Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-[7-Fluoro-3,4-dihydro-4-(3-iodoprop-2-ynyl)-3-oxo-2*H*-1,4-benzoxazin-6-yl]-4,5,6,7-tetrahydro-2*H*-isoindole-1,3-dione

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.007 Å
R factor = 0.030
wR factor = 0.060
Data-to-parameter ratio = 15.4

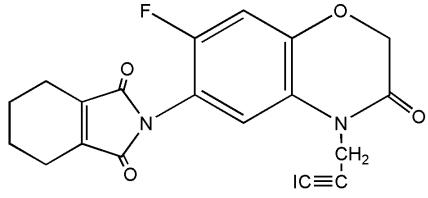
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 18 May 2004
Accepted 5 July 2004
Online 17 July 2004

The title compound, $C_{19}H_{14}FIN_2O_4$, known as a protox inhibitor, was synthesized from flumioxazin. The cyclohexene ring in the molecule is in a distorted chair conformation. The molecules are connected into two-dimensional layers by C=O···I interactions.

Comment

Flumioxazin is a selective post-emergence herbicide which inhibits protoporphyrinogen oxidase (Yoshida *et al.*, 1991). The title compound, (I), is a new flumioxazin analogue. It exhibits excellent control for broadleaf weeds at low dosage and no damage at high dosage for monocotyledon plants, such as wheat and corn (Hou *et al.*, 2003).



(I)

The molecular structure of (I) is illustrated in Fig. 1. The cyclohexene ring in the molecule has a distorted chair conformation. In the crystal structure of (I), screw-related molecules are linked by C=O···I interactions to form layers parallel to the *bc* plane (Fig. 2). Such interactions have been reported previously (Novoselov *et al.*, 1994; de Faria *et al.*, 1999).

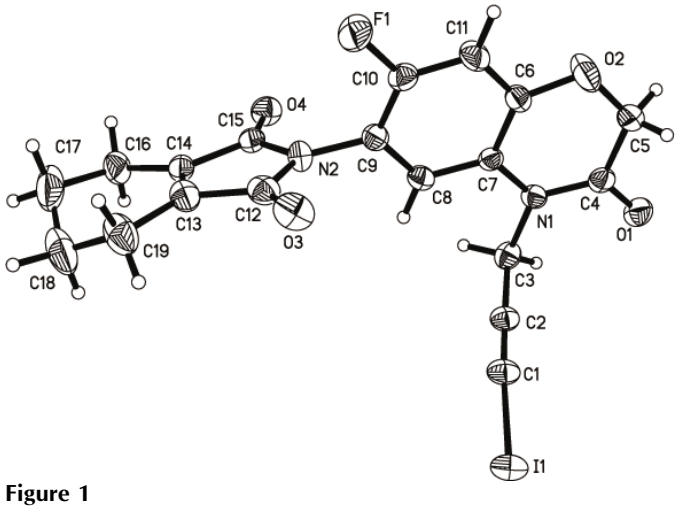


Figure 1
The molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

Experimental

Flumioxazin (10.0 mmol) was mixed with *N*-iodosuccinimide (15.0 mmol) in acetone (10.0 ml) in the presence of an $AgNO_3$ catalyst (0.1 g) at ambient temperature for 24 h (Nagano *et al.*, 1987) (yield 78%). The reaction mixture was poured into iced water. The resulting precipitate was filtered off, washed with water and dried. The crude product was purified by column chromatography (silica gel, petroleum ether–acetone 5:1) to give the title compound, (I) (m.p. 555–556 K). ^{1}H NMR ($CDCl_3$, δ, p.p.m): 1.66 (4H, *m*), 1.99 (4H, *m*), 4.62 (1H, *s*), 4.96 (2H, *s*), 6.76 (1H, *s*), 7.42 (1H, *s*). Compound (I) (20 mg) was dissolved in ethyl acetate (20 ml) for recrystallization. Single crystals of (I), suitable for X-ray analysis, were grown by natural evaporation of the solvent.

Crystal data

$C_{19}H_{14}FIN_2O_4$
M_r = 480.22
Orthorhombic, *Pca*2
a = 9.613 (4) Å
b = 11.759 (5) Å
c = 16.571 (7) Å
V = 1873.1 (14) Å^3
Z = 4
D_x = 1.703 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 908 reflections
θ = 3.7–24.4°
μ = 1.75 mm^{-1}
T = 293 (2) K
Block, colourless
0.42 × 0.38 × 0.24 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.472, T_{max} = 0.658
10 073 measured reflections
3752 independent reflections
2828 reflections with $I > 2\sigma(I)$
R_{int} = 0.033
θ_{max} = 26.4°
$h = -11 \rightarrow 12$
$k = -14 \rightarrow 10$
$l = -20 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.060
S = 0.99
3752 reflections
244 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0247P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.41 e Å^{-3}
Absolute structure: Flack (1983), 1769 Friedel pairs
Flack parameter = 0.01 (2)

H atoms were positioned geometrically, with C—H = 0.93–0.97 Å, and were refined in a riding model, with U_{iso}(H) = 1.2U_{eq}(C).

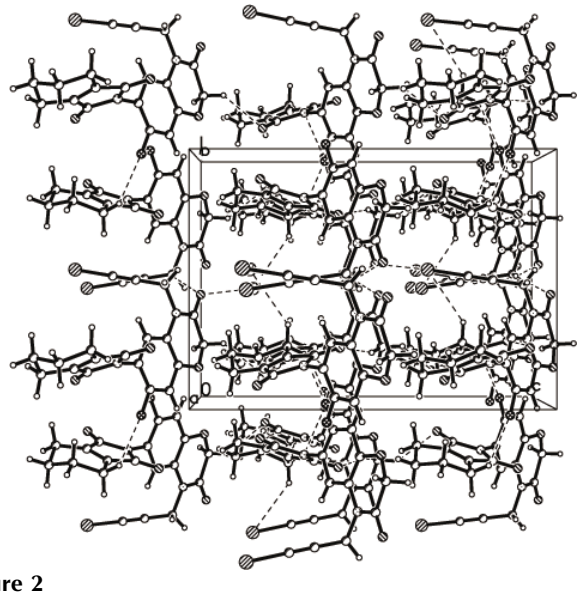


Figure 2
The crystal structure of (I), viewed along the *a* axis. The dashed lines indicate C═O···I interactions.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); 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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