C3—C2	-179.01 (11)	N1—C4—C4 <sup>i</sup> —C5 <sup>i</sup>	-179.76 (11)
C4—N1—C3—C3 <sup>i</sup>	0.75 (17)	C5—C4—C4 <sup>i</sup> —N1 <sup>i</sup>	-179.76 (11)
C3—N1—C4—C5	179.59 (11)	C5—C4—C4 <sup>i</sup> —C5 <sup>i</sup>	0.25 (17)
C11—C1—C1 <sup>i</sup> —C2 <sup>i</sup>	179.08 (10)	C4—C5—C6—C7	-179.86 (12)
C2—C1—C1 <sup>i</sup> —C2 <sup>i</sup>	-1.40 (19)	C10—C5—C6—C7	-0.32 (18)

C11—C1—C1 <sup>i</sup> —C11 <sup>i</sup>	-0.44 (15)	C4—C5—C10—C9	-179.65 (11)
C2—C1—C1 <sup>i</sup> —C11 <sup>i</sup>	179.08 (10)	C4—C5—C10—C10 <sup>i</sup>	0.19 (17)
C1 <sup>i</sup> —C1—C2—C3	0.67 (18)	C6—C5—C10—C9	0.80 (17)
C11—C1—C2—C3	-179.81 (10)	C6—C5—C10—C10 <sup>i</sup>	-179.36 (11)
C1—C2—C3—C3 <sup>i</sup>	0.74 (18)	C5—C6—C7—C8	-0.3 (2)
C1—C2—C3—N1	-179.50 (12)	C6—C7—C8—C9	0.4 (2)
N1—C3—C3 <sup>i</sup> —N1 <sup>i</sup>	-0.96 (18)	C7—C8—C9—C10	0.1 (2)
C2—C3—C3 <sup>i</sup> —C2 <sup>i</sup>	-1.44 (17)	C8—C9—C10—C5	-0.69 (18)
N1—C3—C3 <sup>i</sup> —C2 <sup>i</sup>	178.80 (11)	C8—C9—C10—C10 <sup>i</sup>	179.48 (12)
C2—C3—C3 <sup>i</sup> —N1 <sup>i</sup>	178.80 (11)	C5—C10—C10 <sup>i</sup> —C5 <sup>i</sup>	-0.17 (17)
N1—C4—C5—C6	-0.68 (17)	C5—C10—C10 <sup>i</sup> —C9 <sup>i</sup>	179.66 (11)
N1—C4—C5—C10	179.77 (11)	C9—C10—C10 <sup>i</sup> —C5 <sup>i</sup>	179.66 (11)
C4 <sup>i</sup> —C4—C5—C6	179.32 (11)	C9—C10—C10 <sup>i</sup> —C9 <sup>i</sup>	-0.51 (18)
C4 <sup>i</sup> —C4—C5—C10	-0.24 (17)		

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