Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

A triclinic modification of 5,5′-dinitro-2,2′-dithiodipyridine

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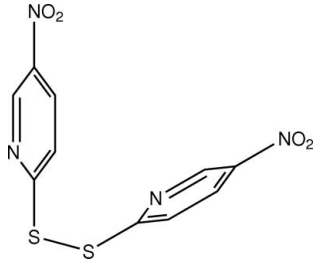
Received 21 November 2007; accepted 23 November 2007

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.006 Å; R factor = 0.087; wR factor = 0.209; data-to-parameter ratio = 13.6.

The asymmetric unit of the title compound, $C_{10}H_6N_4O_4S_2$, contains two independent but similar molecules. The structure is a triclinic polymorph of the monoclinic structure reported previously [Brito, Mundaca, Cárdenas, López-Rodríguez & Vargas (2007). *Acta Cryst.* E**63**, o3351–o3352]. The most obvious difference between the two polymorphs is the C—S—S—C torsion angle [−80.13 (16), −79.8 (2) and 0° for the two molecules of the triclinic polymorph and the monoclinic polymorph, respectively]. The crystal structure of the title compound has two intramolecular C—H···S interactions with average H···S distances of 2.69 Å, whereas this kind of interaction is not evident in the monoclinic polymorph.

Related literature

For related literature, see: Allen *et al.* (1987); Bernstein *et al.* (1995); Brito *et al.* (2007); Glidewell *et al.* (2000); Shefter (1970).



Experimental

Crystal data

$C_{10}H_6N_4O_4S_2$
M_r = 310.31
Triclinic, $P\overline{1}$
a = 7.7832 (12) Å
b = 11.8515 (11) Å
c = 14.513 (2) Å
α = 82.353 (4)°
β = 82.095 (5)°
γ = 72.460 (9)°
V = 1258.4 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.44 mm^{-1}
T = 298 (2) K
0.20 × 0.10 × 0.10 mm

Data collection

Nonius KappaCCD area-detector diffractometer
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)
T_{min} = 0.937, T_{max} = 0.948
11205 measured reflections
4931 independent reflections
4101 reflections with $I > 2\sigma(I)$
R_{int} = 0.062

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.088
$wR(F^2)$ = 0.209
S = 1.16
4931 reflections
362 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

Table 1
Selected torsion angles (°).

C4—S1—S2—C6	−80.13 (16)	S1—S2—C6—C7	−173.5 (3)
C11—S3—S4—C16	−79.8 (2)	S4—S3—C11—N6	−177.7 (3)
S2—S1—C4—N2	173.4 (2)	S4—S3—C11—C12	4.1 (4)
S2—S1—C4—C3	−7.1 (3)	S3—S4—C16—N7	9.5 (4)
S1—S2—C6—N3	6.6 (3)	S3—S4—C16—C17	−172.8 (3)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···S2	0.93	2.68	3.173 (4)	114
C8—H8···O1^{i}	0.93	2.44	3.098 (5)	127
C12—H12···S4	0.93	2.70	3.189 (5)	113
C18—H18···O5^{i}	0.93	2.50	3.171 (6)	129

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

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

This work was supported by a grant from the Universidad de Antofagasta (DI-1324-06). The authors thank the Spanish Research Council (CSIC) for the provision of a free-of-charge licence for the Cambridge Structural Database. AM thanks the Universidad de Antofagasta for a PhD fellowship.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2643).

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supplementary materials

Acta Cryst. (2008). E**64**, o102-o103 [**doi:10.1107/S1600536807062630**]

A triclinic modification of 5,5'-dinitro-2,2'-dithiodipyridine

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Comment

This paper forms part of our continuing study of the synthesis and structural characterization of divalent sulfur compounds (Brito *et al.*, 2007 and references therein). We are particularly interested in the utility of the title compound of as flexible ligand, and its binding modes, for the fabrication of different coordination polymers topologies. We report here the structure of a new polymorph of 5,5'-Dinitro-2,2'-dithiodipyridine isolated during attempts to synthetize coordination polymers with silver trifluoromethanesulfonate of the ligand (Fig. 1, Table 1). The bond lenghts and the conformation are significantly different from those found in the monoclinic modification (Brito *et al.*, 2007). The observed difference between (I) and the previously reported polymorph is in the torsion angle C—S—S—C [80.0 (2) and 0°, respectively]. A database survey of C—S—S—C fragments (Allen *et al.*, 1987) found that S—S bond distances are bimodally distributed: for torsion angles in the ranges 75–105 and 0–20°, the mean S—S bond distances are 2.031 (15) and 2.070 (22) Å, respectively. The corresponding value in the title compound is 2.025 (2) Å and 2.0719 (11) Å for the previously polymorph placing it in the lower quartile for Allen's first set. In both polymorphs the torsion angles *X*—C—S—S (where *X*=N or C) are close to 0 or 180° and within the range found in other substituted aromatic disulfides with an equatorial conformation according to the Shefter classification (Shefter, 1970).

The molecular conformations are dominated by near orthogonality of the lone pairs on the two adjacent S atoms (Glidewell *et al.*, 2000). The molecular packing in the title compound is completely different from that of the monoclinic polymorph. Only in the triclinic form the phenyl rings participant in significant intramolecular C—H···S interactions with average H···S distances of 2.69 Å. These interactions may stabilize the conformation adopted by the molecules in the solid state (Fig. 1). The molecules are linked into chains by two intermolecular C—H···O hydrogen bond. Atoms C8 and C18 in the molecules at (*x*,*y*,*z*) acts as hydrogen bonds donor vía atom H8 and H18 to atoms O1 and O5 in the molecule at (*x*,-1 + *y*,*z*) respectively, so generating by translation two C(12) chains running parallel to [010] direction (Bernstein *et al.*, 1995), (Fig. 2, Table 2). The triclinic modification is much less compact, as noted from the lower density (1.638 Mg m^{-3} compared with 1.725 Mg m^{-3} for the monoclinic form).

Experimental

All reactions were carried out under an atmosphere of purified nitrogen. Solvents were dried and distilled prior to use. 5,5'-dinitro-2,2'-dithiodipyridine and silver trifluoromethanesulfonate were purchased from Aldrich. The title compound was obtained as light yellow prismatic crystals, in an attempts to prepare coordination polymers with silver trifluoromethanesulfonate of the ligand. A mixture of 5,5'-dinitro-2,2'-dithiodipyridine (1 mmol, 310 mg) and silver trifluoromethanesulfonate (1 mmol, 256.9 mg) in methanol (20 ml) was refluxed for 8 h. After slow cooling of the reaction system to room temperature, light yellow prismatic crystals of (I) were formed that were filtered off and washed with cold diethyl ether. FT–IR (KBr pellet, cm^{-1}): ν (w, C—H) 3082, ν(s, N=O of NO_2 asymmetric) 1578, ν (v.s. of NO_2 symmetric) 1356, ν(w, C—H disubstitution 1,4) 1964, ν(s, C—H disubstitution 1,4) 856, ν (w, C—N) 1101, ν(s, C=C) 1600, ν (w, C—H) 1014, (s, C=N) 1512, ν (w, C—S) 743, ν(w S—S) 555.

Refinement

H atoms were positioned geometrically. In the final cycles of the refinement, all H atoms were constrained to ride on their parent atoms, with C—H distances of 0.93 Å and with U_{iso}(H) = 1.2 U_{eq}(C).

The material was difficult to obtain in a suitable crystalline form and the best available specimen was lost during data collection, resulting in 95% completeness.

Figures

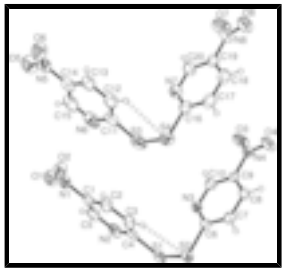

Fig. 1. A view of the molecular structure of (I), showing both molecules in the asymmetric unit with displacement ellipsoids draw at the 30% probability level. Dashed lines indicate the intramolecular C–H···S hydrogen bonds. H atoms are shown as small spheres of arbitrary radii.

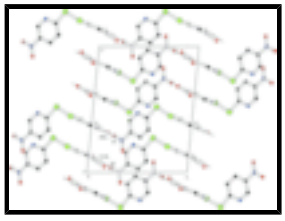

Fig. 2. Part of the crystal structure of (I) showing the formation of two C(12) chains. For the sake of clarity, H atoms not involved in hydrogen bonding have been omitted. [Symmetry codes: (i) 1 − *x*, −*y*, 1 − *z*; (ii) −*x*, −*y*, 1 − *z*].

5,5'-dinitro-2,2'-dithiodipyridine

Crystal data

$C_{10}H_6N_4O_4S_2$	$Z = 4$
$M_r = 310.31$	$F_{000} = 632$
Triclinic, $P\overline{1}$	$D_x = 1.638$ Mg m^{-3}
Hall symbol: -P 1	Melting point: 420 K
$a = 7.7832$ (12) Å	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$b = 11.8515$ (11) Å	Cell parameters from 11236 reflections
$c = 14.513$ (2) Å	$\theta = 1.4$–26.5º
$\alpha = 82.353$ (4)º	$\mu = 0.44$ mm^{-1}
$\beta = 82.095$ (5)º	$T = 298$ (2) K
$\gamma = 72.460$ (9)º	Prismatic, light yellow
$V = 1258.4$ (3) Å^3	0.20 × 0.10 × 0.10 mm

Data collection

Nonius KappaCCD area-detector diffractometer	4101 reflections with $I > 2\sigma(I)$

Radiation source: fine-focus sealed tube
Monochromator: graphite
φ scans, and ω scans with κ offsets
Absorption correction: multi-scan (SORTAV; Blessing, 1995)
T_{min} = 0.937, T_{max} = 0.948
11205 measured reflections
4931 independent reflections

R_{int} = 0.062
θ_{max} = 26.5º
θ_{min} = 1.4º
h = −9→9
k = −12→14
l = −18→18

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.088
$wR(F^2)$ = 0.209
S = 1.16
4931 reflections
362 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0368P)^2 + 1.5007P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.007
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}
Extinction correction: none

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 $F^2 > 2\text{sigma}(F^2)$ 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_{iso}*/U_{eq}
S1	−0.28532 (14)	0.36598 (9)	0.47963 (7)	0.0648 (3)
S2	−0.33244 (12)	0.21846 (9)	0.55311 (8)	0.0597 (3)
S3	0.2159 (2)	0.37139 (15)	0.72593 (9)	0.1008 (5)
S4	0.17109 (18)	0.22078 (14)	0.79400 (11)	0.0932 (5)
O1	−0.1425 (5)	0.7608 (3)	0.7099 (3)	0.0932 (11)
O2	−0.2430 (7)	0.6621 (3)	0.8270 (3)	0.1114 (15)
O3	0.5190 (4)	−0.0383 (3)	0.6160 (3)	0.1013 (14)
O4	0.4123 (4)	−0.1761 (3)	0.6849 (3)	0.0830 (10)
O5	0.3423 (6)	0.7628 (3)	0.9636 (3)	0.1124 (14)
O6	0.2541 (7)	0.6562 (3)	1.0807 (3)	0.1078 (13)
O7	1.0125 (5)	−0.0429 (5)	0.8735 (4)	0.143 (2)
O8	0.8920 (5)	−0.1686 (3)	0.9535 (3)	0.0985 (12)

N1	−0.2025 (5)	0.6797 (3)	0.7443 (3)	0.0695 (10)
N2	−0.2166 (5)	0.5481 (3)	0.5290 (3)	0.0675 (9)
N3	0.0258 (4)	0.1755 (2)	0.5594 (2)	0.0485 (7)
N4	0.3948 (4)	−0.0788 (3)	0.6427 (2)	0.0562 (8)
N5	0.2906 (5)	0.6772 (3)	0.9981 (3)	0.0765 (11)
N6	0.2519 (6)	0.5608 (4)	0.7817 (3)	0.0945 (15)
N7	0.5273 (5)	0.1785 (3)	0.8105 (3)	0.0725 (10)
N8	0.8822 (6)	−0.0777 (4)	0.9035 (3)	0.0798 (11)
C1	−0.2263 (5)	0.5991 (3)	0.6821 (3)	0.0536 (9)
C2	−0.2820 (6)	0.5018 (3)	0.7196 (3)	0.0607 (10)
H2	−0.3062	0.4877	0.784	0.073*
C3	−0.3013 (5)	0.4261 (3)	0.6612 (3)	0.0570 (9)
H3	−0.3366	0.3586	0.6846	0.078 (6)*
C4	−0.2670 (4)	0.4527 (3)	0.5671 (3)	0.0521 (9)
C5	−0.1959 (5)	0.6208 (3)	0.5874 (3)	0.0632 (11)
H5	−0.1598	0.6876	0.5627	0.078 (6)*
C6	−0.1129 (4)	0.1300 (3)	0.5804 (2)	0.0448 (7)
C7	−0.0971 (5)	0.0159 (3)	0.6229 (3)	0.0503 (8)
H7	−0.1982	−0.0121	0.6366	0.078 (6)*
C8	0.0692 (5)	−0.0546 (3)	0.6443 (3)	0.0483 (8)
H8	0.0851	−0.1316	0.6728	0.058*
C9	0.2126 (4)	−0.0072 (3)	0.6222 (2)	0.0425 (7)
C10	0.1881 (5)	0.1059 (3)	0.5803 (3)	0.0505 (8)
H10	0.2877	0.1353	0.5658	0.061*
C11	0.2288 (6)	0.4547 (4)	0.8158 (3)	0.0728 (13)
C12	0.2218 (6)	0.4174 (4)	0.9095 (3)	0.0706 (12)
H12	0.2054	0.3436	0.9313	0.078 (6)*
C13	0.2395 (6)	0.4909 (4)	0.9697 (3)	0.0691 (11)
H13	0.2342	0.4685	1.0337	0.083*
C14	0.2653 (6)	0.5984 (4)	0.9348 (3)	0.0680 (11)
C15	0.2697 (7)	0.6302 (5)	0.8413 (4)	0.0888 (16)
H15	0.286	0.7038	0.8184	0.107*
C16	0.3897 (6)	0.1317 (4)	0.8230 (3)	0.0690 (11)
C17	0.4031 (6)	0.0156 (4)	0.8614 (4)	0.0771 (13)
H17	0.3032	−0.0136	0.8688	0.078 (6)*
C18	0.5647 (6)	−0.0536 (4)	0.8877 (3)	0.0708 (11)
H18	0.5788	−0.1315	0.9137	0.085*
C19	0.7070 (5)	−0.0059 (3)	0.8749 (3)	0.0586 (9)
C20	0.6845 (6)	0.1087 (4)	0.8371 (3)	0.0686 (11)
H20	0.7831	0.1392	0.8298	0.082*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0590 (6)	0.0586 (6)	0.0625 (6)	0.0052 (5)	−0.0153 (5)	0.0013 (5)
S2	0.0394 (5)	0.0547 (6)	0.0817 (7)	−0.0040 (4)	−0.0121 (4)	−0.0117 (5)
S3	0.0942 (10)	0.1143 (11)	0.0657 (8)	0.0208 (8)	−0.0239 (7)	−0.0129 (7)
S4	0.0649 (8)	0.1047 (10)	0.1078 (11)	−0.0014 (7)	−0.0275 (7)	−0.0382 (8)

O1	0.090 (2)	0.0562 (18)	0.140 (3)	−0.0325 (17)	−0.023 (2)	0.0072 (19)
O2	0.192 (5)	0.070 (2)	0.087 (3)	−0.056 (3)	−0.024 (3)	−0.0074 (19)
O3	0.0428 (16)	0.090 (2)	0.167 (4)	−0.0267 (16)	−0.0314 (19)	0.040 (2)
O4	0.0621 (18)	0.0593 (18)	0.115 (3)	−0.0056 (14)	−0.0246 (17)	0.0246 (18)
O5	0.114 (3)	0.083 (2)	0.153 (4)	−0.057 (2)	−0.017 (3)	0.014 (2)
O6	0.157 (4)	0.080 (2)	0.092 (3)	−0.044 (3)	−0.017 (3)	−0.001 (2)
O7	0.064 (2)	0.148 (4)	0.205 (5)	−0.039 (3)	−0.031 (3)	0.056 (4)
O8	0.097 (3)	0.074 (2)	0.113 (3)	−0.0104 (19)	−0.024 (2)	0.013 (2)
N1	0.069 (2)	0.0378 (17)	0.103 (3)	−0.0172 (15)	−0.021 (2)	0.0058 (18)
N2	0.060 (2)	0.057 (2)	0.071 (2)	−0.0063 (16)	−0.0025 (17)	0.0161 (17)
N3	0.0421 (15)	0.0390 (14)	0.0646 (18)	−0.0129 (12)	−0.0090 (13)	0.0010 (13)
N4	0.0443 (17)	0.0510 (18)	0.072 (2)	−0.0121 (14)	−0.0162 (14)	0.0044 (15)
N5	0.071 (2)	0.057 (2)	0.097 (3)	−0.0150 (18)	−0.014 (2)	0.005 (2)
N6	0.095 (3)	0.092 (3)	0.064 (2)	0.003 (2)	−0.002 (2)	0.027 (2)
N7	0.072 (2)	0.065 (2)	0.076 (2)	−0.0121 (18)	−0.0121 (19)	−0.0057 (18)
N8	0.073 (3)	0.070 (2)	0.091 (3)	−0.016 (2)	−0.009 (2)	0.003 (2)
C1	0.0452 (19)	0.0357 (17)	0.075 (3)	−0.0060 (14)	−0.0102 (17)	0.0040 (16)
C2	0.074 (3)	0.0427 (19)	0.063 (2)	−0.0202 (18)	0.0015 (19)	0.0056 (17)
C3	0.067 (2)	0.0396 (18)	0.063 (2)	−0.0177 (17)	−0.0033 (18)	0.0037 (16)
C4	0.0350 (17)	0.0418 (18)	0.068 (2)	0.0017 (14)	−0.0067 (15)	0.0060 (16)
C5	0.054 (2)	0.045 (2)	0.084 (3)	−0.0157 (17)	−0.007 (2)	0.019 (2)
C6	0.0363 (16)	0.0450 (18)	0.0523 (19)	−0.0070 (13)	−0.0070 (14)	−0.0104 (14)
C7	0.0410 (18)	0.0476 (19)	0.065 (2)	−0.0199 (15)	−0.0026 (15)	−0.0033 (16)
C8	0.0481 (19)	0.0377 (17)	0.059 (2)	−0.0153 (14)	−0.0053 (15)	0.0011 (15)
C9	0.0374 (16)	0.0399 (16)	0.0516 (18)	−0.0112 (13)	−0.0096 (14)	−0.0039 (14)
C10	0.0406 (18)	0.0445 (18)	0.070 (2)	−0.0198 (15)	−0.0090 (16)	0.0027 (16)
C11	0.057 (2)	0.077 (3)	0.061 (2)	0.011 (2)	−0.0061 (19)	0.003 (2)
C12	0.082 (3)	0.061 (3)	0.060 (3)	−0.015 (2)	−0.001 (2)	0.005 (2)
C13	0.073 (3)	0.061 (2)	0.063 (3)	−0.013 (2)	−0.003 (2)	0.009 (2)
C14	0.055 (2)	0.061 (2)	0.075 (3)	−0.0069 (19)	−0.003 (2)	0.011 (2)
C15	0.091 (4)	0.072 (3)	0.081 (3)	−0.005 (3)	0.001 (3)	0.021 (3)
C16	0.063 (3)	0.075 (3)	0.068 (3)	−0.009 (2)	−0.006 (2)	−0.029 (2)
C17	0.061 (3)	0.083 (3)	0.096 (3)	−0.030 (2)	−0.001 (2)	−0.029 (3)
C18	0.072 (3)	0.059 (2)	0.084 (3)	−0.023 (2)	−0.002 (2)	−0.014 (2)
C19	0.057 (2)	0.059 (2)	0.059 (2)	−0.0137 (18)	−0.0014 (18)	−0.0117 (18)
C20	0.062 (3)	0.061 (2)	0.082 (3)	−0.018 (2)	−0.004 (2)	−0.005 (2)

Geometric parameters (Å, °)

S1—C4	1.781 (4)	C1—C2	1.372 (5)
S1—S2	2.0275 (15)	C2—C3	1.367 (6)
S2—C6	1.781 (3)	C2—H2	0.93
S3—C11	1.768 (5)	C3—C4	1.368 (5)
S3—S4	2.023 (2)	C3—H3	0.93
S4—C16	1.784 (5)	C5—H5	0.93
O1—N1	1.213 (4)	C6—C7	1.387 (5)
O2—N1	1.205 (5)	C7—C8	1.363 (5)
O3—N4	1.200 (4)	C7—H7	0.93
O4—N4	1.211 (4)	C8—C9	1.376 (4)

O5—N5	1.223 (5)	C8—H8	0.93
O6—N5	1.204 (5)	C9—C10	1.366 (4)
O7—N8	1.213 (5)	C10—H10	0.93
O8—N8	1.204 (5)	C11—C12	1.371 (6)
N1—C1	1.464 (6)	C12—C13	1.360 (6)
N2—C4	1.334 (5)	C12—H12	0.93
N2—C5	1.342 (6)	C13—C14	1.369 (6)
N3—C6	1.329 (4)	C13—H13	0.93
N3—C10	1.333 (4)	C14—C15	1.358 (7)
N4—C9	1.464 (4)	C15—H15	0.93
N5—C14	1.463 (6)	C16—C17	1.393 (6)
N6—C15	1.317 (8)	C17—C18	1.351 (6)
N6—C11	1.343 (6)	C17—H17	0.93
N7—C20	1.329 (6)	C18—C19	1.369 (6)
N7—C16	1.329 (6)	C18—H18	0.93
N8—C19	1.455 (6)	C19—C20	1.365 (5)
C1—C5	1.365 (6)	C20—H20	0.93
C4—S1—S2	104.06 (13)	C6—C7—H7	120.6
C6—S2—S1	103.36 (12)	C7—C8—C9	117.4 (3)
C11—S3—S4	104.51 (17)	C7—C8—H8	121.3
C16—S4—S3	104.02 (18)	C9—C8—H8	121.3
O2—N1—O1	123.3 (4)	C10—C9—C8	120.9 (3)
O2—N1—C1	118.6 (3)	C10—C9—N4	119.0 (3)
O1—N1—C1	118.2 (4)	C8—C9—N4	120.0 (3)
C4—N2—C5	117.4 (3)	N3—C10—C9	122.0 (3)
C6—N3—C10	117.2 (3)	N3—C10—H10	119
O3—N4—O4	123.5 (3)	C9—C10—H10	119
O3—N4—C9	118.3 (3)	N6—C11—C12	122.5 (5)
O4—N4—C9	118.1 (3)	N6—C11—S3	111.9 (4)
O6—N5—O5	123.8 (5)	C12—C11—S3	125.6 (4)
O6—N5—C14	118.2 (4)	C13—C12—C11	118.5 (4)
O5—N5—C14	117.9 (4)	C13—C12—H12	120.8
C15—N6—C11	118.0 (4)	C11—C12—H12	120.8
C20—N7—C16	116.7 (4)	C12—C13—C14	119.0 (4)
O8—N8—O7	123.4 (5)	C12—C13—H13	120.5
O8—N8—C19	119.3 (4)	C14—C13—H13	120.5
O7—N8—C19	117.3 (4)	C15—C14—C13	119.5 (5)
C5—C1—C2	119.9 (4)	C15—C14—N5	120.6 (5)
C5—C1—N1	120.7 (3)	C13—C14—N5	119.9 (4)
C2—C1—N1	119.4 (4)	N6—C15—C14	122.5 (5)
C3—C2—C1	119.1 (4)	N6—C15—H15	118.7
C3—C2—H2	120.5	C14—C15—H15	118.7
C1—C2—H2	120.5	N7—C16—C17	123.7 (4)
C2—C3—C4	117.8 (3)	N7—C16—S4	119.9 (4)
C2—C3—H3	121.1	C17—C16—S4	116.5 (4)
C4—C3—H3	121.1	C18—C17—C16	118.5 (4)
N2—C4—C3	124.1 (4)	C18—C17—H17	120.8
N2—C4—S1	111.2 (3)	C16—C17—H17	120.8
C3—C4—S1	124.7 (3)	C17—C18—C19	118.2 (4)

N2—C5—C1	121.7 (3)	C17—C18—H18	120.9
N2—C5—H5	119.2	C19—C18—H18	120.9
C1—C5—H5	119.2	C20—C19—C18	120.4 (4)
N3—C6—C7	123.6 (3)	C20—C19—N8	120.0 (4)
N3—C6—S2	119.2 (3)	C18—C19—N8	119.5 (4)
C7—C6—S2	117.3 (2)	N7—C20—C19	122.6 (4)
C8—C7—C6	118.8 (3)	N7—C20—H20	118.7
C8—C7—H7	120.6	C19—C20—H20	118.7
C4—S1—S2—C6	−80.13 (16)	N4—C9—C10—N3	179.7 (3)
C11—S3—S4—C16	−79.8 (2)	C15—N6—C11—C12	0.5 (7)
O2—N1—C1—C5	174.8 (4)	C15—N6—C11—S3	−177.8 (4)
O1—N1—C1—C5	−5.3 (5)	S4—S3—C11—N6	−177.7 (3)
O2—N1—C1—C2	−4.4 (6)	S4—S3—C11—C12	4.1 (4)
O1—N1—C1—C2	175.5 (4)	N6—C11—C12—C13	−0.2 (7)
C5—C1—C2—C3	1.8 (6)	S3—C11—C12—C13	177.9 (4)
N1—C1—C2—C3	−179.0 (4)	C11—C12—C13—C14	−0.6 (7)
C1—C2—C3—C4	−1.2 (6)	C12—C13—C14—C15	1.1 (7)
C5—N2—C4—C3	1.1 (5)	C12—C13—C14—N5	−178.0 (4)
C5—N2—C4—S1	−179.4 (3)	O6—N5—C14—C15	167.0 (5)
C2—C3—C4—N2	−0.2 (6)	O5—N5—C14—C15	−10.7 (7)
C2—C3—C4—S1	−179.7 (3)	O6—N5—C14—C13	−13.9 (6)
S2—S1—C4—N2	173.4 (2)	O5—N5—C14—C13	168.3 (4)
S2—S1—C4—C3	−7.1 (3)	C11—N6—C15—C14	−0.1 (8)
C4—N2—C5—C1	−0.5 (6)	C13—C14—C15—N6	−0.7 (8)
C2—C1—C5—N2	−0.9 (6)	N5—C14—C15—N6	178.4 (5)
N1—C1—C5—N2	179.9 (3)	C20—N7—C16—C17	−0.3 (7)
C10—N3—C6—C7	0.6 (5)	C20—N7—C16—S4	177.3 (3)
C10—N3—C6—S2	−179.4 (3)	S3—S4—C16—N7	9.5 (4)
S1—S2—C6—N3	6.6 (3)	S3—S4—C16—C17	−172.8 (3)
S1—S2—C6—C7	−173.5 (3)	N7—C16—C17—C18	0.1 (7)
N3—C6—C7—C8	−0.4 (6)	S4—C16—C17—C18	−177.6 (4)
S2—C6—C7—C8	179.6 (3)	C16—C17—C18—C19	−0.1 (7)
C6—C7—C8—C9	0.1 (5)	C17—C18—C19—C20	0.4 (7)
C7—C8—C9—C10	−0.1 (5)	C17—C18—C19—N8	179.2 (4)
C7—C8—C9—N4	−179.5 (3)	O8—N8—C19—C20	164.9 (4)
O3—N4—C9—C10	−5.0 (6)	O7—N8—C19—C20	−16.6 (7)
O4—N4—C9—C10	175.6 (4)	O8—N8—C19—C18	−14.0 (7)
O3—N4—C9—C8	174.3 (4)	O7—N8—C19—C18	164.5 (5)
O4—N4—C9—C8	−5.0 (5)	C16—N7—C20—C19	0.5 (7)
C6—N3—C10—C9	−0.6 (5)	C18—C19—C20—N7	−0.6 (7)
C8—C9—C10—N3	0.4 (6)	N8—C19—C20—N7	−179.5 (4)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···S2	0.93	2.68	3.173 (4)	114
C8—H8···O1^{i}	0.93	2.44	3.098 (5)	127
C12—H12···S4	0.93	2.70	3.189 (5)	113

C18—H18···O5^{i}	0.93	2.50	3.171 (6)	129

Symmetry codes: (i) *x*, *y*−1, *z*.

Fig. 1

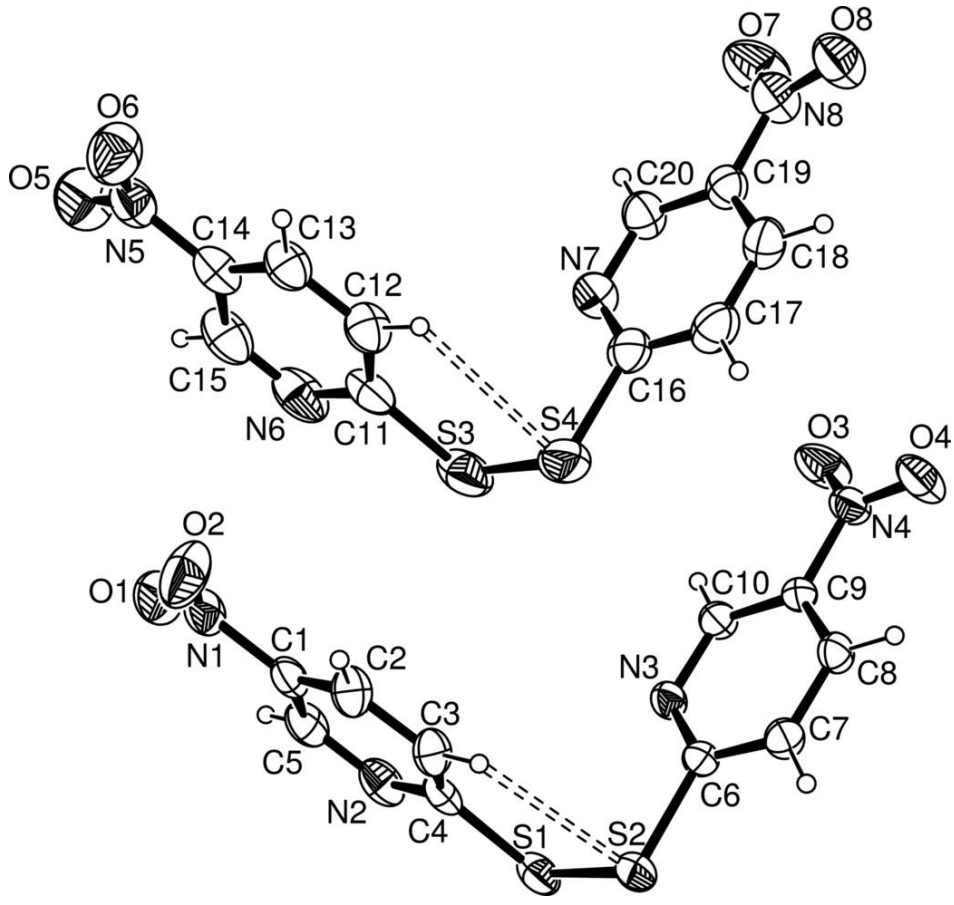

Fig. 2