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3-Chloro-*N*-[7-fluoro-3-oxo-4-(prop-2-ynyl)-3,4-dihydro-2*H*-1,4-benzoxazin-6-yl]-2,2-dimethylpropionamide

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Key indicators

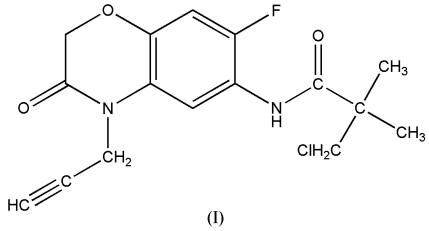
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.037
wR factor = 0.103
Data-to-parameter ratio = 13.4

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

The title compound, $C_{16}H_{16}ClFN_2O_3$, known as a protox inhibitor, was synthesized from 7-fluoro-2*H*-benz[1,4]oxazin-3(4*H*)-one. The bond lengths and angles are unexceptional and the heterocyclic ring adopts a screw-boat conformation.

Comment

The title compound, (I), is a potent inhibitor of the plant enzyme protoporphyrinogen oxidase (protox; Birchfield *et al.*, 1997). Low dosage provides excellent control for broadleaf weeds and, at high dosage, there is no damage for monocotyledonous plants such as wheat and corn (Chamilleri *et al.*, 1988).



(I)

The molecular structure is illustrated in Fig. 1. The conformation of the six-membered heterocyclic ring is close to screw-boat, with atoms C1 and C2 out of the plane of the remaining four atoms by 0.352 (4) and 0.726 (3) Å, respectively. The bond lengths and angles in this ring agree well with those in a related compound (Karolak-Wojciechowska *et al.*, 2001), although the latter adopts a twist-chair conformation.

Experimental

7-Fluoro-2*H*-benz[1,4]oxazin-3(4*H*)-one was prepared according to a previously published method (Terni *et al.*, 1988). It (100 mmol) was then treated with 3-bromoprop-1-yne (100 mmol) to obtain 7-fluoro-

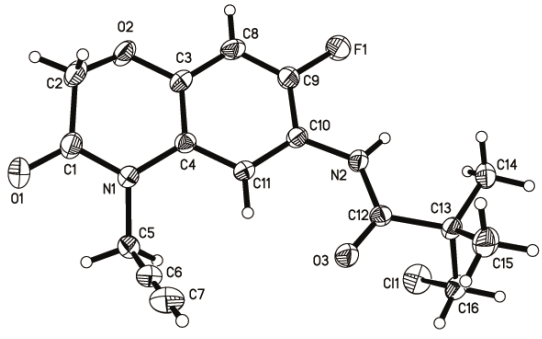


Figure 1
The molecular structure of (I), with displacement ellipsoids drawn at the 30% probability level.

4-(prop-2-ynyl)-2*H*-benz[*b*][1,4]oxazin-3(4*H*)-one. After nitration and reduction, the amide was obtained and mixed with pivaloyl chloride (100 mmol). The reaction mixture was poured into ice water, and the precipitated solid was filtered off, washed with water and then dried. The crude product was purified by column chromatography (silica gel, petroleum ether/acetone 2:1) to give (I). ^{1}H NMR ($CDCl_3$, p.p.m.): 1.29 (3H, *s*), 1.29 (3H, *s*), 1.82 (1H, *s*), 2.60 (2H, *m*), 3.60 (1H, *m*), 3.63 (2H, *m*), 5.10 (2H, *s*), 6.44 (1H, *m*), 7.36 (1H, *m*), 8.00 (1H, *m*). Compound (I) (20 mg) was dissolved in ethyl acetate (20 ml). Single crystals of (I), suitable for X-ray analysis, were grown by natural evaporation of the solvent.

Crystal data

$C_{16}H_{16}ClFN_2O_3$
M_r = 338.76
Monoclinic, $P2_1/c$
a = 13.292 (4) Å
b = 7.529 (2) Å
c = 17.213 (5) Å
β = 111.607 (5)°
V = 1601.5 (8) Å^3
Z = 4
D_x = 1.405 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 903 reflections
θ = 2.6–25.9°
μ = 0.27 mm^{-1}
T = 293 (2) K
Block, colorless
0.26 × 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} = 0.930, T_{max} = 0.948
6555 measured reflections
2813 independent reflections
2157 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 25.0°
$h = -15 \rightarrow 15$
$k = -8 \rightarrow 8$
$l = -20 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.103
S = 1.03
2813 reflections
210 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0478P)^2 + 0.4903P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

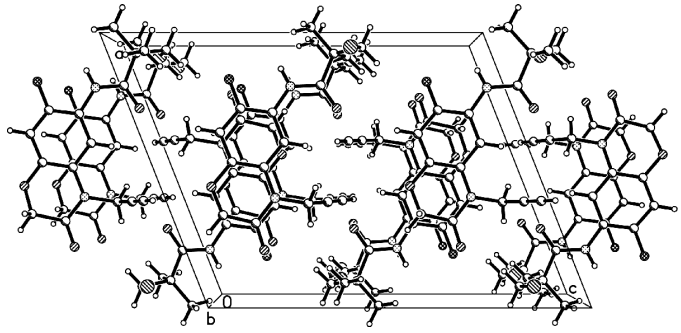


Figure 2
The crystal structure of (I), viewed along the *b* axis.

H atoms were positioned geometrically, with C—H = 0.93–0.97 Å and N—H = 0.86 Å, and refined in the riding-model approximation, with U_{iso}(H) = 1.2U_{eq}(carrier atom).

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*.

References

Birchfied, N. B. & Casida., J. E. (1997). *Pestic. Biochem. Physiol.* **57**, 36–43.
Bruker (1997). *SADABS* (Version 5.0), *SMART* (Version 5.0), *SAINT* (Version 5.0) and *SHELXTL.* (Version 5.10). Bruker AXS Inc., Madison, Wisconsin, USA.
Chamilleri, P., Weaver, K., Clark, M. T., Bowyer, J. R. & Hallahan, B. J. (1988). *J. Agric. Food Chem.* **36**, 1061–1063.
Karolak-Wojciechowska, J., Fruzínski, A. & Mokrosz, M. J. (2001). *J. Mol. Struct. (Theochem)*, **542**, 47–56.
Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.
Terni, P., Rugarli, P. L., Maiorana, S., Pagella, P. G. & Fusco, R. (1988). European Patent Application 252 352 (Cl. C07D513/06), 13 January 1988.