Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

2-Chloro-*N*-(5-chloro-4-fluoro-2-nitrophenyl)-acetamide

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Received 29 July 2004
Accepted 2 September 2004
Online 11 September 2004

Key indicators

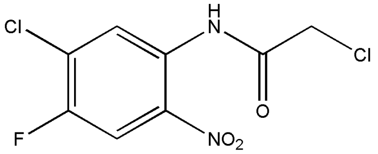
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.035
wR factor = 0.095
Data-to-parameter ratio = 12.4

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

The acetamide fragment and benzene ring in the title compound, $C_8H_5Cl_2FN_2O_3$, are almost coplanar, excluding the H atoms of the chloromethyl group. In the crystal structure, there are two intramolecular hydrogen bonds, one between the amide N atom and a nitro O atom, [N—O = 2.611 (9) Å and N—H···O = 135 (2)°], the other between the amide N atom and the Cl atom of the chloromethyl group [N—Cl = 2.937 (2) Å and N—H···Cl = 120 (2)°].

Comment

The title compound, (I) (Fig. 1), is one of the key intermediates in the preparation of quinoxalinones, which are important precursors for the synthesis of many pharmaceuticals and pesticides (Sakata *et al.*, 1985; Ura & Sakata, 1990). The benzene ring and the acetamide fragment are almost coplanar, excluding the H atoms of the chloromethyl group; the maximum deviation from the least-squares plane is 0.005 Å for atom O1. The bond lengths and angles are unexceptional.


(I)

The structure is stabilized by two intramolecular hydrogen bonds, one between the amide N atom and a nitro O atom (this atom O2 is disordered over two positions O2 and O2′), the other between the amide N atom and the Cl atom of the chloromethyl group (Table 1). No intermolecular hydrogen bond is found in the crystal structure.

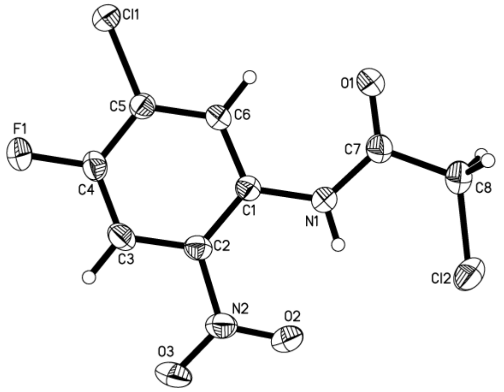


Figure 1
The molecular structure of the title compound, with displacement ellipsoids drawn at the 50% probability level. Only the major disorder component is shown.

Experimental

The title compound was prepared from 4-chloro-3-fluorophenylamine and chloroacetyl chloride through acylation and nitration according to the method described by Li *et al.* (2004). Single crystals of the title compound suitable for X-ray analysis were grown by slow evaporation of an ethanol solution at 298 K.

Crystal data

$C_8H_5Cl_2FN_2O_3$
$M_r = 267.04$
Monoclinic, $P2_1/n$
$a = 11.193\ (3)$ Å
$b = 6.4489\ (19)$ Å
$c = 14.427\ (4)$ Å
$\beta = 93.139\ (5)^\circ$
$V = 1039.8\ (5)$ Å^3
$Z = 4$
$D_x = 1.706$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 860 reflections
$\theta = 3.5–26.2^\circ$
$\mu = 0.63$ mm^{-1}
$T = 293\ (2)$ K
Prism, yellow
0.40 × 0.24 × 0.20 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
$T_{\min} = 0.826$, $T_{\max} = 0.881$
5701 measured reflections
2104 independent reflections
1618 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.024$
$\theta_{\max} = 26.4^\circ$
$h = -13 \rightarrow 12$
$k = -7 \rightarrow 8$
$l = -7 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.095$
$S = 1.02$
2104 reflections
169 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0468P)^2 + 0.356P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.36$ e Å^{-3}
$\Delta\rho_{\min} = -0.40$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.031 (3)

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

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
N1—H1⋯Cl2	0.85 (1)	2.42 (2)	2.937 (2)	120 (2)
N1—H1⋯O2	0.85 (1)	1.94 (2)	2.611 (9)	135 (2)
N1—H1⋯O2′	0.85 (1)	2.10 (4)	2.766 (4)	135 (2)

Atom H1 attached to N1 was located in a difference Fourier map and refined freely. All other H atoms were positioned geometrically and refined in the riding-model approximation. C—H values were set at 0.97 and 0.93 Å for atoms C8 and C3–C6, respectively; $U_{\rm iso}$(H) values were constrained to be $1.5U_{\rm eq}$(carrier) for methyl H and $1.2U_{\rm eq}$(carrier) for the other H atoms. Both O2 and O3 attached to N2 are disordered over two positions. The values of the refined occupancy factors for O2/O3 and O2′/O3′ are 0.81 (3) and 0.19 (3), respectively. The N—O bond lengths were restrained to 1.21 (1) Å.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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