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1,3-Bis(1-cyclohexylethyl)imidazolidine-2-thione

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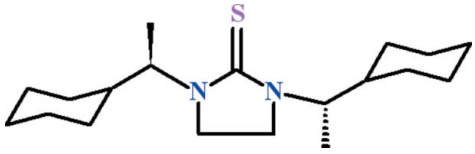
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.005 Å; R factor = 0.060; wR factor = 0.176; data-to-parameter ratio = 24.5.

The complete molecule of the title compound, $C_{19}H_{34}N_2S$, is generated by crystallographic twofold symmetry, with the C=S group lying on the rotation axis. A short C—H···S contact occurs in the molecule. The five-membered ring is twisted and the cyclohexyl ring adopts a chair conformation. The dihedral angle between the mean plane of the five-membered ring and the basal plane of the cyclohexyl ring is 75.32 (13)°.

Related literature

For a related structure, see: Kazak *et al.* (2005).



Experimental

Crystal data

$C_{19}H_{34}N_2S$
M_r = 322.54
Tetragonal, $P4_12_12$
a = 6.1008 (3) Å
c = 53.790 (2) Å
V = 2002.04 (17) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.16 mm^{-1}
T = 296 K
0.30 × 0.25 × 0.20 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.957, T_{max} = 0.966
18805 measured reflections
2500 independent reflections
1357 reflections with $I > 2\sigma(I)$
R_{int} = 0.047

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.060
$wR(F^2)$ = 0.176
S = 1.04
2500 reflections
102 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.12 e Å^{-3}
$\Delta\rho_{min}$ = −0.12 e Å^{-3}
Absolute structure: Flack (1983), with 874 Friedel pairs
Flack parameter: 0.0 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···S1	0.98	2.65	3.174 (3)	114

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2009); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6637).

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supporting information

Acta Cryst. (2012). E**68**, o743 [doi:10.1107/S1600536812006150]

1,3-Bis(1-cyclohexylethyl)imidazolidine-2-thione

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S1. Comment

The title compound (I), (Fig. 1) has been synthesized as a part of our project related to imidazolidinethione.

The crystal structure of 1,3-dibenzoyl-4,5-dihydro-1*H*-imidazole-2(3*H*)-thione (Kazak *et al.*, 2005) has been published which is related to the title compound (I), (Fig. 1).

The molecule has twofold symmetry about the C═S (C1═S1) of imidazolidinethione and therefore, the asymmetric unit is half of the molecule. The asymmetric part of imidazolidinethione moiety A (S1/C1/N1/C2) and the basal plane of cyclohexyl ring B (C6/C7/C9/C10) are almost planar with r.m.s. deviations of 0.036 and 0.004 Å, respectively. The dihedral angle between A/B is 75.32 (13)°. The cyclohexyl adopts chair conformation with apical C-atoms C5 and C8 at a distance of -0.651 (5) and 0.638 (8) Å, respectively from the basal plane B. There exist weak intramolecular H-bondings of C—H···S type (Table 1, Fig. 1) and form S(5) ring motif. No other interaction is found in the crystal.

S2. Experimental

(*S*)-1-cyclohexylethanamine (2.5 equiv.) and 1,2-dibromoethane (1 equiv.) were placed in a pressure vessel and heated at 393 K for 5 h, during which the reaction mixture solidified. The system was cooled to room temperature and NaOH (1 N, 20 ml) and ethyl acetate (20 ml) were added in to the reaction mixture. After dissolving the reaction mixture, the crude product was extracted with ethyl acetate (3 × 25 ml). The combined organic layers were concentrated and subjected to column chromatography. The product obtained from column chromatography (1 equiv.) was added to toluene (0.4 *M*) in pressure vessel and thiocarbonyldiimidazol (1.1 equiv.) was added to it. This mixture was heated about 373 K for 15 h. Again the extraction with ethyl acetate (3 × 25 ml) was carried out by using column chromatography to get the required product. Yield: 90%. Colourless prisms of (I) were obtained by recrystallizing from methanol after 48 h.

S3. Refinement

The H atoms were positioned geometrically (C—H = 0.93–0.98 Å) and refined as riding with U_{iso}(H) = xU_{eq}(C), where x = 1.5 for methyl and x = 1.2 for all other H atoms.

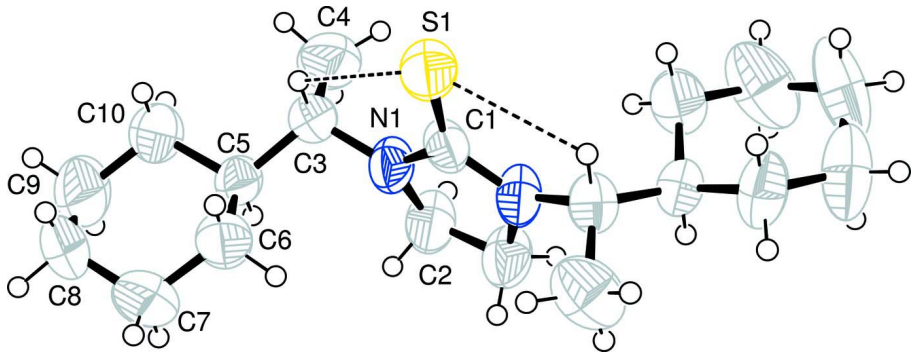

Figure 1

View of the title compound with displacement ellipsoids drawn at the 50% probability level. The dotted lines indicate the short C—H···S contacts.

1,3-Bis(1-cyclohexylethyl)imidazolidine-2-thione

Crystal data

$C_{19}H_{34}N_2S$
$M_r = 322.54$
Tetragonal, $P4_12_12$
Hall symbol: P 4abw 2nw
$a = 6.1008$ (3) Å
$c = 53.790$ (2) Å
$V = 2002.04$ (17) Å^3
$Z = 4$
$F(000) = 712$
$D_x = 1.070$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1358 reflections
$\theta = 3.0$–$28.3°$
$\mu = 0.16$ mm^{-1}
$T = 296$ K
Prism, colourless
0.30 × 0.25 × 0.20 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 7.50 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min} = 0.957$, $T_{max} = 0.966$
18805 measured reflections
2500 independent reflections
1357 reflections with $I > 2\sigma(I)$
$R_{int} = 0.047$
$\theta_{max} = 28.3°$, $\theta_{min} = 3.0°$
$h = -8\rightarrow4$
$k = -7\rightarrow8$
$l = -71\rightarrow65$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.060$
$wR(F^2) = 0.176$
$S = 1.04$
2500 reflections
102 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.069P)^2 + 0.4327P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.12$ e Å^{-3}
$\Delta\rho_{min} = -0.12$ e Å^{-3}
Absolute structure: Flack (1983), with 874 Friedel pairs
Absolute structure parameter: 0.0 (2)

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*-factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.56724 (12)	0.56724 (12)	0.0000	0.0822 (4)
N1	0.2683 (4)	0.2943 (4)	0.02021 (4)	0.0735 (7)
C1	0.3729 (4)	0.3729 (4)	0.0000	0.0652 (10)
C2	0.0922 (6)	0.1442 (6)	0.01337 (5)	0.0868 (10)
H2A	−0.0500	0.2138	0.0149	0.104*
H2B	0.0950	0.0131	0.0236	0.104*
C3	0.2873 (5)	0.3852 (5)	0.04524 (5)	0.0696 (8)
H3	0.4132	0.4849	0.0451	0.083*
C4	0.0864 (7)	0.5222 (6)	0.05128 (8)	0.1131 (13)
H4A	0.1147	0.6093	0.0658	0.170*
H4B	−0.0361	0.4273	0.0544	0.170*
H4C	0.0537	0.6167	0.0375	0.170*
C5	0.3390 (5)	0.2040 (5)	0.06392 (4)	0.0652 (8)
H5	0.2139	0.1032	0.0643	0.078*
C6	0.5406 (6)	0.0737 (6)	0.05665 (6)	0.0915 (10)
H6A	0.5139	0.0013	0.0409	0.110*
H6B	0.6630	0.1732	0.0544	0.110*
C7	0.6000 (9)	−0.0961 (7)	0.07594 (8)	0.1415 (18)
H7A	0.4867	−0.2075	0.0765	0.170*
H7B	0.7363	−0.1666	0.0712	0.170*
C8	0.6254 (10)	0.0031 (7)	0.10136 (8)	0.147 (2)
H8A	0.6529	−0.1123	0.1134	0.177*
H8B	0.7506	0.1011	0.1014	0.177*
C9	0.4288 (10)	0.1250 (8)	0.10869 (7)	0.1370 (18)
H9A	0.4537	0.1940	0.1247	0.164*
H9B	0.3070	0.0240	0.1105	0.164*
C10	0.3709 (7)	0.2961 (6)	0.09005 (5)	0.0976 (12)
H10A	0.4863	0.4054	0.0896	0.117*
H10B	0.2369	0.3685	0.0952	0.117*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0833 (6)	0.0833 (6)	0.0798 (7)	−0.0262 (6)	−0.0097 (5)	0.0097 (5)
N1	0.0914 (18)	0.0764 (16)	0.0526 (12)	−0.0234 (13)	−0.0101 (12)	0.0110 (12)
C1	0.0661 (15)	0.0661 (15)	0.063 (2)	−0.0067 (19)	−0.0152 (14)	0.0152 (14)

C2	0.095 (2)	0.099 (3)	0.0666 (16)	−0.037 (2)	−0.0083 (16)	0.0109 (16)
C3	0.086 (2)	0.0616 (18)	0.0614 (16)	−0.0014 (16)	−0.0048 (15)	0.0017 (13)
C4	0.121 (3)	0.091 (3)	0.128 (3)	0.026 (3)	−0.024 (3)	−0.017 (2)
C5	0.081 (2)	0.0624 (16)	0.0520 (14)	−0.0071 (15)	0.0022 (14)	0.0016 (13)
C6	0.109 (3)	0.087 (2)	0.079 (2)	0.027 (2)	−0.0062 (19)	−0.0082 (18)
C7	0.181 (5)	0.087 (3)	0.157 (4)	0.048 (3)	−0.061 (4)	−0.011 (3)
C8	0.238 (7)	0.094 (3)	0.110 (3)	0.014 (4)	−0.088 (4)	0.021 (2)
C9	0.219 (6)	0.124 (4)	0.068 (2)	−0.022 (4)	−0.019 (3)	0.024 (2)
C10	0.144 (4)	0.094 (2)	0.0548 (16)	0.008 (2)	0.0080 (19)	−0.0014 (17)

Geometric parameters (Å, º)

S1—C1	1.677 (3)	C3—H3	0.9800
N1—C1	1.349 (3)	C4—H4A	0.9600
N1—C2	1.458 (4)	C4—H4B	0.9600
N1—C3	1.461 (4)	C4—H4C	0.9600
C2—C2^{i}	1.506 (4)	C5—H5	0.9800
C3—C4	1.519 (5)	C6—H6A	0.9700
C3—C5	1.527 (4)	C6—H6B	0.9700
C5—C6	1.516 (5)	C7—H7A	0.9700
C5—C10	1.526 (4)	C7—H7B	0.9700
C6—C7	1.510 (6)	C8—H8A	0.9700
C7—C8	1.503 (6)	C8—H8B	0.9700
C8—C9	1.465 (8)	C9—H9A	0.9700
C9—C10	1.490 (6)	C9—H9B	0.9700
C2—H2A	0.9700	C10—H10A	0.9700
C2—H2B	0.9700	C10—H10B	0.9700
C1—N1—C2	111.6 (2)	H4A—C4—H4C	109.00
C1—N1—C3	124.8 (2)	H4B—C4—H4C	109.00
C2—N1—C3	122.0 (2)	C3—C5—H5	108.00
S1—C1—N1	125.87 (13)	C6—C5—H5	108.00
S1—C1—N1^{i}	125.87 (13)	C10—C5—H5	108.00
N1—C1—N1^{i}	108.3 (2)	C5—C6—H6A	109.00
N1—C2—C2^{i}	102.6 (3)	C5—C6—H6B	109.00
N1—C3—C4	110.0 (3)	C7—C6—H6A	109.00
N1—C3—C5	110.4 (2)	C7—C6—H6B	109.00
C4—C3—C5	115.1 (3)	H6A—C6—H6B	108.00
C3—C5—C6	112.2 (2)	C6—C7—H7A	109.00
C3—C5—C10	111.5 (3)	C6—C7—H7B	109.00
C6—C5—C10	109.1 (3)	C8—C7—H7A	109.00
C5—C6—C7	112.2 (3)	C8—C7—H7B	109.00
C6—C7—C8	111.9 (3)	H7A—C7—H7B	108.00
C7—C8—C9	111.4 (4)	C7—C8—H8A	109.00
C8—C9—C10	111.6 (4)	C7—C8—H8B	109.00
C5—C10—C9	113.1 (3)	C9—C8—H8A	109.00
N1—C2—H2A	111.00	C9—C8—H8B	109.00
N1—C2—H2B	111.00	H8A—C8—H8B	108.00

H2A—C2—H2B	109.00	C8—C9—H9A	109.00
C2^{i}—C2—H2A	111.00	C8—C9—H9B	109.00
C2^{i}—C2—H2B	111.00	C10—C9—H9A	109.00
N1—C3—H3	107.00	C10—C9—H9B	109.00
C4—C3—H3	107.00	H9A—C9—H9B	108.00
C5—C3—H3	107.00	C5—C10—H10A	109.00
C3—C4—H4A	109.00	C5—C10—H10B	109.00
C3—C4—H4B	109.00	C9—C10—H10A	109.00
C3—C4—H4C	109.00	C9—C10—H10B	109.00
H4A—C4—H4B	109.00	H10A—C10—H10B	108.00
C2—N1—C1—S1	173.7 (2)	N1—C3—C5—C10	177.2 (3)
C2—N1—C1—N1^{i}	−6.3 (3)	C4—C3—C5—C6	179.8 (3)
C3—N1—C1—S1	8.2 (4)	C4—C3—C5—C10	−57.6 (4)
C3—N1—C1—N1^{i}	−171.8 (2)	C3—C5—C6—C7	176.4 (3)
C1—N1—C2—C2^{i}	15.4 (3)	C10—C5—C6—C7	52.4 (4)
C3—N1—C2—C2^{i}	−178.6 (3)	C3—C5—C10—C9	−178.1 (4)
C1—N1—C3—C4	102.2 (3)	C6—C5—C10—C9	−53.7 (4)
C1—N1—C3—C5	−129.7 (3)	C5—C6—C7—C8	−54.2 (5)
C2—N1—C3—C4	−61.9 (4)	C6—C7—C8—C9	55.0 (5)
C2—N1—C3—C5	66.2 (3)	C7—C8—C9—C10	−55.6 (5)
N1—C2—C2^{i}—N1^{i}	−17.5 (3)	C8—C9—C10—C5	56.3 (5)
N1—C3—C5—C6	54.5 (3)		

Symmetry code: (i) *y*, *x*, −*z*.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···S1	0.98	2.65	3.174 (3)	114