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2-[(1*H*-Imidazol-2-yl)disulfanyl]-1*H*-imidazole

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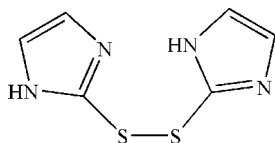
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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.055; wR factor = 0.185; data-to-parameter ratio = 18.0.

In the title molecule, $\text{C}_6\text{H}_6\text{N}_4\text{S}_2$, a twofold rotation axis passes through the mid-point of the S—S bond. The C—S—S—C torsion angle is $83.62(17)^\circ$. π - π stacking between imidazole rings of adjacent molecules is observed in the crystal structure, the centroid-centroid distance being $3.447(2)$ Å. Intermolecular N—H \cdots S hydrogen bonding results in the formation of a linear chain in the c -axis direction.

Related literature

For related imidazole disulfide compounds, see: Robina *et al.* (1990); Figueroa *et al.* (2007); Chernovyants *et al.* (2008).



Experimental

Crystal data

$\text{C}_6\text{H}_6\text{N}_4\text{S}_2$
 $M_r = 198.29$
 Monoclinic, $C2/c$
 $a = 14.083(3)$ Å
 $b = 6.3928(13)$ Å
 $c = 9.922(2)$ Å
 $\beta = 122.29(3)^\circ$

$V = 755.1(4)$ Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.64$ mm⁻¹
 $T = 298$ K
 $0.45 \times 0.25 \times 0.15$ mm

Data collection

STOE IPDS II diffractometer
 Absorption correction: multi-scan
 (*X-RED* and *X-SHAPE*; Stoe & Cie, 2005)
 $T_{\min} = 0.823$, $T_{\max} = 0.906$

4116 measured reflections
 1007 independent reflections
 948 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.112$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.055$
 $wR(F^2) = 0.185$
 $S = 1.18$
 1007 reflections

56 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.82$ e Å⁻³
 $\Delta\rho_{\min} = -0.56$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|---|-------|-------------|-------------|---------------|
| $\text{N2}-\text{H2A}\cdots\text{S1}^i$ | 0.86 | 2.44 | 3.227 (3) | 153 |

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

Data collection: *X-Area* (Stoe & Cie, 2005); cell refinement: *X-Area*; data reduction: *X-Area*; 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); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU5318).

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supplementary materials

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2-[(1*H*-Imidazol-2-yl)disulfanyl]-1*H*-imidazole

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Comment

There have been little attention to crystal structure determination of imidazole disulfides. The crystal structure of 2,2-Dithio-bis(1-*p*-tolyl-1*H*-imidazole-4-carboxaldehyde) (Robina *et al.*, 1990), bis(1-Phenylimidazol-2-yl)disulfide (Figuroa *et al.*, 2007), bis(1 - *t*-Butylimidazol-2-yl)disulfide (Figuroa *et al.*, 2007) and 2,2-Dithiobis(1-methylimidazol-3-ium-2-yl) bis(tri-iodide) di-iodine (Chernovyants *et al.*, 2008) have reported previously. Here we report the crystal structure of 2-(2-(1*H*-imidazol-2-yl)disulfanyl)-1*H*-imidazole. The asymmetric unit of the title compound, (I), contains one half-molecule and a twofold rotation axis passes through the middle of S—S bond (Fig. 1). The S—S bond distance is 2.0713 (14) Å. In this compound the imidazole rings are of course planar and the angle between these rings is 21.83 (19) °. The torsion angle of C1—S1—S1a—C1a (a: $-x, y, -z + 1/2$) is 83.62 (17) °. The ineramolecular N—H···S hydrogen bonds (Table 1) result in the formation of a linear chain in *c*-direction. Further pi-pi interaction between imidazole rings of adjacent chains in *a*-direction (cg···cg distance of 3.4466 (19) Å, *sym* code; $-x, 1 - y, 1 - z$) results in the formation of a supramolecular structure.

Experimental

The title compound has been synthesized during the stirring of 1*H*-imidazole-2-thiol with thallium(I) acetate in 2:1 molar ration in methanol. The suitable crystals for X-ray analysis were obtained by slow evaporation from methanol solution after one week (yield; 75.5%).

Refinement

H atoms were positioned geometrically with C—H = 0.93 and N—H = 0.86 Å, and constrained to ride on their parent atoms with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C}, \text{N})$.

Figures

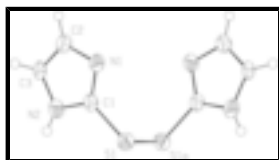


Fig. 1. The molecular structure with the atom-numbering scheme. Displacement ellipsoids are drawn at 30% probability level.

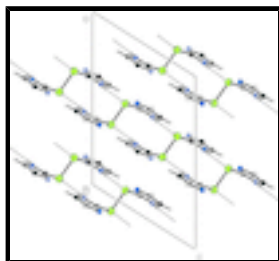


Fig. 2. A packing diagram of (I). Hydrogen bonds are shown as dashed lines.

2-[(1*H*-Imidazol-2-yl)disulfanyl]-1*H*-imidazole

Crystal data

| | |
|-------------------------------|---|
| $C_6H_6N_4S_2$ | $F(000) = 408$ |
| $M_r = 198.29$ | $D_x = 1.744 \text{ Mg m}^{-3}$ |
| Monoclinic, $C2/c$ | Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$ |
| Hall symbol: $-C\ 2yc$ | Cell parameters from 1007 reflections |
| $a = 14.083 (3) \text{ \AA}$ | $\theta = 3.4\text{--}23^\circ$ |
| $b = 6.3928 (13) \text{ \AA}$ | $\mu = 0.64 \text{ mm}^{-1}$ |
| $c = 9.922 (2) \text{ \AA}$ | $T = 298 \text{ K}$ |
| $\beta = 122.29 (3)^\circ$ | Prism, colorless |
| $V = 755.1 (4) \text{ \AA}^3$ | $0.45 \times 0.25 \times 0.15 \text{ mm}$ |
| $Z = 4$ | |

Data collection

| | |
|---|--|
| STOE IPDS II diffractometer | 1007 independent reflections |
| graphite | 948 reflections with $I > 2\sigma(I)$ |
| Detector resolution: $0.15 \text{ pixels mm}^{-1}$ | $R_{\text{int}} = 0.112$ |
| rotation method scans | $\theta_{\text{max}} = 29.2^\circ$, $\theta_{\text{min}} = 3.4^\circ$ |
| Absorption correction: multi-scan (<i>X-RED</i> and <i>X-SHAPE</i> ; Stoe & Cie, 2005) | $h = -19 \rightarrow 19$ |
| $T_{\text{min}} = 0.823$, $T_{\text{max}} = 0.906$ | $k = -8 \rightarrow 6$ |
| 4116 measured reflections | $l = -13 \rightarrow 13$ |

Refinement

| | |
|--|--|
| Refinement on F^2 | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)] = 0.055$ | H-atom parameters constrained |
| $wR(F^2) = 0.185$ | $w = 1/[\sigma^2(F_o^2) + (0.1134P)^2 + 0.6898P]$ |
| $S = 1.18$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| 1007 reflections | $(\Delta\sigma)_{\text{max}} < 0.001$ |
| 56 parameters | $\Delta\rho_{\text{max}} = 0.82 \text{ e \AA}^{-3}$ |
| 0 restraints | $\Delta\rho_{\text{min}} = -0.56 \text{ e \AA}^{-3}$ |
| Primary atom site location: structure-invariant direct methods | Extinction correction: <i>SHELXL</i> |
| | Extinction coefficient: $0.11 (3)$ |

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 (\AA^2)

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|-------------|-------------|----------------------------------|
| S1 | 0.08686 (5) | 0.07449 (9) | 0.32168 (6) | 0.0337 (4) |
| N1 | 0.13136 (17) | 0.4761 (4) | 0.4347 (2) | 0.0319 (5) |
| N2 | 0.11134 (18) | 0.2432 (4) | 0.5892 (2) | 0.0350 (6) |
| H2A | 0.1006 | 0.129 | 0.625 | 0.042* |
| C1 | 0.11109 (17) | 0.2743 (4) | 0.4570 (2) | 0.0281 (5) |
| C2 | 0.1440 (2) | 0.5734 (4) | 0.5611 (3) | 0.0335 (6) |
| H2 | 0.1585 | 0.7155 | 0.5821 | 0.04* |
| C3 | 0.13289 (18) | 0.4382 (4) | 0.6536 (2) | 0.0280 (5) |
| H3 | 0.139 | 0.4723 | 0.7491 | 0.034* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|----|-------------|-------------|-------------|--------------|------------|---------------|
| S1 | 0.0385 (5) | 0.0326 (5) | 0.0294 (5) | 0.00531 (19) | 0.0178 (4) | -0.00166 (17) |
| N1 | 0.0407 (10) | 0.0348 (11) | 0.0256 (9) | -0.0026 (8) | 0.0213 (7) | -0.0020 (7) |
| N2 | 0.0451 (11) | 0.0375 (11) | 0.0271 (10) | 0.0024 (8) | 0.0226 (8) | 0.0042 (7) |
| C1 | 0.0300 (10) | 0.0334 (11) | 0.0222 (10) | 0.0028 (8) | 0.0147 (8) | 0.0011 (7) |
| C2 | 0.0382 (12) | 0.0369 (14) | 0.0260 (11) | -0.0032 (8) | 0.0175 (9) | -0.0040 (7) |
| C3 | 0.0316 (10) | 0.0373 (13) | 0.0180 (9) | 0.0023 (7) | 0.0151 (8) | -0.0010 (7) |

Geometric parameters (\AA , $^\circ$)

| | | | |
|-----------------------|--------------|---------------------------|-------------|
| S1—C1 | 1.750 (2) | N2—C3 | 1.359 (3) |
| S1—S1 ⁱ | 2.0713 (14) | N2—H2A | 0.86 |
| N1—C2 | 1.324 (3) | C2—C3 | 1.329 (3) |
| N1—C1 | 1.364 (3) | C2—H2 | 0.93 |
| N2—C1 | 1.324 (3) | C3—H3 | 0.93 |
| C1—S1—S1 ⁱ | 101.62 (7) | N1—C1—S1 | 122.51 (16) |
| C2—N1—C1 | 102.96 (19) | N1—C2—C3 | 110.0 (2) |
| C1—N2—C3 | 102.12 (19) | N1—C2—H2 | 125 |
| C1—N2—H2A | 128.9 | C3—C2—H2 | 125 |
| C3—N2—H2A | 128.9 | C2—C3—N2 | 110.52 (19) |
| N2—C1—N1 | 114.4 (2) | C2—C3—H3 | 124.7 |
| N2—C1—S1 | 123.13 (18) | N2—C3—H3 | 124.7 |
| C3—N2—C1—N1 | 0.1 (3) | S1 ⁱ —S1—C1—N1 | -93.68 (18) |
| C3—N2—C1—S1 | -179.85 (15) | C1—N1—C2—C3 | 0.5 (3) |
| C2—N1—C1—N2 | -0.4 (3) | N1—C2—C3—N2 | -0.4 (3) |

supplementary materials

| | | | |
|---------------------------|-------------|-------------|---------|
| C2—N1—C1—S1 | 179.60 (17) | C1—N2—C3—C2 | 0.2 (3) |
| S1 ⁱ —S1—C1—N2 | 86.31 (19) | | |

Symmetry codes: (i) $-x, y, -z+1/2$.

Hydrogen-bond geometry (\AA , $^\circ$)

| <i>D</i> —H \cdots <i>A</i> | <i>D</i> —H | H \cdots <i>A</i> | <i>D</i> \cdots <i>A</i> | <i>D</i> —H \cdots <i>A</i> |
|----------------------------------|-------------|---------------------|----------------------------|-------------------------------|
| N2—H2A \cdots S1 ⁱⁱ | 0.86 | 2.44 | 3.227 (3) | 153 |

Symmetry codes: (ii) $x, -y, z+1/2$.

Fig. 1

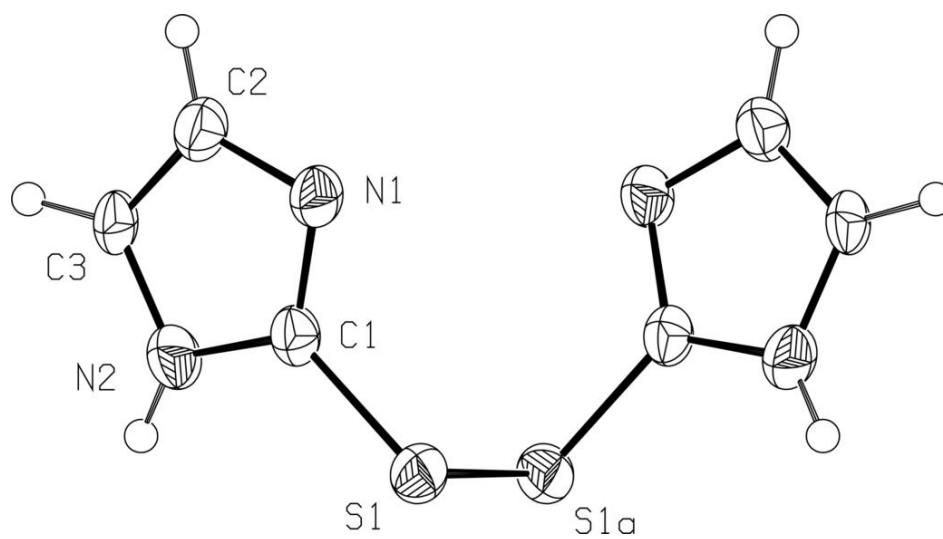


Fig. 2

