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# Crystal structure of 3,3′-diisopropyl-1,1′-(pyridine-2,6-diyl)bis[1*H*-imidazole-2(3*H*)-thione]

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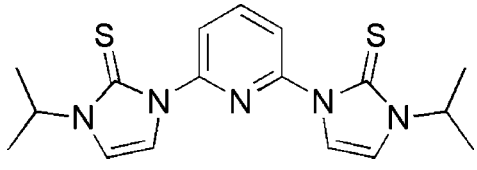
In the title compound, $C_{17}H_{21}N_5S_2$, the dihedral angles between the central pyridine ring and its pendant imidazole rings are 29.40 (9) and 40.77 (9)°; the pendant rings are twisted in an opposite sense with respect to the central ring. In each case, the S atom is approximately anti to the N atom of the pyridine ring. For both substituents, the H atom attached to the central C atom of the isopropyl group is approximately syn to the S atom in the attached ring. In the crystal, molecules are linked by weak C—H···S interactions, generating *C*(5) chains propagating along [001].

**Keywords:** crystal structure; organochalcogen ligand; conformation; C—H···S interactions.

**CCDC reference**: 1054528

## 1. Related literature

For applications of organochalcogen compounds in chemistry, see: Owen (2012). For the synthesis of the starting reagent, 2,6-bis(1-isopropylimidazolium)pyridine dibromide, see: McGuinness *et al.* (2004). For the synthesis of the title compound, see: Jia *et al.* (2009*a*). For the crystal structure of a similar coumpound, see: Jia *et al.* (2009*b*)



## 2. Experimental

### 2.1. Crystal data

$C_{17}H_{21}N_5S_2$
$M_r$ = 359.51
Monoclinic, $P2_1/c$
$a$ = 14.7942 (11) Å
$b$ = 8.9398 (7) Å
$c$ = 13.8194 (11) Å
$\beta$ = 101.675 (1)°
$V$ = 1789.9 (2) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.31 mm$^{-1}$
$T$ = 293 K
0.20 × 0.19 × 0.19 mm

### 2.2. Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
$T_{min}$ = 0.941, $T_{max}$ = 0.944
14858 measured reflections
4083 independent reflections
3131 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.028

### 2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.111
$S$ = 1.02
4083 reflections
217 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.27 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.31 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C13—H13$A$···S2$^{i}$ | 0.93 | 2.81 | 3.7214 (18) | 166 |

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

Data collection: *SMART* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

## Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: HB7387).

## References

Bruker (2008). *SMART*, *SAINT* and *SADABS*. Bruker AXS Inc, Madision, Wisconsin, USA.
Jia, W.-G., Huang, Y.-B. & Jin, G.-X. (2009*a*). *J. Organomet. Chem.* **694**, 4008–4013.
Jia, W.-G., Huang, Y.-B. & Jin, G.-X. (2009*b*). *J. Organomet. Chem.* **694**, 3376–3380.
McGuinness, D. S., Gibson, V. C. & Steed, J. W. (2004). *Organometallics*, **23**, 6288–6292.
Owen, G. R. (2012). *Chem. Soc. Rev.* **41**, 3535–3546.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

# supporting information

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## Crystal structure of 3,3′-diisopropyl-1,1′-(pyridine-2,6-diyl)bis[1*H*-imidazole-2(3*H*)-thione]

**Ying Sun, Hui Wang and Wei-Guo Jia**

### S1. Introduction

The title compound (Fig. 1)was prepared as an intermediate in our ongoing search (Jia *et al.*, 2009*a*) for organochalcogen ligands. The title compound was thermally stable and inert toward air and moisture in the solid state, and was soluble in common organic solvents such as $CH_2Cl_2$, $CHCl_3$ and THF.

The bond lengths and angles are normal and correspond to those observed in the related 2,6-bis(1-*tert*-butylimidazole-2-thione)pyridine (Jia *et al.*, 2009*b*).

### S2. Experimental

#### S2.1. Synthesis and crystallization

The title compound was prepared following the known procedure (Jia *et al.*, 2009*a*). In a 100 mL round-bottomed flask fitted with reflux condenser were placed 2,6-bis(1-isopropylimidazolium)pyridine dibromide (4.65 g, 10 mmol), S (0.64 g, 20 mmol) and 2.8 g $K_2CO_3$ and 50 mL methanol as solvent. The mixture was allowed to reflux for 8 h after which the methanol was removed with a rotary evaporator. The remaining solid was shaken with 2 × 30 mL $CH_2Cl_2$ which was then filtered and rotary evaporated. The product was recrystallized from $CH_2Cl_2$/MeOH to give colorless solid, Yield: (2.80 g 78%).

#### S2.2. Refinement

All hydrogen atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H = 0.93–0.97 Å and $U_{iso}$(H) = 1.2–1.5$U_{eq}$.

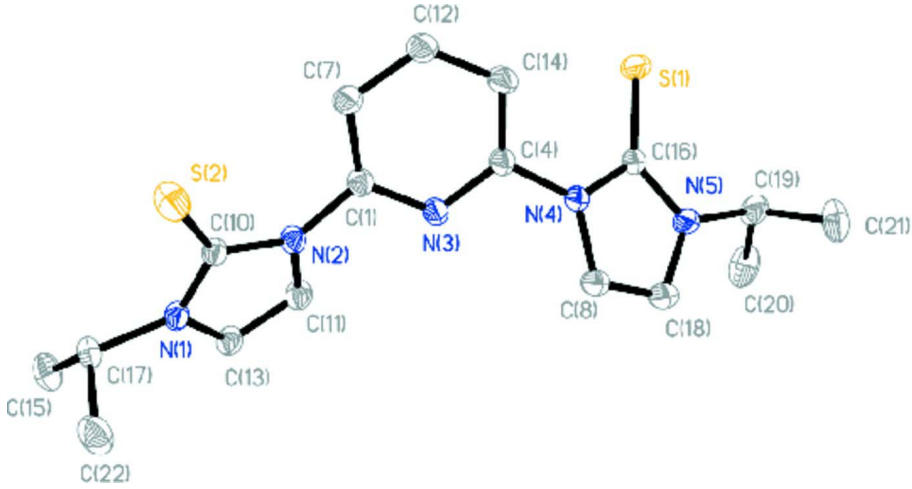


**Figure 1**

The molecular structure of title molecule showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. All hydrogen atoms are omitted for clarity.

**3,3′-Diisopropyl-1,1′-(pyridine-2,6-diyl)bis[1*H*-imidazole-2(3*H*)-thione]**

*Crystal data*

$C_{17}H_{21}N_5S_2$
$M_r = 359.51$
Monoclinic, $P2_1/c$
$a = 14.7942$ (11) Å
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$c = 13.8194$ (11) Å
$\beta = 101.675$ (1)°
$V = 1789.9$ (2) Å$^3$
$Z = 4$
$F(000) = 760$
$D_x = 1.334$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 5873 reflections
$\theta = 2.3$–27.4°
$\mu = 0.31$ mm$^{-1}$
$T = 293$ K
Prism, colorless
0.20 × 0.19 × 0.19 mm

*Data collection*

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
$T_{min} = 0.941$, $T_{max} = 0.944$
14858 measured reflections
4083 independent reflections
3131 reflections with $I > 2\sigma(I)$
$R_{int} = 0.028$
$\theta_{max} = 27.5°$, $\theta_{min} = 1.4°$
$h = -19 \rightarrow 19$
$k = -10 \rightarrow 11$
$l = -16 \rightarrow 17$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.111$
$S = 1.02$
4083 reflections
217 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
$w = 1/[\sigma^2(F_o^2) + (0.0574P)^2 + 0.4683P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.27$ e Å$^{-3}$
$\Delta\rho_{min} = -0.31$ 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 (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| S1 | 0.98780 (3) | 0.42684 (7) | 0.26709 (4) | 0.05246 (17) |
| N1 | 0.70991 (9) | 0.27508 (16) | 0.09090 (10) | 0.0329 (3) |
| C1 | 0.62838 (10) | 0.28936 (19) | 0.11628 (12) | 0.0328 (4) |
| N2 | 0.86809 (8) | 0.25912 (16) | 0.12935 (10) | 0.0317 (3) |
| C2 | 0.61678 (12) | 0.3113 (2) | 0.21163 (13) | 0.0392 (4) |
| H2A | 0.5585 | 0.3252 | 0.2259 | 0.047* |
| C3 | 0.69534 (12) | 0.3118 (2) | 0.28522 (13) | 0.0412 (4) |
| H3A | 0.6904 | 0.3248 | 0.3507 | 0.049* |
| N4 | 0.55290 (9) | 0.28823 (17) | 0.03416 (10) | 0.0338 (3) |
| C4 | 0.78119 (11) | 0.2932 (2) | 0.26209 (13) | 0.0381 (4) |
| H4A | 0.8346 | 0.2907 | 0.3110 | 0.046* |
| N5 | 0.42350 (9) | 0.24388 (18) | −0.06671 (11) | 0.0381 (3) |
| C5 | 0.78467 (10) | 0.27858 (18) | 0.16342 (12) | 0.0320 (3) |
| C6 | 0.87118 (11) | 0.1809 (2) | 0.04339 (12) | 0.0364 (4) |
| H6A | 0.8219 | 0.1327 | 0.0029 | 0.044* |
| C7 | 0.95753 (11) | 0.1872 (2) | 0.02932 (13) | 0.0370 (4) |
| H7A | 0.9795 | 0.1438 | −0.0226 | 0.044* |
| C8 | 0.95469 (10) | 0.31641 (18) | 0.16853 (12) | 0.0321 (4) |
| C9 | 1.15933 (12) | 0.1751 (2) | 0.08642 (16) | 0.0503 (5) |
| H9A | 1.1498 | 0.0880 | 0.1237 | 0.075* |
| H9B | 1.2240 | 0.1981 | 0.0981 | 0.075* |
| H9C | 1.1371 | 0.1562 | 0.0173 | 0.075* |
| C10 | 1.10746 (11) | 0.3058 (2) | 0.11811 (14) | 0.0376 (4) |
| H10A | 1.1317 | 0.3230 | 0.1886 | 0.045* |
| C11 | 1.11964 (14) | 0.4489 (3) | 0.06389 (19) | 0.0612 (6) |
| H11A | 1.0858 | 0.5280 | 0.0873 | 0.092* |
| H11B | 1.0970 | 0.4348 | −0.0057 | 0.092* |
| H11C | 1.1839 | 0.4746 | 0.0756 | 0.092* |
| C12 | 0.55688 (12) | 0.3554 (2) | −0.05536 (13) | 0.0412 (4) |
| H12A | 0.6063 | 0.4095 | −0.0696 | 0.049* |
| C13 | 0.47749 (12) | 0.3283 (2) | −0.11682 (13) | 0.0429 (4) |
| H13A | 0.4610 | 0.3604 | −0.1820 | 0.052* |
| C14 | 0.46937 (11) | 0.2156 (2) | 0.02667 (12) | 0.0343 (4) |
| C15 | 0.33079 (16) | 0.0985 (3) | −0.20167 (18) | 0.0644 (6) |
| H15A | 0.3733 | 0.0167 | −0.1858 | 0.097* |

| | | | | |
|---|---|---|---|---|
| H15B | 0.3496 | 0.1609 | −0.2505 | 0.097* |
| H15C | 0.2700 | 0.0600 | −0.2271 | 0.097* |
| C16 | 0.32985 (12) | 0.1895 (2) | −0.10926 (14) | 0.0461 (5) |
| H16A | 0.3102 | 0.1238 | −0.0606 | 0.055* |
| C17 | 0.26378 (14) | 0.3198 (3) | −0.12819 (18) | 0.0624 (6) |
| H17A | 0.2647 | 0.3725 | −0.0675 | 0.094* |
| H17B | 0.2025 | 0.2835 | −0.1538 | 0.094* |
| H17C | 0.2821 | 0.3862 | −0.1753 | 0.094* |
| S2 | 0.43238 (3) | 0.11047 (6) | 0.11128 (3) | 0.04734 (15) |
| N3 | 1.00857 (9) | 0.26980 (15) | 0.10570 (10) | 0.0320 (3) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| S1 | 0.0442 (3) | 0.0663 (4) | 0.0481 (3) | −0.0154 (2) | 0.0122 (2) | −0.0280 (2) |
| N1 | 0.0281 (6) | 0.0399 (8) | 0.0307 (7) | −0.0005 (6) | 0.0055 (5) | −0.0020 (6) |
| C1 | 0.0289 (7) | 0.0370 (9) | 0.0321 (9) | −0.0014 (6) | 0.0052 (6) | −0.0018 (7) |
| N2 | 0.0271 (6) | 0.0376 (8) | 0.0295 (7) | 0.0002 (5) | 0.0033 (5) | −0.0056 (6) |
| C2 | 0.0333 (8) | 0.0507 (11) | 0.0350 (9) | −0.0015 (7) | 0.0103 (7) | −0.0051 (8) |
| C3 | 0.0415 (9) | 0.0539 (11) | 0.0287 (9) | −0.0068 (8) | 0.0084 (7) | −0.0052 (8) |
| N4 | 0.0273 (6) | 0.0459 (8) | 0.0279 (7) | 0.0001 (6) | 0.0052 (5) | 0.0013 (6) |
| C4 | 0.0343 (8) | 0.0471 (10) | 0.0311 (9) | −0.0035 (7) | 0.0019 (7) | −0.0015 (7) |
| N5 | 0.0317 (7) | 0.0496 (9) | 0.0307 (7) | −0.0029 (6) | 0.0011 (6) | 0.0035 (7) |
| C5 | 0.0294 (7) | 0.0333 (9) | 0.0328 (9) | −0.0013 (6) | 0.0052 (6) | −0.0024 (7) |
| C6 | 0.0332 (8) | 0.0427 (10) | 0.0308 (9) | 0.0003 (7) | 0.0008 (7) | −0.0088 (7) |
| C7 | 0.0355 (8) | 0.0447 (10) | 0.0299 (9) | 0.0041 (7) | 0.0040 (7) | −0.0070 (7) |
| C8 | 0.0293 (7) | 0.0348 (9) | 0.0311 (9) | 0.0004 (6) | 0.0034 (6) | −0.0014 (7) |
| C9 | 0.0335 (9) | 0.0548 (12) | 0.0640 (13) | 0.0066 (8) | 0.0131 (9) | −0.0002 (10) |
| C10 | 0.0257 (7) | 0.0468 (10) | 0.0390 (10) | −0.0017 (7) | 0.0034 (7) | −0.0015 (8) |
| C11 | 0.0445 (11) | 0.0536 (13) | 0.0861 (17) | −0.0055 (9) | 0.0149 (11) | 0.0126 (12) |
| C12 | 0.0363 (9) | 0.0543 (11) | 0.0342 (9) | −0.0048 (8) | 0.0103 (7) | 0.0044 (8) |
| C13 | 0.0413 (9) | 0.0568 (12) | 0.0300 (9) | −0.0008 (8) | 0.0055 (7) | 0.0078 (8) |
| C14 | 0.0276 (7) | 0.0439 (10) | 0.0310 (9) | 0.0014 (7) | 0.0047 (6) | 0.0007 (7) |
| C15 | 0.0577 (13) | 0.0512 (13) | 0.0718 (16) | 0.0040 (10) | −0.0162 (11) | −0.0118 (11) |
| C16 | 0.0344 (9) | 0.0589 (12) | 0.0396 (10) | −0.0094 (8) | −0.0050 (7) | 0.0097 (9) |
| C17 | 0.0375 (10) | 0.0818 (17) | 0.0643 (14) | 0.0058 (10) | 0.0019 (10) | −0.0163 (13) |
| S2 | 0.0384 (2) | 0.0678 (4) | 0.0345 (3) | −0.0096 (2) | 0.00434 (18) | 0.0117 (2) |
| N3 | 0.0275 (6) | 0.0369 (7) | 0.0304 (7) | 0.0015 (5) | 0.0026 (5) | −0.0021 (6) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| S1—C8 | 1.6732 (17) | N5—C14 | 1.355 (2) |
| N1—C1 | 1.329 (2) | N5—C13 | 1.382 (2) |
| N1—C5 | 1.334 (2) | N5—C16 | 1.474 (2) |
| C1—C2 | 1.377 (2) | C6—C7 | 1.332 (2) |
| C1—N4 | 1.422 (2) | C7—N3 | 1.381 (2) |
| N2—C6 | 1.387 (2) | C8—N3 | 1.358 (2) |
| N2—C8 | 1.384 (2) | C9—C10 | 1.511 (3) |

| | | | |
|---|---|---|---|
| N2—C5 | 1.418 (2) | C10—N3 | 1.474 (2) |
| C2—C3 | 1.381 (2) | C10—C11 | 1.511 (3) |
| C3—C4 | 1.381 (2) | C12—C13 | 1.325 (2) |
| N4—C12 | 1.387 (2) | C14—S2 | 1.6759 (18) |
| N4—C14 | 1.381 (2) | C15—C16 | 1.517 (3) |
| C4—C5 | 1.381 (2) | C16—C17 | 1.508 (3) |
| | | | |
| C1—N1—C5 | 117.28 (14) | C7—C6—N2 | 107.61 (14) |
| N1—C1—C2 | 124.19 (15) | C6—C7—N3 | 107.66 (15) |
| N1—C1—N4 | 113.38 (14) | N3—C8—N2 | 104.65 (13) |
| C2—C1—N4 | 122.34 (14) | N3—C8—S1 | 126.06 (12) |
| C6—N2—C8 | 109.47 (13) | N2—C8—S1 | 129.20 (12) |
| C6—N2—C5 | 121.87 (13) | N3—C10—C11 | 110.02 (14) |
| C8—N2—C5 | 128.58 (14) | N3—C10—C9 | 110.36 (15) |
| C3—C2—C1 | 117.12 (15) | C11—C10—C9 | 113.10 (16) |
| C2—C3—C4 | 120.37 (16) | C13—C12—N4 | 107.49 (15) |
| C12—N4—C14 | 109.60 (14) | C12—C13—N5 | 107.80 (16) |
| C12—N4—C1 | 122.72 (13) | N5—C14—N4 | 104.58 (14) |
| C14—N4—C1 | 127.55 (14) | N5—C14—S2 | 126.72 (13) |
| C5—C4—C3 | 117.37 (15) | N4—C14—S2 | 128.67 (13) |
| C14—N5—C13 | 110.50 (14) | N5—C16—C15 | 110.17 (16) |
| C14—N5—C16 | 124.60 (15) | N5—C16—C17 | 109.80 (17) |
| C13—N5—C16 | 124.89 (15) | C15—C16—C17 | 112.60 (16) |
| N1—C5—C4 | 123.57 (14) | C8—N3—C7 | 110.61 (13) |
| N1—C5—N2 | 113.12 (14) | C8—N3—C10 | 123.79 (14) |
| C4—C5—N2 | 123.25 (14) | C7—N3—C10 | 125.60 (14) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C13—H13*A*···S2$^{i}$ | 0.93 | 2.81 | 3.7214 (18) | 166 |

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