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2-({4-[(1*H*-Imidazol-2-ylsulfanyl)methyl]-2,5-dimethylbenzyl}sulfanyl)-1*H*-imidazole

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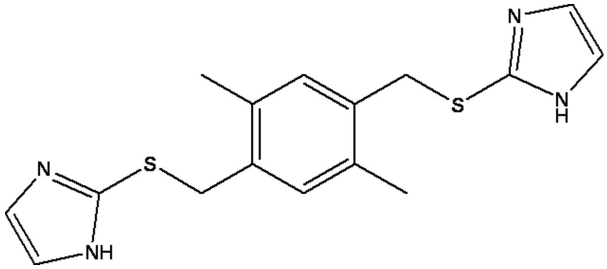
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.005 Å; R factor = 0.058; wR factor = 0.149; data-to-parameter ratio = 18.6.

The title compound, $C_{16}H_{18}N_4S_2$, was prepared by the substitution reaction of two equivalents of 2-mercaptoimidazole for every bromine substituent of 1,4-bis(bromomethyl)-2,5-dimethylbenzene. The molecule is located on a crystallographic centre of inversion and therefore adopts a *trans* configuration with regards to the orientation of the two sulfur atoms. An intermolecular N—H···N hydrogen bond forms layers of molecules parallel to ($\bar{1}$03). The dihedral angle between the central and terminal rings is 174.8 (2)°.

Related literature

For related structures, see: Fan *et al.* (2003); Voo *et al.* (2003).



Experimental

Crystal data

$C_{16}H_{18}N_4S_2$
M_r = 330.46
Monoclinic, $P2_1/n$
a = 6.169 (3) Å
b = 9.491 (5) Å
c = 13.722 (8) Å
β = 95.392 (8)°
V = 799.8 (8) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.33 mm^{-1}
T = 100 K
0.2 × 0.14 × 0.07 mm

Data collection

Bruker APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009) $T_{\min}$ = 0.945, $T_{\max}$ = 0.977
4896 measured reflections
1875 independent reflections
1154 reflections with $I > 2s\sigma(I)$
$R_{\rm int}$ = 0.075

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.149
S = 1.01
1875 reflections
101 parameters
H-atom parameters constrained
$\Delta\rho_{\max}$ = 0.67 e Å^{-3}
$\Delta\rho_{\min}$ = −0.39 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···N4$^{\rm i}$	0.88	1.93	2.791 (4)	165

Symmetry code: (i) $-x+\frac{3}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$.

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); 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: *X-SEED*.

The authors would like to thank the Central Analytical Facility (CAF) at the University of Stellenbosch (US) for the use of their diffractometer as well as the US and NRF for funding.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5352).

References

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Voo, J. K., Lam, K. C., Rheingold, A. L. & Riordan, C. G. (2003). *J. Chem. Soc. Dalton Trans.* pp. 1803–1805.

supplementary materials

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2-({4-[(1*H*-Imidazol-2-ylsulfanyl)methyl]-2,5-dimethylbenzyl}sulfanyl)-1*H*-imidazole

M. J. Applewhite and S. V. Potts

Comment

The molecule adopts a *trans* configuration with regards to the orientation of the two sulfur atoms.

Experimental

2-Mercaptoimidazole (1.60 g, 16 mmol) was added to 1,4-bis(bromomethyl)-2,5-dimethylbenzene (1.30 g, 4 mmol) in 200 mL of MeOH. The resulting solution was refluxed for 24 h.

The solvent was then removed *in vacuo* and K_2CO_3 (6.91 g, 50 mmol) in 100 mL of H_2O was added. The solution was stirred until the product precipitated. The white solid was then filtered, washed with 100 mL of H_2O and left to air dry.

Refinement

All hydrogen atoms were refined in calculated positions, using a riding model (C–H_{ar} = 0.95 Å; C—H = 0.99 Å; N–H = 0.88 Å) with U(H) set to $1.2U_{eq}$ of the parent atom (1.5 for methyl H atoms).

Figures

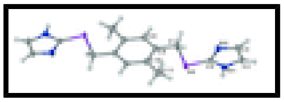

Fig. 1. The molecular structure of the title compound which has 50% probability ellipsoids. The *trans* relationship between the sulfur atoms can clearly be seen.

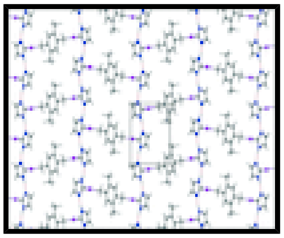

Fig. 2. The crystal packing viewed down the *c* axis shows N—H···N hydrogen bonding occurring to form chains throughout a layer of the crystal structure.

2-({4-[(1*H*-Imidazol-2-ylsulfanyl)methyl]-2,5- dimethylbenzyl}sulfanyl)-1*H*-imidazole

Crystal data

$C_{16}H_{18}N_4S_2$	$F(000) = 348$
$M_r = 330.46$	$D_x = 1.372$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P2yn	Cell parameters from 394 reflections
$a = 6.169$ (3) Å	$\theta = 2.6–28.2°$

$b = 9.491$ (5) Å

$c = 13.722$ (8) Å

$\beta = 95.392$ (8)°

$V = 799.8$ (8) Å^3

$Z = 2$

$\mu = 0.33$ mm^{-1}

$T = 100$ K

Shard, colourless

0.2 × 0.14 × 0.07 mm

Data collection

Bruker APEX CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

graphite

ω scans

Absorption correction: multi-scan (*SADABS*; Bruker, 2009)

$T_{\mathrm{min}} = 0.945$, $T_{\mathrm{max}} = 0.977$

4896 measured reflections

1875 independent reflections

1154 reflections with $I > 2s\sigma(I)$

$R_{\mathrm{int}} = 0.075$

$\theta_{\mathrm{max}} = 28.2°$, $\theta_{\mathrm{min}} = 2.6°$

$h = -8 \rightarrow 7$

$k = -12 \rightarrow 6$

$l = -17 \rightarrow 18$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.058$

$wR(F^2) = 0.149$

$S = 1.01$

1875 reflections

101 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

$w = 1/[\sigma^2(F_o^2) + (0.0617P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\mathrm{max}} < 0.001$

$\Delta\rho_{\mathrm{max}} = 0.67$ e Å^{-3}

$\Delta\rho_{\mathrm{min}} = -0.39$ e Å^{-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	$U_{\mathrm{iso}}*/U_{\mathrm{eq}}$
N1	0.7634 (4)	0.7794 (3)	0.28821 (19)	0.0230 (7)

H1	0.7160	0.6949	0.2705	0.028*
C2	0.8959 (5)	0.8119 (4)	0.3693 (2)	0.0249 (8)
H2	0.9548	0.7482	0.4182	0.030*
C3	0.9285 (5)	0.9529 (4)	0.3674 (2)	0.0257 (8)
H3	1.0158	1.0050	0.4154	0.031*
N4	0.8168 (4)	1.0086 (3)	0.28587 (18)	0.0250 (7)
C5	0.7181 (5)	0.8997 (4)	0.2400 (2)	0.0205 (7)
S6	0.55288 (13)	0.91254 (10)	0.12971 (6)	0.0241 (3)
C7	0.2861 (5)	0.9423 (4)	0.1750 (2)	0.0275 (8)
H7A	0.2403	0.8586	0.2109	0.033*
H7B	0.2914	1.0246	0.2195	0.033*
C8	0.1309 (5)	0.9690 (4)	0.0855 (2)	0.0233 (8)
C9	0.0933 (5)	1.1077 (4)	0.0551 (2)	0.0243 (8)
H9	0.1587	1.1819	0.0939	0.029*
C10	0.0364 (5)	0.8586 (4)	0.0300 (2)	0.0242 (8)
C11	0.0624 (6)	0.7085 (4)	0.0609 (3)	0.0320 (9)
H11A	−0.0269	0.6900	0.1149	0.048*
H11B	0.2156	0.6898	0.0825	0.048*
H11C	0.0154	0.6469	0.0055	0.048*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0274 (15)	0.0207 (16)	0.0199 (14)	−0.0032 (13)	−0.0031 (11)	−0.0010 (12)
C2	0.0244 (19)	0.030 (2)	0.0189 (17)	0.0024 (15)	−0.0067 (14)	0.0045 (15)
C3	0.0272 (19)	0.032 (2)	0.0163 (16)	−0.0024 (16)	−0.0055 (13)	0.0015 (15)
N4	0.0290 (16)	0.0248 (16)	0.0201 (14)	−0.0021 (13)	−0.0035 (12)	−0.0009 (13)
C5	0.0209 (17)	0.0212 (18)	0.0190 (16)	0.0042 (15)	−0.0002 (13)	−0.0013 (14)
S6	0.0221 (5)	0.0314 (5)	0.0177 (4)	0.0031 (4)	−0.0038 (3)	−0.0002 (4)
C7	0.0235 (18)	0.039 (2)	0.0194 (16)	0.0085 (16)	−0.0035 (13)	0.0023 (16)
C8	0.0189 (17)	0.032 (2)	0.0185 (16)	0.0028 (15)	0.0004 (13)	0.0008 (15)
C9	0.0182 (17)	0.029 (2)	0.0249 (17)	0.0009 (15)	0.0002 (13)	−0.0051 (15)
C10	0.0229 (18)	0.0268 (19)	0.0230 (17)	0.0031 (15)	0.0017 (14)	0.0031 (15)
C11	0.034 (2)	0.027 (2)	0.036 (2)	0.0035 (17)	0.0045 (16)	0.0035 (17)

Geometric parameters (Å, °)

N1—C5	1.336 (4)	C7—H7A	0.9900
N1—C2	1.354 (4)	C7—H7B	0.9900
N1—H1	0.8800	C8—C10	1.391 (5)
C2—C3	1.353 (5)	C8—C9	1.394 (5)
C2—H2	0.9500	C9—C10^{i}	1.389 (4)
C3—N4	1.364 (4)	C9—H9	0.9500
C3—H3	0.9500	C10—C9^{i}	1.389 (4)
N4—C5	1.327 (4)	C10—C11	1.491 (5)
C5—S6	1.748 (3)	C11—H11A	0.9800
S6—C7	1.835 (3)	C11—H11B	0.9800
C7—C8	1.506 (4)	C11—H11C	0.9800

C5—N1—C2	107.1 (3)	S6—C7—H7B	110.6
C5—N1—H1	126.5	H7A—C7—H7B	108.7
C2—N1—H1	126.5	C10—C8—C9	119.8 (3)
C3—C2—N1	106.8 (3)	C10—C8—C7	121.4 (3)
C3—C2—H2	126.6	C9—C8—C7	118.6 (3)
N1—C2—H2	126.6	C10^{i}—C9—C8	122.4 (3)
C2—C3—N4	109.6 (3)	C10^{i}—C9—H9	118.8
C2—C3—H3	125.2	C8—C9—H9	118.8
N4—C3—H3	125.2	C9^{i}—C10—C8	117.8 (3)
C5—N4—C3	105.1 (3)	C9^{i}—C10—C11	119.8 (3)
N4—C5—N1	111.5 (3)	C8—C10—C11	122.4 (3)
N4—C5—S6	124.1 (3)	C10—C11—H11A	109.5
N1—C5—S6	124.5 (3)	C10—C11—H11B	109.5
C5—S6—C7	100.70 (15)	H11A—C11—H11B	109.5
C8—C7—S6	105.7 (2)	C10—C11—H11C	109.5
C8—C7—H7A	110.6	H11A—C11—H11C	109.5
S6—C7—H7A	110.6	H11B—C11—H11C	109.5
C8—C7—H7B	110.6		

C5—N1—C2—C3	−0.4 (4)	C5—S6—C7—C8	174.8 (2)
N1—C2—C3—N4	0.2 (4)	S6—C7—C8—C10	83.0 (3)
C2—C3—N4—C5	0.1 (4)	S6—C7—C8—C9	−93.0 (3)
C3—N4—C5—N1	−0.4 (4)	C10—C8—C9—C10^{i}	−0.2 (5)
C3—N4—C5—S6	−179.6 (2)	C7—C8—C9—C10^{i}	175.9 (3)
C2—N1—C5—N4	0.5 (4)	C9—C8—C10—C9^{i}	0.2 (5)
C2—N1—C5—S6	179.7 (2)	C7—C8—C10—C9^{i}	−175.8 (3)
N4—C5—S6—C7	−90.1 (3)	C9—C8—C10—C11	−177.2 (3)
N1—C5—S6—C7	90.8 (3)	C7—C8—C10—C11	6.9 (5)

Symmetry codes: (i) −*x*, −*y*+2, −*z*.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···N4^{ii}	0.88	1.93	2.791 (4)	165

Symmetry codes: (ii) −*x*+3/2, *y*−1/2, −*z*+1/2.

Fig. 1

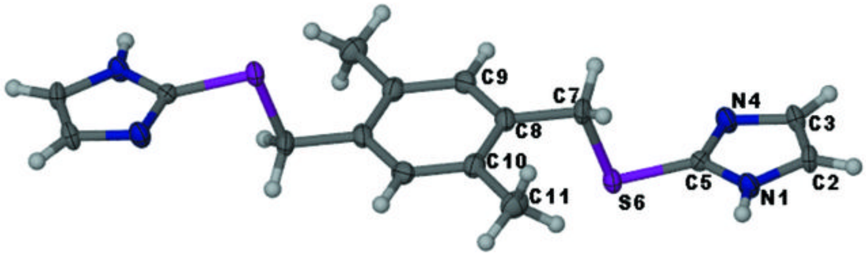

Fig. 2