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(*E*)-(2-Chlorobenzylidene)amino 2-amino-4-chlorobenzoate

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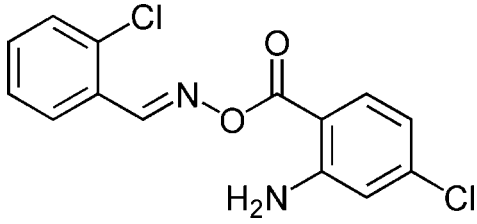
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.001 Å; R factor = 0.028; wR factor = 0.084; data-to-parameter ratio = 21.3.

In the title compound, $C_{14}H_{10}Cl_2N_2O_2$, the configuration about the C=N double bond is *E* and the dihedral angle between the benzene rings is 1.75 (5)°. An intramolecular N—H···O interaction generates an *S*(6) ring. In the crystal, molecules are linked by N—H···O hydrogen bonds, resulting in [101] chains.

Related literature

For background to 2-amino-4-chlorobenzoic acid derivatives, see: Jahnke *et al.* (2010); Lee *et al.* (2005). For a related structure, see: Seong *et al.* (2008).



Experimental

Crystal data

$C_{14}H_{10}Cl_2N_2O_2$
M_r = 309.14
Monoclinic, $P2_1/n$
a = 7.4034 (5) Å
b = 23.8190 (15) Å
c = 7.6362 (5) Å
β = 96.382 (1)°
V = 1338.23 (15) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.49 mm^{-1}
T = 100 K
0.16 × 0.15 × 0.10 mm

Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.926, T_{max} = 0.953
12076 measured reflections
3879 independent reflections
3637 reflections with $I > 2\sigma(I)$
R_{int} = 0.015

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.084
S = 1.05
3879 reflections
182 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.53 e Å^{-3}
$\Delta\rho_{min}$ = −0.53 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2B···O2	0.88	2.02	2.6653 (12)	130
N2—H2A···O2^{i}	0.88	2.19	2.9332 (12)	142

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); 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: HB6586).

References

Bruker (2000). *SMART*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Jahnke, W., Grotzfeld, R. M., Pelle, X., Strauss, A., Fendrich, G., Cowan-Jacob, S. W., Cotesta, S., Fabbro, D., Furet, P., Mestan, D. & Marzinzik, A. L. (2010). *J. Am. Chem. Soc.* **132**, 7043–7048.
Lee, J., Chubb, A. J., Moman, E., McLoughlin, B. M., Sharkey, C. T., Kelly, J. G., Nolan, K. B., Devocelle, M. & Fitzgerald, D. J. (2005). *Org. Biomol. Chem.* **3**, 3678–3685.
Seong, C. M., Park, W. K., Park, C. M., Kong, J. Y. & Park, N. S. (2008). *Bioorg. Med. Chem. Lett.* **18**, 738–743.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supplementary materials

Acta Cryst. (2012). E**68**, o770 [doi:10.1107/S1600536812003844]

(*E*)-(2-Chlorobenzylidene)amino 2-amino-4-chlorobenzoate

Weiyan Yin, Zhi Wang, Ying Liang and Zi-Wen Yang

Comment

2-Amino-4-chlorobenzoic acid derivatives show diverse biological properties such as inhibitor of the prostaglandin H2 synthase peroxidase activity (Lee *et al.*, 2005) and allosteric inhibitors of Bcr-Abl (Jahnke *et al.*, 2010). As a part of our studies of 2-aminobenzoic acid-containing compounds with potential biological activities, we report here the crystal structure of the title compound, (I) (Fig. 1).

The conformation of the N—H and the C=O bonds in the 2-aminobenzoic acid segment is similar to that observed in other 2-aminobenzoic acid compound (Seong *et al.*, 2008). The dihedral angles between the two phenyl rings is 1.75 (5)°. The molecular structure is linked by N—H···O hydrogen-bonds (Table 1).

Experimental

Dicyclohexylcarbodiimide (1.1 g, 5.0 mmol) and 4-dimethylamiopryidine(0.25 g, 1.0 mmol) was added to a mixture of 2-chlorobenzaldehyde oxime (0.78 g, 5.0 mmol) and 2-amino-4-chlorobenzoic acid (0.86 g, 5.0 mmol) in dichloromethane (30 ml). The reaction mixture was stirred for 14 h at 353 k. The product was collected by filtration give a gray solid and recrystallization from its ether solution yielded colourless prisms of (I) after a few days.

Refinement

The H atoms were placed in calculated positions (C—H = 0.93–0.97Å and N—H = 0.86 Å), and refined as riding with U_{iso} (H) = 1.2U_{eq}(C, N).

Computing details

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT* (Bruker, 2000); 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).

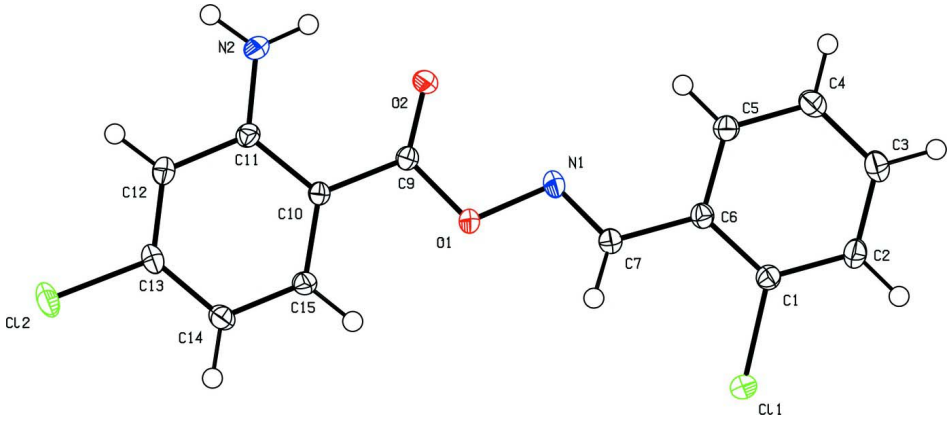

Figure 1

The molecular structure of the title compound, showing 30% probability displacement ellipsoids.

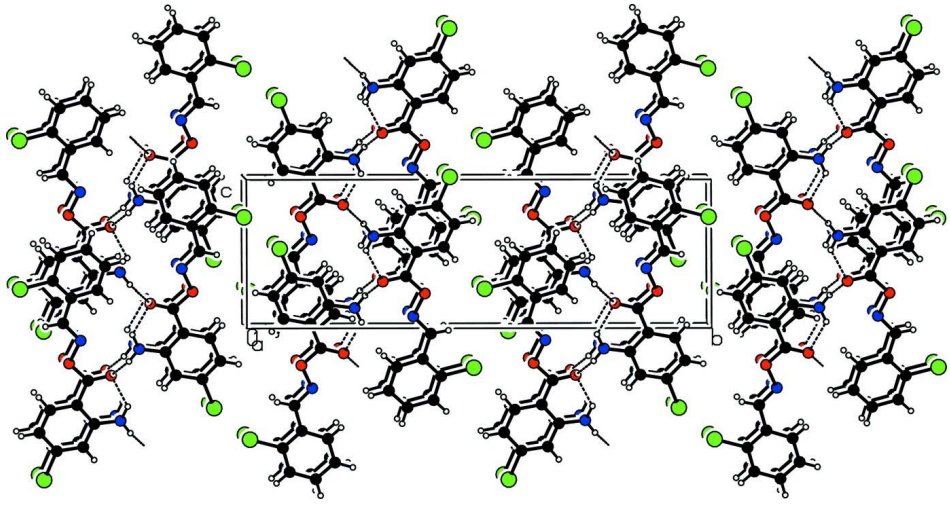

Figure 2

Crystal Packing diagram of (I). Hydrogen bonds are shown as dashed lines.

(*E*)-(2-Chlorobenzylidene)amino 2-amino-4-chlorobenzoate

Crystal data

$C_{14}H_{10}Cl_2N_2O_2$
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Monoclinic, $P2_1/n$
$a = 7.4034$ (5) Å
$b = 23.8190$ (15) Å
$c = 7.6362$ (5) Å
$\beta = 96.382$ (1)°
$V = 1338.23$ (15) Å^3
$Z = 4$

$F(000) = 632$
$D_x = 1.534$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 9848 reflections
$\theta = 2.8$–$32.0°$
$\mu = 0.49$ mm^{-1}
$T = 100$ K
Block, colorless
0.16 × 0.15 × 0.10 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, 2000)
T_{min} = 0.926, T_{max} = 0.953
12076 measured reflections
3879 independent reflections
3637 reflections with $I > 2\sigma(I)$

R_{int} = 0.015
θ_{max} = 30.0°, θ_{min} = 1.7°
h = −10→10
k = −33→32
l = −10→10

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.084
S = 1.05
3879 reflections
182 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
$w = 1/[\sigma^2(F_o^2) + (0.0495P)^2 + 0.5103P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.53 e Å^{-3}
$\Delta\rho_{min}$ = −0.53 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0048 (10)

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 (Å²)

	x	y	z	U_{iso}*/U_{eq}
C1	0.45478 (14)	0.08217 (4)	0.17648 (13)	0.01523 (18)
C2	0.36816 (14)	0.09752 (5)	0.01219 (13)	0.01805 (19)
H2	0.3133	0.0698	−0.0659	0.022*
C3	0.36279 (15)	0.15379 (5)	−0.03640 (14)	0.0197 (2)
H3	0.3043	0.1646	−0.1483	0.024*
C4	0.44295 (15)	0.19433 (5)	0.07849 (14)	0.0190 (2)
H4	0.4383	0.2328	0.0453	0.023*
C5	0.52956 (14)	0.17850 (4)	0.24135 (13)	0.01681 (19)
H5	0.5854	0.2063	0.3184	0.020*
C6	0.53611 (13)	0.12204 (4)	0.29440 (13)	0.01445 (18)
C7	0.62742 (13)	0.10574 (4)	0.46828 (13)	0.01528 (18)
H7	0.6660	0.0682	0.4918	0.018*
C9	0.80555 (13)	0.16068 (4)	0.86039 (13)	0.01431 (18)
C10	0.89159 (13)	0.13801 (4)	1.02756 (13)	0.01340 (17)
C11	0.97488 (13)	0.17569 (4)	1.15683 (13)	0.01466 (18)
C12	1.05960 (14)	0.15271 (4)	1.31596 (13)	0.01651 (19)
H12	1.1176	0.1767	1.4044	0.020*
C13	1.05789 (14)	0.09568 (4)	1.34250 (13)	0.01679 (19)

C14	0.97615 (14)	0.05787 (4)	1.21752 (14)	0.01768 (19)
H14	0.9772	0.0186	1.2394	0.021*
C15	0.89387 (13)	0.07991 (4)	1.06104 (13)	0.01523 (18)
H15	0.8374	0.0552	0.9739	0.018*
Cl1	0.45712 (4)	0.011502 (10)	0.23325 (3)	0.01985 (8)
Cl2	1.16302 (4)	0.069412 (12)	1.54105 (3)	0.02424 (8)
N1	0.65290 (13)	0.14358 (4)	0.58580 (12)	0.01819 (18)
N2	0.97461 (13)	0.23234 (4)	1.13648 (13)	0.02025 (19)
H2A	1.0264	0.2539	1.2213	0.024*
H2B	0.9226	0.2475	1.0385	0.024*
O1	0.74622 (10)	0.11954 (3)	0.74289 (10)	0.01663 (15)
O2	0.78875 (12)	0.21027 (3)	0.82487 (11)	0.02169 (17)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0162 (4)	0.0151 (4)	0.0144 (4)	0.0010 (3)	0.0017 (3)	−0.0007 (3)
C2	0.0188 (4)	0.0214 (5)	0.0136 (4)	0.0015 (4)	−0.0002 (3)	−0.0026 (3)
C3	0.0209 (5)	0.0236 (5)	0.0145 (4)	0.0046 (4)	0.0011 (4)	0.0016 (4)
C4	0.0220 (5)	0.0179 (4)	0.0173 (4)	0.0025 (4)	0.0034 (4)	0.0030 (4)
C5	0.0192 (4)	0.0158 (4)	0.0156 (4)	−0.0005 (3)	0.0027 (3)	−0.0001 (3)
C6	0.0147 (4)	0.0159 (4)	0.0127 (4)	0.0010 (3)	0.0018 (3)	0.0001 (3)
C7	0.0158 (4)	0.0157 (4)	0.0140 (4)	−0.0002 (3)	0.0002 (3)	0.0006 (3)
C9	0.0131 (4)	0.0141 (4)	0.0151 (4)	−0.0011 (3)	−0.0016 (3)	0.0003 (3)
C10	0.0129 (4)	0.0133 (4)	0.0134 (4)	−0.0004 (3)	−0.0014 (3)	0.0004 (3)
C11	0.0134 (4)	0.0142 (4)	0.0160 (4)	−0.0009 (3)	−0.0001 (3)	−0.0010 (3)
C12	0.0155 (4)	0.0195 (5)	0.0139 (4)	−0.0010 (3)	−0.0008 (3)	−0.0010 (3)
C13	0.0150 (4)	0.0217 (5)	0.0133 (4)	0.0003 (3)	−0.0002 (3)	0.0041 (3)
C14	0.0181 (4)	0.0157 (4)	0.0187 (5)	−0.0008 (3)	−0.0003 (4)	0.0040 (3)
C15	0.0152 (4)	0.0134 (4)	0.0166 (4)	−0.0012 (3)	−0.0005 (3)	0.0009 (3)
Cl1	0.02618 (14)	0.01429 (12)	0.01833 (13)	−0.00055 (8)	−0.00089 (9)	−0.00137 (8)
Cl2	0.02529 (14)	0.03026 (15)	0.01581 (13)	−0.00042 (10)	−0.00378 (10)	0.00817 (9)
N1	0.0215 (4)	0.0180 (4)	0.0137 (4)	0.0028 (3)	−0.0036 (3)	0.0012 (3)
N2	0.0244 (4)	0.0134 (4)	0.0210 (4)	−0.0021 (3)	−0.0062 (3)	−0.0016 (3)
O1	0.0210 (4)	0.0146 (3)	0.0130 (3)	0.0009 (3)	−0.0041 (3)	−0.0002 (2)
O2	0.0264 (4)	0.0138 (3)	0.0223 (4)	−0.0026 (3)	−0.0090 (3)	0.0035 (3)

Geometric parameters (Å, º)

C1—C2	1.3928 (14)	C9—C10	1.4649 (13)
C1—C6	1.3980 (14)	C10—C15	1.4071 (13)
C1—Cl1	1.7378 (10)	C10—C11	1.4230 (13)
C2—C3	1.3902 (15)	C11—N2	1.3581 (13)
C2—H2	0.9500	C11—C12	1.4143 (14)
C3—C4	1.3923 (15)	C12—C13	1.3736 (14)
C3—H3	0.9500	C12—H12	0.9500
C4—C5	1.3866 (14)	C13—C14	1.3999 (15)
C4—H4	0.9500	C13—Cl2	1.7417 (10)
C5—C6	1.4039 (14)	C14—C15	1.3829 (14)
C5—H5	0.9500	C14—H14	0.9500

C6—C7	1.4735 (14)	C15—H15	0.9500
C7—N1	1.2712 (13)	N1—O1	1.4352 (11)
C7—H7	0.9500	N2—H2A	0.8800
C9—O2	1.2151 (12)	N2—H2B	0.8800
C9—O1	1.3677 (12)		
C2—C1—C6	121.62 (9)	C15—C10—C11	119.92 (9)
C2—C1—Cl1	118.01 (8)	C15—C10—C9	121.07 (9)
C6—C1—Cl1	120.37 (8)	C11—C10—C9	119.01 (9)
C3—C2—C1	119.34 (10)	N2—C11—C12	118.53 (9)
C3—C2—H2	120.3	N2—C11—C10	123.52 (9)
C1—C2—H2	120.3	C12—C11—C10	117.94 (9)
C2—C3—C4	120.20 (10)	C13—C12—C11	119.94 (9)
C2—C3—H3	119.9	C13—C12—H12	120.0
C4—C3—H3	119.9	C11—C12—H12	120.0
C5—C4—C3	119.92 (10)	C12—C13—C14	123.09 (9)
C5—C4—H4	120.0	C12—C13—Cl2	118.24 (8)
C3—C4—H4	120.0	C14—C13—Cl2	118.67 (8)
C4—C5—C6	121.13 (10)	C15—C14—C13	117.41 (9)
C4—C5—H5	119.4	C15—C14—H14	121.3
C6—C5—H5	119.4	C13—C14—H14	121.3
C1—C6—C5	117.79 (9)	C14—C15—C10	121.70 (9)
C1—C6—C7	121.54 (9)	C14—C15—H15	119.1
C5—C6—C7	120.67 (9)	C10—C15—H15	119.1
N1—C7—C6	117.81 (9)	C7—N1—O1	109.09 (8)
N1—C7—H7	121.1	C11—N2—H2A	120.0
C6—C7—H7	121.1	C11—N2—H2B	120.0
O2—C9—O1	122.17 (9)	H2A—N2—H2B	120.0
O2—C9—C10	125.21 (9)	C9—O1—N1	110.62 (7)
O1—C9—C10	112.61 (8)		
C6—C1—C2—C3	0.25 (16)	C15—C10—C11—N2	−178.31 (10)
Cl1—C1—C2—C3	179.50 (8)	C9—C10—C11—N2	2.12 (15)
C1—C2—C3—C4	−0.16 (16)	C15—C10—C11—C12	0.59 (14)
C2—C3—C4—C5	0.48 (16)	C9—C10—C11—C12	−178.99 (9)
C3—C4—C5—C6	−0.89 (16)	N2—C11—C12—C13	178.24 (10)
C2—C1—C6—C5	−0.63 (15)	C10—C11—C12—C13	−0.71 (15)
Cl1—C1—C6—C5	−179.86 (8)	C11—C12—C13—C14	0.47 (16)
C2—C1—C6—C7	179.90 (9)	C11—C12—C13—Cl2	−179.67 (8)
Cl1—C1—C6—C7	0.66 (14)	C12—C13—C14—C15	−0.08 (16)
C4—C5—C6—C1	0.95 (15)	Cl2—C13—C14—C15	−179.94 (8)
C4—C5—C6—C7	−179.57 (9)	C13—C14—C15—C10	−0.04 (15)
C1—C6—C7—N1	−160.77 (10)	C11—C10—C15—C14	−0.22 (15)
C5—C6—C7—N1	19.77 (14)	C9—C10—C15—C14	179.35 (9)
O2—C9—C10—C15	175.68 (10)	C6—C7—N1—O1	−178.86 (8)
O1—C9—C10—C15	−5.26 (14)	O2—C9—O1—N1	−4.58 (14)
O2—C9—C10—C11	−4.74 (16)	C10—C9—O1—N1	176.34 (8)
O1—C9—C10—C11	174.31 (9)	C7—N1—O1—C9	168.81 (9)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*B*···O2	0.88	2.02	2.6653 (12)	130
N2—H2*A*···O2^{i}	0.88	2.19	2.9332 (12)	142

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