Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N^1-(2,4-Dichloroanilino)acetamidine

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Received 21 August 2006
Accepted 2 September 2006

Key indicators

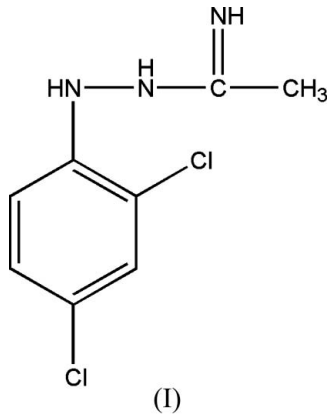
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.007 Å
R factor = 0.070
wR factor = 0.214
Data-to-parameter ratio = 16.4

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

The title compound, $C_8H_9Cl_2N_3$, was synthesized by the reaction of ethyl acetimidate hydrochloride and 2,4-dichlorophenylhydrazine. Both intramolecular N—H···Cl and intermolecular N—H···N hydrogen-bond interactions are present, resulting in a one-dimensional supramolecular chain structure.

Comment

1-Aryl-1,2,4-triazolin-5-one derivatives exhibit herbicidal activity (Mitsuru *et al.*, 1982), for example, with soybeans (Keifer & Tymonko, 1990), by preventing or destroying undesired plant growth (Theodoridis, 1991). The title compound, (I), is an important intermediate in the preparation of triazolinones (Goudar, 2003) and shows good photoconductivity (Chi *et al.*, 2001). We report here the crystal structure of the title compound, (I).



(I)

In the molecule of (I), all bond lengths and angles are in normal ranges. The molecular structure of (I) is shown in Fig. 1, where the dashed line indicates an intramolecular N—H···Cl hydrogen bond (Table 1). The dihedral angle between the ring plane and the N2/C7/C8/N3 plane is 67.0 (2)°.

In the crystal structure, intermolecular N—H···N hydrogen bonds (Fig. 2 and Table 1) link the molecules into one-dimensional supramolecular chains.

Experimental

A stirred solution of 13.6 g (0.11 mol) of ethyl acetimidate hydrochloride and 13.2 g (0.13 mol) of triethylamine in 100 g of dichloromethane was cooled to about 273 K for 5 min and 17.7 g (0.10 mol) of 2,4-dichlorophenylhydrazine was added. When the addition was complete, the reaction mixture was stirred at 273 K for about 1 h; it was then allowed to warm to ambient temperature and stirred for about 2 h. The reaction mixture was then washed with about 20 ml

water and dried with magnesium sulfate. The mixture was filtered and the filtrate was concentrated under reduced pressure. The residue was triturated with 20 ml of hexane, and the resultant solid was collected by filtration. The solid was washed with 50 ml of hexane and dried, yielding 20.4 g of 2-(2,4-dichlorophenyl)hydrazidethaneimidic acid. The compound was crystallized twice by slow evaporation of an ethanol solution and crystals suitable for X-ray diffraction were obtained.

Crystal data

$C_8H_9Cl_2N_3$
M_r = 218.08
Monoclinic, $C2/c$
a = 22.832 (5) Å
b = 4.756 (1) Å
c = 20.515 (4) Å
β = 115.74 (3)°
V = 2006.7 (9) Å^3
Z = 8
D_x = 1.444 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.60 mm^{-1}
T = 293 (2) K
Plate, colourless
0.40 × 0.30 × 0.10 mm

Data collection

Enraf-Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.795, T_{max} = 0.942
2013 measured reflections
1966 independent reflections
1178 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
θ_{max} = 26.0°
3 standard reflections
every 200 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.070
$wR(F^2)$ = 0.214
S = 1.04
1966 reflections
120 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0946P)^2 + 3.9518P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.027
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0105 (16)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···Cl1	0.90	2.53	2.963 (4)	110
N2—H2···N3^{i}	0.86	2.13	2.788 (5)	134

Symmetry code: (i) $x, y-1, z$.

The H atoms attached to N were located in a difference map and then refined as riding (N—H = 0.83–0.90 Å). Carbon-bound H atoms were placed in idealized positions and refined as riding, with C—H = 0.93–0.96 Å; for all H atoms, U_{iso}(H) = 1.2 or 1.5 times U_{eq}(carrier atom).

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: *SHELXL*.

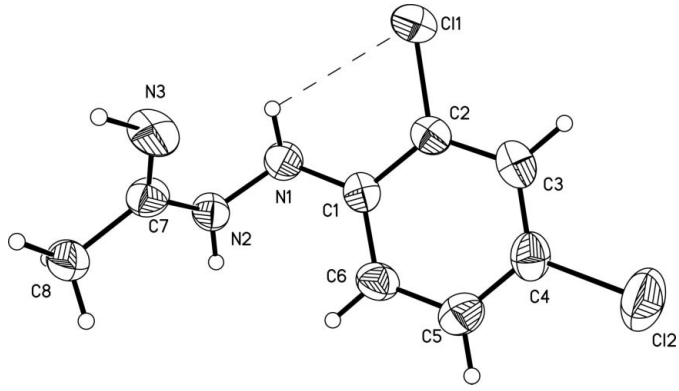


Figure 1
A view of the molecular structure, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. The dashed line indicates a hydrogen bond.

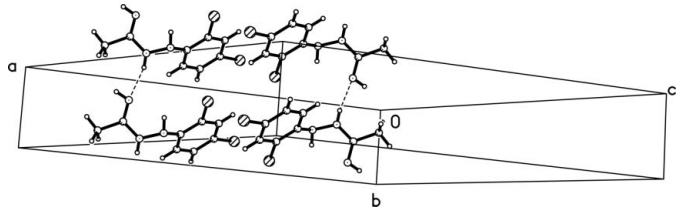


Figure 2
A partial packing diagram of (I). Hydrogen bonds are shown as dashed lines.

The authors thank the Center for Testing and Analysis, Nanjing University, for support.

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