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## Structure Reports

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# *N,N'*-Bis(2,5-dichlorophenyl)-isophthalamide

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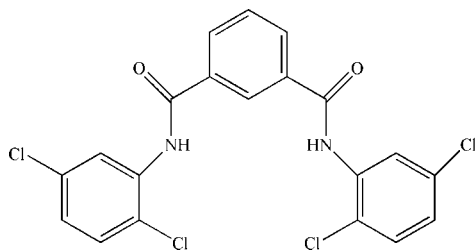
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 Key indicators: single-crystal X-ray study;  $T = 298$  K; mean  $\sigma(\text{C}-\text{C}) = 0.004$  Å;  $R$  factor = 0.042;  $wR$  factor = 0.110; data-to-parameter ratio = 13.2.

The asymmetric unit of the title compound,  $\text{C}_{20}\text{H}_{12}\text{Cl}_4\text{N}_2\text{O}_2$ , contains one half-molecule with a center of symmetry along a  $\text{C}\cdots\text{C}$  axis of the central benzene ring. The two  $\text{C}=\text{O}$  groups adopt an *anti* orientation and the two amide groups are twisted away from the central benzene ring by  $27.38$  (3) and  $27.62$  (4)°. The mean planes of the dichloro-substituted benzene rings are twisted by  $7.95$  (4)° with respect to the benzene ring. The crystal packing is stabilized by weak intermolecular  $\text{N}-\text{H}\cdots\text{O}$  interactions.

## Related literature

For the design of artificial receptors related to isophthalamide, see: Gale (2006). For related structures, see: Light *et al.* (2006); Kavallieratos *et al.* (1997, 1999). For standard bond lengths, see: Allen *et al.* (1987).



## Experimental

### Crystal data

 $\text{C}_{20}\text{H}_{12}\text{Cl}_4\text{N}_2\text{O}_2$ 
 $M_r = 454.12$ 

 Monoclinic,  $P2_1/c$   
 $a = 11.3661$  (11) Å  
 $b = 10.0239$  (9) Å  
 $c = 8.9470$  (7) Å  
 $\beta = 109.988$  (1)°  
 $V = 957.95$  (15) Å<sup>3</sup>
 $Z = 2$   
 Mo  $K\alpha$  radiation  
 $\mu = 0.64$  mm<sup>-1</sup>  
 $T = 298$  K  
 $0.49 \times 0.20 \times 0.10$  mm

### Data collection

 Bruker SMART CCD area-detector diffractometer  
 Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)  
 $T_{\min} = 0.745$ ,  $T_{\max} = 0.939$ 

 4668 measured reflections  
 1687 independent reflections  
 1152 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.030$ 

### Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.042$   
 $wR(F^2) = 0.110$   
 $S = 1.01$   
 1687 reflections

 128 parameters  
 H-atom parameters constrained  
 $\Delta\rho_{\max} = 0.27$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.30$  e Å<sup>-3</sup>
**Table 1**

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1}\cdots\text{O1}^i$	0.86	2.22	3.046 (3)	160

 Symmetry code: (i)  $x, -y + 1, z - \frac{1}{2}$ .

Data collection: *SMART* (Bruker, 2007); cell refinement: *SAINTE* (Bruker, 2007); 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 *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: JJ2095).

## References

- Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
- Bruker (2007). *SAINTE* and *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Gale, P. A. (2006). *Acc. Chem. Res.* **36**, 465–475.
- Kavallieratos, K., Bertao, C. M. & Grabtee, R. H. (1999). *J. Org. Chem.* **64**, 1675–1683.
- Kavallieratos, K., Gala, S. R., Austin, D. J. & Grabtee, R. H. (1997). *J. Am. Chem. Soc.* **119**, 2325–2326.
- Light, M. E., Gale, P. A. & Navakhun, K. (2006). *Acta Cryst.* **E62**, o1097–o1098.
- Sheldrick, G. M. (2004). *SADABS*. University of Göttingen, Germany.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.

## supporting information

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***N,N'*-Bis(2,5-dichlorophenyl)isophthalamide****Ruitao Zhu, Jinlong Dong, Yu Zuo and Yuehong Ren****S1. Comment**

Anion recognition is an area of growing interest in supramolecular chemistry due to its important role in a wide range of environmental, clinical, chemical, and biological applications. Considerable attention has been focused on the design of artificial receptors that are able to selectively recognize and sense anion species (Gale, 2006). Artificial receptors, containing the isophthalamide core function as effective receptors for halide anions in very simple systems (Kavallieratos *et al.*, 1997, 1999; Light *et al.* 2006). We report here the crystal structure of the title compound, C<sub>20</sub>H<sub>12</sub>Cl<sub>4</sub>N<sub>2</sub>O<sub>2</sub>, (I), related to these types of receptors.

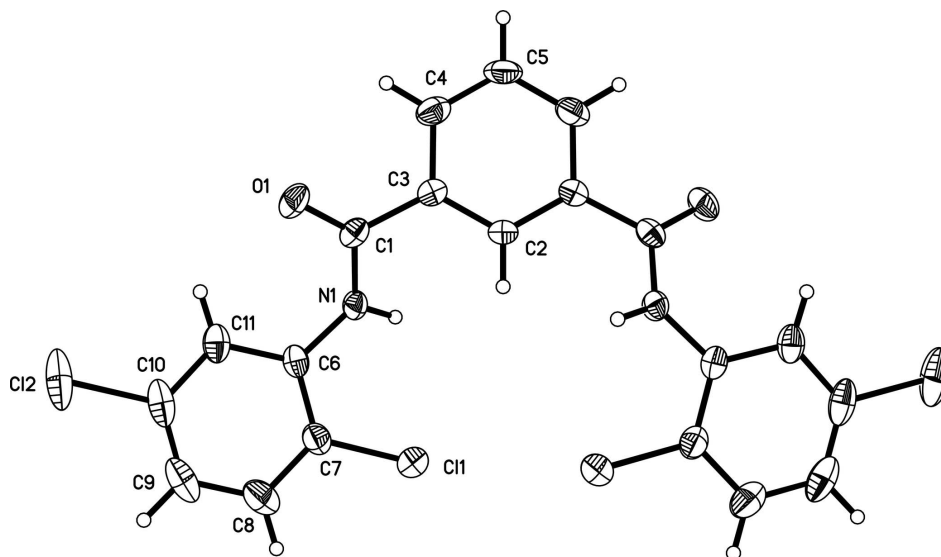
In the title compound, (I), the asymmetric unit containing one-half of the molecule crystallizes with a center of symmetry along the C2—C5 axis in the benzene ring thereby producing the desired structure (Fig. 1). In the molecule the two C=O groups adopt an *anti* orientation and the two amide groups are twisted away from the center benzene ring by 27.38 (3) ° and 27.62 (4) °, respectively. The mean planes of the dichloro substituted benzene rings are twisted by 7.95 (4) ° with that of the benzene ring. Bond lengths are in normal ranges (Allen *et al.*, 1987). Crystal packing is stabilized by weak N—H···O intermolecular interactions (Table 1).

**S2. Experimental**

*N,N*-Bis(2,5-dichlorophenyl)isophthalamide (I) was prepared according to literature procedures (Kavallieratos *et al.*, 1997, 1999). To dichloromethane (20 ml) in a 100 ml flask was added 2,5-dichloroaniline (1.62 g, 10 mmol) with magnetic stirring. Isophthaloyl chloride (1.01 g, 5 mmol) was added gradually, and the reaction mixture was stirred at room temperature for 2 h and then poured into ice water (100 ml). The product was precipitated as a white powder, which was washed three times with water. Recrystallization from dimethyl sulfoxide solution produced the crystals of the title compound.

**S3. Refinement**

H atoms were placed in idealized positions and allowed to ride on their respective parent atoms, with C—H = 0.93–0.96 Å, N—H = 0.86 Å and  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C}, \text{N})$  or  $1.5U_{\text{eq}}(\text{C}_{\text{methyl}})$ .

**Figure 1**

The molecular structure of the title compound with displacement ellipsoids are drawn at the 30% probability level.

### *N,N'*-bis(2,5-dichlorophenyl)benzene-1,3-dicarboxamide

#### Crystal data

$C_{20}H_{12}Cl_4N_2O_2$

$M_r = 454.12$

Monoclinic,  $P2_1/c$

Hall symbol:  $-P\ 2_1/c$

$a = 11.3661$  (11) Å

$b = 10.0239$  (9) Å

$c = 8.9470$  (7) Å

$\beta = 109.988$  (1)°

$V = 957.95$  (15) Å<sup>3</sup>

$Z = 2$

$F(000) = 460$

$D_x = 1.574$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 1505 reflections

$\theta = 2.8$ – $27.4$ °

$\mu = 0.64$  mm<sup>-1</sup>

$T = 298$  K

Prism, colorless

$0.49 \times 0.20 \times 0.10$  mm

#### Data collection

Bruker SMART CCD area-detector  
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

$\varphi$  and  $\omega$  scans

Absorption correction: multi-scan

(*SADABS*; Sheldrick, 2004)

$T_{\min} = 0.745$ ,  $T_{\max} = 0.939$

4668 measured reflections

1687 independent reflections

1152 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.030$

$\theta_{\max} = 25.0$ °,  $\theta_{\min} = 2.8$ °

$h = -13 \rightarrow 6$

$k = -11 \rightarrow 11$

$l = -10 \rightarrow 10$

#### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.042$

$wR(F^2) = 0.110$

$S = 1.01$

1687 reflections

128 parameters

0 restraints

Primary atom site location: structure-invariant  
direct methods

Secondary atom site location: difference Fourier  
map

Hydrogen site location: inferred from  
neighbouring sites

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0416P)^2 + 0.6846P]$$

where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} < 0.001$

$$\Delta\rho_{\max} = 0.27 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.30 \text{ e } \text{\AA}^{-3}$$

*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>	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl1	0.64968 (9)	0.85140 (8)	0.38003 (10)	0.0726 (3)
Cl2	0.99324 (10)	0.70144 (15)	1.06068 (10)	0.1021 (5)
N1	0.7062 (2)	0.5769 (2)	0.4952 (2)	0.0412 (6)
H1	0.6911	0.5986	0.3974	0.049*
O1	0.70204 (19)	0.4014 (2)	0.6539 (2)	0.0544 (6)
C1	0.6692 (2)	0.4532 (3)	0.5224 (3)	0.0392 (7)
C2	0.5000	0.4520 (4)	0.2500	0.0362 (9)
H2	0.5000	0.5448	0.2500	0.043*
C3	0.5811 (2)	0.3842 (3)	0.3794 (3)	0.0372 (6)
C4	0.5787 (3)	0.2456 (3)	0.3786 (4)	0.0537 (8)
H4	0.6306	0.1985	0.4657	0.064*
C5	0.5000	0.1782 (4)	0.2500	0.0638 (13)
H5	0.5000	0.0854	0.2500	0.077*
C6	0.7666 (2)	0.6736 (3)	0.6099 (3)	0.0401 (7)
C7	0.7463 (3)	0.8080 (3)	0.5689 (3)	0.0489 (8)
C8	0.8009 (3)	0.9072 (4)	0.6768 (4)	0.0672 (10)
H8	0.7873	0.9962	0.6468	0.081*
C9	0.8753 (3)	0.8746 (4)	0.8289 (4)	0.0732 (11)
H9	0.9104	0.9410	0.9034	0.088*
C10	0.8969 (3)	0.7431 (4)	0.8689 (4)	0.0640 (10)
C11	0.8451 (3)	0.6423 (3)	0.7628 (3)	0.0513 (8)
H11	0.8624	0.5536	0.7932	0.062*

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Cl1	0.0832 (7)	0.0488 (5)	0.0643 (6)	0.0049 (4)	-0.0025 (5)	0.0043 (4)
Cl2	0.0748 (7)	0.1852 (13)	0.0349 (5)	-0.0308 (7)	0.0039 (4)	-0.0066 (6)
N1	0.0485 (14)	0.0452 (14)	0.0276 (11)	-0.0025 (11)	0.0101 (10)	0.0034 (10)
O1	0.0655 (14)	0.0594 (13)	0.0382 (11)	0.0103 (11)	0.0177 (10)	0.0161 (10)
C1	0.0390 (15)	0.0439 (16)	0.0397 (16)	0.0121 (13)	0.0197 (13)	0.0064 (13)
C2	0.041 (2)	0.0292 (19)	0.042 (2)	0.000	0.0197 (18)	0.000

C3	0.0366 (15)	0.0367 (15)	0.0427 (16)	0.0029 (12)	0.0191 (13)	0.0041 (12)
C4	0.0489 (18)	0.0418 (17)	0.069 (2)	0.0079 (15)	0.0184 (16)	0.0109 (15)
C5	0.069 (3)	0.027 (2)	0.089 (4)	0.000	0.019 (3)	0.000
C6	0.0347 (15)	0.0546 (18)	0.0327 (15)	-0.0042 (13)	0.0139 (12)	-0.0040 (13)
C7	0.0422 (17)	0.0531 (18)	0.0475 (17)	-0.0007 (14)	0.0101 (14)	-0.0103 (14)
C8	0.061 (2)	0.064 (2)	0.075 (2)	-0.0116 (18)	0.020 (2)	-0.0223 (19)
C9	0.056 (2)	0.102 (3)	0.061 (2)	-0.020 (2)	0.0185 (19)	-0.040 (2)
C10	0.0418 (18)	0.115 (3)	0.0355 (17)	-0.016 (2)	0.0131 (14)	-0.0138 (19)
C11	0.0395 (16)	0.077 (2)	0.0369 (16)	-0.0049 (16)	0.0128 (14)	0.0011 (16)

*Geometric parameters (Å, °)*

C11—C7	1.727 (3)	C4—H4	0.9300
C12—C10	1.742 (3)	C5—C4 <sup>i</sup>	1.370 (4)
N1—C1	1.358 (3)	C5—H5	0.9300
N1—C6	1.408 (3)	C6—C11	1.390 (4)
N1—H1	0.8600	C6—C7	1.395 (4)
O1—C1	1.222 (3)	C7—C8	1.376 (4)
C1—C3	1.498 (4)	C8—C9	1.373 (5)
C2—C3 <sup>i</sup>	1.387 (3)	C8—H8	0.9300
C2—C3	1.387 (3)	C9—C10	1.366 (5)
C2—H2	0.9300	C9—H9	0.9300
C3—C4	1.389 (4)	C10—C11	1.374 (5)
C4—C5	1.370 (4)	C11—H11	0.9300
C1—N1—C6	127.0 (2)	C11—C6—C7	118.0 (3)
C1—N1—H1	116.5	C11—C6—N1	123.5 (3)
C6—N1—H1	116.5	C7—C6—N1	118.5 (2)
O1—C1—N1	123.3 (3)	C8—C7—C6	121.2 (3)
O1—C1—C3	121.4 (3)	C8—C7—C11	119.2 (3)
N1—C1—C3	115.2 (2)	C6—C7—C11	119.6 (2)
C3 <sup>i</sup> —C2—C3	121.2 (3)	C9—C8—C7	120.0 (3)
C3 <sup>i</sup> —C2—H2	119.4	C9—C8—H8	120.0
C3—C2—H2	119.4	C7—C8—H8	120.0
C2—C3—C4	118.7 (3)	C10—C9—C8	118.9 (3)
C2—C3—C1	123.1 (2)	C10—C9—H9	120.5
C4—C3—C1	118.2 (2)	C8—C9—H9	120.5
C5—C4—C3	120.2 (3)	C9—C10—C11	122.2 (3)
C5—C4—H4	119.9	C9—C10—C12	119.1 (3)
C3—C4—H4	119.9	C11—C10—C12	118.7 (3)
C4—C5—C4 <sup>i</sup>	120.9 (4)	C10—C11—C6	119.5 (3)
C4—C5—H5	119.6	C10—C11—H11	120.2
C4 <sup>i</sup> —C5—H5	119.6	C6—C11—H11	120.2
C6—N1—C1—O1	-12.3 (4)	C11—C6—C7—C8	-0.8 (5)
C6—N1—C1—C3	166.2 (2)	N1—C6—C7—C8	178.8 (3)
C3 <sup>i</sup> —C2—C3—C4	-0.9 (2)	C11—C6—C7—C11	179.9 (2)
C3 <sup>i</sup> —C2—C3—C1	-179.2 (3)	N1—C6—C7—C11	-0.6 (4)

O1—C1—C3—C2	151.0 (2)	C6—C7—C8—C9	-1.0 (5)
N1—C1—C3—C2	-27.5 (3)	C11—C7—C8—C9	178.3 (3)
O1—C1—C3—C4	-27.4 (4)	C7—C8—C9—C10	2.0 (5)
N1—C1—C3—C4	154.2 (3)	C8—C9—C10—C11	-1.1 (6)
C2—C3—C4—C5	1.9 (4)	C8—C9—C10—C12	179.0 (3)
C1—C3—C4—C5	-179.7 (2)	C9—C10—C11—C6	-0.8 (5)
C3—C4—C5—C4 <sup>i</sup>	-1.0 (2)	C12—C10—C11—C6	179.1 (2)
C1—N1—C6—C11	29.4 (4)	C7—C6—C11—C10	1.7 (4)
C1—N1—C6—C7	-150.1 (3)	N1—C6—C11—C10	-177.8 (3)

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

*Hydrogen-bond geometry (Å, °)*

<i>D—H...A</i>	<i>D—H</i>	<i>H...A</i>	<i>D...A</i>	<i>D—H...A</i>
N1—H1...O1 <sup>ii</sup>	0.86	2.22	3.046 (3)	160

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