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(4*R*,5*R*)-2,2-Dimethyl-4,5-bis[2-(3-methyl-2-thioxo-2,3-dihydro-1*H*-imidazol-2-yl)ethyl]-1,3-dioxolane

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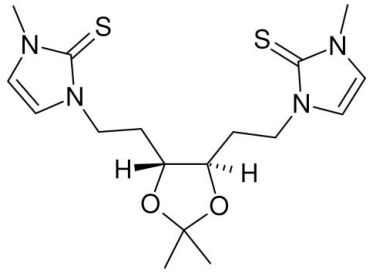
Key indicators: single-crystal X-ray study; T = 120 K; mean σ(C–C) = 0.012 Å; R factor = 0.097; wR factor = 0.241; data-to-parameter ratio = 18.6.

Two molecules make up the asymmetric unit of the chiral title compound, $C_{17}H_{26}N_4O_2S_2$. The dihedral angles between the imidazolethione rings are 23.7 (5) and 31.4 (5)° in the two molecules. The packing appears to be consolidated by pseudo-aromatic stacking interactions involving the imidazolethione rings [centroid-to-centroid separations = 3.669 (8) and 3.675 (8) Å]. Short C—H···S and C—H···O contacts are also apparent in the crystal structure.

Related literature

For background, see: Marshall & Harrison (2007); Williamson *et al.* (2006). For reference structural data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{17}H_{26}N_4O_2S_2$
M_r = 382.54
Monoclinic, $P2_1$
a = 6.827 (5) Å
b = 20.732 (9) Å
c = 14.071 (8) Å
β = 93.68 (2)°
V = 1988 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.29 mm^{-1}
T = 120 (2) K
0.15 × 0.07 × 0.03 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: none
21365 measured reflections
8499 independent reflections
4275 reflections with $I > 2\sigma(I)$
R_{int} = 0.131

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.097
$wR(F^2)$ = 0.241
S = 1.01
8499 reflections
458 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}
Absolute structure: Flack (1983), with 3717 Friedel pairs
Flack parameter: 0.03 (14)

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

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2···S1^{i}	0.95	2.86	3.703 (8)	148
C16—H16*C*···O3	0.98	2.35	3.321 (10)	171
C33—H33*C*···O1^{ii}	0.98	2.49	3.424 (10)	160
C34—H34*C*···O2^{iii}	0.98	2.33	3.266 (10)	160

Symmetry codes: (i) $x-1, y, z$; (ii) $x-1, y, z-1$; (iii) $x, y, z-1$.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *HKL SCALEPACK* (Otwinowski & Minor 1997); data reduction: *HKL DENZO* (Otwinowski & Minor 1997), *HKL SCALEPACK* and *SORTAV* (Blessing 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2406).

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supplementary materials

Acta Cryst. (2007). E**63**, o4878 [**doi:10.1107/S160053680706148X**]

(4*R*,5*R*)-2,2-Dimethyl-4,5-bis[2-(3-methyl-2-thioxo-2,3-dihydro-1*H*-imidazol-2-yl)ethyl]-1,3-dioxolane

C. Marshall and W. T. A. Harrison

Comment

As part of our ongoing investigations of chiral, C_2-symmetric catalysts (Marshall & Harrison, 2007), the title compound, (I), which is an intermediate in such materials, has been synthesized and structurally characterized.

There are two molecules of (I) in the asymmetric unit (Fig. 1). All the chiral centres (C6, C7, C23, C24) possess their expected *R* configuarations. The dihedral angle between the N1/N2/C1/C2/C3 (IM1) and N3/N4/C10/C11/C12 (IM2) imidazole rings is 23.7 (5)°; that between the N5/N6/C18/C19/C20 (IM3) and N7/N8/C27/C28/C29 (IM4) planes is 31.4 (5)°. The thio-imidazole rings show their expected geometries (Marshall & Harrison, 2007; Williamson *et al.*, 2006) and indeed all the geometrical parameters of the two molecules may be regarded as normal (Allen *et al.*, 1987).

The crystal packing for (I) features short face-to-face contacts between the imidazole rings with a centroid-centroid separation of 3.675 (8) Å for IM1···IM2^{i} (i = 2 − *x*, 1/2 + *y*, 1 − *z*) and 3.669 (8) Å for IM3···IM4^{ii} (ii = 1 − *x*, −1/2 + *y*, −*z*). Such contacts are usually referred to as aromatic π–π stacking. However, the aromatic nature of the thio-imidazole ring is questionable (Williamson *et al.*, 2006). If these really represent attractive intermolecular forces in the crystal, then [010] chains of molecules arise (Fig. 2). Some short C—H···S and C—H···O contacts (Table 1) also occur in the crystal.

Experimental

A mixture of (4*R*,5*R*)-4,5-bis(1-methylimidazolium-3-ethyl)-2,2-dimethly-1,3-dioxolane dibromide (0.46 g, 0.96 mmol), methanol (10 ml), pyridine (0.5 ml), sulfur (0.09 g, 2.89 mmol), and 1,8-diazabicyclo[5.4.0]undec-7-ene (0.64 g, 4.23 mmol) was heated at 338 K for 18 h. Once cooled to room temperature the mixture was opened to water (40 ml) and extracted with chloroform (3 × 20 ml). The combined extracts were dried over magnesium sulfate, filtered and concentrated under reduced pressure to leave a brown residue. The crude product was purified by column chromatography (SiO_2, ethyl acetate: petroleum ether *v*/*v* 1:1, loaded as a dichloromethane solution) to give the title compound (0.34 g, 93%) as a colourless solid which was recrystallized from diethyl ether, acetone (*v*/*v* 1:1) to give colourless blades of (I); mp 390 K.

Refinement

The small crystal used for data collection was a weak scatterer, which may correlate with the rather high residuals.

The H atoms were placed in calculated positions (C—H = 0.95–1.00 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C). The methyl groups were allowed to rotate, but not to tip, to best fit the electron density.

Figures

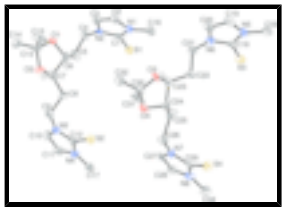

Fig. 1. View of the molecular structure of (I) showing 50% displacement ellipsoids. All the H atoms are omitted for clarity except those attached to the chiral C atoms, which are represented as arbitrary spheres.

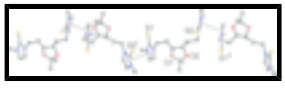

Fig. 2. Part of a chain of C1-containing molecules in the crystal of (I), with the dashed lines indicating the pseudo-aromatic stacking contacts between the centroids of the 5-membered rings (see text). All H atoms omitted for clarity. Symmetry codes: (i) $2-x$, $1/2+y$, $1-z$; (ii) $2-x$, $1/2+y$, $1-z$. The C18-containing molecule forms similar chains.

(4*R*,5*R*)-2,2-Dimethyl-4,5-bis[2-(3-methyl-2-thioxo-2,3-dihydro-1*H*- imidazol-2-yl)ethyl]-1,3-dioxolane

Crystal data

$C_{17}H_{26}N_4O_2S_2$	$F_{000} = 816$
$M_r = 382.54$	$D_x = 1.278$ Mg m^{-3}
Monoclinic, $P2_1$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: P 2yb	Cell parameters from 10746 reflections
$a = 6.827$ (5) Å	$\theta = 2.9$–27.5º
$b = 20.732$ (9) Å	$\mu = 0.29$ mm^{-1}
$c = 14.071$ (8) Å	$T = 120$ (2) K
$\beta = 93.68$ (2)º	Blade, colourless
$V = 1988$ (4) Å^3	0.15 × 0.07 × 0.03 mm
$Z = 4$	

Data collection

Nonius KappaCCD diffractometer	4275 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{int} = 0.131$
Monochromator: graphite	$\theta_{max} = 27.7$º
$T = 120(2)$ K	$\theta_{min} = 3.0$º
ω and φ scans	$h = -8\rightarrow8$
Absorption correction: none	$k = -26\rightarrow26$
21365 measured reflections	$l = -18\rightarrow18$
8499 independent reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.097$	$w = 1/[\sigma^2(F_o^2) + (0.1095P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$wR(F^2) = 0.241$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.01$	$\Delta\rho_{max} = 1.19$ e Å^{-3}
8499 reflections	$\Delta\rho_{min} = -0.38$ e Å^{-3}
458 parameters	Extinction correction: none
1 restraint	Absolute structure: Flack (1983), with 3717 Friedel pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: 0.03 (14)
Secondary atom site location: difference Fourier map	

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	1.0766 (3)	−0.09444 (11)	0.37993 (15)	0.0358 (6)
S2	1.1011 (3)	0.25588 (10)	0.38446 (15)	0.0379 (6)
O1	0.9082 (7)	0.0267 (2)	0.7294 (3)	0.0281 (13)
O2	0.9200 (8)	0.1377 (2)	0.7253 (4)	0.0319 (14)
N1	0.6913 (9)	−0.0893 (3)	0.4156 (4)	0.0287 (15)
N2	0.8869 (8)	−0.0746 (3)	0.5447 (4)	0.0225 (15)
N3	1.3291 (10)	0.2429 (3)	0.5522 (4)	0.0322 (16)
N4	1.4920 (9)	0.2678 (3)	0.4321 (4)	0.0313 (16)
C1	0.8820 (11)	−0.0868 (4)	0.4470 (6)	0.0286 (19)
C2	0.5784 (11)	−0.0793 (4)	0.4942 (6)	0.0283 (19)
H2	0.4391	−0.0788	0.4916	0.034*
C3	0.6915 (12)	−0.0707 (4)	0.5710 (6)	0.034 (2)
H3	0.6502	−0.0633	0.6333	0.041*
C4	1.0701 (12)	−0.0718 (4)	0.6069 (6)	0.033 (2)
H4A	1.1718	−0.0987	0.5790	0.039*
H4B	1.0455	−0.0898	0.6702	0.039*
C5	1.1441 (12)	−0.0030 (4)	0.6182 (6)	0.034 (2)
H5A	1.2525	−0.0024	0.6684	0.041*
H5B	1.1993	0.0104	0.5579	0.041*
C6	0.9947 (13)	0.0455 (4)	0.6431 (5)	0.034 (2)
H6	0.8902	0.0486	0.5901	0.041*
C7	1.0671 (13)	0.1117 (4)	0.6699 (6)	0.035 (2)
H7	1.1940	0.1086	0.7093	0.042*

C8	1.0929 (14)	0.1560 (4)	0.5824 (6)	0.039 (2)
H8A	1.1866	0.1354	0.5410	0.046*
H8B	0.9652	0.1597	0.5454	0.046*
C9	1.1676 (12)	0.2245 (4)	0.6086 (6)	0.033 (2)
H9A	1.0589	0.2558	0.5977	0.039*
H9B	1.2114	0.2259	0.6770	0.039*
C10	1.5189 (14)	0.2460 (4)	0.5853 (6)	0.039 (2)
H10	1.5697	0.2378	0.6486	0.046*
C11	1.6230 (11)	0.2632 (4)	0.5099 (6)	0.037 (2)
H11	1.7604	0.2707	0.5110	0.044*
C12	1.3064 (11)	0.2562 (4)	0.4560 (6)	0.0285 (19)
C13	0.8483 (12)	0.0842 (4)	0.7781 (5)	0.034 (2)
C14	0.9443 (16)	0.0811 (5)	0.8777 (6)	0.056 (3)
H14A	0.9187	0.1213	0.9114	0.084*
H14B	1.0862	0.0754	0.8745	0.084*
H14C	0.8903	0.0447	0.9119	0.084*
C15	0.6255 (13)	0.0889 (4)	0.7705 (7)	0.050 (3)
H15A	0.5843	0.1265	0.8064	0.076*
H15B	0.5694	0.0497	0.7966	0.076*
H15C	0.5792	0.0935	0.7034	0.076*
C16	0.6178 (12)	−0.1033 (4)	0.3189 (6)	0.039 (2)
H16A	0.4858	−0.1218	0.3195	0.058*
H16B	0.7054	−0.1341	0.2903	0.058*
H16C	0.6126	−0.0633	0.2815	0.058*
C17	1.5424 (12)	0.2842 (4)	0.3361 (6)	0.039 (2)
H17A	1.6842	0.2913	0.3356	0.058*
H17B	1.4727	0.3235	0.3152	0.058*
H17C	1.5042	0.2487	0.2927	0.058*
S3	0.3242 (3)	−0.08682 (10)	−0.11152 (15)	0.0346 (5)
S4	0.4476 (3)	0.27361 (10)	−0.13945 (14)	0.0315 (5)
O3	0.6325 (8)	0.0390 (2)	0.2112 (4)	0.0354 (14)
O4	0.6722 (7)	0.1490 (2)	0.2102 (4)	0.0318 (14)
N5	−0.0519 (9)	−0.0961 (3)	−0.0621 (4)	0.0286 (16)
N6	0.1467 (9)	−0.0640 (3)	0.0544 (5)	0.0290 (16)
N7	0.6796 (9)	0.2483 (3)	0.0229 (4)	0.0239 (15)
N8	0.8460 (9)	0.2680 (3)	−0.1002 (4)	0.0284 (15)
C18	0.1379 (11)	−0.0834 (4)	−0.0398 (5)	0.0272 (18)
C19	−0.1644 (12)	−0.0857 (4)	0.0162 (6)	0.037 (2)
H19	−0.3020	−0.0916	0.0184	0.044*
C20	−0.0388 (11)	−0.0657 (4)	0.0878 (6)	0.030 (2)
H20	−0.0720	−0.0547	0.1504	0.036*
C21	0.3238 (12)	−0.0426 (4)	0.1091 (6)	0.034 (2)
H21A	0.4264	−0.0762	0.1054	0.041*
H21B	0.2945	−0.0381	0.1768	0.041*
C22	0.4043 (12)	0.0216 (4)	0.0743 (6)	0.034 (2)
H22A	0.5163	0.0129	0.0349	0.041*
H22B	0.3009	0.0434	0.0334	0.041*
C23	0.4715 (11)	0.0666 (4)	0.1552 (5)	0.0289 (19)
H23	0.3603	0.0758	0.1961	0.035*

C24	0.5638 (12)	0.1292 (3)	0.1247 (5)	0.0260 (19)
H24	0.6549	0.1209	0.0732	0.031*
C25	0.4178 (11)	0.1814 (4)	0.0942 (5)	0.0258 (18)
H25A	0.3175	0.1847	0.1418	0.031*
H25B	0.3501	0.1692	0.0325	0.031*
C26	0.5161 (11)	0.2471 (4)	0.0840 (6)	0.0288 (19)
H26A	0.4158	0.2782	0.0587	0.035*
H26B	0.5635	0.2622	0.1482	0.035*
C27	0.8729 (13)	0.2427 (4)	0.0525 (6)	0.036 (2)
H27	0.9249	0.2326	0.1150	0.044*
C28	0.9747 (11)	0.2542 (4)	−0.0237 (6)	0.0288 (19)
H28	1.1136	0.2530	−0.0248	0.035*
C29	0.6561 (11)	0.2632 (4)	−0.0717 (5)	0.0283 (19)
C30	0.7339 (12)	0.0893 (4)	0.2601 (6)	0.037 (2)
C31	0.9550 (12)	0.0815 (5)	0.2540 (7)	0.055 (3)
H31A	1.0230	0.1175	0.2872	0.082*
H31B	0.9866	0.0814	0.1871	0.082*
H31C	0.9974	0.0408	0.2840	0.082*
C32	0.6749 (17)	0.0963 (5)	0.3611 (6)	0.060 (3)
H32A	0.7504	0.1312	0.3927	0.090*
H32B	0.7014	0.0558	0.3957	0.090*
H32C	0.5345	0.1062	0.3606	0.090*
C33	−0.1337 (12)	−0.1177 (4)	−0.1555 (6)	0.039 (2)
H33A	−0.2612	−0.1382	−0.1485	0.058*
H33B	−0.0440	−0.1487	−0.1823	0.058*
H33C	−0.1507	−0.0805	−0.1982	0.058*
C34	0.8937 (12)	0.2850 (4)	−0.1942 (6)	0.035 (2)
H34A	1.0232	0.3055	−0.1917	0.053*
H34B	0.7950	0.3151	−0.2219	0.053*
H34C	0.8956	0.2461	−0.2336	0.053*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0335 (13)	0.0382 (13)	0.0357 (12)	−0.0003 (11)	0.0030 (10)	−0.0042 (10)
S2	0.0347 (13)	0.0385 (14)	0.0395 (13)	−0.0065 (10)	−0.0047 (10)	0.0006 (10)
O1	0.034 (3)	0.018 (3)	0.033 (3)	−0.004 (2)	0.010 (3)	0.000 (2)
O2	0.032 (3)	0.024 (3)	0.042 (3)	−0.002 (2)	0.014 (3)	0.007 (3)
N1	0.040 (4)	0.010 (3)	0.035 (4)	0.007 (3)	−0.004 (3)	0.005 (3)
N2	0.018 (3)	0.023 (4)	0.027 (3)	−0.009 (3)	0.006 (3)	−0.001 (3)
N3	0.039 (4)	0.023 (4)	0.034 (4)	0.000 (3)	0.005 (4)	−0.004 (3)
N4	0.030 (4)	0.033 (4)	0.033 (4)	0.001 (3)	0.013 (3)	−0.001 (3)
C1	0.023 (4)	0.010 (4)	0.051 (5)	−0.003 (3)	−0.012 (4)	0.007 (4)
C2	0.017 (4)	0.027 (5)	0.041 (5)	0.000 (4)	0.002 (4)	−0.002 (4)
C3	0.038 (5)	0.029 (5)	0.037 (5)	−0.002 (4)	0.021 (4)	−0.004 (4)
C4	0.038 (5)	0.024 (5)	0.035 (5)	0.010 (4)	−0.007 (4)	−0.004 (4)
C5	0.039 (5)	0.035 (5)	0.029 (5)	0.002 (4)	0.001 (4)	−0.009 (4)
C6	0.052 (6)	0.030 (5)	0.021 (4)	0.008 (4)	0.000 (4)	−0.007 (4)

C7	0.052 (6)	0.023 (5)	0.031 (5)	0.005 (4)	0.006 (4)	−0.002 (4)
C8	0.048 (6)	0.036 (5)	0.031 (5)	−0.015 (4)	0.003 (4)	0.009 (4)
C9	0.036 (5)	0.032 (5)	0.031 (5)	−0.005 (4)	0.004 (4)	−0.007 (4)
C10	0.064 (7)	0.022 (5)	0.028 (5)	0.012 (4)	−0.011 (5)	0.000 (4)
C11	0.018 (4)	0.040 (6)	0.052 (6)	−0.010 (4)	0.003 (4)	−0.005 (5)
C12	0.025 (4)	0.020 (4)	0.043 (5)	−0.001 (4)	0.019 (4)	0.003 (4)
C13	0.046 (5)	0.023 (5)	0.036 (5)	−0.003 (4)	0.016 (4)	0.000 (4)
C14	0.096 (8)	0.039 (6)	0.035 (5)	−0.009 (6)	0.018 (5)	0.000 (5)
C15	0.042 (6)	0.021 (5)	0.090 (7)	−0.001 (4)	0.023 (5)	0.002 (5)
C16	0.029 (5)	0.044 (6)	0.044 (5)	−0.008 (4)	0.007 (4)	−0.011 (4)
C17	0.031 (5)	0.040 (6)	0.046 (5)	−0.012 (4)	0.008 (4)	−0.010 (4)
S3	0.0304 (12)	0.0326 (12)	0.0412 (12)	−0.0007 (10)	0.0048 (9)	−0.0001 (10)
S4	0.0220 (11)	0.0397 (13)	0.0323 (11)	0.0040 (10)	−0.0029 (9)	0.0048 (10)
O3	0.042 (3)	0.014 (3)	0.048 (4)	−0.001 (2)	−0.015 (3)	−0.001 (3)
O4	0.029 (3)	0.026 (3)	0.038 (3)	0.003 (2)	−0.015 (3)	0.001 (2)
N5	0.034 (4)	0.017 (4)	0.033 (4)	0.002 (3)	−0.006 (3)	−0.003 (3)
N6	0.029 (4)	0.023 (4)	0.034 (4)	0.001 (3)	−0.006 (3)	0.006 (3)
N7	0.021 (4)	0.029 (4)	0.021 (3)	0.002 (3)	0.002 (3)	−0.004 (3)
N8	0.028 (4)	0.019 (4)	0.038 (4)	0.005 (3)	0.003 (3)	0.003 (3)
C18	0.038 (5)	0.015 (4)	0.028 (4)	−0.009 (4)	0.002 (4)	0.006 (4)
C19	0.033 (5)	0.028 (5)	0.049 (5)	−0.007 (4)	−0.001 (4)	−0.005 (4)
C20	0.021 (5)	0.030 (5)	0.040 (5)	−0.004 (4)	0.009 (4)	−0.003 (4)
C21	0.038 (5)	0.025 (5)	0.037 (5)	−0.002 (4)	−0.005 (4)	0.007 (4)
C22	0.037 (5)	0.023 (5)	0.042 (5)	−0.012 (4)	0.002 (4)	0.001 (4)
C23	0.026 (5)	0.025 (5)	0.035 (5)	0.003 (3)	−0.005 (4)	−0.002 (4)
C24	0.034 (5)	0.024 (5)	0.021 (4)	0.010 (4)	0.007 (4)	−0.005 (3)
C25	0.026 (4)	0.026 (5)	0.024 (4)	0.000 (3)	−0.010 (3)	−0.003 (3)
C26	0.028 (5)	0.020 (4)	0.039 (5)	0.000 (3)	0.005 (4)	0.002 (4)
C27	0.057 (6)	0.019 (5)	0.031 (5)	−0.011 (4)	−0.013 (4)	0.004 (4)
C28	0.020 (4)	0.022 (4)	0.045 (5)	0.004 (4)	0.001 (4)	0.008 (4)
C29	0.029 (5)	0.017 (4)	0.041 (5)	−0.016 (4)	0.017 (4)	−0.015 (4)
C30	0.041 (5)	0.027 (5)	0.039 (5)	0.001 (4)	−0.023 (4)	0.007 (4)
C31	0.045 (6)	0.029 (5)	0.086 (7)	−0.016 (5)	−0.025 (5)	0.000 (6)
C32	0.102 (9)	0.035 (6)	0.041 (6)	−0.014 (5)	−0.012 (6)	0.007 (4)
C33	0.035 (5)	0.030 (5)	0.050 (6)	−0.011 (4)	−0.002 (4)	0.005 (4)
C34	0.041 (5)	0.027 (5)	0.040 (5)	0.013 (4)	0.018 (4)	−0.004 (4)

Geometric parameters (Å, °)

S1—C1	1.685 (9)	S3—C18	1.675 (8)
S2—C12	1.672 (9)	S4—C29	1.675 (9)
O1—C6	1.437 (9)	O3—C30	1.406 (9)
O1—C13	1.447 (9)	O3—C23	1.430 (9)
O2—C7	1.417 (10)	O4—C24	1.431 (9)
O2—C13	1.439 (9)	O4—C30	1.471 (9)
N1—C1	1.349 (9)	N5—C18	1.339 (9)
N1—C2	1.403 (10)	N5—C19	1.400 (10)
N1—C16	1.449 (10)	N5—C33	1.465 (10)
N2—C1	1.397 (9)	N6—C20	1.379 (10)

N2—C3	1.409 (10)	N6—C18	1.383 (9)
N2—C4	1.480 (9)	N6—C21	1.460 (9)
N3—C10	1.350 (11)	N7—C27	1.363 (10)
N3—C12	1.381 (9)	N7—C29	1.366 (9)
N3—C9	1.450 (10)	N7—C26	1.452 (9)
N4—C12	1.353 (9)	N8—C28	1.375 (9)
N4—C11	1.372 (10)	N8—C29	1.386 (9)
N4—C17	1.456 (10)	N8—C34	1.426 (10)
C2—C3	1.299 (10)	C19—C20	1.346 (10)
C2—H2	0.9500	C19—H19	0.9500
C3—H3	0.9500	C20—H20	0.9500
C4—C5	1.518 (11)	C21—C22	1.533 (10)
C4—H4A	0.9900	C21—H21A	0.9900
C4—H4B	0.9900	C21—H21B	0.9900
C5—C6	1.490 (11)	C22—C23	1.520 (10)
C5—H5A	0.9900	C22—H22A	0.9900
C5—H5B	0.9900	C22—H22B	0.9900
C6—C7	1.500 (11)	C23—C24	1.517 (11)
C6—H6	1.0000	C23—H23	1.0000
C7—C8	1.554 (11)	C24—C25	1.515 (10)
C7—H7	1.0000	C24—H24	1.0000
C8—C9	1.546 (11)	C25—C26	1.529 (11)
C8—H8A	0.9900	C25—H25A	0.9900
C8—H8B	0.9900	C25—H25B	0.9900
C9—H9A	0.9900	C26—H26A	0.9900
C9—H9B	0.9900	C26—H26B	0.9900
C10—C11	1.362 (11)	C27—C28	1.336 (11)
C10—H10	0.9500	C27—H27	0.9500
C11—H11	0.9500	C28—H28	0.9500
C13—C14	1.511 (11)	C30—C32	1.510 (12)
C13—C15	1.521 (11)	C30—C31	1.526 (12)
C14—H14A	0.9800	C31—H31A	0.9800
C14—H14B	0.9800	C31—H31B	0.9800
C14—H14C	0.9800	C31—H31C	0.9800
C15—H15A	0.9800	C32—H32A	0.9800
C15—H15B	0.9800	C32—H32B	0.9800
C15—H15C	0.9800	C32—H32C	0.9800
C16—H16A	0.9800	C33—H33A	0.9800
C16—H16B	0.9800	C33—H33B	0.9800
C16—H16C	0.9800	C33—H33C	0.9800
C17—H17A	0.9800	C34—H34A	0.9800
C17—H17B	0.9800	C34—H34B	0.9800
C17—H17C	0.9800	C34—H34C	0.9800
C6—O1—C13	108.7 (5)	C30—O3—C23	108.0 (5)
C7—O2—C13	105.5 (6)	C24—O4—C30	106.1 (5)
C1—N1—C2	107.7 (6)	C18—N5—C19	110.9 (6)
C1—N1—C16	125.7 (7)	C18—N5—C33	125.3 (7)
C2—N1—C16	126.5 (6)	C19—N5—C33	123.8 (7)
C1—N2—C3	107.8 (6)	C20—N6—C18	109.6 (6)

C1—N2—C4	123.7 (6)	C20—N6—C21	125.1 (7)
C3—N2—C4	128.4 (6)	C18—N6—C21	125.3 (7)
C10—N3—C12	111.9 (7)	C27—N7—C29	111.6 (6)
C10—N3—C9	124.8 (7)	C27—N7—C26	125.7 (6)
C12—N3—C9	123.2 (7)	C29—N7—C26	122.3 (6)
C12—N4—C11	111.3 (6)	C28—N8—C29	108.7 (6)
C12—N4—C17	123.5 (7)	C28—N8—C34	127.2 (6)
C11—N4—C17	125.2 (6)	C29—N8—C34	124.1 (6)
N1—C1—N2	106.9 (7)	N5—C18—N6	105.3 (6)
N1—C1—S1	126.3 (6)	N5—C18—S3	127.7 (6)
N2—C1—S1	126.8 (5)	N6—C18—S3	127.0 (6)
C3—C2—N1	110.4 (7)	C20—C19—N5	106.3 (7)
C3—C2—H2	124.8	C20—C19—H19	126.8
N1—C2—H2	124.8	N5—C19—H19	126.8
C2—C3—N2	107.2 (7)	C19—C20—N6	107.9 (7)
C2—C3—H3	126.4	C19—C20—H20	126.1
N2—C3—H3	126.4	N6—C20—H20	126.1
N2—C4—C5	111.2 (6)	N6—C21—C22	113.3 (6)
N2—C4—H4A	109.4	N6—C21—H21A	108.9
C5—C4—H4A	109.4	C22—C21—H21A	108.9
N2—C4—H4B	109.4	N6—C21—H21B	108.9
C5—C4—H4B	109.4	C22—C21—H21B	108.9
H4A—C4—H4B	108.0	H21A—C21—H21B	107.7
C6—C5—C4	115.5 (7)	C23—C22—C21	113.0 (7)
C6—C5—H5A	108.4	C23—C22—H22A	109.0
C4—C5—H5A	108.4	C21—C22—H22A	109.0
C6—C5—H5B	108.4	C23—C22—H22B	109.0
C4—C5—H5B	108.4	C21—C22—H22B	109.0
H5A—C5—H5B	107.5	H22A—C22—H22B	107.8
O1—C6—C5	109.8 (7)	O3—C23—C24	100.4 (6)
O1—C6—C7	100.6 (6)	O3—C23—C22	110.6 (6)
C5—C6—C7	117.2 (7)	C24—C23—C22	115.1 (7)
O1—C6—H6	109.6	O3—C23—H23	110.1
C5—C6—H6	109.6	C24—C23—H23	110.1
C7—C6—H6	109.6	C22—C23—H23	110.1
O2—C7—C6	104.6 (7)	O4—C24—C25	109.2 (6)
O2—C7—C8	109.4 (7)	O4—C24—C23	102.1 (6)
C6—C7—C8	113.2 (7)	C25—C24—C23	114.5 (7)
O2—C7—H7	109.8	O4—C24—H24	110.3
C6—C7—H7	109.8	C25—C24—H24	110.3
C8—C7—H7	109.8	C23—C24—H24	110.3
C9—C8—C7	114.0 (7)	C24—C25—C26	112.2 (6)
C9—C8—H8A	108.8	C24—C25—H25A	109.2
C7—C8—H8A	108.8	C26—C25—H25A	109.2
C9—C8—H8B	108.8	C24—C25—H25B	109.2
C7—C8—H8B	108.8	C26—C25—H25B	109.2
H8A—C8—H8B	107.6	H25A—C25—H25B	107.9
N3—C9—C8	111.2 (6)	N7—C26—C25	115.4 (6)
N3—C9—H9A	109.4	N7—C26—H26A	108.4

C8—C9—H9A	109.4	C25—C26—H26A	108.4
N3—C9—H9B	109.4	N7—C26—H26B	108.4
C8—C9—H9B	109.4	C25—C26—H26B	108.4
H9A—C9—H9B	108.0	H26A—C26—H26B	107.5
N3—C10—C11	106.3 (7)	C28—C27—N7	106.4 (7)
N3—C10—H10	126.8	C28—C27—H27	126.8
C11—C10—H10	126.8	N7—C27—H27	126.8
C10—C11—N4	107.1 (7)	C27—C28—N8	109.1 (7)
C10—C11—H11	126.5	C27—C28—H28	125.5
N4—C11—H11	126.5	N8—C28—H28	125.5
N4—C12—N3	103.3 (7)	N7—C29—N8	104.2 (6)
N4—C12—S2	127.9 (6)	N7—C29—S4	128.8 (6)
N3—C12—S2	128.8 (6)	N8—C29—S4	127.0 (6)
O2—C13—O1	106.0 (6)	O3—C30—O4	105.9 (5)
O2—C13—C14	111.7 (7)	O3—C30—C32	112.3 (7)
O1—C13—C14	106.6 (7)	O4—C30—C32	106.5 (7)
O2—C13—C15	106.6 (7)	O3—C30—C31	110.6 (7)
O1—C13—C15	109.4 (6)	O4—C30—C31	108.4 (7)
C14—C13—C15	116.1 (8)	C32—C30—C31	112.8 (7)
C13—C14—H14A	109.5	C30—C31—H31A	109.5
C13—C14—H14B	109.5	C30—C31—H31B	109.5
H14A—C14—H14B	109.5	H31A—C31—H31B	109.5
C13—C14—H14C	109.5	C30—C31—H31C	109.5
H14A—C14—H14C	109.5	H31A—C31—H31C	109.5
H14B—C14—H14C	109.5	H31B—C31—H31C	109.5
C13—C15—H15A	109.5	C30—C32—H32A	109.5
C13—C15—H15B	109.5	C30—C32—H32B	109.5
H15A—C15—H15B	109.5	H32A—C32—H32B	109.5
C13—C15—H15C	109.5	C30—C32—H32C	109.5
H15A—C15—H15C	109.5	H32A—C32—H32C	109.5
H15B—C15—H15C	109.5	H32B—C32—H32C	109.5
N1—C16—H16A	109.5	N5—C33—H33A	109.5
N1—C16—H16B	109.5	N5—C33—H33B	109.5
H16A—C16—H16B	109.5	H33A—C33—H33B	109.5
N1—C16—H16C	109.5	N5—C33—H33C	109.5
H16A—C16—H16C	109.5	H33A—C33—H33C	109.5
H16B—C16—H16C	109.5	H33B—C33—H33C	109.5
N4—C17—H17A	109.5	N8—C34—H34A	109.5
N4—C17—H17B	109.5	N8—C34—H34B	109.5
H17A—C17—H17B	109.5	H34A—C34—H34B	109.5
N4—C17—H17C	109.5	N8—C34—H34C	109.5
H17A—C17—H17C	109.5	H34A—C34—H34C	109.5
H17B—C17—H17C	109.5	H34B—C34—H34C	109.5
C2—N1—C1—N2	−0.6 (8)	C19—N5—C18—N6	−0.5 (8)
C16—N1—C1—N2	−177.6 (7)	C33—N5—C18—N6	179.2 (7)
C2—N1—C1—S1	−178.7 (6)	C19—N5—C18—S3	−178.0 (6)
C16—N1—C1—S1	4.3 (11)	C33—N5—C18—S3	1.7 (11)
C3—N2—C1—N1	0.8 (8)	C20—N6—C18—N5	0.4 (8)
C4—N2—C1—N1	176.6 (6)	C21—N6—C18—N5	−177.5 (6)

C3—N2—C1—S1	178.9 (6)	C20—N6—C18—S3	177.9 (6)
C4—N2—C1—S1	−5.4 (10)	C21—N6—C18—S3	0.0 (11)
C1—N1—C2—C3	0.2 (9)	C18—N5—C19—C20	0.4 (9)
C16—N1—C2—C3	177.1 (7)	C33—N5—C19—C20	−179.3 (7)
N1—C2—C3—N2	0.3 (9)	N5—C19—C20—N6	−0.1 (9)
C1—N2—C3—C2	−0.7 (8)	C18—N6—C20—C19	−0.2 (9)
C4—N2—C3—C2	−176.2 (7)	C21—N6—C20—C19	177.8 (7)
C1—N2—C4—C5	94.4 (8)	C20—N6—C21—C22	−110.2 (8)
C3—N2—C4—C5	−90.7 (9)	C18—N6—C21—C22	67.4 (10)
N2—C4—C5—C6	48.6 (9)	N6—C21—C22—C23	137.8 (7)
C13—O1—C6—C5	150.7 (6)	C30—O3—C23—C24	36.5 (8)
C13—O1—C6—C7	26.5 (7)	C30—O3—C23—C22	158.5 (7)
C4—C5—C6—O1	56.9 (9)	C21—C22—C23—O3	63.6 (9)
C4—C5—C6—C7	170.7 (7)	C21—C22—C23—C24	176.5 (7)
C13—O2—C7—C6	35.2 (7)	C30—O4—C24—C25	153.0 (6)
C13—O2—C7—C8	156.8 (6)	C30—O4—C24—C23	31.5 (7)
O1—C6—C7—O2	−37.8 (7)	O3—C23—C24—O4	−41.2 (7)
C5—C6—C7—O2	−156.7 (7)	C22—C23—C24—O4	−159.9 (6)
O1—C6—C7—C8	−156.8 (7)	O3—C23—C24—C25	−159.1 (6)
C5—C6—C7—C8	84.3 (9)	C22—C23—C24—C25	82.2 (9)
O2—C7—C8—C9	63.8 (9)	O4—C24—C25—C26	54.5 (8)
C6—C7—C8—C9	−180.0 (7)	C23—C24—C25—C26	168.2 (6)
C10—N3—C9—C8	−106.2 (8)	C27—N7—C26—C25	−95.3 (9)
C12—N3—C9—C8	70.3 (9)	C29—N7—C26—C25	92.9 (8)
C7—C8—C9—N3	132.0 (8)	C24—C25—C26—N7	53.4 (9)
C12—N3—C10—C11	1.5 (9)	C29—N7—C27—C28	0.0 (9)
C9—N3—C10—C11	178.4 (7)	C26—N7—C27—C28	−172.5 (7)
N3—C10—C11—N4	−2.1 (9)	N7—C27—C28—N8	1.0 (9)
C12—N4—C11—C10	2.1 (9)	C29—N8—C28—C27	−1.7 (8)
C17—N4—C11—C10	−179.0 (8)	C34—N8—C28—C27	177.9 (7)
C11—N4—C12—N3	−1.1 (8)	C27—N7—C29—N8	−1.1 (8)
C17—N4—C12—N3	179.9 (7)	C26—N7—C29—N8	171.8 (6)
C11—N4—C12—S2	−179.4 (6)	C27—N7—C29—S4	178.9 (6)
C17—N4—C12—S2	1.6 (11)	C26—N7—C29—S4	−8.3 (11)
C10—N3—C12—N4	−0.3 (8)	C28—N8—C29—N7	1.7 (7)
C9—N3—C12—N4	−177.2 (7)	C34—N8—C29—N7	−178.0 (7)
C10—N3—C12—S2	178.0 (6)	C28—N8—C29—S4	−178.3 (6)
C9—N3—C12—S2	1.1 (11)	C34—N8—C29—S4	2.0 (11)
C7—O2—C13—O1	−18.3 (7)	C23—O3—C30—O4	−17.9 (8)
C7—O2—C13—C14	97.4 (8)	C23—O3—C30—C32	97.9 (8)
C7—O2—C13—C15	−134.8 (7)	C23—O3—C30—C31	−135.1 (7)
C6—O1—C13—O2	−6.3 (7)	C24—O4—C30—O3	−9.8 (8)
C6—O1—C13—C14	−125.4 (7)	C24—O4—C30—C32	−129.5 (7)
C6—O1—C13—C15	108.2 (7)	C24—O4—C30—C31	108.9 (7)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···S1^{i}	0.95	2.86	3.703 (8)	148

C16—H16C···O3	0.98	2.35	3.321 (10)	171
C33—H33C···O1^{ii}	0.98	2.49	3.424 (10)	160
C34—H34C···O2^{iii}	0.98	2.33	3.266 (10)	160

Symmetry codes: (i) *x*−1, *y*, *z*; (ii) *x*−1, *y*, *z*−1; (iii) *x*, *y*, *z*−1.

Fig. 1

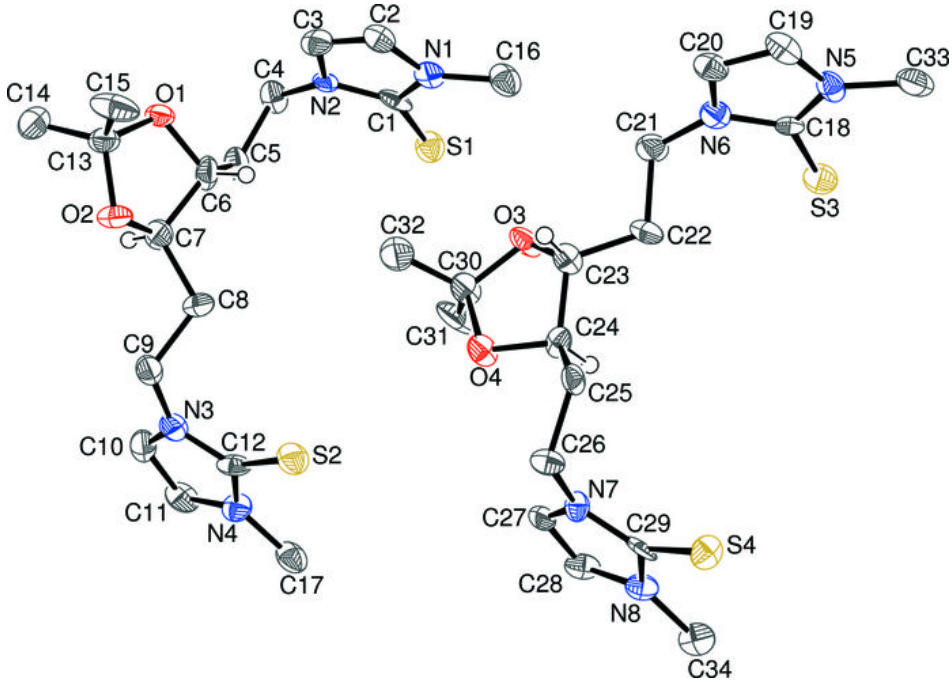
C3
C2
N1
C16
C4
N2
C1
S1
C14
C15
O1
C13
C5
C6
O2
C7
C8
C9
N3
C10
C12
S2
C11
N4
C17
C19
C20
N5
C33
C21
N6
C18
S3
C32
O3
C22
C30
C23
C31
C24
O4
C25
C26
N7
C27
C29
S4
C28
N8
C34

Fig. 2