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4-Chlorobenzamidoxime

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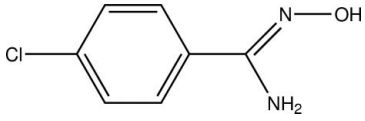
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; R factor = 0.061; wR factor = 0.179; data-to-parameter ratio = 15.4.

In the title compound, $C_7H_7ClN_2O$, an intramolecular N—H···O hydrogen bond helps to establish the conformation. In the crystal structure, intermolecular N—H···O and O—H···N hydrogen bonds occur.

Related literature

For related literature, see: Chertanova *et al.* (1994).



Experimental

Crystal data

$C_7H_7ClN_2O$
M_r = 170.60
Monoclinic, $P2_1/c$
a = 7.9930 (16) Å
b = 12.806 (3) Å
c = 7.6740 (15) Å
β = 90.96 (3)°
V = 785.4 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.43 mm^{-1}
T = 293 (2) K
0.20 × 0.20 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.920, T_{max} = 0.959
1653 measured reflections
1544 independent reflections
978 reflections with $I > 2\sigma(I)$
R_{int} = 0.045
3 standard reflections
every 200 reflections
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.061
$wR(F^2)$ = 0.179
S = 1.09
1544 reflections
100 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1C···N2^{i}	0.82	2.10	2.756 (4)	136
N1—H1B···O1^{ii}	0.86	2.43	3.130 (4)	140
N1—H1A···O1	0.86	2.24	2.551 (4)	101

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

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Siemens, 1996); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2638).

References

Chertanova, L., Pascard, C. & Sheremetev, A. (1994). *Acta Cryst.* B**50**, 708–716.
Enraf–Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
Harms, K. & Wocadlo, S. (1995). *XCAD4*. University of Marburg, Germany.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.
Siemens (1996). *SHELXTL*. Version 5.06. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

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supplementary materials

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4-Chlorobenzamidoxime

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Comment

We report here the crystal structure of the benzonitrile derived title compound, (I), (Fig. 1). The dihedral angle between N2/C7/N1/O and the adjacent bezene ring is 16.30 (11)°. The conformation is stablisied by an intramolecular N—H···O hydrogen bond (Table 1). In the crystal, intermolecular N—H···O and O—H···N interactions occur.

Experimental

4-Chloro-benzonitrile (20 mmol) was dissolved in ethanol (8 ml); hydroxylamine hydrochloride (20 mmol) was dissolved in ethanol (6 ml) and potassium carbonate (10 mmol) was dissolved in water (10 ml). The three solutions were mixed and the resulting mixture was refluxed for 24 h. After cooling and filtrating, the crude title compound was obtained and purified by crystallizing from a mixture of ethanol (6 ml) and water (2 ml). Colourless blocks of (I) were obstained by slow evaporation of an ethanol solution. ^{1}H NMR ($CDCl_3$, δ, p.p.m.): 7.31–7.32 (m, 2H), 7.62–7.64 (m, 2H), 2.28 (s, 1H), 2.06 (s, 2H).

Refinement

The H atoms were geometrically placed (C—H = 0.93–0.97 Å, N—H = 0.86 Å, O—H = 0.82 Å) and refined as riding with U_{iso}(H) = 1.2 or 1.5U_{eq} of the carrier atom.

Figures

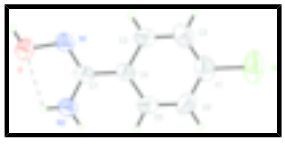

Fig. 1. A view of the molecular structure of (I), showing displacement ellipsoids at the 30% probability level. The dashed line indicates the N—H···O hydrogen bond.

4-Chlorobenzamidoxime

Crystal data

$C_7H_7ClN_2O$	F_{000} = 352
M_r = 170.60	D_x = 1.443 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 25 reflections
a = 7.9930 (16) Å	θ = 10–13°
b = 12.806 (3) Å	μ = 0.43 mm^{-1}
c = 7.6740 (15) Å	T = 293 (2) K
β = 90.96 (3)°	Block, colourless

V = 785.4 (3) Å^3	0.20 × 0.20 × 0.10 mm
Z = 4	

Data collection

Nonius CAD4 diffractometer	R_{int} = 0.045
Radiation source: fine-focus sealed tube	θ_{max} = 26.0º
Monochromator: graphite	θ_{min} = 2.6º
T = 293(2) K	h = −9→9
ω/2θ scans	k = 0→15
Absorption correction: ψ scan (North *et al.*, 1968)	l = 0→9
T_{min} = 0.920, T_{max} = 0.959	3 standard reflections
1653 measured reflections	every 200 reflections
1544 independent reflections	intensity decay: none
978 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.061	H-atom parameters constrained
$wR(F^2)$ = 0.179	$w = 1/[\sigma^2(F_o^2) + (0.0834P)^2 + 0.2545P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.09	$(\Delta/\sigma)_{max} < 0.001$
1544 reflections	$\Delta\rho_{max}$ = 0.39 e Å^{-3}
100 parameters	$\Delta\rho_{min}$ = −0.27 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 $F^2 > 2\text{sigma}(F^2)$ 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}
Cl	0.45717 (14)	0.11469 (9)	0.18287 (15)	0.0774 (5)

O1	1.1252 (3)	0.09855 (19)	0.9789 (3)	0.0563 (7)
H1C	1.1124	0.0652	1.0689	0.084*
N1	1.1427 (3)	0.1907 (2)	0.6852 (4)	0.0517 (8)
H1A	1.2254	0.1929	0.7582	0.062*
H1B	1.1478	0.2242	0.5883	0.062*
N2	0.9851 (4)	0.0818 (2)	0.8643 (4)	0.0472 (7)
C1	0.6165 (5)	0.1198 (3)	0.3401 (5)	0.0533 (10)
C2	0.7787 (4)	0.1202 (3)	0.2888 (5)	0.0503 (9)
H2B	0.8045	0.1171	0.1712	0.060*
C3	0.9031 (5)	0.1252 (3)	0.4131 (4)	0.0490 (9)
H3A	1.0139	0.1254	0.3779	0.059*
C4	0.8697 (4)	0.1299 (2)	0.5918 (4)	0.0403 (8)
C5	0.7012 (4)	0.1283 (3)	0.6391 (5)	0.0507 (9)
H5A	0.6740	0.1308	0.7563	0.061*
C6	0.5760 (5)	0.1232 (3)	0.5154 (5)	0.0565 (10)
H6A	0.4647	0.1219	0.5486	0.068*
C7	1.0036 (4)	0.1342 (3)	0.7230 (4)	0.0436 (8)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl	0.0770 (8)	0.0708 (8)	0.0837 (9)	−0.0060 (6)	−0.0208 (6)	0.0085 (6)
O1	0.0697 (17)	0.0537 (16)	0.0454 (14)	−0.0116 (13)	0.0017 (12)	0.0051 (11)
N1	0.0437 (16)	0.063 (2)	0.0482 (16)	−0.0116 (14)	0.0011 (12)	0.0066 (14)
N2	0.0613 (18)	0.0428 (16)	0.0377 (15)	−0.0034 (14)	0.0069 (13)	0.0029 (13)
C1	0.062 (2)	0.0389 (19)	0.059 (2)	0.0016 (17)	−0.0042 (18)	0.0020 (17)
C2	0.060 (2)	0.051 (2)	0.0407 (19)	0.0009 (17)	0.0009 (16)	−0.0010 (16)
C3	0.052 (2)	0.047 (2)	0.048 (2)	0.0031 (16)	0.0121 (15)	−0.0020 (16)
C4	0.0494 (18)	0.0310 (17)	0.0407 (18)	0.0001 (14)	0.0049 (14)	0.0033 (13)
C5	0.053 (2)	0.050 (2)	0.050 (2)	−0.0008 (17)	0.0153 (16)	−0.0027 (17)
C6	0.052 (2)	0.047 (2)	0.072 (3)	−0.0026 (17)	0.0166 (18)	−0.0060 (19)
C7	0.0502 (19)	0.0400 (18)	0.0409 (19)	0.0065 (15)	0.0139 (15)	−0.0044 (15)

Geometric parameters (Å, °)

Cl—C1	1.741 (4)	C2—C3	1.368 (5)
O1—N2	1.428 (4)	C2—H2B	0.9300
O1—H1C	0.8200	C3—C4	1.402 (5)
N1—C7	1.362 (4)	C3—H3A	0.9300
N1—H1A	0.8600	C4—C5	1.401 (5)
N1—H1B	0.8600	C4—C7	1.458 (5)
N2—C7	1.285 (4)	C5—C6	1.369 (5)
C1—C2	1.361 (5)	C5—H5A	0.9300
C1—C6	1.390 (5)	C6—H6A	0.9300
N2—O1—H1C	109.5	C4—C3—H3A	118.8
C7—N1—H1A	120.0	C5—C4—C3	116.9 (3)
C7—N1—H1B	120.0	C5—C4—C7	121.3 (3)
H1A—N1—H1B	120.0	C3—C4—C7	121.8 (3)

C7—N2—O1	110.0 (3)	C6—C5—C4	121.1 (3)
C2—C1—C6	121.2 (3)	C6—C5—H5A	119.5
C2—C1—Cl	119.3 (3)	C4—C5—H5A	119.5
C6—C1—Cl	119.5 (3)	C5—C6—C1	119.6 (4)
C1—C2—C3	118.9 (3)	C5—C6—H6A	120.2
C1—C2—H2B	120.6	C1—C6—H6A	120.2
C3—C2—H2B	120.6	N2—C7—N1	124.2 (3)
C2—C3—C4	122.4 (3)	N2—C7—C4	118.0 (3)
C2—C3—H3A	118.8	N1—C7—C4	117.7 (3)
C6—C1—C2—C3	−0.8 (5)	C2—C1—C6—C5	0.9 (5)
Cl—C1—C2—C3	179.4 (3)	Cl—C1—C6—C5	−179.3 (3)
C1—C2—C3—C4	0.0 (5)	O1—N2—C7—N1	4.4 (4)
C2—C3—C4—C5	0.6 (5)	O1—N2—C7—C4	−177.9 (3)
C2—C3—C4—C7	179.4 (3)	C5—C4—C7—N2	38.0 (5)
C3—C4—C5—C6	−0.5 (5)	C3—C4—C7—N2	−140.7 (3)
C7—C4—C5—C6	−179.3 (3)	C5—C4—C7—N1	−144.2 (3)
C4—C5—C6—C1	−0.2 (5)	C3—C4—C7—N1	37.1 (5)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1C···N2^{i}	0.82	2.10	2.756 (4)	136
N1—H1B···O1^{ii}	0.86	2.43	3.130 (4)	140
N1—H1A···O1	0.86	2.24	2.551 (4)	101

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

Fig. 1