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N-(1-Cyano-1-methylbutyl)-1,2,3-benzothiadiazole-7-carboxamide

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.005 Å
R factor = 0.040
wR factor = 0.107
Data-to-parameter ratio = 14.2

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

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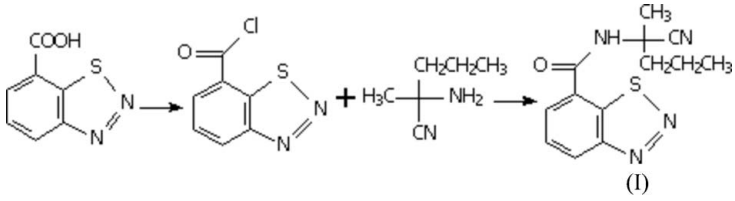
In the title compound, $C_{13}H_{14}N_4OS$, which was synthesized as a candidate plant activator by the reaction of 1-amino-1-methyl-1-propylacetonitrile and 1,2,3-benzothiadiazole-7-carbonyl chloride, the benzothiadiazole moiety is essentially planar, forming a dihedral angle of 14.5 (4)° with the amide group. In the crystal structure, molecules are linked by intermolecular N—H···O hydrogen bonds [H···O = 2.20 (4) Å] to form extended chains in the *a*-axis direction.

Comment

Derivatives of acibenzolar-*S*-methyl or BTH (*S*-methyl 1,2,3-benzothiadiazole-7-carbothioate) possess systemic acquired resistance against fungi and virus in agricultural practice. They are potential plant activators with environmentally friendly characteristics (Gozzo, 2003). Many studies have modified the structure (Bao, Liu *et al.*, 2005) in order to find more powerful plant activator candidates that can be applied to the prevention and cure of plant diseases and viruses, especially the tobacco mosaic virus (TMV). 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 and we have recently determined the structures of three more (Bao, Fan *et al.*, 2005; Liu *et al.*, 2005; Zhao *et al.*, 2005). We have now determined the structure of the title compound, (I), to investigate its quantitative structure–activity relationship (QSAR).



(I)

The molecular structure of (I) is shown in Fig. 1. The benzothiadiazole moiety is essentially planar (r.m.s. deviation 0.011 Å), forming a dihedral angle with the C7, O1 and N3 plane of atoms of 14.5 (4)°. The approximate coplanarity of the amine and benzothiadiazole groups, in conjunction with the observed bond lengths and angles (Table 1), suggests that an extended π-conjugated system exists in this part of the molecule.

In the crystal structure of (I), extended one-dimensional chains are formed *via* intermolecular N—H···O hydrogen bonds (Table 2).

Experimental

1,2,3-Benzothiadiazole-7-carboxylic acid was synthesized according to the description of Fan *et al.* (2005). The title compound was

prepared by reacting 1-amino-1-methyl-1-propylacetonitrile with 1,2,3-benzothiadiazole-7-carbonyl chloride (Bao, Fan *et al.*, 2005). The product obtained after silica-gel column chromatography was recrystallized from a mixture of petroleum ether (333–363 K) and ethyl acetate (2:1 *v*/*v*) at room temperature to yield colourless crystals suitable for X-ray diffraction analysis.

Crystal data

$C_{13}H_{14}N_4OS$
M_r = 274.34
Monoclinic, $P2_1$
a = 6.236 (2) Å
b = 13.263 (4) Å
c = 8.667 (3) Å
β = 110.740 (5)°
V = 670.4 (4) Å^3
Z = 2
D_x = 1.359 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2025 reflections
θ = 2.5–24.6°
μ = 0.24 mm^{-1}
T = 294 (2) K
Block, colourless
0.32 × 0.24 × 0.20 mm

Data collection

Bruker SMART-CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.920, T_{max} = 0.953
3879 measured reflections
2534 independent reflections
2262 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 26.4°
$h = -7 \rightarrow 7$
$k = -13 \rightarrow 16$
$l = -7 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.107
S = 1.07
2534 reflections
179 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0645P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}
Absolute structure: Flack (1983), with 1097 Friedel pairs
Flack parameter: 0.03 (12)

Table 1
Selected geometric parameters (Å, °).

S1—C2	1.702 (3)	N3—C8	1.467 (4)
S1—N1	1.703 (4)	O1—C7	1.222 (4)
N1—N2	1.278 (5)	C1—C6	1.380 (4)
N2—C3	1.377 (5)	C1—C7	1.494 (4)
N3—C7	1.344 (4)	C2—C3	1.393 (5)
C2—S1—N1	92.29 (17)	O1—C7—C1	120.1 (3)
C7—N3—C8	123.0 (3)	N3—C7—C1	116.9 (3)
O1—C7—N3	122.9 (3)		
C13—C8—C10—C11	174.1 (3)	C8—C10—C11—C12	173.7 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3···N4^{i}	0.89 (4)	2.20 (4)	3.083 (4)	173 (3)

Symmetry code: (i) $x + 1, y, z$.

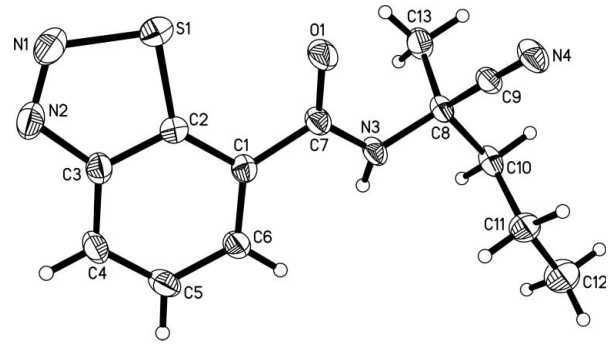


Figure 1
A view of (I), with displacement ellipsoids drawn at the 30% probabilty level. H atoms are shown as spheres of arbitrary radii.

The position of the amine H atom was refined independently with an isotropic displacement parameter. All H atoms bonded to C atoms were placed in idealized positions and constrained to ride on their parent atoms, with C—H distances ranging from 0.93 to 0.97 Å, and with U_{iso}(H) = 1.2U_{eq}(C), or 1.5U_{eq}(C_{methyl}).

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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