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

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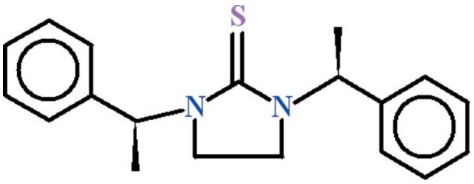
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.007 Å; R factor = 0.070; wR factor = 0.170; data-to-parameter ratio = 16.2.

The complete molecule of the title compound, $C_{19}H_{22}N_2S$, is generated by crystallographic twofold symmetry with the C=S group lying on the rotation axis. The imidazolidine ring adopts a flattened twist conformation. The dihedral angle between the asymmetric part of the imidazolidine-2-thione fragment and the benzene ring is 89.49 (17)°.

Related literature

For a related structure, see: Umar *et al.* (2012).



Experimental

Crystal data

$C_{19}H_{22}N_2S$
M_r = 310.45
Tetragonal, $P4_32_12$
a = 5.8692 (5) Å
c = 50.637 (5) Å
V = 1744.3 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.18 mm^{-1}
T = 296 K
0.28 × 0.24 × 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
18956 measured reflections
1717 independent reflections
1150 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.062

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.070
$wR(F^2)$ = 0.170
S = 1.11
1717 reflections
106 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\max}$ = 0.18 e Å^{-3}
$\Delta\rho_{\min}$ = −0.17 e Å^{-3}
Absolute structure: Flack (1983), 569 Friedel pairs
Flack parameter: 0.1 (3)

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: GK2471).

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supplementary materials

Acta Cryst. (2012). E**68**, o1188 [doi:10.1107/S160053681201224X]

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

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Comment

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

Recently we have reported the crystal structure of 1,3-bis(1-cyclohexylethyl)imidazolidine (Umar *et al.*, 2012) that is related to the title compound.

The molecule has twofold rotation symmetry about the C=S bond of imidazolidinethione fragment and therefore the asymmetric unit consists of half of the molecule. The asymmetric part of imidazolidinethione fragment A (S1/C1/N1/C2) and the benzene ring B (C6/C7/C9/C10) form the dihedral angle of 89.49 (17)°.

Experimental

(*S*)-1-Phenylethanamine (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 into 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 at 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: 80%).White prisms of of the title compound were obtained by recrystalization from methanol during 48 h (m.p. 416 K).

Refinement

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

Computing details

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT* (Bruker, 2007); 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* (Spek, 2009).

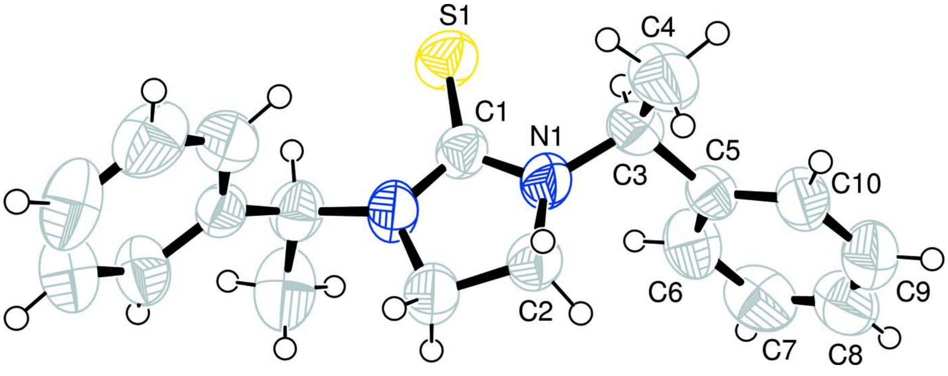

Figure 1

View of the title molecule with displacement ellipsoids drawn at the 50% probability level. H atoms are shown by small circles of arbitrary radii.

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

Crystal data

$C_{19}H_{22}N_2S$
$M_r = 310.45$
Tetragonal, $P4_32_12$
Hall symbol: P 4nw 2abw
$a = 5.8692$ (5) Å
$c = 50.637$ (5) Å
$V = 1744.3$ (3) Å^3
$Z = 4$
$F(000) = 664$

$D_x = 1.182$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1150 reflections
$\theta = 3.2$–26.0°
$\mu = 0.18$ mm^{-1}
$T = 296$ K
Prism, white
0.28 × 0.24 × 0.20 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 7.80 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min} = 0.957$, $T_{max} = 0.966$

18956 measured reflections
1717 independent reflections
1150 reflections with $I > 2\sigma(I)$
$R_{int} = 0.062$
$\theta_{max} = 26.0°$, $\theta_{min} = 3.2°$
$h = -3 \rightarrow 7$
$k = -7 \rightarrow 7$
$l = -62 \rightarrow 62$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.070$
$wR(F^2) = 0.170$
$S = 1.11$
1717 reflections
106 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0459P)^2 + 1.2139P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.18$ e Å^{-3}
$\Delta\rho_{min} = -0.17$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.011 (3)
Absolute structure: Flack (1983), 569 Friedel pairs
Flack parameter: 0.1 (3)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

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 (Å^2)

	x	y	z	$U_{\rm iso}$*/$U_{\rm eq}$
S1	1.12952 (18)	1.12952 (18)	0.0000	0.0805 (6)
N1	0.8146 (6)	0.8492 (6)	0.02136 (5)	0.0692 (10)
C1	0.9276 (6)	0.9276 (6)	0.0000	0.0598 (14)
C2	0.6343 (8)	0.6919 (7)	0.01411 (7)	0.0723 (12)
H2A	0.4858	0.7636	0.0154	0.087*
H2B	0.6367	0.5571	0.0252	0.087*
C3	0.8267 (8)	0.9498 (8)	0.04762 (8)	0.0687 (12)
H3	0.962 (7)	1.038 (7)	0.0469 (8)	0.082*
C4	0.6154 (10)	1.0894 (8)	0.05338 (9)	0.1018 (18)
H4A	0.5884	1.1932	0.0391	0.153*
H4B	0.6372	1.1738	0.0694	0.153*
H4C	0.4868	0.9897	0.0553	0.153*
C5	0.8783 (7)	0.7647 (7)	0.06798 (7)	0.0579 (10)
C6	1.0569 (8)	0.6131 (9)	0.06414 (9)	0.0843 (14)
H6	1.1454	0.6219	0.0489	0.101*
C7	1.1034 (9)	0.4468 (9)	0.08326 (11)	0.0963 (17)
H7	1.2213	0.3437	0.0805	0.116*
C8	0.9810 (11)	0.4343 (9)	0.10538 (10)	0.1002 (19)
H8	1.0161	0.3253	0.1181	0.120*
C9	0.8049 (10)	0.5808 (9)	0.10942 (9)	0.0949 (17)
H9	0.7157	0.5700	0.1246	0.114*
C10	0.7614 (8)	0.7429 (8)	0.09100 (7)	0.0764 (12)
H10	0.6442	0.8456	0.0943	0.092*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0776 (8)	0.0776 (8)	0.0861 (11)	−0.0227 (10)	−0.0077 (7)	0.0077 (7)
N1	0.085 (3)	0.070 (2)	0.0521 (17)	−0.0229 (18)	−0.0062 (16)	0.0039 (17)
C1	0.062 (2)	0.062 (2)	0.056 (3)	−0.002 (3)	−0.007 (2)	0.007 (2)
C2	0.084 (3)	0.073 (3)	0.060 (2)	−0.024 (2)	−0.003 (2)	0.0031 (19)
C3	0.080 (3)	0.063 (3)	0.063 (2)	−0.005 (2)	−0.007 (2)	−0.006 (2)
C4	0.138 (5)	0.081 (4)	0.086 (3)	0.046 (4)	−0.015 (3)	−0.008 (3)
C5	0.057 (2)	0.063 (2)	0.053 (2)	0.001 (2)	−0.005 (2)	−0.0054 (18)
C6	0.070 (3)	0.108 (4)	0.074 (3)	0.012 (3)	0.003 (2)	−0.006 (3)
C7	0.089 (4)	0.090 (4)	0.110 (4)	0.037 (3)	−0.024 (3)	−0.012 (3)
C8	0.140 (5)	0.084 (4)	0.077 (3)	0.021 (4)	−0.034 (3)	−0.002 (3)

C9	0.123 (5)	0.097 (4)	0.065 (3)	0.006 (4)	0.003 (3)	0.006 (3)
C10	0.092 (3)	0.079 (3)	0.058 (2)	0.017 (2)	0.004 (2)	−0.001 (2)

Geometric parameters (Å, º)

S1—C1	1.676 (5)	C4—H4C	0.9600
N1—C1	1.350 (4)	C5—C10	1.359 (5)
N1—C2	1.451 (5)	C5—C6	1.388 (6)
N1—C3	1.456 (5)	C6—C7	1.402 (7)
C1—N1$^{\mathrm{i}}$	1.350 (4)	C6—H6	0.9300
C2—C2$^{\mathrm{i}}$	1.507 (7)	C7—C8	1.333 (7)
C2—H2A	0.9700	C7—H7	0.9300
C2—H2B	0.9700	C8—C9	1.360 (7)
C3—C4	1.515 (6)	C8—H8	0.9300
C3—C5	1.528 (6)	C9—C10	1.357 (6)
C3—H3	0.95 (4)	C9—H9	0.9300
C4—H4A	0.9600	C10—H10	0.9300
C4—H4B	0.9600		

C1—N1—C2	111.9 (3)	C3—C4—H4C	109.5
C1—N1—C3	124.7 (3)	H4A—C4—H4C	109.5
C2—N1—C3	121.6 (3)	H4B—C4—H4C	109.5
N1$^{\mathrm{i}}$—C1—N1	107.9 (4)	C10—C5—C6	116.2 (4)
N1$^{\mathrm{i}}$—C1—S1	126.1 (2)	C10—C5—C3	123.1 (4)
N1—C1—S1	126.1 (2)	C6—C5—C3	120.7 (4)
N1—C2—C2$^{\mathrm{i}}$	102.7 (2)	C5—C6—C7	119.8 (4)
N1—C2—H2A	111.2	C5—C6—H6	120.1
C2$^{\mathrm{i}}$—C2—H2A	111.2	C7—C6—H6	120.1
N1—C2—H2B	111.2	C8—C7—C6	120.9 (5)
C2$^{\mathrm{i}}$—C2—H2B	111.2	C8—C7—H7	119.5
H2A—C2—H2B	109.1	C6—C7—H7	119.5
N1—C3—C4	110.8 (4)	C7—C8—C9	120.1 (5)
N1—C3—C5	109.7 (3)	C7—C8—H8	120.0
C4—C3—C5	114.6 (4)	C9—C8—H8	120.0
N1—C3—H3	103 (2)	C10—C9—C8	118.9 (5)
C4—C3—H3	113 (3)	C10—C9—H9	120.6
C5—C3—H3	104 (3)	C8—C9—H9	120.6
C3—C4—H4A	109.5	C9—C10—C5	124.1 (5)
C3—C4—H4B	109.5	C9—C10—H10	117.9
H4A—C4—H4B	109.5	C5—C10—H10	117.9

C2—N1—C1—N1$^{\mathrm{i}}$	−6.1 (2)	C4—C3—C5—C10	−7.4 (6)
C3—N1—C1—N1$^{\mathrm{i}}$	−171.0 (5)	N1—C3—C5—C6	49.8 (5)
C2—N1—C1—S1	173.9 (2)	C4—C3—C5—C6	175.2 (4)
C3—N1—C1—S1	9.0 (5)	C10—C5—C6—C7	1.4 (7)
C1—N1—C2—C2$^{\mathrm{i}}$	14.8 (5)	C3—C5—C6—C7	179.1 (4)
C3—N1—C2—C2$^{\mathrm{i}}$	−179.8 (4)	C5—C6—C7—C8	−1.2 (8)
C1—N1—C3—C4	102.8 (5)	C6—C7—C8—C9	1.5 (8)
C2—N1—C3—C4	−60.7 (5)	C7—C8—C9—C10	−2.0 (8)
C1—N1—C3—C5	−129.6 (4)	C8—C9—C10—C5	2.4 (8)

C2—N1—C3—C5	66.9 (5)	C6—C5—C10—C9	−2.1 (7)
N1—C3—C5—C10	−132.8 (4)	C3—C5—C10—C9	−179.7 (4)

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	0.95 (4)	2.63 (4)	3.176 (4)	117 (3)