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2-(1,3-Dioxo-4,5,6,7-tetrahydro-1*H*-isoindol-2-yl)-*N*-[7-fluoro-3-oxo-4-(prop-2-ynyl)-3,4-dihydro-2*H*-benzoxazin-6-yl]-acetamide monohydrate

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In the title compound, $C_{21}H_{18}FN_3O_5\cdot H_2O$, the cyclohexene ring exhibits a distorted chair conformation. The crystal packing is stabilized by intra- and intermolecular hydrogen bonds.

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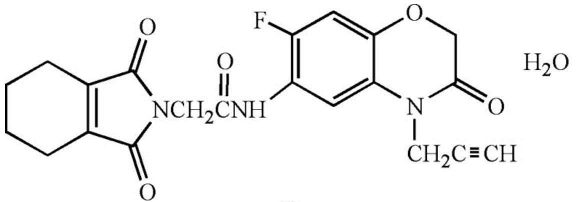
Key indicators

Single-crystal X-ray study
T = 292 K
Mean σ(C–C) = 0.004 Å
Disorder in main residue
R factor = 0.062
wR factor = 0.167
Data-to-parameter ratio = 11.3

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

Comment

Herbicides inhibiting protoporphyrinogen oxidase (protox) have been sold commercially for nearly 40 years (Dayan & Duke, 1997). The title compound, (I), may belong to this family of protox-inhibiting herbicides and we present its crystal structure here.



(I)

The molecular stucture of (I) is shown in Fig. 1. The C10—N2 bond is shorter than the normal value of C—N [1.47 (2) Å; Sasada, 1984]. The bond length of C2—C3 is slightly greater than the normal value of C–C [1.54 (3) Å; Sasada, 1984]. The torsion angles C1—C2—C3—C4 and C2—C3—C4—C5 indicate a distorted chair conformation of the cyclohexene ring. The sum of the C8—N1—C7, C8—N1—C9 and C7—N1—C9 angles is 359.9°, the sum of the C10—N2—C11, C10—N2—H2 and C11—N2—H2 angles is 359.1° and the sum of the C17—N3—C15, C17—N3—C19 and C15—N3—C19 angles is 360.0°.

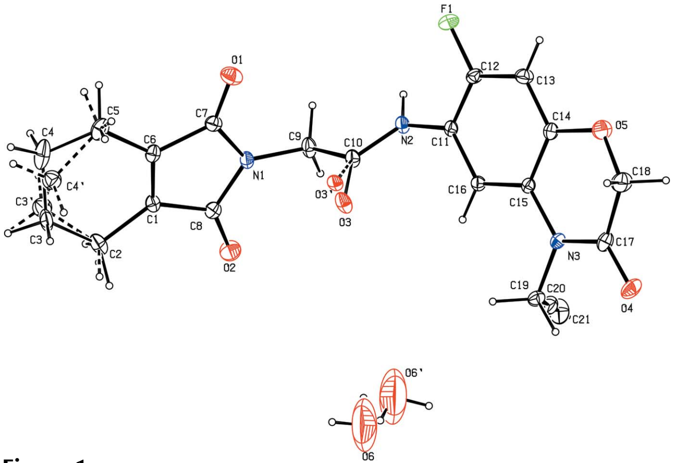


Figure 1
The molecular structure of (I), showing displacement ellipsoids drawn at the 50% probability level. All disorder components are shown.

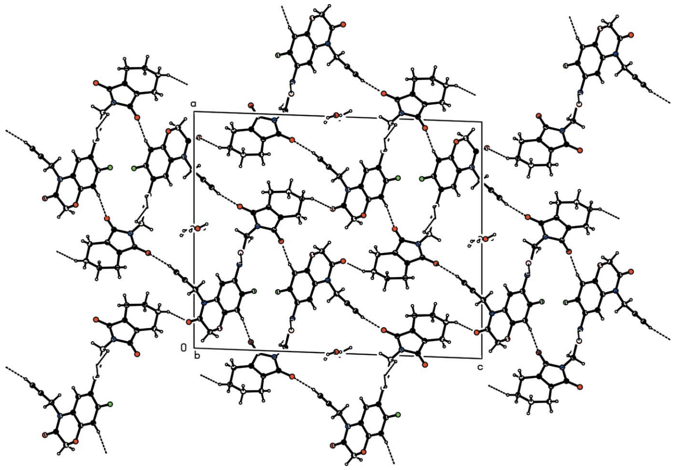


Figure 2
The packing of (I), with hydrogen bonds shown as dashed lines. Only one component of each disordered group is shown.

Therefore, atoms N1, N2 and N3 are sp^2 hybridized. The molecules of (I) form two-dimensional layers through hydrogen bonds in the *ac* plane (Table 2 and Fig. 2).

Experimental

2-[1,3-Dioxo-4,5,6,7-tetrahydro-1*H*-isoindol-2-yl]acetyl choride (1.2 mmol) in dry toluene (10 ml) was added dropwise to a solution of 6-amino-7-fluoro-4-(prop-2-ynyl)-2*H*-benzoxazin-3(4*H*)-one (1 mmol) and triethylamine (1.2 mmol) in dry toluene (10 ml) under N_2 at room temperature, and the resulting mixture was stirred for 2 h. After filtration, the solid was washed with water and recrystallized from petroleum ether and methanol (4:1 *v*/*v*). Colorless plate-shaped crystals of (I) were obtained by evaporation of the solvent over a period of two weeks.

Crystal data

$C_{21}H_{18}FN_3O_5\cdot H_2O$
$M_r = 429.40$
Monoclinic, $P2_1/n$
$a = 19.2192$ (19) Å
$b = 4.7354$ (5) Å
$c = 23.421$ (2) Å
$\beta = 92.091$ (2)°
$V = 2130.2$ (4) Å^3
$Z = 4$
$D_x = 1.339$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2160 reflections
$\theta = 2.7$–21.4°
$\mu = 0.11$ mm^{-1}
$T = 292$ (2) K
Plate, colorless
0.40 × 0.10 × 0.02 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{\min} = 0.959$, $T_{\max} = 0.998$
14145 measured reflections
3719 independent reflections
2411 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.050$
$\theta_{\max} = 25.0°$
$h = -21 \rightarrow 22$
$k = -5 \rightarrow 5$
$l = -27 \rightarrow 27$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.062$
$wR(F^2) = 0.167$
$S = 1.04$
3719 reflections
328 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0734P)^2 + 0.5971P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.26$ e Å^{-3}
$\Delta\rho_{\min} = -0.21$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C2—C3	1.560 (7)	C10—N2	1.318 (4)
C8—N1—C7	109.7 (2)	C17—N3—C15	120.9 (3)
C8—N1—C9	126.7 (3)	C17—N3—C19	118.9 (2)
C7—N1—C9	123.5 (3)	C15—N3—C19	120.2 (2)
C10—N2—C11	126.1 (2)		
C1—C2—C3—C4	31.5 (10)	C2—C3—C4—C5	−52.1 (12)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C21—H21···O2$^{\rm i}$	0.93	2.33	3.244 (5)	168
C19—H19*B*···O4	0.97	2.30	2.735 (4)	106
C13—H13···O1$^{\rm ii}$	0.93	2.44	3.354 (4)	170
C9—H9*A*···O3$^{\rm iii}$	0.97	2.48	3.189 (8)	130
C4—H4*B*···O4$^{\rm iv}$	0.97	2.54	3.454 (8)	157
N2—H2···O3$^{\rm iii}$	0.851 (10)	2.04 (2)	2.847 (9)	158 (3)

Symmetry codes: (i) $-x+1, -y, -z$; (ii) $-x+\frac{1}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$; (iii) $x, y-1, z$; (iv) $x+\frac{1}{2}, -y+\frac{3}{2}, z+\frac{1}{2}$.

The amide and water H atoms were located in a difference map and were refined with the restraints N—H = 0.86 (1) Å and O—H = 0.82 Å, and with $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(carrier) for H1 and 1.5$U_{\rm eq}$(carrier) for water H atoms. Other H atoms were positioned geometrically, with C—H = 0.93 or 0.97 Å, and refined in a riding model, with $U_{\rm iso}$(H)= 1.2$U_{\rm eq}$(C) or 1.5$U_{\rm eq}$(methyl C). Two of the C atoms in the cyclohexene ring were disordered over two positions, and the occupancy factors for disordered positions C3/C3′ and C4/C4′ were refined to 0.709 (12) and 0.291 (12). Atom O3/O3′ was disordered over two positions, with occupancies of 0.77 (9) and 0.23 (9). Water atoms O6 and O6′, with partial occupancies of 0.50 [initially refined to 0.504 (1)], were assigned tentatively, based only on the crystallographic evidence; the water probably derives from the methanol solvent used for recrystallization.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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