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6-Methoxy-1,3-benzoxazol-2(3*H*)-one

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.032
wR factor = 0.100
Data-to-parameter ratio = 11.5

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

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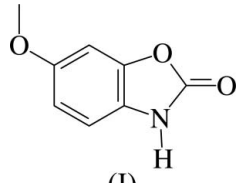
The molecule of the title compound, $C_8H_7NO_3$, isolated from the plant *Scoparia dulcis L.*, is essentially planar. Intermolecular N—H···O hydrogen bonds link the molecules into a chain running along the *b* axis.

Comment

The plant *Scoparia dulcis L.* has been explored for the treatment of diabetes, tumors and plasmodial, bacterial and viral diseases. The compounds isolated from this plant have been tested for their efficacy as drugs for various diseases. Nishino *et al.* (1993) demonstrated the antitumor effect of scopadulcic acid B fraction. The anti-HIV property of betulinic acid has been investigated by De Clercq (2001). The title compound, (I), commonly known as coixol, is used in traditional medicines in China and Japan for the treatment of arthritis and as a tonic. The spectroscopic analysis of (I) has been reported by Chen & Chen (1976) and Li *et al.* (1981). Compound (I) exhibits antifungal, antihistaminic, muscle-relaxant with an anti-convulsant effect in rats (Gomita *et al.*, 1981) and fever-reducing properties (Shin *et al.*, 2005). Wang & Ng (2002) have reported that the title compound is most potent in inhibiting HIV-1 reverse transcriptase. As the title compound is of high pharmaceutical importance, we have undertaken its crystal structure determination by X-ray diffraction.



(I)

The bond lengths and angles in (I) (Fig. 1) are comparable with the literature values (Allen *et al.*, 1987). The O1—C5 and N3—C4 distances are longer than the O1—C2 and N3—C2 distances, respectively (Table 1). The fused five- and six-membered rings are coplanar, making a dihedral angle of 0.2 (1)°. Atom O2 deviates by 0.007 (1) Å from the plane of the five-membered ring. The methoxy group is almost coplanar with the attached benzene ring, the C10—O3—C7—C6 torsion angle being 3.7 (2)°.

The crystal structure of (I) is stabilized by N—H···O intermolecular hydrogen bonds (Table 2). Atom N3 acts as a donor to atom O2 at $(\frac{1}{2}-x, -\frac{1}{2}+y, \frac{3}{2}-z)$, generating a *C*(4) chain running along the *b* axis (Fig. 2). No hydrogen-bonding interactions are observed between adjacent chains along the *a* axis.

Experimental

The aerial parts of *Scoparia dulcis* were collected, washed in distilled water and shade-dried. Using a mechanical pulverizer, the dried materials were powdered. Five different extracts were prepared by soaking the plant powder in organic solvents (petroleum ether, hexane, chloroform, ethyl acetate, methanol) in order of increasing polarity. The extracts were concentrated to 2% of their original volume using a rotary evaporator. The evaporated extracts were subjected to column chromatography. For packing the column, silica gel of 100–200 mesh size was used. The chloroform extract when eluted with a mixture of chloroform–ethyl acetate (7:3) yielded a pale-yellow crystalline compound. The compound was recrystallized from ethyl acetate by slow evaporation.

Crystal data

$C_8H_7NO_3$
M_r = 165.15
Monoclinic, $P2_1/n$
a = 12.4251 (12) Å
b = 3.9154 (4) Å
c = 16.0231 (16) Å
β = 108.796 (2)°
V = 737.94 (13) Å^3
Z = 4
D_x = 1.486 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 293 (2) K
Block, pale yellow
0.23 × 0.23 × 0.21 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: none
6554 measured reflections
1306 independent reflections
1231 reflections with $I > 2\sigma(I)$
R_{int} = 0.020
θ_{max} = 25.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.100
S = 1.11
1306 reflections
114 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0597P)^2 + 0.0945P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.12 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O2—C2	1.217 (2)	C4—C5	1.375 (2)
O3—C7	1.366 (2)	C4—C9	1.387 (2)
O3—C10	1.422 (2)	C5—C6	1.375 (2)
O1—C2	1.366 (2)	C6—C7	1.396 (2)
O1—C5	1.391 (1)	C7—C8	1.393 (2)
C2—N3	1.346 (2)	C8—C9	1.384 (2)
N3—C4	1.398 (2)		
C7—O3—C10	117.8 (1)	C2—N3—C4	109.5 (1)
C2—O1—C5	107.3 (1)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3···O2^{i}	0.94 (2)	1.86 (2)	2.792 (2)	171 (2)

Symmetry code: (i) $-x+\frac{1}{2}, y-\frac{1}{2}, -z+\frac{3}{2}$.

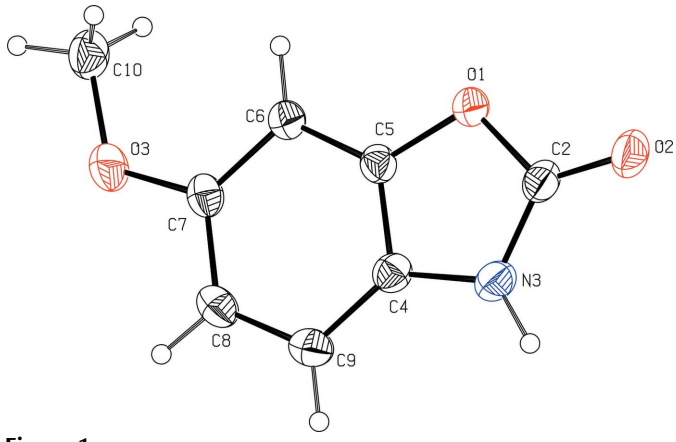


Figure 1
The molecular structure of (I), showing 30% probability displacement ellipsoids and the atomic numbering.

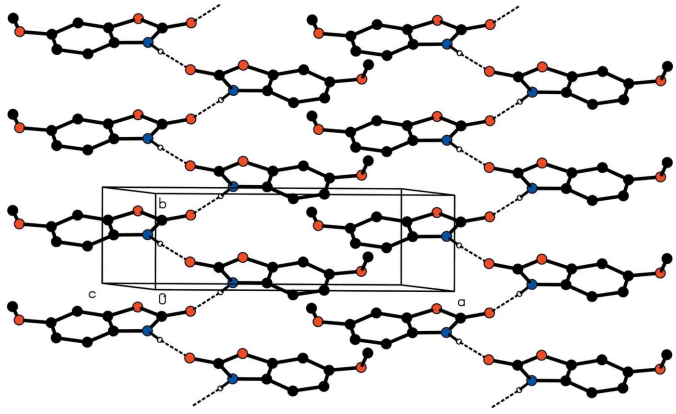


Figure 2
The crystal structure of (I), showing the hydrogen-bonded (dashed lines) chains. For clarity, only H atoms involved in hydrogen bonding are shown.

The N-bound H atom was located in a difference map and refined freely. C-bound H atoms were positioned geometrically and refined using a riding model, with C—H = 0.93 or 0.96 Å and U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C). A rotating-group model was used for the methyl group.

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: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PARST* (Nardelli, 1995).

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