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Ethyl 1-(1,2,3-benzothiadiazol-7-ylcarbonyl)-piperidine-4-carboxylate

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The title compound, $C_{15}H_{17}N_3O_3S$, a potent new plant elicitor, was synthesized by reaction between 1,2,3-benzothiadiazole-7-carbonyl chloride and ethyl piperidine-4-carboxylate. The piperidine ring adopts a chair conformation. Weak intermolecular C—H···O hydrogen bonds stabilize the crystal packing.

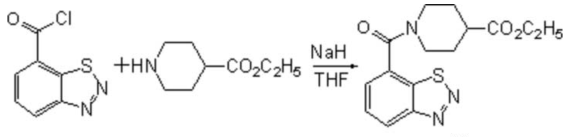
Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.004 Å
Disorder in main residue
R factor = 0.048
wR factor = 0.162
Data-to-parameter ratio = 13.9

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

Comment

A plant elicitor can induce systemic acquired resistance (SAR) in the treated plant against pathogen attack with a new mode of action. Benzothiadiazole (BTH), 2,6-dichloroisonicotinic acid, β-aminobutyric acid and salicylic acid and its derivatives are known as plant elicitors (Gozzo, 2003; Bao, Liu & Fan, 2005; Yasuda *et al.*, 2004). In a continuation of our systematic search for novel BTH derivatives exhibiting SAR activity (Bao, Fan *et al.*, 2005; Ai *et al.*, 2005, 2006; Liu *et al.*, 2005; Zhao *et al.*, 2006), we present here the crystal structure of the title compound, (I), prepared recently by our group.



(I)

The bond lengths and angles in (I) are normal (Table 1). The benzothiadiazole moiety is essentially planar, with an r.m.s. deviation of 0.0221 Å for the non-H atoms. The mean planes of the benzothiadiazole and the C7/O1/N3 group (Fig. 1) make a dihedral angle of 37.8 (1)°. The piperidine ring adopts a chair conformation. Weak intermolecular C—H···O hydrogen bonds (Table 2) stabilize the crystal packing.

Experimental

Compound (I) was prepared according to a reported precedure (Fan *et al.*, 2005). Single crystals suitable for X-ray diffraction were obtained by recrystallization and slow evaporation of a petroleum ether and ethyl acetate (5:1 *v*/*v*) solution at room temperature.

Crystal data

$C_{15}H_{17}N_3O_3S$
$M_r = 319.38$
Monoclinic, $P2_1/c$
$a = 15.465\ (3)$ Å
$b = 12.284\ (2)$ Å
$c = 8.1513\ (15)$ Å
$\beta = 96.094\ (3)^\circ$
$V = 1539.8\ (5)$ Å^3
$Z = 4$
$D_x = 1.378$ Mg m^{-3}
Mo *K*α radiation
Cell parameters from 2419 reflections
$\theta = 2.7–23.7^\circ$
$\mu = 0.23$ mm^{-1}
$T = 294\ (2)$ K
Prism, colourless
0.32 × 0.28 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.92$, $T_{\max} = 0.951$
8514 measured reflections

3160 independent reflections
1849 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.032$
$\theta_{\max} = 26.5°$
$h = -19 \rightarrow 19$
$k = -15 \rightarrow 14$
$l = -10 \rightarrow 6$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.048$
$wR(F^2) = 0.162$
$S = 0.98$
3160 reflections
227 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0854P)^2 + 0.2872P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.29$ e Å^{-3}
$\Delta\rho_{\min} = -0.25$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O2—C13	1.186 (3)	C5—H5*A*	0.9300
N3—C12	1.468 (3)	C10—C13	1.506 (4)
C4—H4*A*	0.9300		
C12—N3—C8	112.9 (2)	C8—C9—C10	110.8 (2)
C1—C2—C3	121.1 (2)	C14—C15—H15*B*	109.5
C5—C6—C7	125.9 (2)		
S1—C1—C2—C3	−176.7 (2)	C7—N3—C8—C9	−139.4 (3)
N2—C2—C3—C4	−178.4 (2)	C11—C10—C13—O3	−71.4 (5)
C4—C5—C6—C7	178.1 (2)	C13—O3—C14—C15	−90.0 (8)

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

D—H$\cdots$*A*	*D*—H	H$\cdots$*A*	*D*$\cdots$*A*	*D*—H$\cdots$*A*
C9—H9*B*$\cdots$O2$^{\text{i}}$	0.97	2.59	3.542 (3)	169
C15—H15*C*$\cdots$O1$^{\text{ii}}$	0.96	2.57	3.401 (8)	145

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

All H atoms were placed in idealized positions and constrained to ride on their parent atoms, with C—H = 0.93–0.98 Å and U_{iso}(H) = 1.2U_{eq}(aromatic C) or 1.5U_{eq}(methyl C). The disordered ethoxy group was split into two positions with different site occupancies, which refined to values of 0.552 (9) and 0.448 (9).

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); 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, 1999); software used to prepare material for publication: *SHELXTL*.

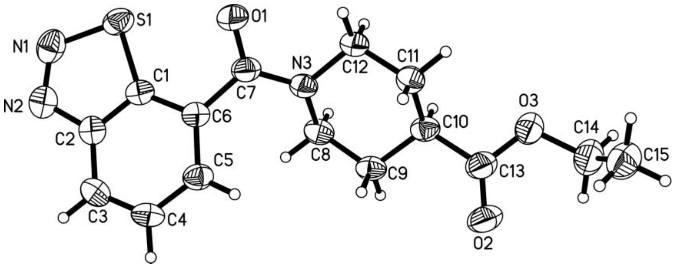


Figure 1
View of (I), showing the atom-numbering scheme and displacement ellipsoids drawn at the 30% probability level. The minor component of the disordered ethoxy group has been omitted for clarity.

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