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Pyrrolo[2,1-*c*][1,4]benzodiazepine-5,11-dithione

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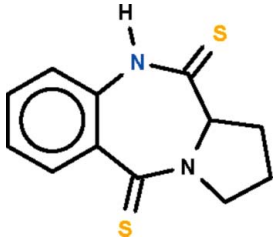
Key indicators: single-crystal X-ray study; T = 200 K; mean σ(C–C) = 0.003 Å; R factor = 0.042; wR factor = 0.107; data-to-parameter ratio = 15.6.

The seven-membered fused-ring in the title compound, $C_{12}H_{12}N_2S_2$, adopts a boat conformation (with the two phenylene C atoms representing the stern and the methine C atom the prow). This methine C atom and the tertiary N atom also belong to a five-membered ring, which has an envelope conformation. In the crystal structure, molecules are linked about a center of inversion by pairs of N—H···S hydrogen bonds.

Related literature

For background to pyrrolo[2,1-*c*][1,4]benzodiazepine-5,11-dione, see: Antonow *et al.* (2007); Kamal *et al.* (2007). For a related structure, Neidle *et al.* (1991).



Experimental

Crystal data

$C_{12}H_{12}N_2S_2$
M_r = 248.36
Monoclinic, $P2_1/c$
a = 13.9831 (5) Å
b = 10.0134 (3) Å
c = 8.2670 (3) Å
β = 97.089 (1)°
V = 1148.68 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.44 mm^{-1}
T = 200 K
0.12 × 0.10 × 0.07 mm

Data collection

Bruker X8 APEXII diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.950, T_{max} = 0.970
14013 measured reflections
3017 independent reflections
2117 reflections with $I > 2\sigma(I)$
R_{int} = 0.055

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.107
S = 1.01
3017 reflections
193 parameters
12 restraints
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···S1^{i}	0.86 (1)	2.58 (1)	3.411 (2)	166 (2)

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

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2774).

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supporting information

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Pyrrolo[2,1-*c*][1,4]benzodiazepine-5,11-dithione

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S1. Comment

Pyrrolo[2,1-*c*][1,4]benzodiazepine-5,11-dione is the homolog of a class of compounds that are active against mycobacterium tuberculosis (Kamal *et al.*, 2007). Other C-2 aryl substituted derivatives are cytotoxic (Antonow *et al.*, 2007). The crystal structure of the parent compound has not been reported although that the (11*aS*)-1,2,3,10,11,11*a*-hexahydro has bee published (Neidle *et al.*, 1997). The structure of the parent compound is probably similar to that of the isoelectronic dithione (Scheme I, Fig. 1). The seven-membered fused-ring in $C_{12}H_{12}N_2S_2$ adopts a boat conformation (with the two phenylene carbons representing the stern and the methine carbon atom the prow). This methine C atom and the tertiary N atom also belong to a five-membered ring, which has an envelope shape. Two $C_{12}H_{12}N_2O_2$ molecules are linked about a center-of-inversion by *N*–*H*···$O_{carbonyl}$ hydrogen bonds.

S2. Experimental

Pyrrolo[2,1-*c*][1,4]benzodiazepine-5,11-dithione (1 g, 4.62 mmol) and phosphorus pentasulfide (2.05 g, 9.24 mmol) are heated in pyridine (60 ml) for 4 h. The pyridine was evaporated under reduced pressure and the residue heated in water (100 ml). The suspension was set aside for a day. The insoluble product was recrystallized from ethanol to furnish colorless crystals (90% yield).

S3. Refinement

The nitrogen- and carbon-bound H-atoms were refined with restraints (C–H 0.95±0.01 Å for the aromatic atoms and 0.99±0.01 Å for the aliphatic atoms; N–H 0.86±0.01 Å). Their temperature factors were freely refined.

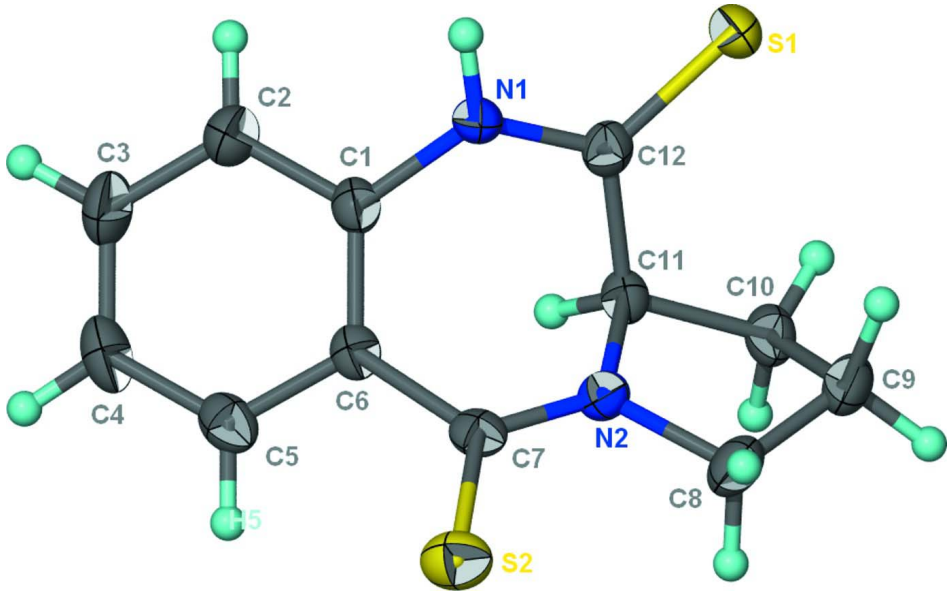

Figure 1

Thermal ellipsoid plot (Barbour, 2001) of the molecule of $C_{12}H_{12}N_2S_2$ at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

Pyrrolo[2,1-*c*][1,4]benzodiazepine-5,11-dithione

Crystal data

$C_{12}H_{12}N_2S_2$
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Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 13.9831 (5) Å
b = 10.0134 (3) Å
c = 8.2670 (3) Å
β = 97.089 (1)°
V = 1148.68 (7) Å^3
Z = 4
$F(000)$ = 520
D_x = 1.436 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2753 reflections
θ = 2.5–26.5°
μ = 0.44 mm^{-1}
T = 200 K
Prism, colorless
0.12 × 0.10 × 0.07 mm

Data collection

Bruker X8 APEXII diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.950, T_{max} = 0.970
14013 measured reflections
3017 independent reflections
2117 reflections with $I > 2\sigma(I)$
R_{int} = 0.055
θ_{max} = 28.9°, θ_{min} = 1.5°
h = −18→18
k = −12→13
l = −11→11

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.107
S = 1.01
3017 reflections
193 parameters
12 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites
All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.047P)^2 + 0.5062P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.39$ e Å^{-3}
$\Delta\rho_{min} = -0.28$ e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.60817 (4)	0.35830 (6)	0.60020 (8)	0.02972 (16)
S2	0.92223 (4)	0.75918 (6)	0.51650 (7)	0.02991 (16)
N1	0.62240 (12)	0.61932 (18)	0.5884 (2)	0.0221 (4)
N2	0.82707 (11)	0.57256 (17)	0.6540 (2)	0.0196 (4)
C1	0.65476 (14)	0.7511 (2)	0.6273 (2)	0.0205 (4)
C2	0.58339 (16)	0.8462 (2)	0.6410 (3)	0.0277 (5)
C3	0.60718 (17)	0.9769 (2)	0.6798 (3)	0.0318 (5)
C4	0.70347 (18)	1.0143 (2)	0.7079 (3)	0.0307 (5)
C5	0.77396 (16)	0.9220 (2)	0.6894 (3)	0.0251 (5)
C6	0.75250 (14)	0.7891 (2)	0.6467 (2)	0.0191 (4)
C7	0.83258 (14)	0.7001 (2)	0.6109 (2)	0.0194 (4)
C8	0.90083 (16)	0.4717 (2)	0.6291 (3)	0.0274 (5)
C9	0.86119 (16)	0.3432 (2)	0.6929 (3)	0.0284 (5)
C10	0.80322 (16)	0.3926 (2)	0.8260 (3)	0.0251 (5)
C11	0.75591 (14)	0.5204 (2)	0.7555 (2)	0.0190 (4)
C12	0.66077 (14)	0.5040 (2)	0.6465 (2)	0.0205 (4)
H1	0.5667 (10)	0.611 (2)	0.534 (2)	0.027 (6)*
H2	0.5176 (8)	0.822 (2)	0.628 (3)	0.034 (7)*
H3	0.5592 (13)	1.0406 (19)	0.694 (3)	0.030 (6)*
H4	0.7233 (16)	1.1027 (13)	0.736 (3)	0.032 (7)*
H5	0.8403 (8)	0.948 (2)	0.708 (3)	0.021 (6)*
H81	0.9608 (11)	0.498 (2)	0.697 (2)	0.030 (6)*
H82	0.9116 (16)	0.471 (2)	0.5138 (14)	0.034 (7)*
H91	0.8196 (14)	0.296 (2)	0.606 (2)	0.029 (6)*
H92	0.9130 (13)	0.281 (2)	0.736 (3)	0.037 (7)*
H11	0.7457 (14)	0.5865 (16)	0.8397 (19)	0.017 (5)*
H101	0.8467 (13)	0.421 (2)	0.9245 (19)	0.029 (6)*
H102	0.7565 (13)	0.3271 (18)	0.859 (2)	0.025 (6)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0233 (3)	0.0180 (3)	0.0459 (3)	−0.0022 (2)	−0.0034 (2)	0.0011 (3)
S2	0.0248 (3)	0.0352 (4)	0.0300 (3)	−0.0034 (2)	0.0043 (2)	0.0035 (2)
N1	0.0172 (9)	0.0175 (10)	0.0299 (9)	−0.0008 (7)	−0.0039 (7)	−0.0007 (7)
N2	0.0172 (8)	0.0202 (10)	0.0211 (8)	0.0010 (7)	0.0012 (7)	−0.0007 (7)
C1	0.0238 (10)	0.0169 (11)	0.0202 (9)	0.0007 (8)	0.0004 (8)	−0.0004 (8)
C2	0.0244 (11)	0.0242 (12)	0.0344 (12)	0.0036 (9)	0.0037 (9)	−0.0019 (10)
C3	0.0360 (13)	0.0219 (13)	0.0382 (13)	0.0085 (10)	0.0074 (10)	−0.0027 (10)
C4	0.0455 (14)	0.0177 (12)	0.0289 (11)	−0.0002 (10)	0.0041 (10)	−0.0019 (9)

C5	0.0300 (12)	0.0222 (12)	0.0220 (10)	−0.0046 (9)	−0.0008 (9)	0.0023 (9)
C6	0.0232 (10)	0.0180 (11)	0.0157 (9)	0.0001 (8)	0.0013 (8)	0.0009 (8)
C7	0.0191 (10)	0.0229 (11)	0.0150 (9)	−0.0025 (8)	−0.0027 (7)	−0.0005 (8)
C8	0.0224 (11)	0.0276 (13)	0.0317 (12)	0.0081 (10)	0.0015 (9)	−0.0012 (10)
C9	0.0259 (11)	0.0223 (12)	0.0351 (12)	0.0073 (9)	−0.0042 (10)	−0.0028 (10)
C10	0.0296 (12)	0.0187 (11)	0.0254 (10)	0.0021 (9)	−0.0035 (9)	0.0012 (9)
C11	0.0221 (10)	0.0162 (10)	0.0184 (9)	0.0004 (8)	0.0016 (8)	0.0000 (8)
C12	0.0203 (10)	0.0197 (11)	0.0224 (10)	0.0019 (8)	0.0065 (8)	−0.0010 (8)

Geometric parameters (Å, º)

S1—C12	1.657 (2)	C4—H4	0.947 (10)
S2—C7	1.665 (2)	C5—C6	1.400 (3)
N1—C12	1.337 (3)	C5—H5	0.956 (9)
N1—C1	1.419 (3)	C6—C7	1.489 (3)
N1—H1	0.855 (10)	C8—C9	1.520 (3)
N2—C7	1.330 (3)	C8—H81	0.984 (10)
N2—C11	1.474 (2)	C8—H82	0.984 (9)
N2—C8	1.476 (3)	C9—C10	1.527 (3)
C1—C2	1.394 (3)	C9—H91	0.986 (10)
C1—C6	1.409 (3)	C9—H92	0.986 (10)
C2—C3	1.379 (3)	C10—C11	1.523 (3)
C2—H2	0.944 (10)	C10—H101	0.996 (10)
C3—C4	1.389 (3)	C10—H102	0.986 (9)
C3—H3	0.942 (10)	C11—C12	1.521 (3)
C4—C5	1.374 (3)	C11—H11	0.984 (9)
C12—N1—C1	128.23 (17)	N2—C8—C9	103.87 (17)
C12—N1—H1	113.8 (16)	N2—C8—H81	107.5 (14)
C1—N1—H1	117.1 (16)	C9—C8—H81	110.5 (14)
C7—N2—C11	123.94 (16)	N2—C8—H82	109.3 (14)
C7—N2—C8	123.70 (17)	C9—C8—H82	116.0 (15)
C11—N2—C8	111.65 (16)	H81—C8—H82	109.2 (19)
C2—C1—C6	120.0 (2)	C8—C9—C10	102.99 (18)
C2—C1—N1	116.23 (18)	C8—C9—H91	110.8 (14)
C6—C1—N1	123.69 (18)	C10—C9—H91	111.3 (13)
C3—C2—C1	120.8 (2)	C8—C9—H92	112.0 (14)
C3—C2—H2	118.2 (16)	C10—C9—H92	112.0 (14)
C1—C2—H2	120.9 (16)	H91—C9—H92	108 (2)
C2—C3—C4	119.7 (2)	C11—C10—C9	103.89 (17)
C2—C3—H3	121.1 (15)	C11—C10—H101	105.2 (14)
C4—C3—H3	119.1 (15)	C9—C10—H101	111.0 (13)
C5—C4—C3	119.6 (2)	C11—C10—H102	113.1 (13)
C5—C4—H4	117.6 (15)	C9—C10—H102	114.2 (13)
C3—C4—H4	122.8 (15)	H101—C10—H102	109.0 (18)
C4—C5—C6	122.2 (2)	N2—C11—C12	107.68 (15)
C4—C5—H5	119.9 (14)	N2—C11—C10	102.94 (16)
C6—C5—H5	117.9 (14)	C12—C11—C10	116.32 (17)

C5—C6—C1	117.39 (19)	N2—C11—H11	109.3 (12)
C5—C6—C7	118.44 (18)	C12—C11—H11	107.4 (12)
C1—C6—C7	123.96 (19)	C10—C11—H11	112.9 (12)
N2—C7—C6	116.84 (17)	N1—C12—C11	113.76 (17)
N2—C7—S2	122.59 (16)	N1—C12—S1	122.01 (15)
C6—C7—S2	120.55 (16)	C11—C12—S1	124.22 (15)

C12—N1—C1—C2	−140.9 (2)	C5—C6—C7—S2	−36.1 (2)
C12—N1—C1—C6	41.2 (3)	C1—C6—C7—S2	138.49 (17)
C6—C1—C2—C3	−2.6 (3)	C7—N2—C8—C9	−178.51 (18)
N1—C1—C2—C3	179.4 (2)	C11—N2—C8—C9	10.8 (2)
C1—C2—C3—C4	−0.9 (3)	N2—C8—C9—C10	−30.2 (2)
C2—C3—C4—C5	3.0 (3)	C8—C9—C10—C11	38.8 (2)
C3—C4—C5—C6	−1.6 (3)	C7—N2—C11—C12	79.1 (2)
C4—C5—C6—C1	−1.8 (3)	C8—N2—C11—C12	−110.28 (18)
C4—C5—C6—C7	173.16 (19)	C7—N2—C11—C10	−157.51 (18)
C2—C1—C6—C5	3.8 (3)	C8—N2—C11—C10	13.1 (2)
N1—C1—C6—C5	−178.33 (18)	C9—C10—C11—N2	−31.7 (2)
C2—C1—C6—C7	−170.78 (19)	C9—C10—C11—C12	85.7 (2)
N1—C1—C6—C7	7.0 (3)	C1—N1—C12—C11	−6.1 (3)
C11—N2—C7—C6	−9.7 (3)	C1—N1—C12—S1	174.50 (16)
C8—N2—C7—C6	−179.26 (17)	N2—C11—C12—N1	−65.3 (2)
C11—N2—C7—S2	171.99 (14)	C10—C11—C12—N1	179.85 (18)
C8—N2—C7—S2	2.5 (3)	N2—C11—C12—S1	114.07 (17)
C5—C6—C7—N2	145.61 (19)	C10—C11—C12—S1	−0.8 (3)
C1—C6—C7—N2	−39.8 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···S1^{i}	0.86 (1)	2.58 (1)	3.411 (2)	166 (2)

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