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2,2′-Dichloro-*N*,*N*′-[1,3-phenylenebis(methylene)]diacetamide

Hong-Xin Cai and Wei-Na Wu*

Department of Physics and Chemistry, Henan Polytechnic University, Jiaozuo 454000, People's Republic of China
Correspondence e-mail: wuwn08@hpu.edu.cn

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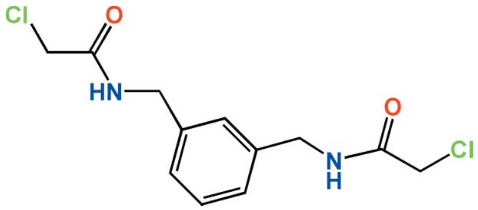
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.004 Å; R factor = 0.055; wR factor = 0.157; data-to-parameter ratio = 19.0.

The complete molecule of the title compound, $C_{12}H_{14}Cl_2N_2O_2$, is generated by a crystallographic twofold axis with two C atoms of the central benzene ring lying on the axis. In the crystal, N—H···O hydrogen bonds link the molecules into chains parallel to the c axis.

Related literature

For the synthesis of lanthanide complexes with amide-type ligands, see: Wu *et al.* (2008). For a related structure, see: Yuan *et al.* (2010).



Experimental

Crystal data

$C_{12}H_{14}Cl_2N_2O_2$
M_r = 289.15
Monoclinic, $C2/c$
a = 20.62 (2) Å
b = 7.464 (8) Å
c = 9.485 (11) Å
β = 110.362 (11)°
V = 1369 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.47 mm^{-1}
T = 296 K
0.27 × 0.23 × 0.22 mm

Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2007)
T_{min} = 0.881, T_{max} = 0.902
6996 measured reflections
1574 independent reflections
1288 reflections with $I > 2\sigma(I)$
R_{int} = 0.080

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.157
S = 1.06
1574 reflections
83 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1*C*···O1^i	0.86	2.03	2.864 (3)	163

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

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); 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*).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: VM2160).

References

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Yuan, M.-S., Li, Z. & Wang, Q. (2010). *Acta Cryst.* E**66**, o2017.

supplementary materials

Acta Cryst. (2012). E**68**, o933   [doi:10.1107/S1600536812008653]

2,2′-Dichloro-*N*,*N*′-[1,3-phenylenebis(methylene)]diacetamide

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Comment

The luminescent properties of the lanthanide complexes with amide type ligands have been investigated in our previous work (Wu *et al.*, 2008). As part of our ongoing studies of the amide type ligands, the title compound was synthesized and characterized by X-ray diffraction.

The complete molecule of the title compound (Fig. 1) is generated by a crystallographic twofold axis with atoms C5 and C7 of the central phenyl group lying on the axis. All the bond lengths are comparable with those observed in a similar compound (Yuan *et al.*, 2010). In the crystal, intermolecular N—H···O hydrogen bonds link the molecules into chains parallel to the *c* axis (Table 1).

Experimental

A chloroform solution containing chloroacetyl chloride (2.26 g, 0.02 mol) was added dropwise to a solution of (3-(aminomethyl)phenyl)methanamine (1.36 g, 0.01 mol) and pyridine (1.60 g, 0.02 mol) in chloroform (20 ml) under stirring on a ice-water bath. Then, the reaction mixture was stirred at room temperature for 3.5 h. A solid product was separated from the solution by suction filtration, purified by washing with water, 0.5 mol/*L* HCl, 0.5 mol/*L* NaOH and distilled water, respectively. Colourless prism crystals were obtained by slow evaporation of the acetone solution at room temperature.

Refinement

The H atoms were placed at calculated positions and refined in riding mode, with the carrier atom-H distances = 0.93 Å for aryl, 0.97 Å for methylene and 0.86 Å for the secondary amine H atoms. The $U_{\rm iso}$ values were constrained to be $1.2U_{\rm eq}$ of the carrier atom for the H atoms.

Computing details

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT* (Bruker, 2007); 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* (Sheldrick, 2008).

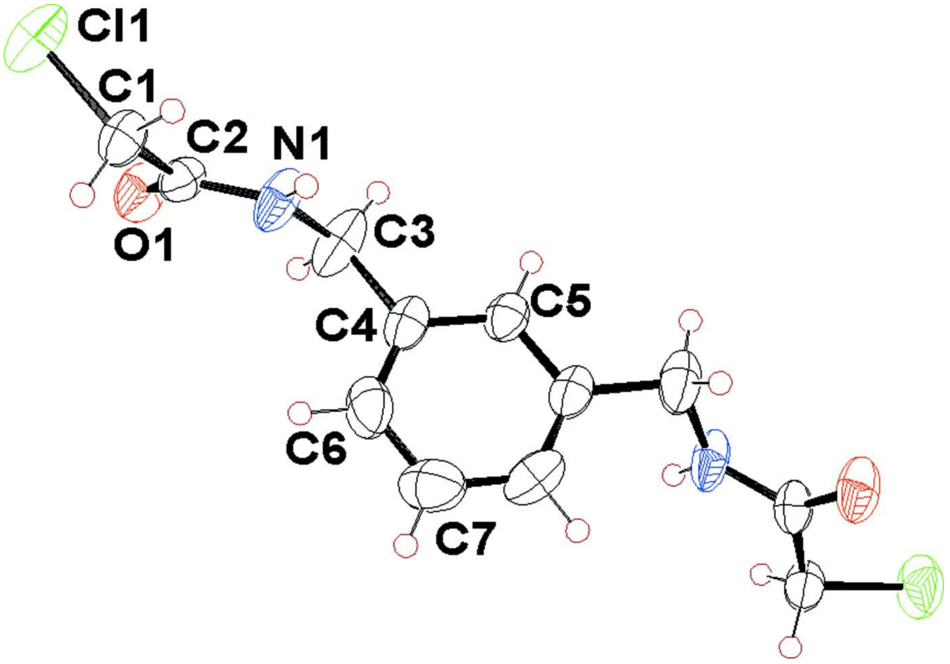

Figure 1

The molecular structure shown with 50% probability displacement ellipsoids. Unlabelled atoms are related with the labelled ones by symmetry operation (-*x*, *y*, -*z* - 1/2).

2,2′-Dichloro-*N*,*N*′-[1,3-phenylenebis(methylene)]diacetamide

Crystal data

$C_{12}H_{14}Cl_2N_2O_2$
$M_r = 289.15$
Monoclinic, $C2/c$
Hall symbol: -C 2yc
$a = 20.62$ (2) Å
$b = 7.464$ (8) Å
$c = 9.485$ (11) Å
$\beta = 110.362$ (11)°
$V = 1369$ (3) Å^3
$Z = 4$

$F(000) = 600$
$D_x = 1.403$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1673 reflections
$\theta = 2.1$–27.5°
$\mu = 0.47$ mm^{-1}
$T = 296$ K
Prism, colorless
0.27 × 0.23 × 0.22 mm

Data collection

Bruker SMART APEX CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2007)
$T_{\min} = 0.881$, $T_{\max} = 0.902$

6996 measured reflections
1574 independent reflections
1288 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.080$
$\theta_{\max} = 27.5°$, $\theta_{\min} = 2.9°$
$h = -26 \rightarrow 26$
$k = -9 \rightarrow 9$
$l = -12 \rightarrow 12$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.055$	H-atom parameters constrained
$wR(F^2) = 0.157$	$w = 1/[\sigma^2(F_o^2) + (0.0746P)^2 + 0.9706P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.06$	$(\Delta/\sigma)_{\max} < 0.001$
1574 reflections	$\Delta\rho_{\max} = 0.46$ e Å^{-3}
83 parameters	$\Delta\rho_{\min} = -0.31$ e Å^{-3}
0 restraints	
Primary atom site location: structure-invariant direct methods	

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 (Å^2)

	x	*y*	*z*	$U_{\rm iso}$*/$U_{\rm eq}$
C1	0.28959 (11)	−0.0305 (3)	0.6316 (2)	0.0460 (5)
H1A	0.2913	0.0011	0.5338	0.055*
H1B	0.2936	−0.1597	0.6422	0.055*
C2	0.22101 (10)	0.0300 (3)	0.6421 (2)	0.0399 (5)
C3	0.10062 (11)	0.1072 (4)	0.4959 (3)	0.0635 (8)
H3A	0.0938	0.2349	0.4780	0.076*
H3B	0.0931	0.0795	0.5889	0.076*
C4	0.04867 (10)	0.0058 (3)	0.3676 (2)	0.0443 (5)
C5	0.0000	0.0960 (4)	0.2500	0.0429 (7)
H5	0.0000	0.2206	0.2500	0.052*
C6	0.04839 (12)	−0.1788 (4)	0.3656 (3)	0.0599 (7)
H6	0.0810	−0.2418	0.4427	0.072*
C7	0.0000	−0.2710 (5)	0.2500	0.0701 (11)
H7	0.0000	−0.3956	0.2500	0.084*
N1	0.17173 (9)	0.0598 (3)	0.51078 (19)	0.0526 (6)
H1C	0.1819	0.0510	0.4305	0.063*
O1	0.21270 (9)	0.0449 (3)	0.76351 (17)	0.0564 (5)
Cl1	0.35925 (3)	0.07271 (11)	0.77441 (8)	0.0728 (3)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0388 (11)	0.0610 (13)	0.0346 (10)	−0.0002 (9)	0.0081 (8)	−0.0056 (9)
C2	0.0372 (10)	0.0537 (12)	0.0294 (9)	−0.0106 (8)	0.0123 (8)	−0.0037 (8)
C3	0.0341 (11)	0.113 (2)	0.0410 (11)	0.0022 (12)	0.0095 (9)	−0.0225 (13)
C4	0.0299 (9)	0.0698 (14)	0.0344 (10)	0.0022 (9)	0.0129 (8)	−0.0017 (9)

C5	0.0319 (14)	0.0550 (18)	0.0414 (14)	0.000	0.0120 (12)	0.000
C6	0.0376 (11)	0.0767 (18)	0.0620 (14)	0.0108 (11)	0.0133 (10)	0.0208 (12)
C7	0.0496 (19)	0.052 (2)	0.108 (3)	0.000	0.027 (2)	0.000
N1	0.0323 (9)	0.0990 (17)	0.0264 (8)	−0.0014 (9)	0.0101 (7)	−0.0088 (8)
O1	0.0523 (10)	0.0911 (14)	0.0281 (7)	−0.0061 (8)	0.0170 (7)	−0.0022 (7)
Cl1	0.0404 (4)	0.1002 (6)	0.0649 (5)	−0.0068 (3)	0.0021 (3)	−0.0226 (4)

Geometric parameters (Å, º)

C1—C2	1.520 (3)	C4—C6	1.378 (4)
C1—Cl1	1.771 (2)	C4—C5	1.388 (3)
C1—H1A	0.9700	C5—C4^{i}	1.388 (3)
C1—H1B	0.9700	C5—H5	0.9300
C2—O1	1.227 (3)	C6—C7	1.382 (3)
C2—N1	1.324 (3)	C6—H6	0.9300
C3—N1	1.467 (3)	C7—C6^{i}	1.382 (3)
C3—C4	1.514 (3)	C7—H7	0.9300
C3—H3A	0.9700	N1—H1C	0.8600
C3—H3B	0.9700		
C2—C1—Cl1	110.26 (16)	C6—C4—C5	118.4 (2)
C2—C1—H1A	109.6	C6—C4—C3	120.6 (2)
Cl1—C1—H1A	109.6	C5—C4—C3	121.0 (2)
C2—C1—H1B	109.6	C4^{i}—C5—C4	122.0 (3)
Cl1—C1—H1B	109.6	C4^{i}—C5—H5	119.0
H1A—C1—H1B	108.1	C4—C5—H5	119.0
O1—C2—N1	123.7 (2)	C4—C6—C7	120.5 (2)
O1—C2—C1	121.7 (2)	C4—C6—H6	119.8
N1—C2—C1	114.53 (19)	C7—C6—H6	119.8
N1—C3—C4	111.2 (2)	C6—C7—C6^{i}	120.3 (4)
N1—C3—H3A	109.4	C6—C7—H7	119.9
C4—C3—H3A	109.4	C6^{i}—C7—H7	119.9
N1—C3—H3B	109.4	C2—N1—C3	123.07 (19)
C4—C3—H3B	109.4	C2—N1—H1C	118.5
H3A—C3—H3B	108.0	C3—N1—H1C	118.5
Cl1—C1—C2—O1	−41.4 (3)	C5—C4—C6—C7	−0.9 (3)
Cl1—C1—C2—N1	140.17 (19)	C3—C4—C6—C7	178.68 (18)
N1—C3—C4—C6	58.1 (3)	C4—C6—C7—C6^{i}	0.47 (15)
N1—C3—C4—C5	−122.3 (2)	O1—C2—N1—C3	−2.6 (4)
C6—C4—C5—C4^{i}	0.46 (14)	C1—C2—N1—C3	175.8 (2)
C3—C4—C5—C4^{i}	−179.1 (2)	C4—C3—N1—C2	−137.0 (2)

Symmetry code: (i) −*x*, *y*, −*z*+1/2.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*C*···O1^{ii}	0.86	2.03	2.864 (3)	163

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