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N-(1-Cyanocyclohexyl)-1,2,3-benzothiadiazole-7-carboxamide

Feng-Li Liu,[a] Zhi-Jin Fan,[a,b]* Hai-Bin Song,[a] Xiu-Feng Liu[a] and Yong-Gang Zhang[a]

[a]State Key Laboratory of Elemento-Organic Chemistry, Nankai University, Tianjin 300071, People's Republic of China, and [b]Key Laboratory of Pesticide Chemistry & Application Technology of the Ministry of Agriculture, Beijin 100094, People's Republic of China

Correspondence e-mail: fanzj@nankai.edu.cn

Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.003 Å
R factor = 0.039
wR factor = 0.103
Data-to-parameter ratio = 12.9

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

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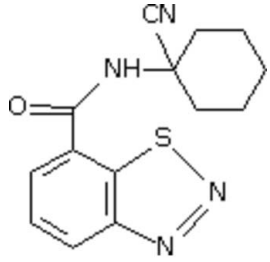
The title compound, $C_{14}H_{14}N_4OS$, is a derivative of acibenzolar-*S*-methyl or BTH, a plant elicitor. The asymmetric unit contains two independent molecules in each of which the benzothiadiazole ring system is planar and the cyclohexane ring adopts a chair conformation. In the crystal structure, molecules are linked by N—H···N and C—H···N hydrogen bonds.

Comment

Acibenzolar-*S*-methyl (also called BTH) is a leading plant activator, a kind of environmentally benign agrochemical developed in recent years with a new mode of action (Gozzo, 2003). Many studies have been carried out on the structural modification of this compound (Bao, Liu *et al.*, 2005; Kunz *et al.*, 1997). In order to find more powerful plant activators that can be applied to prevent and cure diseases caused by viruses, and to understand the quantitative structure–activity relationship (QSAR) of this new kind of pesticide, the title compound, (I), was synthesized. A search of the Cambridge Structural Database (Version 5.26 with updates to August 2005; Allen, 2002), revealed only seven structures containing the benzothiadiazole group. Recently, we have reported the structures of *N*-phenyl-*N*′-(1,3-thiazol-2-yl)-1,2,3-benzothiadiazole-7-carboxamidine (Bao, Fan *et al.*, 2005) and *N*-(1-cyano-1-methylbutyl)-1,2,3-benzothiadiazole-7-carboxamide (Ai *et al.*, 2005). We now report here the crystal structure of *N*-(1-cyanocyclohexyl)-1,2,3-benzothiadiazole-7-carboxamide, (I).



(I)

The asymmetric unit of (I) contains two crystallographically independent molecules (Fig. 1). Bond lengths and angles in (I) are normal, and values for the two independent molecules agree well with each other (Table 1). In both independent molecules, the benzothiadiazole ring system is essentially planar and the cyclohexane ring adopts a chair conformation. In one of the molecules, the amide plane and benzothiadiazole ring system are coplanar, whereas in the other (containing atom S2), they are twisted by 14.3 (1)°. The crystal structure is

stabilized by N—H···N and C—H···N hydrogen bonds (Table 2).

Experimental

Compound (I) was prepared according to the reported procedure of Fan *et al.* (2005). Single crystals suitable for X-ray diffraction were obtained by recrystallization from a mixture of petroleum ether and ethyl acetate (2:1 *v*/*v*).

Crystal data

$C_{14}H_{14}N_4OS$
M_r = 286.35
Triclinic, $P\overline{1}$
a = 11.0871 (17) Å
b = 11.8713 (19) Å
c = 12.0846 (19) Å
α = 117.308 (4)°
β = 96.217 (6)°
γ = 100.195 (5)°
V = 1357.9 (4) Å^3
Z = 4
D_x = 1.401 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 956 reflections
θ = 3.4–26.2°
μ = 0.24 mm^{-1}
T = 294 (2) K
Block, colourless
0.30 × 0.24 × 0.14 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.920, T_{max} = 0.967
7142 measured reflections
4777 independent reflections
3527 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
θ_{max} = 25.0°
$h = -13 \rightarrow 11$
$k = -13 \rightarrow 14$
$l = -14 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.103
S = 1.02
4777 reflections
370 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0462P)^2 + 0.3245P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.028 (2)

Table 1
Selected bond lengths (Å).

S1—C1	1.704 (2)	N3—C7	1.351 (3)
S1—N1	1.707 (2)	N3—C8	1.460 (3)
S2—C15	1.704 (2)	N4—C14	1.140 (3)
S2—N5	1.713 (2)	N5—N6	1.273 (3)
O1—C7	1.230 (2)	N6—C16	1.393 (3)
O2—C21	1.227 (2)	N7—C21	1.343 (3)
N1—N2	1.282 (3)	N7—C22	1.466 (3)
N2—C2	1.386 (3)	N8—C28	1.129 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3A···N8^{i}	0.84 (2)	2.33 (2)	3.135 (3)	163 (2)
C10—H10B···N5^{ii}	0.97	2.53	3.384 (4)	147

Symmetry codes: (i) $x+1, y, z$; (ii) $x+1, y+1, z$.

The amine H atoms were located in a difference Fourier map and refined isotropically [N—H = 0.78 (2) and 0.84 (2) Å]. H atoms attached to C atoms were placed in idealized positions and constrained to ride on their parent atoms, with C—H = 0.93–0.97 Å and U_{iso}(H) = 1.2–1.5U_{eq}(C).

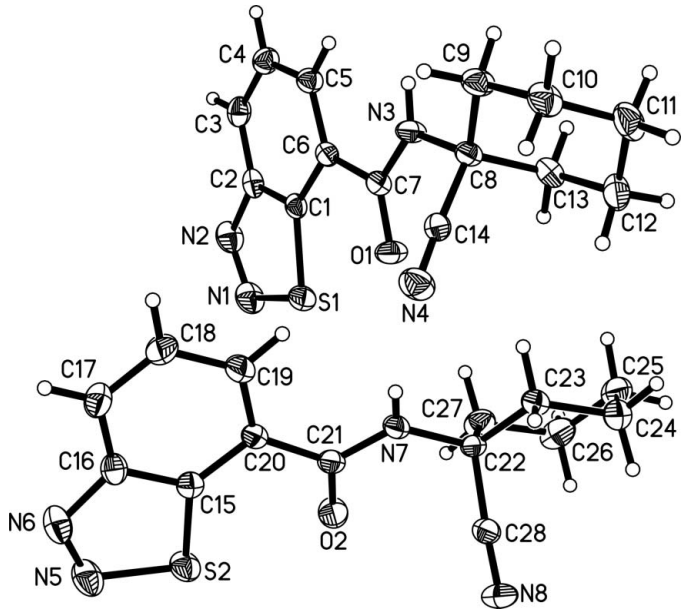


Figure 1
The aymmetric unit of (I), showing the atomic numbering. Displacement ellipsoids are drawn at the 30% probability level.

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*.

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References

Ai, Y.-W., Liu, F.-L., Fan, Z.-J., Song, H. B. & Nie, K. S. (2005). *Acta Cryst.* E**61**, o3891–o3892.
Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.
Bao, L.-L., Fan, Z.-J., Song, H.-B & Nie, K.-S. (2005). *Acta Cryst.* E**61**, o3817–o3818.
Bao, L.-L., Liu, F.-L. & Fan, Z.-J. (2005). *Chin. J. of Pestic. Sci.* **7**, 201–209. (In Chinese.)
Bruker (1998). *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (1999). *SAINT* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Fan, Z.-J., Liu, F.-L. & Liu, X.-F. (2005). Chin. Patent CN1 680 342A. (In Chinese.)
Gozzo, F. (2003). *J. Agric. Food. Chem.* **51**, 4487–4503.
Kunz, W., Schurter, R. & Maetzke, T. (1997). *Pestic. Sci.* **50**, 275–282.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.