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

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The title compound, $C_{11}H_{10}N_4OS$ was synthesized as a candidate plant activator. In the crystal structure, the benzene ring and the thiadiazole ring are nearly coplanar, making a dihedral angle of 1.6 (5)°. Molecules are linked by intermolecular N—H···O hydrogen bonds [H···O = 2.27 (2) Å].

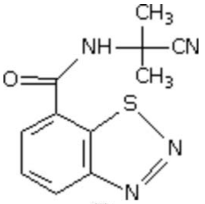
Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.003 Å
R factor = 0.031
wR factor = 0.078
Data-to-parameter ratio = 15.2

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

Comment

Plant activators are environmentally benign plant protection chemicals with a new mode of action. Of this group, acibenzolar-*S*-methyl (BTH) has been the most successful (Gozzo, 2003; Fan *et al.*, 2005). Many derivatives of BTH possess induction of systemic acquired resistance (SAR) (Bao *et al.*, 2005). In order to find more powerful plant activators, novel derivatives of BTH were synthesized for screening (Bao *et al.*, 2005; Ai *et al.*, 2005, 2006; Liu *et al.*, 2005; Zhao *et al.*, 2006). We report here the crystal structure of the title compound, (I).



(I)

The molecular structure of (I) is shown in Fig. 1. The benzene ring and thiadiazole ring are nearly coplanar with a dihedral angle of 1.6 (5)°; the benzothiadiazole moiety forms a dihedral angle of 5.7 (1)° with the C7/O1/N3 plane. The approximate coplanarity of the amine and benzothiadiazole groups, in conjunction with the observed bond lengths and angles (Table 1), suggest that an extended π-conjugated system exists in this part of the molecule. In the crystal structure, extended one-dimensional chains are formed *via* intermolecular N—H···O hydrogen bonds (Table 2).

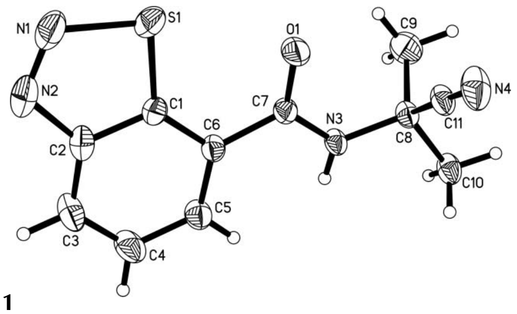


Figure 1
View of the title compound, (I), with displacement ellipsoids drawn at the 30% probability level.

Experimental

Compound (I) was prepared by reacting (reflux, 3 h) 2-amino-2,2-dimethylacetonitrile (0.42 g) with 1,2,3-benzothiadiazole-7-carbonyl chloride (1 g) in a mixture of CH_2Cl_2 (20 ml) and triethylamine (0.7 ml). The crude product was obtained by washing the reaction mixture with dilute hydrochloric acid, saturated $NaHCO_3$ and water, and removing the solvent *via* vacuum. Colorless crystals of the title compound were obtained by column chromatography on silica gel and recrystallization from petroleum ether (333–363 K) and ethyl acetate (2:1 *v*/*v*) at room temperature.

Crystal data

$C_{11}H_{10}N_4OS$
$M_r = 246.29$
Orthorhombic, $Pna2_1$
$a = 8.996$ (2) Å
$b = 21.630$ (6) Å
$c = 6.1600$ (16) Å
$V = 1198.6$ (5) Å^3
$Z = 4$
$D_x = 1.365$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3339 reflections
$\theta = 3.4$–$26.3°$
$\mu = 0.26$ mm^{-1}
$T = 294$ (2) K
Block, colorless
0.26 × 0.22 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.928$, $T_{\max} = 0.954$
6784 measured reflections
2436 independent reflections
2120 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.022$
$\theta_{\max} = 26.5°$
$h = -9 \rightarrow 11$
$k = -27 \rightarrow 26$
$l = -7 \rightarrow 7$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.031$
$wR(F^2) = 0.078$
$S = 1.05$
2436 reflections
160 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0421P)^2 + 0.0902P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.12$ e Å^{-3}
$\Delta\rho_{\min} = -0.18$ e Å^{-3}
Absolute structure: Flack (1983), 1064 Friedel pairs
Flack parameter: 0.01 (8)

Table 1
Selected geometric parameters (Å, °).

S1—N1	1.704 (2)	N2—C2	1.394 (3)
S1—C1	1.712 (2)	N3—C7	1.347 (2)
O1—C7	1.225 (2)	N3—C8	1.466 (2)
N1—N2	1.275 (3)		
N1—S1—C1	92.31 (10)	O1—C7—C6	119.29 (17)
C7—N3—C8	122.87 (16)	N3—C7—C6	118.49 (17)
O1—C7—N3	122.21 (18)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3···N4$^{\rm i}$	0.83 (2)	2.27 (2)	3.083 (3)	166.8 (19)

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

All H atoms were placed in idealized positions and constrained to ride on their parent atoms, with N—H = 0.86 Å and C—H = 0.93 or 0.96 Å, and $U_{\rm iso}$(H) = $1.2U_{\rm eq}$(C) or $1.5U_{\rm eq}$(methyl C). The position of the amine H atom was refined independently with an isotropic displacement parameter.

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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