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2-[(4-Carboxyphenyl)amino]-4-chlorobenzoic acid monohydrate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.050
wR factor = 0.153
Data-to-parameter ratio = 11.9

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $C_{14}H_{12}ClNO_5{\cdot}H_2O$, is a derivative of aminobenzoic acid and forms an extensive hydrogen-bond network within the crystal.

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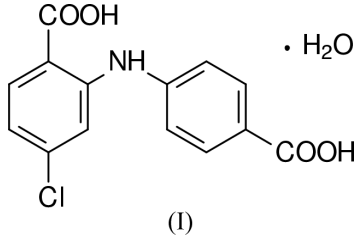
Comment

2-(4-Carboxyphenyl)amino-4-chlorobenzoic acid, used as an intermediate in the synthesis of acridones, crystallized as a monohydrate, (I). Fig. 1 shows the atom-numbering scheme. The aromatic rings are planar and the dihedral angle between the two planes is 26.9 (1)°. An internal N—H···O hydrogen bond with the imino N atom as donor and the carbonyl O atom as acceptor is present. The imino group is not involved in intermolecular interactions, which is a commom feature of related compounds, such as fenamates (Dhanaraj & Vijayan, 1988). The carboxyl groups are, therefore, the only site for intermolecular interactions. The water molecule is involved in three hydrogen bonds of the O—H···O type with another water molecule and the two carboxylic acid groups. The O9 atom of one carboxylic acid group, as donor, forms an intermolecular hydrogen bond with the O18 atom of the other carboxylic acid group acting as acceptor.



(I)

Experimental

The synthesis of (I) was carried out by the reaction of 4-chlorobenzoic acid with 4-aminobenzoic acid, in the presence of K_2CO_3, dimethylformamide and Cu powder. In the recrystallization from ethanol–water (1:1), colourless prisms of the title compound were obtained as the monohydrate.

Crystal data

$C_{14}H_{10}ClNO_4{\cdot}H_2O$
M_r = 309.70
Monoclinic, $P2_1/c$
a = 16.0658 (9) Å
b = 3.9597 (2) Å
c = 21.473 (1) Å
β = 90.619 (6)°
V = 1365.9 (1) Å^3
Z = 4
D_x = 1.506 Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 41 reflections
θ = 4.1–40.3°
μ = 2.69 mm^{-1}
T = 293 K
Prism, colourless
0.64 × 0.10 × 0.03 mm

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Data collection

Siemens *P*4 four-circle diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.553$, $T_{max} = 0.922$
3920 measured reflections
2394 independent reflections
1831 reflections with $F^2 > 2\sigma(F^2)$

$R_{int} = 0.042$
$\theta_{max} = 68.9°$
$h = -19 \rightarrow 1$
$k = -4 \rightarrow 1$
$l = -25 \rightarrow 25$
3 standard reflections every 100 reflections intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.153$
$S = 1.13$
2394 reflections
202 parameters
H atoms treated by a mixture of constrained and independent refinement

$w = 1/[\sigma^2(F_o^2) + (0.0774P)^2 + 0.5504P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.30$ e Å^{-3}
$\Delta\rho_{min} = -0.39$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0035 (6)

Table 1
Selected geometric parameters (Å, °).

Cl4—C4	1.737 (3)	O19—C17	1.326 (3)
O8—C7	1.227 (3)	N10—C2	1.380 (3)
O9—C7	1.310 (4)	N10—C11	1.393 (3)
O18—C17	1.217 (3)		
C2—N10—C11	132.7 (2)	O9—C7—C1	114.8 (2)
N10—C2—C1	119.4 (2)	N10—C11—C12	116.0 (2)
N10—C2—C3	122.4 (2)	N10—C11—C16	125.4 (2)
Cl4—C4—C3	118.8 (2)	O18—C17—C14	125.2 (2)
Cl4—C4—C5	118.41 (19)	O19—C17—C14	113.6 (2)
O8—C7—O9	121.5 (2)	O18—C17—O19	121.2 (2)
O8—C7—C1	123.7 (2)		

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O9—H9···O18^{i}	0.82	1.83	2.654 (3)	177
N10—H10···O8	0.86 (3)	1.89 (3)	2.638 (3)	144 (3)
O19—H19···O20	0.82	1.84	2.659 (3)	172
O20—H20*A*···O20^{ii}	0.83 (4)	1.96 (4)	2.768 (4)	163 (4)
O20—H20*B*···O8^{iii}	0.87 (5)	1.91 (5)	2.733 (3)	158 (4)

Symmetry codes: (i) $x, \frac{1}{2} - y, z - \frac{1}{2}$; (ii) $2 - x, y - \frac{1}{2}, \frac{5}{2} - z$; (iii) $2 - x, -y, 2 - z$.

The positions of the H atoms of the imino group and those of the water molecule were obtained from a difference Fourier synthesis and the coordinates were allowed to refine. The OH groups were found from a circular difference Fourier synthesis and allowed to refine as a 'rotating group'. The remaining H atoms were calculated geometrically and included in the refinement, but were constrained to ride on their parent atoms. The isotropic displacement parameters of the H atoms were fixed to $1.2U_{eq}$ of their parent atoms.

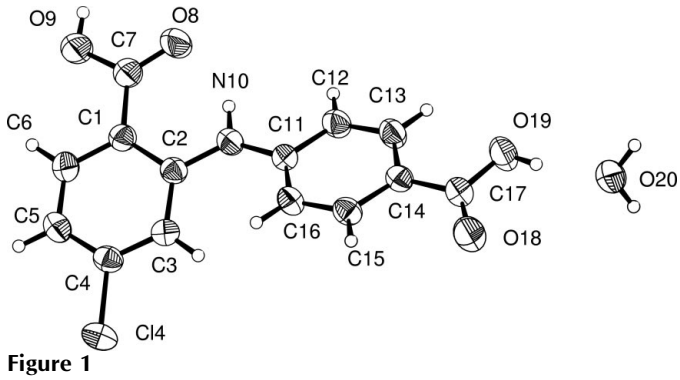

Figure 1
Plot showing the atomic numbering scheme for (I). Displacement ellipsoids are drawn at the 50% probability level for non-H atoms.

Data collection: *XSCANS* (Siemens, 1996); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *DIAMOND* (Bergerhoff, 1996); software used to prepare material for publication: *PARST* (Nardelli, 1983, 1991, 1995) and *PLATON* (Spek, 1990).

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