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N,*N*′-(Ethane-1,2-diyl)bis(2-chlorobenzamide)

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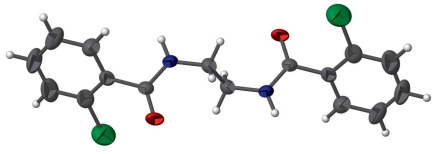
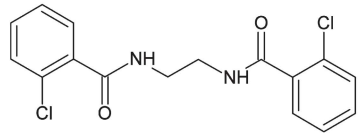
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_{16}H_{14}Cl_2N_2O_2$, crystallized with one half-molecule in the asymmetric unit; the whole molecule is generated by inversion symmetry, the center of inversion being situated at the middle of the bridging $-CH_2-CH_2-$ bond. The dihedral angle between the amide group and the benzene ring is 52.4 (2)°. In the crystal, molecules are linked by two pairs of N—H···O hydrogen bonds forming a ladder-like structure propagating along the *a*-axis direction and enclosing $R_2^2(14)$ ring motifs. The compound was refined as a two-component twin [BASF = 0.18 (1)].

3D view



Chemical scheme



Structure description

Ethylenediamine, having two amines, is bifunctional and readily forms heterocycles such as imidazolidines. It is also widely used as a precursor to form various polymers (Wang *et al.*, 2013). It is an ingredient in the common bronchodilator drug aminophylline, where it serves to solubilize the active ingredient theophylline. It has also been used in dermatologic preparations (Hogan, 1990). Ethylenediamine is one of the most frequent contact sensitizers (Zuidema, 1985). It is used as a solvent, miscible with water, oxygenated and aromatic solvents (Ashford, 1994). Ethylenediamine dihydroiodide (EDDI) has been added to animal feeds as a source of iodide (Lyday, 2000). *N*-substituted benzamides are well known anticancer compounds (Olsson *et al.*, 2002), they exhibit potent anti-emetic activity (Vega-Noverola *et al.*, 1989) and inhibit the activity of nuclear factor-B and the nuclear factor of activated T cells while inducing activator protein 1 activity in T-lymphocytes (Lindgren *et al.*, 2001). In view of this interest we have synthesized the title compound and describe herein its crystal structure.

The bond lengths and bond angles in the title compound, Fig. 1, are close to those observed for similar compounds, for example in *N*,*N*′-ethane-1,2-diylbis(4-methoxy-

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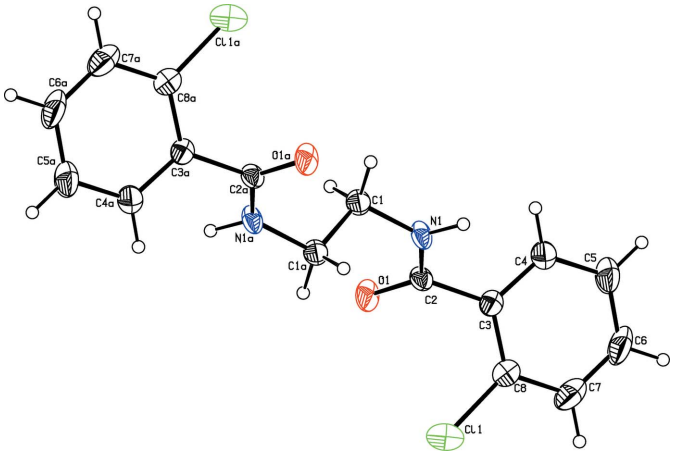

Figure 1
The molecular structure of the title compound, with atom labelling [symmetry code: (*a*) $-x+1, -y, -z+1$]. Displacement ellipsoids are drawn at the 30% probability level.

benzamide) (Aparicio *et al.*, 2014), 4,4′-(ethane-1,2-diyldicarbamoyl)dibenzoic acid (Guarda *et al.*, 2012) and 1,2-bis[(2-aminobenzoyl)amino]ethane (Bertolasi *et al.*, 2009). In the title compound, the dihedral angle between the amide group (O1/N1/C2/C3) and the benzene ring (C3–C8) is 52.4 (2)°. In the above mentioned compounds, this dihedral angle is *ca* 25.9, 27.5 and 30.6°, respectively.

In the crystal, molecules are linked by two pairs of N—H···O hydrogen bonds, forming a ladder-like structure propagating along the *a*-axis direction and enclosing $R_2^2(14)$ loops (Table 1 and Fig. 2).

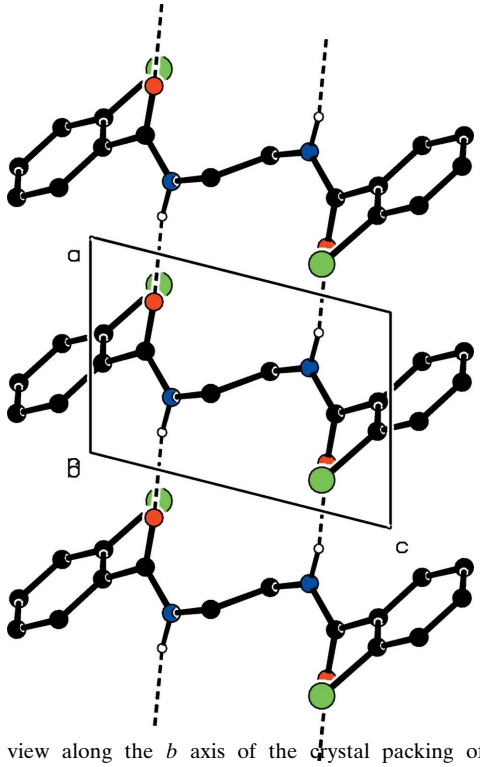

Figure 2
A partial view along the *b* axis of the crystal packing of the title compound. The dashed lines indicate the hydrogen bonds (see Table 1). For clarity only the H atoms involved in interactions have been included.

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^{i}	0.90 (5)	1.99 (5)	2.801 (4)	149 (4)

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{16}H_{14}Cl_2N_2O_2$
M_r	337.19
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	293
a, b, c (Å)	4.9667 (6), 23.701 (3), 7.1113 (8)
β (°)	104.189 (4)
V (Å^3)	811.59 (17)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.41
Crystal size (mm)	0.30 × 0.25 × 0.20
Data collection	
Diffractometer	Bruker Kappa APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2004)
T_{min}, T_{max}	0.885, 0.922
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	11558, 1429, 1429
R_{int}	0.061
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.595
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.064, 0.156, 1.17
No. of reflections	11558
No. of parameters	105
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.40, −0.32

Computer programs: *APEX2*, *SAINT* and *XPREP* (Bruker, 2004), *SIR92* (Altomare *et al.*, 1993), *SHELXL2014* (Sheldrick, 2015) and *ORTEP-3 for Windows* (Farrugia, 2012).

Synthesis and crystallization

The title compound was synthesized following a published procedure (Revathi *et al.*, 2015). In a 250 ml round-bottomed flask, 25ml of ethylmethylketone was added to ethylenediamine (0.01mol) and stirred at room temperature. After 10 min, triethylamine (0.04 mol) was added and the mixture was stirred for 15 min. 2-Chlorobenzoyl chloride (0.04 mol) was then added and the reaction mixture was stirred at room temperature for 2 h. The precipitate that formed was filtered off and the filtrate evaporated to give crude title product. It was recrystallized twice from ethylmethylketone to give yellow block-like crystals of the title compound.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The compound was refined as a two-component twin [BASF = 0.18 (1)].

Acknowledgements

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full crystallographic data

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Crystal data

$C_{16}H_{14}Cl_2N_2O_2$
M_r = 337.19
Monoclinic, $P2_1/c$
a = 4.9667 (6) Å
b = 23.701 (3) Å
c = 7.1113 (8) Å
β = 104.189 (4)°
V = 811.59 (17) Å^3
Z = 2
$F(000)$ = 348
D_x = 1.380 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 6198 reflections
θ = 2.6–29.3°
μ = 0.41 mm^{-1}
T = 293 K
Block, yellow
0.30 × 0.25 × 0.20 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω and φ scan
Absorption correction: multi-scan (SADABS; Bruker, 2004)
T_{min} = 0.885, T_{max} = 0.922
11558 measured reflections
1429 independent reflections
1429 reflections with $I > 2\sigma(I)$
R_{int} = 0.061
θ_{max} = 25.0°, θ_{min} = 3.1°
h = −5→5
k = −28→28
l = −8→8

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.064
$wR(F^2)$ = 0.156
S = 1.17
11558 reflections
105 parameters
0 restraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + 1.3152P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.40 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

Special details

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

Refinement. Refined as a 2-component twin.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.5857 (8)	−0.00874 (16)	0.5990 (6)	0.0425 (11)
H1A	0.4843	−0.0367	0.6536	0.051*
H1B	0.7574	−0.0259	0.5855	0.051*
C2	0.4659 (8)	0.06093 (16)	0.8173 (6)	0.0365 (10)
C3	0.5714 (8)	0.10688 (16)	0.9594 (6)	0.0371 (10)
C4	0.8101 (9)	0.09869 (18)	1.1056 (7)	0.0508 (12)
H4	0.9088	0.0652	1.1089	0.061*
C5	0.9046 (11)	0.1387 (2)	1.2456 (7)	0.0681 (15)
H5	1.0635	0.1321	1.3439	0.082*
C6	0.7609 (13)	0.1887 (2)	1.2385 (9)	0.0773 (17)
H6	0.8235	0.2160	1.3331	0.093*
C7	0.5285 (12)	0.1986 (2)	1.0949 (9)	0.0700 (16)
H7	0.4339	0.2327	1.0904	0.084*
C8	0.4345 (9)	0.15789 (17)	0.9560 (7)	0.0475 (11)
N1	0.6494 (7)	0.03913 (15)	0.7298 (5)	0.0407 (9)
O1	0.2278 (6)	0.04316 (12)	0.7882 (5)	0.0564 (9)
Cl1	0.1434 (3)	0.17296 (5)	0.7711 (2)	0.0786 (6)
H1	0.822 (10)	0.0537 (18)	0.761 (7)	0.069 (15)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.034 (2)	0.046 (2)	0.048 (3)	0.0037 (19)	0.0094 (19)	−0.013 (2)
C2	0.026 (2)	0.043 (2)	0.041 (3)	0.0015 (17)	0.0086 (19)	−0.0049 (19)
C3	0.033 (2)	0.039 (2)	0.044 (3)	−0.0043 (18)	0.018 (2)	−0.0056 (19)
C4	0.045 (3)	0.050 (3)	0.054 (3)	−0.001 (2)	0.006 (2)	−0.008 (2)
C5	0.067 (4)	0.071 (4)	0.056 (4)	−0.015 (3)	−0.004 (3)	−0.018 (3)
C6	0.088 (4)	0.059 (4)	0.081 (5)	−0.024 (3)	0.014 (4)	−0.037 (3)
C7	0.086 (4)	0.042 (3)	0.085 (5)	−0.002 (3)	0.028 (4)	−0.016 (3)
C8	0.052 (3)	0.041 (2)	0.050 (3)	0.000 (2)	0.015 (2)	−0.001 (2)
N1	0.0215 (18)	0.056 (2)	0.045 (2)	−0.0040 (16)	0.0076 (16)	−0.0165 (17)
O1	0.0235 (16)	0.0612 (19)	0.089 (3)	−0.0107 (14)	0.0222 (16)	−0.0276 (17)
Cl1	0.0734 (10)	0.0652 (9)	0.0913 (12)	0.0220 (7)	0.0088 (8)	0.0099 (8)

Geometric parameters (Å, º)

C1—N1	1.452 (5)	C4—H4	0.9300
C1—C1^{i}	1.513 (8)	C5—C6	1.379 (7)
C1—H1A	0.9700	C5—H5	0.9300
C1—H1B	0.9700	C6—C7	1.362 (8)
C2—O1	1.224 (4)	C6—H6	0.9300
C2—N1	1.328 (5)	C7—C8	1.378 (6)
C2—C3	1.490 (5)	C7—H7	0.9300
C3—C8	1.384 (5)	C8—Cl1	1.736 (5)
C3—C4	1.386 (6)	N1—H1	0.90 (5)

C4—C5	1.371 (6)		
N1—C1—C1^{i}	111.5 (4)	C4—C5—C6	119.1 (5)
N1—C1—H1A	109.3	C4—C5—H5	120.5
C1^{i}—C1—H1A	109.3	C6—C5—H5	120.5
N1—C1—H1B	109.3	C7—C6—C5	120.8 (5)
C1^{i}—C1—H1B	109.3	C7—C6—H6	119.6
H1A—C1—H1B	108.0	C5—C6—H6	119.6
O1—C2—N1	122.2 (4)	C6—C7—C8	119.5 (5)
O1—C2—C3	122.0 (3)	C6—C7—H7	120.3
N1—C2—C3	115.7 (3)	C8—C7—H7	120.3
C8—C3—C4	117.5 (4)	C7—C8—C3	121.4 (5)
C8—C3—C2	122.5 (4)	C7—C8—Cl1	118.1 (4)
C4—C3—C2	120.0 (4)	C3—C8—Cl1	120.5 (3)
C5—C4—C3	121.7 (4)	C2—N1—C1	122.5 (3)
C5—C4—H4	119.1	C2—N1—H1	117 (3)
C3—C4—H4	119.1	C1—N1—H1	120 (3)
O1—C2—C3—C8	−52.3 (6)	C6—C7—C8—C3	0.0 (8)
N1—C2—C3—C8	130.1 (4)	C6—C7—C8—Cl1	−178.1 (4)
O1—C2—C3—C4	125.5 (5)	C4—C3—C8—C7	−1.2 (6)
N1—C2—C3—C4	−52.1 (5)	C2—C3—C8—C7	176.7 (4)
C8—C3—C4—C5	1.8 (7)	C4—C3—C8—Cl1	176.9 (3)
C2—C3—C4—C5	−176.2 (4)	C2—C3—C8—Cl1	−5.2 (5)
C3—C4—C5—C6	−1.1 (7)	O1—C2—N1—C1	−2.5 (6)
C4—C5—C6—C7	−0.1 (8)	C3—C2—N1—C1	175.2 (4)
C5—C6—C7—C8	0.7 (8)	C1^{i}—C1—N1—C2	78.1 (6)

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{ii}	0.90 (5)	1.99 (5)	2.801 (4)	149 (4)

Symmetry code: (ii) *x*+1, *y*, *z*.