

## 3-(1,3-Dithiolan-2-ylidene)-1-phenylpyridine-2,4(1*H*,3*H*)-dione

Lei-Jiao Li, Yan Li, Xi-Yun Hao and Xiu-Yun Sun\*

School of Chemical and Pharmaceutical Engineering, Jilin Institute of Chemical Technology, Jilin 132022, People's Republic of China  
Correspondence e-mail: xiuyuns@sina.com

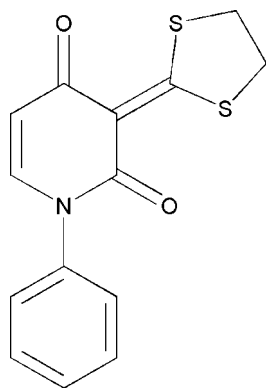
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Key indicators: single-crystal X-ray study;  $T = 292$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å; disorder in main residue;  $R$  factor = 0.029;  $wR$  factor = 0.071; data-to-parameter ratio = 13.3.

The title compound,  $\text{C}_{14}\text{H}_{11}\text{NO}_2\text{S}_2$ , was synthesized by reaction of 2-(1,3-dithiolan-2-ylidene)-3-oxo-*N*-phenylbutanamide with *N,N'*-dimethylformamide dimethyl acetal in *N,N'*-dimethylformamide. The molecule exhibits a V-shaped conformation in the crystal, with a dihedral angle of  $65.9$  ( $2$ ) $^\circ$  between the benzene and pyridine rings. In the crystal,  $\text{C}-\text{H}\cdots\text{O}$  and  $\text{C}-\text{H}\cdots\text{S}$  interactions are observed. Two C atoms of the dithiolane ring are disordered with occupancies in the ratio 0.541 (13)/0.459 (13).

### Related literature

For the synthesis, see Li *et al.*, (2008).



### Experimental

#### Crystal data

$\text{C}_{14}\text{H}_{11}\text{NO}_2\text{S}_2$

$M_r = 289.36$

Monoclinic,  $P2_1$   
 $a = 5.7708$  (17) Å  
 $b = 12.033$  (4) Å  
 $c = 9.624$  (3) Å  
 $\beta = 101.094$  (3) $^\circ$   
 $V = 655.8$  (4) Å $^3$

$Z = 2$   
Mo  $K\alpha$  radiation  
 $\mu = 0.40$  mm $^{-1}$   
 $T = 292$  K  
 $0.35 \times 0.29 \times 0.28$  mm

#### Data collection

Bruker APEXII CCD diffractometer  
Absorption correction: multi-scan (SADABS; Bruker, 2002)  
 $T_{\min} = 0.870$ ,  $T_{\max} = 0.894$

5598 measured reflections  
2543 independent reflections  
2361 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.050$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.029$   
 $wR(F^2) = 0.071$   
 $S = 1.04$   
2543 reflections  
191 parameters  
1 restraint

H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 0.17$  e Å $^{-3}$   
 $\Delta\rho_{\text{min}} = -0.21$  e Å $^{-3}$   
Absolute structure: Flack (1983),  
1181 Friedel pairs  
Flack parameter:  $-0.05$  (5)

**Table 1**

Hydrogen-bond geometry (Å,  $^\circ$ ).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C1}'-\text{H1}'A\cdots\text{O1}^i$	0.97	2.43	3.300 (17)	148
$\text{C2}-\text{H2}B\cdots\text{O1}^i$	0.97	2.69	3.388 (8)	129
$\text{C7}-\text{H7}\cdots\text{O2}^{ii}$	0.93	2.36	3.259 (3)	163
$\text{C14}-\text{H14}\cdots\text{O2}^{iii}$	0.93	2.46	3.293 (3)	149
$\text{C11}-\text{H11}\cdots\text{S1}^{iv}$	0.93	2.90	3.756 (3)	153

Symmetry codes: (i)  $-x+1, y-\frac{1}{2}, -z+1$ ; (ii)  $-x, y+\frac{1}{2}, -z+2$ ; (iii)  $-x+1, y+\frac{1}{2}, -z+2$ ; (iv)  $-x, y+\frac{1}{2}, -z+1$ .

Data collection: APEX2 (Bruker, 2007); cell refinement: SAINT (Bruker, 2007); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); software used to prepare material for publication: SHELXTL.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RK2145).

### References

- Bruker (2002). *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.  
Bruker (2007). *APEX2*, and *S SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.  
Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.  
Li, Y.-H., Li, W.-L., Zhang, R., Zhou, Y. & Dong, D.-W. (2008). *Synthesis*, **21**, 3411–3414.  
Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.

**supplementary materials**

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### 3-(1,3-Dithiolan-2-ylidene)-1-phenylpyridine-2,4(1*H*,3*H*)-dione

L.-J. Li, Y. Li, X.-Y. Hao and X.-Y. Sun

#### Comment

*N*-Substituted pyridone compounds have versatile physiological activity. For instance, they can sterilize, ease pain, resist tumour and cure parkinsonism and so forth. These compounds can cure  $\beta$ -thalassemia beyond compare, because they have powerful ability to form coordination compounds. In the molecule of the title compound, (Fig. 1), C1 and C2 are disordered in ratio 0.541 (13)/0.459 (13). The molecule exhibits a V-shaped conformation in the crystal with a dihedral angle of 65.9 (2) $^\circ$  between the benzene ring and the pyridine ring. The dihedral angle between the pyridine ring and the dithiolane ring is 2.6 (8) $^\circ$ .

#### Experimental

The title compound, with M.P. 497 K, was synthesized according to the literature (Li *et al.*, 2008). It was dissolved in ethyl acetate at room temperature and hexane was added. The solution was kept at room temperature in a sealed flask for a few days to give single crystals suitable for single crystal X-ray analysis.

#### Refinement

All H atoms bound to C atoms were generated geometrically and refined as riding atoms with C–H = 0.93 Å for aromatic H and 0.97 Å for CH<sub>2</sub> groups, with  $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$ .

#### Figures

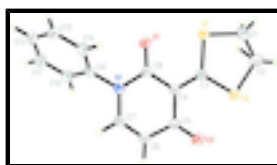


Fig. 1. Molecular structure of the title compound with atom numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are presented as a small spheres of arbitrary radius. Only major fragment of disordered cycle is presented.

### 3-(1,3-Dithiolan-2-ylidene)-1-phenylpyridine-2,4(1*H*,3*H*)-dione

#### Crystal data

C<sub>14</sub>H<sub>11</sub>NO<sub>2</sub>S<sub>2</sub>

$M_r = 289.36$

Monoclinic,  $P2_1$

Hall symbol: P 2yb

$a = 5.7708$  (17) Å

$b = 12.033$  (4) Å

$F_{000} = 300$

$D_x = 1.465$  Mg m<sup>-3</sup>

Melting point: 497 K

Mo  $K\alpha$  radiation

$\lambda = 0.71073$  Å

Cell parameters from 3388 reflections

$\theta = 2.7\text{--}26.1^\circ$

# supplementary materials

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$c = 9.624 (3) \text{ \AA}$	$\mu = 0.40 \text{ mm}^{-1}$
$\beta = 101.094 (3)^\circ$	$T = 292 \text{ K}$
$V = 655.8 (4) \text{ \AA}^3$	Block, yellow
$Z = 2$	$0.35 \times 0.29 \times 0.28 \text{ mm}$

## Data collection

Bruker APEXII CCD diffractometer	2543 independent reflections
Radiation source: fine-focus sealed tube	2361 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.050$
$T = 292 \text{ K}$	$\theta_{\text{max}} = 26.1^\circ$
$\omega$ scans	$\theta_{\text{min}} = 2.7^\circ$
Absorption correction: multi-scan (SADABS; Bruker, 2002)	$h = -7 \rightarrow 7$
$T_{\text{min}} = 0.870, T_{\text{max}} = 0.894$	$k = -14 \rightarrow 14$
5598 measured reflections	$l = -11 \rightarrow 11$

## Refinement

Refinement on $F^2$	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.029$	$w = 1/[\sigma^2(F_o^2) + (0.035P)^2]$
$wR(F^2) = 0.071$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.04$	$(\Delta/\sigma)_{\text{max}} = 0.001$
2543 reflections	$\Delta\rho_{\text{max}} = 0.17 \text{ e \AA}^{-3}$
191 parameters	$\Delta\rho_{\text{min}} = -0.21 \text{ e \AA}^{-3}$
1 restraint	Extinction correction: none
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983), 1181 Friedel pairs
Secondary atom site location: difference Fourier map	Flack parameter: $-0.05 (5)$

## Special details

**Geometry.** All s.u.'s (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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  $F^2 > 2\sigma(F^2)$  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 ( $\text{\AA}^2$ )

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
S1	0.64100 (9)	0.15466 (4)	0.63757 (5)	0.04732 (14)	
S2	0.47667 (10)	-0.01817 (4)	0.80937 (6)	0.05463 (16)	
C1	0.775 (3)	0.0231 (13)	0.6391 (16)	0.063 (3)	0.541 (13)
H1A	0.8165	0.0099	0.5476	0.076*	0.541 (13)
H1B	0.9197	0.0230	0.7097	0.076*	0.541 (13)
C2	0.6217 (17)	-0.0684 (4)	0.6704 (9)	0.0580 (18)	0.541 (13)
H2A	0.7152	-0.1339	0.7017	0.070*	0.541 (13)
H2B	0.5059	-0.0874	0.5866	0.070*	0.541 (13)
C1'	0.742 (3)	0.0127 (13)	0.6019 (19)	0.064 (4)	0.459 (13)
H1'A	0.6365	-0.0202	0.5217	0.076*	0.459 (13)
H1'B	0.9000	0.0149	0.5818	0.076*	0.459 (13)
C2'	0.7381 (17)	-0.0516 (5)	0.7321 (11)	0.057 (2)	0.459 (13)
H2'A	0.8802	-0.0357	0.8011	0.069*	0.459 (13)
H2'B	0.7374	-0.1303	0.7104	0.069*	0.459 (13)
C3	0.4657 (3)	0.12007 (16)	0.75830 (19)	0.0385 (4)	
C4	0.3248 (3)	0.19662 (16)	0.8105 (2)	0.0384 (4)	
C5	0.1840 (3)	0.16281 (18)	0.91489 (18)	0.0419 (4)	
C6	0.0463 (4)	0.24818 (18)	0.9638 (2)	0.0495 (5)	
H6	-0.0410	0.2314	1.0328	0.059*	
C7	0.0409 (3)	0.35039 (17)	0.9126 (2)	0.0477 (5)	
H7	-0.0534	0.4027	0.9462	0.057*	
C8	0.3154 (3)	0.31026 (17)	0.7579 (2)	0.0422 (4)	
C9	0.1463 (3)	0.49614 (15)	0.7608 (2)	0.0402 (4)	
C10	-0.0659 (3)	0.53405 (19)	0.6854 (2)	0.0517 (5)	
H10	-0.1967	0.4874	0.6672	0.062*	
C11	-0.0823 (4)	0.6429 (2)	0.6368 (2)	0.0613 (6)	
H11	-0.2248	0.6693	0.5854	0.074*	
C12	0.1097 (5)	0.71177 (19)	0.6640 (3)	0.0646 (6)	
H12	0.0981	0.7844	0.6301	0.078*	
C13	0.3191 (4)	0.67360 (19)	0.7411 (3)	0.0604 (6)	
H13	0.4486	0.7209	0.7608	0.072*	
C14	0.3393 (3)	0.56575 (17)	0.7898 (2)	0.0494 (5)	
H14	0.4820	0.5401	0.8419	0.059*	
N1	0.1675 (3)	0.38340 (13)	0.81222 (16)	0.0422 (4)	
O1	0.4251 (3)	0.34287 (12)	0.66800 (17)	0.0611 (4)	
O2	0.1866 (2)	0.06549 (12)	0.95739 (17)	0.0562 (4)	

Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
S1	0.0552 (3)	0.0452 (3)	0.0482 (3)	0.0055 (2)	0.0264 (2)	0.0024 (2)
S2	0.0693 (3)	0.0355 (2)	0.0669 (3)	0.0066 (2)	0.0325 (3)	0.0060 (3)
C1	0.071 (4)	0.066 (6)	0.064 (7)	0.005 (3)	0.039 (4)	-0.022 (4)
C2	0.067 (4)	0.040 (2)	0.074 (4)	0.004 (2)	0.030 (3)	-0.007 (2)

## supplementary materials

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C1'	0.111 (10)	0.034 (4)	0.059 (8)	0.014 (4)	0.048 (6)	-0.012 (4)
C2'	0.059 (4)	0.035 (3)	0.081 (5)	0.005 (3)	0.022 (4)	0.001 (3)
C3	0.0437 (10)	0.0379 (10)	0.0358 (9)	-0.0007 (8)	0.0123 (7)	0.0000 (7)
C4	0.0437 (9)	0.0369 (9)	0.0381 (9)	-0.0002 (7)	0.0167 (7)	0.0008 (8)
C5	0.0482 (10)	0.0415 (10)	0.0387 (9)	-0.0055 (9)	0.0154 (7)	0.0024 (9)
C6	0.0597 (12)	0.0503 (12)	0.0469 (11)	0.0006 (10)	0.0313 (9)	0.0002 (9)
C7	0.0556 (11)	0.0459 (12)	0.0484 (11)	0.0022 (9)	0.0272 (9)	-0.0053 (9)
C8	0.0501 (11)	0.0399 (10)	0.0412 (10)	0.0042 (8)	0.0199 (8)	0.0025 (8)
C9	0.0458 (9)	0.0376 (10)	0.0417 (9)	0.0042 (8)	0.0194 (7)	0.0006 (8)
C10	0.0436 (10)	0.0543 (12)	0.0589 (13)	0.0057 (9)	0.0144 (9)	-0.0045 (10)
C11	0.0611 (12)	0.0638 (15)	0.0593 (12)	0.0260 (12)	0.0127 (10)	0.0049 (12)
C12	0.0907 (17)	0.0430 (11)	0.0712 (14)	0.0188 (12)	0.0433 (13)	0.0104 (11)
C13	0.0627 (13)	0.0433 (12)	0.0831 (15)	-0.0024 (10)	0.0341 (11)	-0.0004 (11)
C14	0.0463 (10)	0.0441 (11)	0.0600 (14)	0.0013 (9)	0.0153 (9)	0.0000 (9)
N1	0.0508 (9)	0.0347 (8)	0.0465 (9)	0.0038 (7)	0.0227 (7)	-0.0003 (7)
O1	0.0833 (10)	0.0434 (8)	0.0717 (10)	0.0140 (8)	0.0525 (8)	0.0151 (7)
O2	0.0678 (9)	0.0442 (8)	0.0644 (9)	-0.0033 (7)	0.0323 (7)	0.0112 (7)

### *Geometric parameters (Å, °)*

S1—C3	1.732 (2)	C5—C6	1.432 (3)
S1—C1	1.761 (15)	C6—C7	1.323 (3)
S1—C1'	1.857 (16)	C6—H6	0.9300
S2—C3	1.732 (2)	C7—N1	1.377 (2)
S2—C2	1.813 (5)	C7—H7	0.9300
S2—C2'	1.850 (6)	C8—O1	1.231 (2)
C1—C2	1.479 (19)	C8—N1	1.396 (2)
C1—H1A	0.9700	C9—C10	1.376 (3)
C1—H1B	0.9700	C9—C14	1.379 (3)
C2—H2A	0.9700	C9—N1	1.441 (2)
C2—H2B	0.9700	C10—C11	1.388 (3)
C1'—C2'	1.48 (2)	C10—H10	0.9300
C1'—H1'A	0.9700	C11—C12	1.367 (4)
C1'—H1'B	0.9700	C11—H11	0.9300
C2'—H2'A	0.9700	C12—C13	1.370 (3)
C2'—H2'B	0.9700	C12—H12	0.9300
C3—C4	1.385 (3)	C13—C14	1.377 (3)
C4—C8	1.455 (3)	C13—H13	0.9300
C4—C5	1.466 (2)	C14—H14	0.9300
C5—O2	1.240 (2)		
C3—S1—C1	95.2 (5)	C8—C4—C5	120.70 (16)
C3—S1—C1'	98.4 (7)	O2—C5—C6	122.80 (17)
C1—S1—C1'	12.1 (9)	O2—C5—C4	121.01 (18)
C3—S2—C2	96.19 (18)	C6—C5—C4	116.20 (18)
C3—S2—C2'	94.75 (19)	C7—C6—C5	121.31 (18)
C2—S2—C2'	26.22 (19)	C7—C6—H6	119.3
C2—C1—S1	113.1 (9)	C5—C6—H6	119.3
C2—C1—H1A	109.0	C6—C7—N1	123.46 (17)
S1—C1—H1A	109.0	C6—C7—H7	118.3

C2—C1—H1B	109.0	N1—C7—H7	118.3
S1—C1—H1B	109.0	O1—C8—N1	119.78 (18)
H1A—C1—H1B	107.8	O1—C8—C4	123.65 (17)
C1—C2—S2	106.3 (7)	N1—C8—C4	116.55 (16)
C1—C2—H2A	110.5	C10—C9—C14	120.52 (19)
S2—C2—H2A	110.5	C10—C9—N1	120.09 (17)
C1—C2—H2B	110.5	C14—C9—N1	119.38 (17)
S2—C2—H2B	110.5	C9—C10—C11	119.1 (2)
H2A—C2—H2B	108.7	C9—C10—H10	120.4
C2'—C1'—S1	105.6 (11)	C11—C10—H10	120.4
C2'—C1'—H1'A	110.6	C12—C11—C10	120.5 (2)
S1—C1'—H1'A	110.6	C12—C11—H11	119.8
C2'—C1'—H1'B	110.6	C10—C11—H11	119.8
S1—C1'—H1'B	110.6	C11—C12—C13	119.9 (2)
H1'A—C1'—H1'B	108.7	C11—C12—H12	120.1
C1'—C2'—S2	111.9 (9)	C13—C12—H12	120.1
C1'—C2'—H2'A	109.2	C12—C13—C14	120.6 (2)
S2—C2'—H2'A	109.2	C12—C13—H13	119.7
C1'—C2'—H2'B	109.2	C14—C13—H13	119.7
S2—C2'—H2'B	109.2	C13—C14—C9	119.4 (2)
H2'A—C2'—H2'B	107.9	C13—C14—H14	120.3
C4—C3—S1	122.87 (14)	C9—C14—H14	120.3
C4—C3—S2	121.84 (15)	C7—N1—C8	121.74 (16)
S1—C3—S2	115.28 (11)	C7—N1—C9	119.40 (15)
C3—C4—C8	118.94 (16)	C8—N1—C9	118.86 (15)
C3—C4—C5	120.35 (18)		
C3—S1—C1—C2	29.1 (11)	O2—C5—C6—C7	-177.6 (2)
C1'—S1—C1—C2	-77 (5)	C4—C5—C6—C7	2.6 (3)
S1—C1—C2—S2	-40.8 (12)	C5—C6—C7—N1	-1.2 (3)
C3—S2—C2—C1	31.6 (9)	C3—C4—C8—O1	0.5 (3)
C2'—S2—C2—C1	-56.5 (9)	C5—C4—C8—O1	-178.25 (19)
C3—S1—C1'—C2'	-28.1 (12)	C3—C4—C8—N1	178.91 (17)
C1—S1—C1'—C2'	47 (5)	C5—C4—C8—N1	0.1 (3)
S1—C1'—C2'—S2	40.1 (13)	C14—C9—C10—C11	1.1 (3)
C3—S2—C2'—C1'	-33.7 (10)	N1—C9—C10—C11	-179.94 (18)
C2—S2—C2'—C1'	60.7 (11)	C9—C10—C11—C12	-0.3 (3)
C1—S1—C3—C4	175.0 (5)	C10—C11—C12—C13	-0.8 (4)
C1'—S1—C3—C4	-173.3 (6)	C11—C12—C13—C14	1.0 (4)
C1—S1—C3—S2	-5.4 (5)	C12—C13—C14—C9	-0.3 (3)
C1'—S1—C3—S2	6.3 (6)	C10—C9—C14—C13	-0.8 (3)
C2—S2—C3—C4	165.9 (4)	N1—C9—C14—C13	-179.81 (18)
C2'—S2—C3—C4	-167.8 (4)	C6—C7—N1—C8	-0.9 (3)
C2—S2—C3—S1	-13.8 (4)	C6—C7—N1—C9	178.73 (18)
C2'—S2—C3—S1	12.5 (4)	O1—C8—N1—C7	179.79 (18)
S1—C3—C4—C8	2.5 (3)	C4—C8—N1—C7	1.4 (3)
S2—C3—C4—C8	-177.09 (14)	O1—C8—N1—C9	0.2 (3)
S1—C3—C4—C5	-178.70 (14)	C4—C8—N1—C9	-178.25 (16)
S2—C3—C4—C5	1.7 (2)	C10—C9—N1—C7	-65.0 (2)
C3—C4—C5—O2	-0.6 (3)	C14—C9—N1—C7	114.0 (2)

## supplementary materials

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C8—C4—C5—O2	178.16 (18)	C10—C9—N1—C8	114.7 (2)
C3—C4—C5—C6	179.24 (18)	C14—C9—N1—C8	-66.3 (2)
C8—C4—C5—C6	-2.0 (3)		

### Hydrogen-bond geometry ( $\text{\AA}$ , $^\circ$ )

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C1'—H1'A $\cdots$ O1 <sup>i</sup>	0.97	2.43	3.300 (17)	148
C2—H2B $\cdots$ O1 <sup>i</sup>	0.97	2.69	3.388 (8)	129
C7—H7 $\cdots$ O2 <sup>ii</sup>	0.93	2.36	3.259 (3)	163
C14—H14 $\cdots$ O2 <sup>iii</sup>	0.93	2.46	3.293 (3)	149
C11—H11 $\cdots$ S1 <sup>iv</sup>	0.93	2.90	3.756 (3)	153

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



Fig. 1

