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2-(2-Chlorobenzamido)benzoic acid

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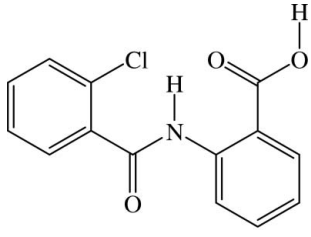
Key indicators: single-crystal X-ray study; T = 283 K; mean σ(C–C) = 0.003 Å; R factor = 0.045; wR factor = 0.109; data-to-parameter ratio = 12.2.

The title compound, $C_{14}H_{10}ClNO_3$, was synthesized by reacting stoichiometric amounts of anthranilic acid and 2-chlorobenzoyl chloride at ambient temperature. The dihedral angle between the two rings is 63.19 (12)°. The molecules are stabilized by four intramolecular hydrogen bonds, two C—H···O, one N—H···O and one N—H···Cl. Intermolecular O—H···O hydrogen bonding links the molecules in parallel layers along the *ac* plane.

Related literature

For related literature, see: Allen *et al.* (1987); Bernstein *et al.* (1995); Isaji *et al.* (1997); Levin *et al.* (2001); Manley *et al.* (2002); Mistry *et al.* (2001); Mo *et al.* (2000); Pavlidis & Perry (1994); Roe *et al.* (1999); Sebolt-Leopold *et al.* (1999).



Experimental

Crystal data

$C_{14}H_{10}ClNO_3$
M_r = 275.68
Monoclinic, $P2_1/c$
a = 10.7808 (7) Å
b = 15.9978 (11) Å
c = 7.2944 (5) Å
β = 97.122 (1)°
V = 1248.35 (15) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.31 mm^{-1}
T = 283 (2) K
0.35 × 0.33 × 0.08 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.900, T_{max} = 0.976
6230 measured reflections
2188 independent reflections
1725 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.109
S = 1.07
2188 reflections
180 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N1···Cl1	0.82 (2)	2.82 (2)	3.108 (2)	103 (3)
N1—H1N1···O2	0.82 (2)	1.92 (2)	2.638 (3)	145 (2)
O3—H1O3···O1^{i}	0.87 (3)	1.79 (3)	2.656 (3)	173 (3)
C10—H10A···O3	0.93	2.36	2.694 (3)	101
C13—H13A···O1	0.93	2.29	2.872 (3)	120

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

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: KP2128).

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supplementary materials

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2-(2-Chlorobenzamido)benzoic acid

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Comment

The molecules designed on anthranilic acid scaffold for new anticancer drugs have attracted great interest in recent years (Roe *et al.*, 1999; Mistry *et al.*, 2001; Manley *et al.*, 2002; Levin *et al.*, 2001). Among them there are: Tranilast (*N*-[3,4-dimethoxycinnamoyl]-anthranilic acid), a well known anti-allergic drug (Isaji *et al.*, 1997); farnesyl anthranilate suppresses the growth of murine melanomas in *in vivo* and *in vitro* (Mo *et al.*,2000); the anthranilamide PD 184352 (CI-1040), developed by Parke-Davis, is an inhibitor of both mitogen activated extracellular kinases (MEK1 and MEK2). In vivo, PD 184352 was shown to inhibit the growth of colon and pancreatic tumors (Sebolt-Leopold *et al.*, 1999). Therefore, precedent examples of drugs with anthranilic scaffold stimulated us to continue our research with anticancer activity based on the anthranilic acid scaffold. The title compound I, is expected to possess some interesting biological activities. The present paper describes the X-ray structure analysis that will be of help in molecular modelling and future drug design.

The title compound (I), was synthesized in moderate yield (Pavlidis & Perry, 1994) by reacting stichiometric amounts of anthranilic acid and 2-chlorobenzoylchloride at ambient temperature, using pyridine as a solvent. All bond lengths in the title compound (I) show normal values (Allen *et al.*, 1987). The two aromatic rings are completely planar and the acute angle between them is 63.19 (12)°. The title compound (I) (Fig. 1) shows intramolecular N—H···Cl, N—H···O and C—H···O hydrogen bonds generating three S(6) graph set motifs, while C—H···O hydrogen bond generates an S(5) graph sett motif (Bernstein *et al.*, 1995). These intramolecular hydrogen bonds influence the widening and shrinkage (from 120°) of the the exocyclic angles C7—N1—C8 [129.7 (2) °] and N1—C7—C1 [116.3 (2) °], respectively. An intermolecular O—H···O hydrogen bond keeps molecules in parallel layers along the *c* axis (Fig. 2).

Experimental

To a solution of anthranilic acid (1.0 g, 7.29 mmol) in pyridine (25 ml) was added 2-chlorobenzoylchloride (1.27 g, 7.29 mmol). The mixture was shaken for 5 min and keep at room temperature for a further 25 min with occasional shaking. The reaction mixture was poured into ice water (200 ml) and stirred for a few minutes; the precipitate was filtered off; the residue was washed with cold water (3x 60 ml). The title compound (I), was crystallized from dichloromethane in 62% overall yield (1.24 g).

Refinement

H atoms bonded to oxygen or nitrogen were refined freely to account for inter- and intra-molecular hydrogen bonds. However, the rest of atoms were placed in calculated positions with a C—H distances in 0.93 Å and U_{iso}(H) = 1.5U_{eq}(N) for amide H and 1.2U_{eq}(C) for others.

Figures

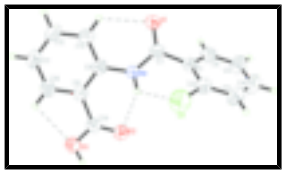

Fig. 1. The structure of (I) showing the 50% probability displacement ellipsoids and the atom-numbering scheme. A dashed lines indicate the intramolecular hydrogen bonds.

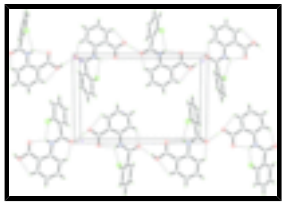

Fig. 2. The crystal packing of (I), viewed down the *c* axis.

2-(2-Chlorobenzamido)benzoic acid

Crystal data

$C_{14}H_{10}ClNO_3$	F_{000} = 568
M_r = 275.68	D_x = 1.467 Mg m^{-3}
Monoclinic, $P2_1/c$	Melting point: 379 K
Hall symbol: -P 2ybc	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 10.7808 (7) Å	Cell parameters from 1776 reflections
b = 15.9978 (11) Å	θ = 2.6–27.2º
c = 7.2944 (5) Å	μ = 0.31 mm^{-1}
β = 97.1220 (10)º	T = 283 (2) K
V = 1248.35 (15) Å^3	Block, colourless
Z = 4	0.35 × 0.33 × 0.08 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer	2188 independent reflections
Radiation source: fine-focus sealed tube	1725 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.026
Detector resolution: 8.33 pixels mm^{-1}	θ_{max} = 25.0º
T = 283(2) K	θ_{min} = 1.9º
ω scans	h = −11→12
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	k = −19→18
T_{min} = 0.900, T_{max} = 0.976	l = −8→8
6230 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map

Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.045$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.109$	$w = 1/[\sigma^2(F_o^2) + (0.0432P)^2 + 0.495P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.07$	$(\Delta/\sigma)_{max} < 0.001$
2188 reflections	$\Delta\rho_{max} = 0.23$ e Å^{-3}
180 parameters	$\Delta\rho_{min} = -0.32$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 $F^2 > 2$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_{iso}*/U_{eq}
Cl1	0.74177 (7)	0.65886 (5)	−0.01354 (10)	0.0683 (3)
O1	0.97574 (15)	0.51058 (10)	−0.2858 (3)	0.0541 (5)
N1	0.98919 (18)	0.64494 (13)	−0.1894 (3)	0.0415 (5)
O3	1.15398 (17)	0.87729 (11)	−0.0861 (3)	0.0559 (5)
O2	0.97761 (16)	0.80934 (10)	−0.1690 (3)	0.0584 (5)
C2	0.7035 (2)	0.62740 (15)	−0.2423 (3)	0.0433 (6)
C3	0.5802 (2)	0.63371 (18)	−0.3190 (4)	0.0553 (7)
H3A	0.5211	0.6563	−0.2506	0.066*
C4	0.5451 (2)	0.60673 (19)	−0.4956 (4)	0.0597 (8)
H4A	0.4619	0.6106	−0.5466	0.072*
C5	0.6319 (2)	0.57403 (18)	−0.5981 (4)	0.0560 (7)
H5A	0.6079	0.5567	−0.7190	0.067*
C6	0.7549 (2)	0.56701 (16)	−0.5213 (3)	0.0468 (6)
H6A	0.8132	0.5442	−0.5910	0.056*
C1	0.7934 (2)	0.59333 (13)	−0.3420 (3)	0.0378 (6)
C7	0.9274 (2)	0.57911 (14)	−0.2679 (3)	0.0387 (6)
C8	1.1139 (2)	0.65116 (14)	−0.1069 (3)	0.0367 (5)
C13	1.1887 (2)	0.58132 (15)	−0.0640 (3)	0.0458 (6)
H13A	1.1569	0.5281	−0.0906	0.055*
C12	1.3100 (2)	0.59119 (17)	0.0180 (4)	0.0526 (7)
H12A	1.3596	0.5442	0.0460	0.063*

C11	1.3593 (2)	0.66937 (17)	0.0593 (4)	0.0507 (7)
H11A	1.4414	0.6754	0.1138	0.061*
C10	1.2851 (2)	0.73829 (16)	0.0186 (3)	0.0439 (6)
H10A	1.3179	0.7911	0.0479	0.053*
C9	1.1627 (2)	0.73155 (14)	−0.0651 (3)	0.0346 (5)
C14	1.0881 (2)	0.80848 (14)	−0.1115 (3)	0.0389 (6)
H1N1	0.954 (2)	0.6908 (15)	−0.193 (3)	0.039 (7)*
H1O3	1.107 (3)	0.920 (2)	−0.122 (4)	0.080 (11)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0647 (5)	0.0891 (6)	0.0524 (4)	0.0047 (4)	0.0126 (3)	−0.0174 (4)
O1	0.0418 (10)	0.0363 (10)	0.0818 (14)	0.0031 (8)	−0.0015 (9)	−0.0151 (9)
N1	0.0367 (11)	0.0271 (11)	0.0585 (14)	0.0046 (9)	−0.0022 (9)	−0.0054 (9)
O3	0.0494 (11)	0.0327 (10)	0.0819 (14)	−0.0050 (9)	−0.0063 (10)	0.0057 (9)
O2	0.0420 (11)	0.0351 (10)	0.0942 (15)	0.0026 (8)	−0.0064 (10)	−0.0016 (9)
C2	0.0447 (14)	0.0401 (14)	0.0458 (14)	0.0017 (11)	0.0087 (11)	0.0007 (11)
C3	0.0434 (15)	0.0641 (18)	0.0602 (18)	0.0108 (13)	0.0132 (13)	0.0032 (14)
C4	0.0383 (15)	0.074 (2)	0.0647 (19)	0.0074 (14)	−0.0006 (13)	0.0034 (15)
C5	0.0490 (17)	0.0667 (18)	0.0503 (16)	0.0018 (14)	−0.0020 (13)	−0.0028 (14)
C6	0.0437 (15)	0.0478 (15)	0.0494 (15)	0.0000 (11)	0.0072 (12)	−0.0064 (12)
C1	0.0392 (13)	0.0284 (12)	0.0460 (14)	−0.0014 (10)	0.0065 (11)	0.0000 (10)
C7	0.0400 (14)	0.0319 (13)	0.0447 (14)	0.0017 (10)	0.0073 (11)	−0.0015 (10)
C8	0.0348 (13)	0.0384 (13)	0.0372 (13)	0.0022 (10)	0.0050 (10)	−0.0003 (10)
C13	0.0488 (15)	0.0356 (13)	0.0512 (15)	0.0040 (11)	−0.0010 (12)	−0.0019 (11)
C12	0.0435 (15)	0.0531 (17)	0.0591 (17)	0.0150 (13)	−0.0017 (12)	0.0053 (13)
C11	0.0338 (14)	0.0629 (18)	0.0529 (16)	0.0012 (12)	−0.0042 (11)	0.0054 (13)
C10	0.0422 (14)	0.0438 (14)	0.0449 (14)	−0.0070 (11)	0.0024 (11)	0.0035 (11)
C9	0.0353 (12)	0.0365 (13)	0.0323 (12)	0.0002 (10)	0.0058 (9)	0.0017 (10)
C14	0.0401 (14)	0.0352 (13)	0.0410 (14)	−0.0010 (11)	0.0033 (11)	−0.0013 (10)

Geometric parameters (Å, °)

Cl1—C2	1.743 (3)	C5—H5A	0.9300
O1—C7	1.228 (3)	C6—C1	1.388 (3)
N1—C7	1.336 (3)	C6—H6A	0.9300
N1—C8	1.407 (3)	C1—C7	1.496 (3)
N1—H1N1	0.83 (2)	C8—C13	1.390 (3)
O3—C14	1.311 (3)	C8—C9	1.408 (3)
O3—H1O3	0.87 (3)	C13—C12	1.378 (3)
O2—C14	1.213 (3)	C13—H13A	0.9300
C2—C3	1.381 (4)	C12—C11	1.377 (4)
C2—C1	1.393 (3)	C12—H12A	0.9300
C3—C4	1.367 (4)	C11—C10	1.373 (3)
C3—H3A	0.9300	C11—H11A	0.9300
C4—C5	1.372 (4)	C10—C9	1.388 (3)
C4—H4A	0.9300	C10—H10A	0.9300
C5—C6	1.379 (3)	C9—C14	1.486 (3)

C7—N1—C8	129.7 (2)	O1—C7—C1	120.1 (2)
C7—N1—H1N1	118.8 (16)	N1—C7—C1	116.3 (2)
C8—N1—H1N1	111.4 (16)	C13—C8—N1	122.4 (2)
C14—O3—H1O3	109 (2)	C13—C8—C9	119.7 (2)
C3—C2—C1	121.0 (2)	N1—C8—C9	117.9 (2)
C3—C2—Cl1	117.9 (2)	C12—C13—C8	119.9 (2)
C1—C2—Cl1	121.02 (18)	C12—C13—H13A	120.1
C4—C3—C2	119.9 (3)	C8—C13—H13A	120.1
C4—C3—H3A	120.0	C11—C12—C13	121.2 (2)
C2—C3—H3A	120.0	C11—C12—H12A	119.4
C3—C4—C5	120.4 (2)	C13—C12—H12A	119.4
C3—C4—H4A	119.8	C10—C11—C12	118.9 (2)
C5—C4—H4A	119.8	C10—C11—H11A	120.5
C4—C5—C6	119.8 (3)	C12—C11—H11A	120.5
C4—C5—H5A	120.1	C11—C10—C9	122.0 (2)
C6—C5—H5A	120.1	C11—C10—H10A	119.0
C5—C6—C1	121.2 (2)	C9—C10—H10A	119.0
C5—C6—H6A	119.4	C10—C9—C8	118.4 (2)
C1—C6—H6A	119.4	C10—C9—C14	119.6 (2)
C6—C1—C2	117.7 (2)	C8—C9—C14	122.0 (2)
C6—C1—C7	117.2 (2)	O2—C14—O3	122.1 (2)
C2—C1—C7	125.0 (2)	O2—C14—C9	124.7 (2)
O1—C7—N1	123.6 (2)	O3—C14—C9	113.3 (2)
C1—C2—C3—C4	−0.4 (4)	C7—N1—C8—C13	−12.4 (4)
Cl1—C2—C3—C4	−177.5 (2)	C7—N1—C8—C9	168.4 (2)
C2—C3—C4—C5	−0.6 (4)	N1—C8—C13—C12	−179.5 (2)
C3—C4—C5—C6	1.1 (4)	C9—C8—C13—C12	−0.4 (4)
C4—C5—C6—C1	−0.7 (4)	C8—C13—C12—C11	0.2 (4)
C5—C6—C1—C2	−0.2 (4)	C13—C12—C11—C10	0.4 (4)
C5—C6—C1—C7	177.0 (2)	C12—C11—C10—C9	−0.9 (4)
C3—C2—C1—C6	0.8 (4)	C11—C10—C9—C8	0.7 (4)
Cl1—C2—C1—C6	177.76 (18)	C11—C10—C9—C14	−178.0 (2)
C3—C2—C1—C7	−176.2 (2)	C13—C8—C9—C10	0.0 (3)
Cl1—C2—C1—C7	0.8 (3)	N1—C8—C9—C10	179.2 (2)
C8—N1—C7—O1	−2.8 (4)	C13—C8—C9—C14	178.6 (2)
C8—N1—C7—C1	179.4 (2)	N1—C8—C9—C14	−2.2 (3)
C6—C1—C7—O1	−47.6 (3)	C10—C9—C14—O2	−173.0 (2)
C2—C1—C7—O1	129.3 (3)	C8—C9—C14—O2	8.4 (4)
C6—C1—C7—N1	130.3 (2)	C10—C9—C14—O3	7.6 (3)
C2—C1—C7—N1	−52.8 (3)	C8—C9—C14—O3	−171.0 (2)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1N1···Cl1	0.82 (2)	2.82 (2)	3.108 (2)	103 (3)
N1—H1N1···O2	0.82 (2)	1.92 (2)	2.638 (3)	145 (2)
O3—H1O3···O1^{i}	0.87 (3)	1.79 (3)	2.656 (3)	173 (3)
C10—H10A···O3	0.93	2.36	2.694 (3)	101

C13—H13A···O1	0.93	2.29	2.872 (3)	120

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

Fig. 1

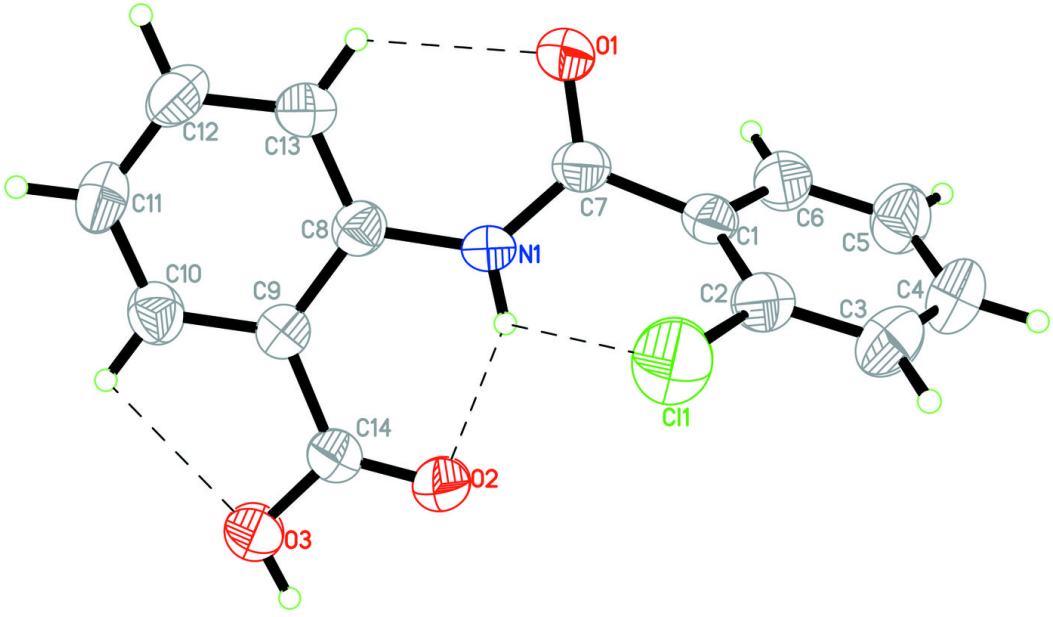

Fig. 2