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Key indicators

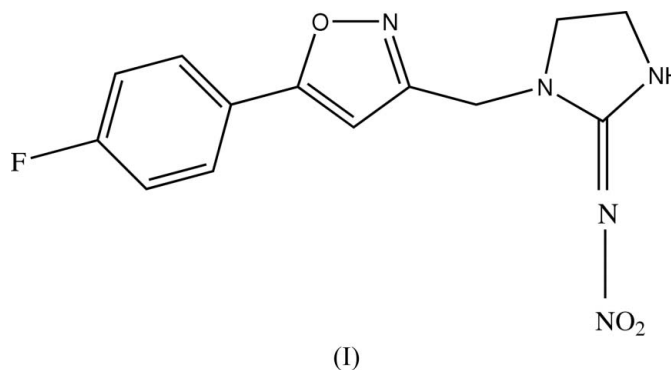
Single-crystal X-ray study
T = 273 K
Mean $\sigma(C-C)$ = 0.002 Å
R factor = 0.034
wR factor = 0.095
Data-to-parameter ratio = 11.6For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.1-[5-(4-Fluorophenyl)-1,2-oxazol-3-ylmethyl]-
2-nitroimidazolidine

In the title compound, $C_{13}H_{12}FN_5O_3$, the five-membered imidazolidine ring adopts an envelope conformation. The molecular structure is stabilized by an intramolecular N—H···O hydrogen bond. The crystal structure shows C—H···O and C—H···N interactions.

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Comment

Isoxazoles play an important role in the synthesis of many complex natural products. They are often used as pharmacophores in medicinal chemistry (Aicher *et al.*, 1998). We recently reported the crystal structures of three substituted imidazolidine derivatives (Ravikumar *et al.*, 2006). In continuation of the structural elucidation of this series of compounds, the crystal structure determination of the title compound, (I), was undertaken and the results are presented here.



The bond lengths and angles of (I) are comparable to those in similar structures (Kyziol *et al.*, 2000; Xu *et al.*, 2004). The benzene ring is inclined at an angle of 9.6 (1)° to the plane of the isoxazole ring.

The imidazolidine ring adopts an envelope conformation with atom C5 displaced by 0.279 (2) Å from the plane of the other four atoms, and is almost perpendicular to both the isoxazole and the phenyl rings [dihedral angles 85.6 (1) and 79.4 (1)°, respectively]. The dihedral angle between the nitrimino group (N4/N5/O2/O3) and the imidazolidine ring (N2/C5/C6/N3/C7) is 8.3 (1)°.

An intramolecular N—H···O hydrogen bond is observed between atom N3 of the imidazolidine ring and atom O3 of the nitrimino group, forming a six-membered ring [Ettér symbol *S*(6); Bernstein *et al.*, 1995]. C—H···O and C—H···N interactions are also present in the crystal packing (Table 1). In the crystal structure, centrosymmetric π – π stacking $Cg1 \cdots Cg2^{iii}$ [3.740 (1) Å; symmetry code: (iii) 1 – x, –y, 1 – z; *Cg1* is the

centroid of the isoxazole ring and $Cg2$ is the centroid of the fluorophenyl ring] is also observed.

Experimental

The title compound was synthesized as follows: 0.5 g (0.002 mol) of isoxazolyl methyl chloride, 0.67 g (0.005 mol) of 2-nitroiminoimidazole and 0.33 g (0.002 mol) of K_2CO_3 in 10 ml acetonitrile were refluxed for 3 h. Completion of the reaction was monitored by TLC and was indicated by the disappearance of the isoxazolyl methyl chloride spot and the appearance of a new polar spot which corresponded to the title compound. After completion of the reaction acetonitrile was removed *in vacuo* and the crude product was obtained as a solid, which was washed with water (2×20 ml) and recrystallized from methanol to afford colorless crystals (yield 62%).

Crystal data

$C_{13}H_{12}FN_5O_3$	$Z = 4$
$M_r = 305.28$	$D_x = 1.516 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation
$a = 7.8575$ (6) Å	$\mu = 0.12 \text{ mm}^{-1}$
$b = 15.6860$ (12) Å	$T = 273$ (2) K
$c = 11.4098$ (9) Å	Block, colorless
$\beta = 107.945$ (1)°	$0.20 \times 0.17 \times 0.12 \text{ mm}$
$V = 1337.88$ (18) Å ³	

Data collection

Bruker SMART APEX CCD area-detector diffractometer	2359 independent reflections
ω scans	2179 reflections with $I > 2\sigma(I)$
Absorption correction: none	$R_{\text{int}} = 0.020$
12586 measured reflections	$\theta_{\text{max}} = 25.0^\circ$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0516P)^2 + 0.2286P]$
$R[F^2 > 2\sigma(F^2)] = 0.034$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.095$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.07$	$\Delta\rho_{\text{max}} = 0.15 \text{ e } \text{Å}^{-3}$
2359 reflections	$\Delta\rho_{\text{min}} = -0.17 \text{ e } \text{Å}^{-3}$
203 parameters	
H atoms treated by a mixture of independent and constrained refinement	

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

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$N3-H3N \cdots O3$	0.856 (18)	2.070 (17)	2.594 (2)	119 (1)
$C2-H2 \cdots O2^i$	0.93	2.54	3.368 (2)	149
$C2-H2 \cdots O3^i$	0.93	2.56	3.236 (2)	130
$C6-H6A \cdots O1^{ii}$	0.97	2.50	3.346 (2)	146
$C6-H6A \cdots N1^{ii}$	0.97	2.60	3.488 (2)	153

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

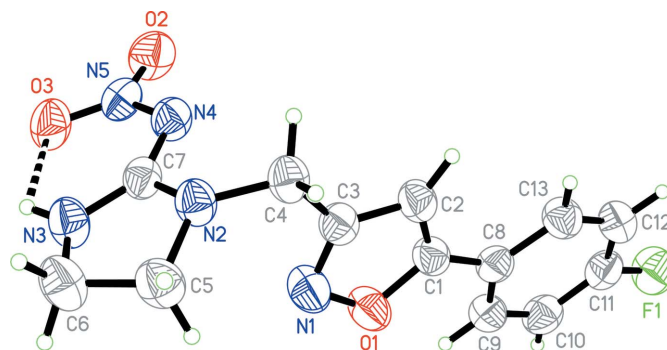


Figure 1

A view of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii. The intramolecular hydrogen bond is drawn as a dashed line.

The H atom on N3 was located in a difference density map and refined freely. All other H atoms were positioned geometrically and treated as riding, with C–H distances in the range 0.93–0.97 Å and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL/PC* (Sheldrick, 1990) and *MERCURY* (Bruno *et al.*, 2002); software used to prepare material for publication: *SHELXL97*.

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