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N-(4-Chlorophenyl)-*N*-(4,5-dihydro-1*H*-imidazol-2-yl)amine formamide solvate

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.003 Å
R factor = 0.042
wR factor = 0.111
Data-to-parameter ratio = 18.3

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

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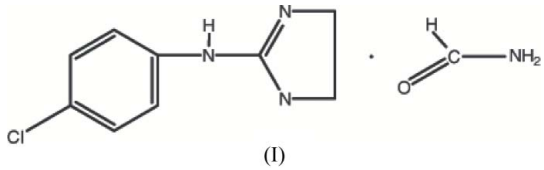
In the title compound, $C_9H_{10}ClN_3\cdot HC(O)NH_2$, the benzene and imidazole rings make a dihedral angle of 43.3 (1)°. In the crystal structure, molecules are linked through weak intermolecular N—H···O, N—H···N and C—H···N hydrogen bonds, forming a three-dimensional framework.

Comment

N-Mono- and *N*,*N*-disubstituted dithiocarbamate derivatives have shown antibacterial, antiviral and antifungal activities (Kaplancikli *et al.*, 2004). 2-Arylamino-2-imidazolines have an interesting chemistry and are effective pharmacophores in medicinal chemistry. 2-Arylamino-2-imidazolines, in particular 2,6-dichlorophenylamino-2-imidazoline (*clonidine*), have a pronounced hypotensive action which is coupled with a sedative action (Saczewski *et al.*, 2003).



(I)

The molecular structure of the title compound, (I), is shown in Fig. 1. The bond lengths and angles are within normal ranges and compare well with those reported in similar compounds (Yıldırım, Akkurt, Danışman *et al.*, 2006;Yıldırım, Akkurt, Genç *et al.*, 2006; Öztürk *et al.*, 2004; Akkurt *et al.*,

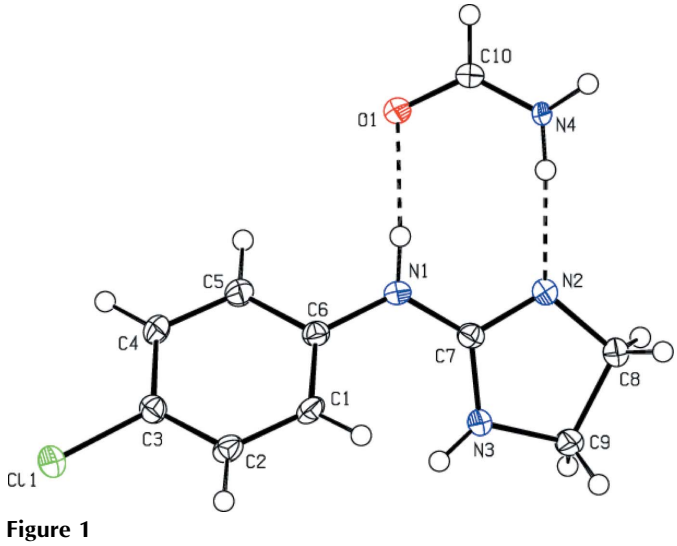


Figure 1
The molecular structure of the title compound with the atom-numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 50% probability level. Dashed lines indicate hydrogen bonds.

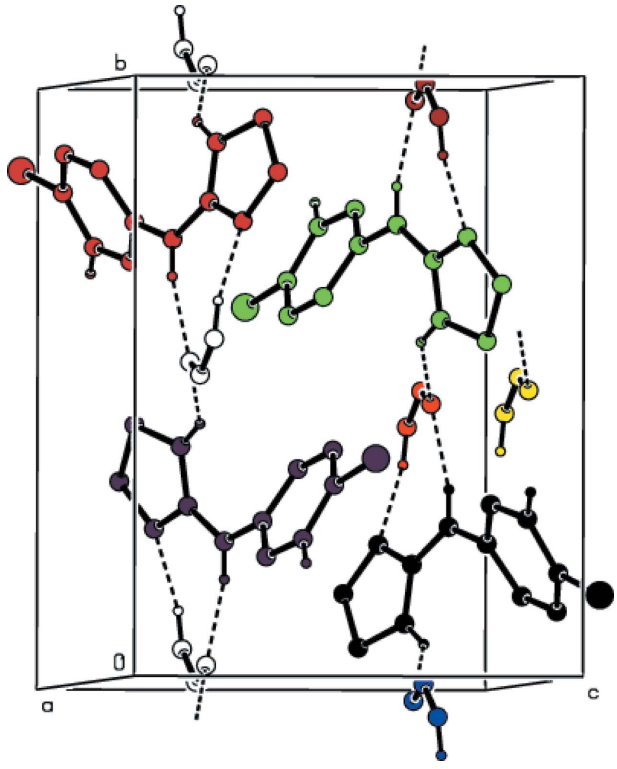


Figure 2
View of the packing and hydrogen bonding of (I). Dashed lines indicate hydrogen bonds. For clarity, H atoms not involved hydrogen bonding have been omitted.

2004). The lengths of the two C—N bonds [C6—N1 = 1.415 (2) and C7—N1 = 1.342 (2) Å] are consistent with those found in related compounds (Allen *et al.*, 1987). The main molecule of the title compound, (I), is non-planar, the substituted benzene ring and the imidazole ring making a dihedral angle of 43.3 (1)°.

In the crystal structure, molecules of (I) are linked into a three-dimensional framework by intermolecular N—H···O, N—H···N and C—H···N hydrogen bonds (Fig. 2 and Table 1). The hydrogen bonds reflect the fact that the proton at N3 is the most acidic one, which is also in agreement with the molecular structures of deprotonated 2-arylamino-2-imidazolines (Carpy *et al.*, 1980; Ghosh *et al.*, 1992).

Experimental

The syntheses of dimethyl *N*-(4-chlorophenyl)dithioimidocarbonate and *N*-(4-chlorophenyl)-*N*-(4,5-dihydro-1*H*-imidazol-2-yl)amine were performed according to literature procedures (Genc & Servi, 2005; Servi *et al.*, 2005). A solution of dimethyl *N*-(4-chlorophenyl)-dithioimidocarbonate (0.04 mol) in DMF (15 ml) was added to a solution of ethylenediamine (5.4 ml, d = 0.897 Mg m^{-3}, 0.08 mol) in DMF (15 ml), with stirring, at room temperature. The reaction mixture was refluxed at 383 K for approximatly 6–8 h, cooled, then added to ice-cold water. The resulting solid was washed with water and dried. The crude products were recrystallized from a formamide solution. Block-shaped colourless crystals of (I) suitable for X-ray analysis were obtained after six months by slow evaporation of formamide at room temperature (m.p. 420–422 K; yield 71%). IR (KBr, cm^{-1}, ν_{max}): 3284 (imidazoline N–H stretching), 3245 (N–H stretching), 1681 (C=N stretching), 1594 (N–H bending); ^{1}H NMR (200 MHz, $CDCl_3$): δ 3.67 (*s*, 1H, NH), 3.76 (*s*, 4H, imidazoline CH_2), 6.24 (broad peak, 1H, imidazoline NH), 7.05–7.40 (*m*, 4H, Ar—H).

Crystal data

$C_9H_{10}ClN_3\cdot CH_3NO$
M_r = 240.69
Monoclinic, $P2_1/c$
a = 8.5840 (5) Å
b = 13.2740 (4) Å
c = 9.9080 (7) Å
β = 105.789 (5)°
V = 1086.4 (1) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.34 mm^{-1}
T = 150 K
0.32 × 0.09 × 0.07 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2002)
T_{min} = 0.900, T_{max} = 0.977
15967 measured reflections
2655 independent reflections
2034 reflections with $I > 2\sigma(I)$
R_{int} = 0.044

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.111
S = 1.01
2655 reflections
145 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.51 e Å^{-3}
$\Delta\rho_{min}$ = −0.57 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O1	0.86	1.94	2.7759 (19)	164
N3—H3···O1^{i}	0.86	2.05	2.793 (2)	144
N4—H4A···N2	0.86	1.97	2.809 (2)	166
C4—H4···N4^{ii}	0.93	2.56	3.457 (2)	162

Symmetry codes: (i) $-x+1, y-\frac{1}{2}, -z+\frac{3}{2}$; (ii) $x-1, y, z$.

H atoms were positioned geometrically and were refined as riding, with C—H = 0.93–0.96 and N—H = 0.86 Å, and with U_{iso}(H) = 1.2U_{eq}(C,N).

Data collection: *COLLECT* (Nonius, 1999); cell refinement: *EVALCCD* (Duisenberg *et al.*, 2003); data reduction: *EVALCCD* and*SADABS* (Sheldrick, 2002); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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