

**(5''E)-1''-Benzyl-1'-methyl-5''-[4-(methylsulfanyl)benzylidene]-4'-[4-(methylsulfanyl)phenyl]dispiro[indoline-3,2'-pyrrolidine-3',3''-piperidine]-2,4''-dione dichloromethane solvate**

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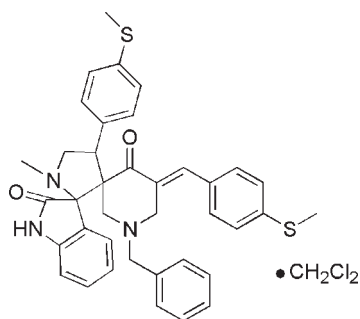
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Key indicators: single-crystal X-ray study;  $T = 113$  K; mean  $\sigma(\text{C}-\text{C}) = 0.006$  Å; disorder in solvent or counterion;  $R$  factor = 0.059;  $wR$  factor = 0.154; data-to-parameter ratio = 13.8.

In the title compound,  $\text{C}_{38}\text{H}_{36}\text{N}_3\text{O}_2\text{S}_2 \cdot \text{CH}_2\text{Cl}_2$ , the 2-oxindole ring is almost planar (r.m.s. deviation = 0.032 Å), the pyrrolidine ring adopts a twist conformation and the piperidone ring exists as a chair. Three short  $\text{C}-\text{H} \cdots \text{O}$  intramolecular contacts occur. In the crystal, molecules are linked by  $\text{C}-\text{H} \cdots \text{O}$  and  $\text{C}-\text{H} \cdots \text{N}$  interactions. The dichloromethane solvent molecule is disordered over two orientations in a 0.765 (11):0.235 (11) ratio.

Related literature

For background to dispiro ring systems, see: Kobayashi *et al.* (1991).



Experimental

Crystal data

$\text{C}_{38}\text{H}_{36}\text{N}_3\text{O}_2\text{S}_2 \cdot \text{CH}_2\text{Cl}_2$   
 $M_r = 715.74$   
Monoclinic,  $C2/c$   
 $a = 36.456$  (7) Å  
 $b = 8.6164$  (17) Å  
 $c = 26.184$  (5) Å  
 $\beta = 119.58$  (3)°

$V = 7153$  (2) Å<sup>3</sup>  
 $Z = 8$   
Mo  $K\alpha$  radiation  
 $\mu = 0.34$  mm<sup>-1</sup>  
 $T = 113$  K  
 $0.20 \times 0.18 \times 0.12$  mm

Data collection

Rigaku Saturn CCD diffractometer  
Absorption correction: multi-scan  
(*CrystalClear*; Rigaku, 2005)  
 $T_{\min} = 0.936$ ,  $T_{\max} = 0.961$

20568 measured reflections  
6263 independent reflections  
2890 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.116$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.059$   
 $wR(F^2) = 0.154$   
 $S = 1.01$   
6263 reflections  
455 parameters

40 restraints  
H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 0.44$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.46$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$\text{C1}-\text{H1A} \cdots \text{O2}$	0.97	2.53	3.149 (5)	122
$\text{C7}-\text{H7B} \cdots \text{O2}$	0.97	2.37	2.971 (5)	119
$\text{C10}-\text{H10} \cdots \text{O1}$	0.93	2.57	3.220 (5)	127
$\text{C39}-\text{H39A} \cdots \text{N3}$	0.97	2.44	3.405 (10)	175
$\text{C39}-\text{H39B} \cdots \text{O1}$	0.97	2.53	3.140 (7)	121

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; 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*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5259).

References

Kobayashi, J., Tsuda, M., Agemi, K. & Vacelet, J. (1991). *Tetrahedron*, **47**, 6617–6622.  
Rigaku (2005). *CrystalClear*. Rigaku Corporation, Tokyo, Japan.  
Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.

**supplementary materials**

Acta Cryst. (2010). E66, o244 [ doi:10.1107/S1600536809054774 ]

**(5''E)-1''-Benzyl-1'-methyl-5''-[4-(methylsulfanyl)benzylidene]-4'-[4-(methylsulfanyl)phenyl]dispiro[indoline-3,2'-pyrrolidine-3',3''-piperidine]-2,4''-dione dichloromethane solvate**

**Y. Hou**

**Comment**

In this paper, the structure of the title compound, (I), is reported. The molecular structure of (I) is illustrated in Fig. 1. There exists a dispiro rings in the molecule which consists of a 2-oxindole ring, a pyrrolidine ring and a piperidone rings. The pyrrolidine ring is not planar, with twist conformations. 2-oxindole ring (C15/N2/C14/C13/C12/C11/C10/C9/C8) is nearly planar that the mean deviation from this plane is 0.0148 (3)%A.

The piperidone ring has the usual chair conformation. There exist intermolecular O—H—N hydrogen bonds.

**Experimental**

A mixture of 1-benzyl-3,5-bis-(4-methylsulfanyl-benzylidene)-piperidin-4-one (2 mmol), isatin (2 mmol) and sarcosine (2 mmol) were refluxed in methanol (80 ml) until the disappearance of the starting material as evidenced by the TLC. After the reaction was over the solvent was removed *in vacuo* and the residue was separated by column chromatography (silica gel, petroleum ether/ethyl acetate = 5:1) to give the title compound. 20 mg of the title compound was dissolved in 15 ml dichloromethane; the solution was kept at room temperature for 15 d by natural evaporation to give colourless blocks of (I).

**Refinement**

All H atoms were fixed geometrically and treated as riding with C—H = 0.96Å (methyl), 0.97Å (methylene), 0.98Å (methine) with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$  or  $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{methine})$ .

**Figures**

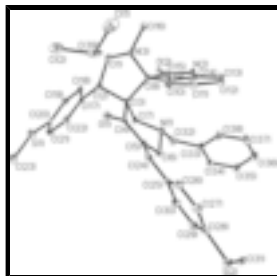


Fig. 1. The molecular structure of (I), drawn with 30% probability ellipsoids

# supplementary materials

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## (5<sup>E</sup>)-1<sup>H</sup>-Benzyl-1<sup>H</sup>-methyl-5<sup>H</sup>-[4-(methylsulfonyl)benzylidene]-4<sup>H</sup>-[4-(methylsulfonyl)phenyl]dispiro[indoline-3,2<sup>H</sup>-pyrrolidine-3<sup>H</sup>,3<sup>H</sup>- piperidine]-2,4<sup>H</sup>-dione dichloromethane solvate

### Crystal data

C<sub>38</sub>H<sub>36</sub>N<sub>3</sub>O<sub>2</sub>S<sub>2</sub>·CH<sub>2</sub>Cl<sub>2</sub>

*M<sub>r</sub>* = 715.74

Monoclinic, *C2/c*

Hall symbol: -C 2yc

*a* = 36.456 (7) Å

*b* = 8.6164 (17) Å

*c* = 26.184 (5) Å

β = 119.58 (3)°

*V* = 7153 (2) Å<sup>3</sup>

*Z* = 8

*F*(000) = 3000

*D<sub>x</sub>* = 1.329 Mg m<sup>-3</sup>

Melting point: 473 K

Mo *K*α radiation, λ = 0.71073 Å

Cell parameters from 8719 reflections

θ = 1.6–27.9°

μ = 0.34 mm<sup>-1</sup>

*T* = 113 K

Block, colourless

0.20 × 0.18 × 0.12 mm

### Data collection

Rigaku Saturn CCD  
diffractometer

Radiation source: rotating anode  
confocal

Detector resolution: 7.31 pixels mm<sup>-1</sup>

ω and φ scans

Absorption correction: multi-scan  
(*CrystalClear*; Rigaku, 2005)

*T<sub>min</sub>* = 0.936, *T<sub>max</sub>* = 0.961

20568 measured reflections

6263 independent reflections

2890 reflections with *I* > 2σ(*I*)

*R<sub>int</sub>* = 0.116

θ<sub>max</sub> = 25.0°, θ<sub>min</sub> = 2.3°

*h* = -36→43

*k* = -10→10

*l* = -31→30

### Refinement

Refinement on *F*<sup>2</sup>

Least-squares matrix: full

*R* [*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.059

*wR* (*F*<sup>2</sup>) = 0.154

*S* = 1.01

6263 reflections

455 parameters

40 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/[σ<sup>2</sup>(*F<sub>o</sub>*<sup>2</sup>) + (0.028*P*)<sup>2</sup>]

where *P* = (*F<sub>o</sub>*<sup>2</sup> + 2*F<sub>c</sub>*<sup>2</sup>)/3

(Δ/σ)<sub>max</sub> = 0.001

Δρ<sub>max</sub> = 0.44 e Å<sup>-3</sup>

Δρ<sub>min</sub> = -0.46 e Å<sup>-3</sup>

*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 ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
S1	0.81245 (4)	-0.42806 (12)	0.32967 (5)	0.0300 (3)	
S2	1.03858 (4)	-0.26067 (12)	0.01917 (5)	0.0282 (3)	
O1	0.93859 (8)	0.1218 (3)	0.23595 (11)	0.0217 (7)	
O2	0.77000 (8)	0.1837 (3)	0.07939 (11)	0.0206 (6)	
N1	0.84168 (10)	-0.0525 (3)	0.08359 (13)	0.0167 (7)	
N2	0.80248 (10)	0.2974 (3)	0.03269 (13)	0.0161 (8)	
N3	0.84549 (10)	0.3613 (3)	0.17922 (13)	0.0162 (7)	
C1	0.83077 (13)	0.2676 (4)	0.21221 (16)	0.0197 (9)	
H1A	0.8014	0.2384	0.1879	0.024*	
H1B	0.8342	0.3225	0.2466	0.024*	
C2	0.85950 (12)	0.1269 (4)	0.22927 (17)	0.0188 (9)	
H2	0.8873	0.1605	0.2604	0.023*	
C3	0.86374 (12)	0.0969 (4)	0.17302 (15)	0.0123 (8)	
C4	0.90973 (12)	0.0694 (4)	0.18967 (16)	0.0161 (9)	
C5	0.91820 (12)	-0.0144 (4)	0.14716 (16)	0.0148 (9)	
C6	0.88378 (11)	-0.1081 (4)	0.09815 (16)	0.0178 (9)	
H6A	0.8870	-0.1016	0.0636	0.021*	
H6B	0.8866	-0.2162	0.1099	0.021*	
C7	0.83915 (12)	-0.0466 (4)	0.13777 (15)	0.0163 (9)	
H7A	0.8512	-0.1400	0.1607	0.020*	
H7B	0.8099	-0.0395	0.1284	0.020*	
C8	0.84714 (12)	0.2525 (4)	0.13602 (16)	0.0154 (9)	
C9	0.87097 (11)	0.3202 (4)	0.10800 (16)	0.0151 (9)	
C10	0.91286 (12)	0.3682 (4)	0.13121 (17)	0.0194 (9)	
H10	0.9318	0.3559	0.1711	0.023*	
C11	0.92606 (12)	0.4327 (4)	0.09561 (17)	0.0211 (10)	
H11	0.9539	0.4651	0.1116	0.025*	
C12	0.89836 (13)	0.4514 (4)	0.03508 (18)	0.0246 (10)	
H12	0.9081	0.4924	0.0111	0.029*	
C13	0.85601 (12)	0.4080 (4)	0.01113 (17)	0.0177 (9)	
H13	0.8370	0.4218	-0.0286	0.021*	
C14	0.84364 (12)	0.3449 (4)	0.04796 (16)	0.0163 (9)	
C15	0.80162 (12)	0.2343 (4)	0.08046 (16)	0.0192 (9)	

## supplementary materials

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C16	0.82163 (14)	0.5041 (4)	0.15488 (17)	0.0275 (11)	
H16A	0.7928	0.4790	0.1275	0.041*	
H16B	0.8336	0.5608	0.1350	0.041*	
H16C	0.8228	0.5664	0.1861	0.041*	
C17	0.84726 (12)	-0.0104 (4)	0.25245 (15)	0.0145 (9)	
C18	0.80632 (12)	-0.0352 (5)	0.24449 (16)	0.0223 (10)	
H18	0.7851	0.0364	0.2230	0.027*	
C19	0.79734 (13)	-0.1614 (5)	0.26757 (17)	0.0234 (10)	
H19	0.7700	-0.1745	0.2607	0.028*	
C20	0.82776 (13)	-0.2718 (4)	0.30111 (16)	0.0203 (10)	
C21	0.86876 (13)	-0.2481 (4)	0.31048 (17)	0.0233 (10)	
H21	0.8900	-0.3187	0.3329	0.028*	
C22	0.87787 (13)	-0.1194 (5)	0.28639 (17)	0.0247 (10)	
H22	0.9052	-0.1060	0.2932	0.030*	
C23	0.86044 (12)	-0.5343 (5)	0.37319 (17)	0.0286 (11)	
H23A	0.8693	-0.5819	0.3479	0.043*	
H23B	0.8557	-0.6130	0.3952	0.043*	
H23C	0.8820	-0.4646	0.3997	0.043*	
C24	0.95764 (12)	0.0014 (4)	0.15326 (17)	0.0207 (10)	
H24	0.9759	0.0637	0.1846	0.025*	
C25	0.97545 (12)	-0.0613 (4)	0.11979 (16)	0.0165 (9)	
C26	1.00906 (12)	0.0163 (4)	0.11886 (17)	0.0233 (10)	
H26	1.0188	0.1081	0.1400	0.028*	
C27	1.02810 (12)	-0.0368 (5)	0.08835 (18)	0.0254 (10)	
H27	1.0496	0.0210	0.0883	0.031*	
C28	1.01570 (12)	-0.1768 (4)	0.05726 (17)	0.0226 (10)	
C29	0.98184 (13)	-0.2554 (4)	0.05714 (18)	0.0249 (10)	
H29	0.9723	-0.3472	0.0359	0.030*	
C30	0.96232 (13)	-0.2024 (4)	0.08701 (18)	0.0232 (10)	
H30	0.9402	-0.2590	0.0859	0.028*	
C31	1.07740 (13)	-0.1205 (4)	0.02636 (19)	0.0291 (11)	
H31A	1.0637	-0.0238	0.0093	0.044*	
H31B	1.0918	-0.1576	0.0064	0.044*	
H31C	1.0974	-0.1052	0.0672	0.044*	
C32	0.80877 (13)	-0.1550 (4)	0.03998 (16)	0.0247 (10)	
H32A	0.7820	-0.1289	0.0374	0.030*	
H32B	0.8154	-0.2614	0.0537	0.030*	
C33	0.80392 (12)	-0.1451 (4)	-0.02036 (16)	0.0191 (9)	
C34	0.78570 (11)	-0.2681 (4)	-0.05875 (17)	0.0180 (9)	
H34	0.7792	-0.3589	-0.0457	0.022*	
C35	0.77693 (13)	-0.2570 (5)	-0.11688 (18)	0.0288 (11)	
H35	0.7646	-0.3402	-0.1423	0.035*	
C36	0.78647 (13)	-0.1237 (5)	-0.13671 (18)	0.0306 (11)	
H36	0.7800	-0.1160	-0.1757	0.037*	
C37	0.80560 (13)	-0.0015 (5)	-0.09880 (18)	0.0288 (11)	
H37	0.8126	0.0880	-0.1119	0.035*	
C38	0.81437 (13)	-0.0121 (5)	-0.04127 (17)	0.0244 (10)	
H38	0.8275	0.0706	-0.0159	0.029*	
Cl1	0.95562 (15)	0.6560 (5)	0.26917 (14)	0.0621 (10)	0.765 (11)

C39	0.9453 (3)	0.4623 (7)	0.2825 (3)	0.050 (2)	0.765 (11)
H39A	0.9163	0.4362	0.2546	0.060*	0.765 (11)
H39B	0.9634	0.3917	0.2762	0.060*	0.765 (11)
Cl1'	0.9728 (4)	0.5901 (17)	0.2710 (4)	0.060 (3)	0.235 (11)
C39'	0.9325 (4)	0.508 (4)	0.2821 (6)	0.049 (5)	0.235 (11)
H39C	0.9115	0.5868	0.2750	0.058*	0.235 (11)
H39D	0.9188	0.4246	0.2543	0.058*	0.235 (11)
Cl2	0.95372 (4)	0.43615 (14)	0.35449 (6)	0.0482 (4)	

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
S1	0.0261 (7)	0.0307 (6)	0.0351 (7)	-0.0029 (6)	0.0166 (6)	0.0082 (6)
S2	0.0301 (7)	0.0283 (6)	0.0334 (7)	0.0046 (5)	0.0213 (6)	0.0022 (6)
O1	0.0125 (15)	0.0322 (16)	0.0118 (15)	0.0005 (13)	-0.0006 (13)	-0.0042 (13)
O2	0.0099 (15)	0.0301 (15)	0.0189 (16)	-0.0067 (13)	0.0049 (13)	0.0066 (13)
N1	0.0142 (18)	0.0185 (16)	0.0123 (18)	-0.0033 (15)	0.0027 (15)	-0.0038 (15)
N2	0.0154 (18)	0.0216 (17)	0.0151 (18)	-0.0049 (15)	0.0104 (15)	0.0053 (15)
N3	0.0218 (19)	0.0154 (16)	0.0154 (18)	0.0015 (15)	0.0122 (16)	0.0002 (15)
C1	0.028 (3)	0.018 (2)	0.017 (2)	0.0012 (19)	0.013 (2)	-0.0018 (18)
C2	0.015 (2)	0.024 (2)	0.015 (2)	-0.0008 (19)	0.0060 (19)	-0.0012 (19)
C3	0.014 (2)	0.0155 (19)	0.009 (2)	0.0007 (18)	0.0065 (17)	0.0044 (17)
C4	0.018 (2)	0.0125 (19)	0.015 (2)	0.0009 (18)	0.007 (2)	0.0051 (18)
C5	0.012 (2)	0.022 (2)	0.013 (2)	-0.0010 (18)	0.0078 (18)	0.0014 (18)
C6	0.021 (2)	0.017 (2)	0.019 (2)	-0.0050 (19)	0.012 (2)	-0.0017 (18)
C7	0.017 (2)	0.025 (2)	0.010 (2)	0.0001 (19)	0.0094 (18)	0.0006 (18)
C8	0.013 (2)	0.022 (2)	0.011 (2)	0.0007 (19)	0.0057 (18)	-0.0002 (18)
C9	0.013 (2)	0.0135 (19)	0.017 (2)	0.0002 (18)	0.0057 (19)	0.0039 (18)
C10	0.016 (2)	0.013 (2)	0.019 (2)	0.0025 (18)	0.0008 (19)	0.0029 (18)
C11	0.013 (2)	0.019 (2)	0.030 (3)	-0.0009 (19)	0.009 (2)	0.002 (2)
C12	0.029 (3)	0.019 (2)	0.029 (3)	0.003 (2)	0.017 (2)	0.002 (2)
C13	0.018 (2)	0.021 (2)	0.015 (2)	0.0021 (19)	0.0090 (19)	0.0025 (18)
C14	0.015 (2)	0.016 (2)	0.013 (2)	0.0060 (18)	0.0032 (18)	-0.0002 (18)
C15	0.014 (2)	0.026 (2)	0.011 (2)	0.004 (2)	0.0020 (18)	0.0010 (19)
C16	0.041 (3)	0.028 (2)	0.022 (3)	0.008 (2)	0.022 (2)	0.006 (2)
C17	0.016 (2)	0.0167 (19)	0.008 (2)	-0.0033 (18)	0.0042 (18)	-0.0037 (17)
C18	0.019 (2)	0.032 (2)	0.011 (2)	0.002 (2)	0.0042 (19)	-0.001 (2)
C19	0.017 (2)	0.029 (2)	0.023 (3)	-0.001 (2)	0.010 (2)	-0.008 (2)
C20	0.025 (2)	0.029 (2)	0.011 (2)	-0.007 (2)	0.0120 (19)	-0.0068 (19)
C21	0.018 (2)	0.026 (2)	0.024 (2)	0.004 (2)	0.009 (2)	0.006 (2)
C22	0.016 (2)	0.038 (3)	0.017 (2)	-0.006 (2)	0.006 (2)	-0.003 (2)
C23	0.031 (3)	0.036 (3)	0.024 (3)	-0.007 (2)	0.018 (2)	0.003 (2)
C24	0.017 (2)	0.017 (2)	0.017 (2)	-0.0038 (19)	-0.0003 (19)	0.0012 (19)
C25	0.015 (2)	0.018 (2)	0.016 (2)	0.0048 (18)	0.0077 (19)	0.0028 (18)
C26	0.018 (2)	0.024 (2)	0.023 (2)	-0.001 (2)	0.006 (2)	-0.004 (2)
C27	0.014 (2)	0.029 (2)	0.029 (3)	-0.002 (2)	0.007 (2)	-0.002 (2)
C28	0.019 (2)	0.023 (2)	0.022 (2)	0.002 (2)	0.008 (2)	0.007 (2)
C29	0.024 (3)	0.022 (2)	0.027 (3)	0.000 (2)	0.011 (2)	-0.001 (2)

## supplementary materials

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C30	0.019 (2)	0.017 (2)	0.033 (3)	-0.0006 (19)	0.013 (2)	0.002 (2)
C31	0.038 (3)	0.027 (2)	0.042 (3)	0.007 (2)	0.035 (3)	0.005 (2)
C32	0.020 (2)	0.029 (2)	0.019 (2)	-0.011 (2)	0.004 (2)	-0.004 (2)
C33	0.014 (2)	0.030 (2)	0.012 (2)	-0.001 (2)	0.0049 (19)	0.000 (2)
C34	0.014 (2)	0.017 (2)	0.024 (2)	-0.0007 (18)	0.0102 (19)	-0.0014 (19)
C35	0.025 (3)	0.037 (3)	0.020 (2)	0.000 (2)	0.008 (2)	-0.011 (2)
C36	0.034 (3)	0.033 (3)	0.019 (3)	-0.001 (2)	0.009 (2)	-0.002 (2)
C37	0.030 (3)	0.034 (2)	0.025 (3)	-0.002 (2)	0.015 (2)	0.005 (2)
C38	0.027 (2)	0.028 (2)	0.014 (2)	-0.009 (2)	0.006 (2)	-0.009 (2)
Cl1	0.062 (2)	0.0615 (18)	0.0748 (16)	-0.0259 (16)	0.0424 (15)	-0.0276 (14)
C39	0.034 (5)	0.037 (4)	0.050 (4)	0.012 (3)	-0.001 (3)	-0.027 (3)
Cl1'	0.054 (5)	0.069 (6)	0.040 (4)	-0.019 (4)	0.010 (3)	-0.005 (4)
C39'	0.040 (9)	0.053 (9)	0.047 (8)	0.001 (8)	0.017 (7)	-0.017 (8)
Cl2	0.0323 (7)	0.0441 (7)	0.0626 (9)	0.0007 (6)	0.0191 (7)	-0.0107 (7)

### *Geometric parameters (Å, °)*

S1—C20	1.760 (4)	C18—H18	0.9300
S1—C23	1.797 (4)	C19—C20	1.394 (5)
S2—C28	1.742 (4)	C19—H19	0.9300
S2—C31	1.799 (4)	C20—C21	1.405 (5)
O1—C4	1.233 (4)	C21—C22	1.394 (5)
O2—C15	1.220 (4)	C21—H21	0.9300
N1—C6	1.467 (4)	C22—H22	0.9300
N1—C7	1.467 (4)	C23—H23A	0.9600
N1—C32	1.474 (4)	C23—H23B	0.9600
N2—C15	1.378 (4)	C23—H23C	0.9600
N2—C14	1.409 (4)	C24—C25	1.429 (5)
N3—C16	1.459 (4)	C24—H24	0.9300
N3—C1	1.464 (4)	C25—C26	1.406 (5)
N3—C8	1.493 (4)	C25—C30	1.428 (5)
C1—C2	1.518 (5)	C26—C27	1.370 (5)
C1—H1A	0.9700	C26—H26	0.9300
C1—H1B	0.9700	C27—C28	1.399 (5)
C2—C17	1.495 (5)	C27—H27	0.9300
C2—C3	1.576 (5)	C28—C29	1.406 (5)
C2—H2	0.9800	C29—C30	1.370 (5)
C3—C4	1.528 (5)	C29—H29	0.9300
C3—C7	1.539 (5)	C30—H30	0.9300
C3—C8	1.588 (5)	C31—H31A	0.9600
C4—C5	1.482 (5)	C31—H31B	0.9600
C5—C24	1.373 (5)	C31—H31C	0.9600
C5—C6	1.511 (5)	C32—C33	1.503 (5)
C6—H6A	0.9700	C32—H32A	0.9700
C6—H6B	0.9700	C32—H32B	0.9700
C7—H7A	0.9700	C33—C34	1.384 (5)
C7—H7B	0.9700	C33—C38	1.402 (5)
C8—C9	1.504 (5)	C34—C35	1.395 (5)
C8—C15	1.585 (5)	C34—H34	0.9300



C9—C10	1.399 (5)	C35—C36	1.374 (5)
C9—C14	1.401 (5)	C35—H35	0.9300
C10—C11	1.361 (5)	C36—C37	1.377 (5)
C10—H10	0.9300	C36—H36	0.9300
C11—C12	1.406 (5)	C37—C38	1.380 (5)
C11—H11	0.9300	C37—H37	0.9300
C12—C13	1.400 (5)	C38—H38	0.9300
C12—H12	0.9300	C11—C39	1.783 (6)
C13—C14	1.363 (5)	C39—C12	1.767 (6)
C13—H13	0.9300	C39—H39A	0.9700
C16—H16A	0.9600	C39—H39B	0.9700
C16—H16B	0.9600	C11'—C39'	1.778 (10)
C16—H16C	0.9600	C39'—C12	1.770 (10)
C17—C22	1.392 (5)	C39'—H39C	0.9700
C17—C18	1.418 (5)	C39'—H39D	0.9700
C18—C19	1.360 (5)		
C20—S1—C23	104.20 (19)	C17—C18—H18	119.3
C28—S2—C31	103.65 (18)	C18—C19—C20	122.3 (4)
C6—N1—C7	108.5 (3)	C18—C19—H19	118.9
C6—N1—C32	110.6 (3)	C20—C19—H19	118.9
C7—N1—C32	110.7 (3)	C19—C20—C21	117.2 (4)
C15—N2—C14	110.8 (3)	C19—C20—S1	118.0 (3)
C16—N3—C1	114.2 (3)	C21—C20—S1	124.8 (3)
C16—N3—C8	115.9 (3)	C22—C21—C20	120.5 (4)
C1—N3—C8	105.3 (3)	C22—C21—H21	119.8
N3—C1—C2	102.3 (3)	C20—C21—H21	119.8
N3—C1—H1A	111.3	C17—C22—C21	122.0 (4)
C2—C1—H1A	111.3	C17—C22—H22	119.0
N3—C1—H1B	111.3	C21—C22—H22	119.0
C2—C1—H1B	111.3	S1—C23—H23A	109.5
H1A—C1—H1B	109.2	S1—C23—H23B	109.5
C17—C2—C1	116.8 (3)	H23A—C23—H23B	109.5
C17—C2—C3	115.9 (3)	S1—C23—H23C	109.5
C1—C2—C3	103.1 (3)	H23A—C23—H23C	109.5
C17—C2—H2	106.8	H23B—C23—H23C	109.5
C1—C2—H2	106.8	C5—C24—C25	131.1 (4)
C3—C2—H2	106.8	C5—C24—H24	114.4
C4—C3—C7	106.2 (3)	C25—C24—H24	114.4
C4—C3—C2	111.0 (3)	C26—C25—C30	115.5 (3)
C7—C3—C2	113.3 (3)	C26—C25—C24	119.6 (3)
C4—C3—C8	109.8 (3)	C30—C25—C24	124.9 (4)
C7—C3—C8	112.2 (3)	C27—C26—C25	123.2 (4)
C2—C3—C8	104.4 (3)	C27—C26—H26	118.4
O1—C4—C5	121.6 (3)	C25—C26—H26	118.4
O1—C4—C3	120.5 (3)	C26—C27—C28	121.1 (4)
C5—C4—C3	117.9 (3)	C26—C27—H27	119.5
C24—C5—C4	117.3 (4)	C28—C27—H27	119.5
C24—C5—C6	122.3 (3)	C27—C28—C29	116.6 (4)
C4—C5—C6	120.3 (3)	C27—C28—S2	125.6 (3)

## supplementary materials

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N1—C6—C5	111.7 (3)	C29—C28—S2	117.8 (3)
N1—C6—H6A	109.3	C30—C29—C28	122.7 (4)
C5—C6—H6A	109.3	C30—C29—H29	118.6
N1—C6—H6B	109.3	C28—C29—H29	118.6
C5—C6—H6B	109.3	C29—C30—C25	120.9 (4)
H6A—C6—H6B	107.9	C29—C30—H30	119.6
N1—C7—C3	108.1 (3)	C25—C30—H30	119.6
N1—C7—H7A	110.1	S2—C31—H31A	109.5
C3—C7—H7A	110.1	S2—C31—H31B	109.5
N1—C7—H7B	110.1	H31A—C31—H31B	109.5
C3—C7—H7B	110.1	S2—C31—H31C	109.5
H7A—C7—H7B	108.4	H31A—C31—H31C	109.5
N3—C8—C9	112.0 (3)	H31B—C31—H31C	109.5
N3—C8—C15	110.0 (3)	N1—C32—C33	114.1 (3)
C9—C8—C15	100.7 (3)	N1—C32—H32A	108.7
N3—C8—C3	102.3 (3)	C33—C32—H32A	108.7
C9—C8—C3	119.1 (3)	N1—C32—H32B	108.7
C15—C8—C3	112.8 (3)	C33—C32—H32B	108.7
C10—C9—C14	117.5 (3)	H32A—C32—H32B	107.6
C10—C9—C8	132.2 (3)	C34—C33—C38	117.9 (3)
C14—C9—C8	110.1 (3)	C34—C33—C32	119.0 (3)
C11—C10—C9	120.2 (4)	C38—C33—C32	122.9 (4)
C11—C10—H10	119.9	C33—C34—C35	120.6 (4)
C9—C10—H10	119.9	C33—C34—H34	119.7
C10—C11—C12	121.3 (4)	C35—C34—H34	119.7
C10—C11—H11	119.4	C36—C35—C34	120.3 (4)
C12—C11—H11	119.4	C36—C35—H35	119.8
C13—C12—C11	119.6 (4)	C34—C35—H35	119.8
C13—C12—H12	120.2	C35—C36—C37	119.9 (4)
C11—C12—H12	120.2	C35—C36—H36	120.0
C14—C13—C12	117.8 (4)	C37—C36—H36	120.0
C14—C13—H13	121.1	C36—C37—C38	119.9 (4)
C12—C13—H13	121.1	C36—C37—H37	120.1
C13—C14—C9	123.6 (4)	C38—C37—H37	120.1
C13—C14—N2	126.4 (3)	C37—C38—C33	121.3 (4)
C9—C14—N2	110.0 (3)	C37—C38—H38	119.4
O2—C15—N2	124.4 (4)	C33—C38—H38	119.4
O2—C15—C8	127.3 (3)	Cl2—C39—Cl1	112.7 (3)
N2—C15—C8	108.2 (3)	Cl2—C39—H39A	109.0
N3—C16—H16A	109.5	Cl1—C39—H39A	109.0
N3—C16—H16B	109.5	Cl2—C39—H39B	109.0
H16A—C16—H16B	109.5	Cl1—C39—H39B	109.0
N3—C16—H16C	109.5	H39A—C39—H39B	107.8
H16A—C16—H16C	109.5	Cl2—C39'—Cl1'	110.8 (8)
H16B—C16—H16C	109.5	Cl2—C39'—H39C	109.5
C22—C17—C18	116.5 (3)	Cl1'—C39'—H39C	109.5
C22—C17—C2	119.2 (3)	Cl2—C39'—H39D	109.5
C18—C17—C2	124.3 (4)	Cl1'—C39'—H39D	109.5
C19—C18—C17	121.5 (4)	H39C—C39'—H39D	108.1

C19—C18—H18	119.3	C39—C12—C39'	19.7 (8)
C16—N3—C1—C2	177.5 (3)	C10—C9—C14—N2	-177.0 (3)
C8—N3—C1—C2	49.3 (3)	C8—C9—C14—N2	-0.4 (4)
N3—C1—C2—C17	-166.9 (3)	C15—N2—C14—C13	178.1 (3)
N3—C1—C2—C3	-38.6 (3)	C15—N2—C14—C9	-2.8 (4)
C17—C2—C3—C4	-97.5 (4)	C14—N2—C15—O2	-179.9 (3)
C1—C2—C3—C4	133.6 (3)	C14—N2—C15—C8	4.6 (4)
C17—C2—C3—C7	21.8 (5)	N3—C8—C15—O2	-61.5 (5)
C1—C2—C3—C7	-107.1 (3)	C9—C8—C15—O2	-179.9 (4)
C17—C2—C3—C8	144.2 (3)	C3—C8—C15—O2	52.0 (5)
C1—C2—C3—C8	15.3 (4)	N3—C8—C15—N2	113.8 (3)
C7—C3—C4—O1	-148.3 (3)	C9—C8—C15—N2	-4.5 (4)
C2—C3—C4—O1	-24.8 (5)	C3—C8—C15—N2	-132.6 (3)
C8—C3—C4—O1	90.1 (4)	C1—C2—C17—C22	-158.7 (3)
C7—C3—C4—C5	34.9 (4)	C3—C2—C17—C22	79.4 (4)
C2—C3—C4—C5	158.4 (3)	C1—C2—C17—C18	18.3 (5)
C8—C3—C4—C5	-86.7 (4)	C3—C2—C17—C18	-103.5 (4)
O1—C4—C5—C24	-16.0 (5)	C22—C17—C18—C19	-1.8 (5)
C3—C4—C5—C24	160.8 (3)	C2—C17—C18—C19	-178.9 (3)
O1—C4—C5—C6	166.1 (3)	C17—C18—C19—C20	1.2 (6)
C3—C4—C5—C6	-17.1 (5)	C18—C19—C20—C21	0.0 (6)
C7—N1—C6—C5	-52.7 (4)	C18—C19—C20—S1	178.9 (3)
C32—N1—C6—C5	-174.3 (3)	C23—S1—C20—C19	-176.4 (3)
C24—C5—C6—N1	-153.3 (3)	C23—S1—C20—C21	2.4 (4)
C4—C5—C6—N1	24.5 (5)	C19—C20—C21—C22	-0.5 (6)
C6—N1—C7—C3	76.1 (3)	S1—C20—C21—C22	-179.3 (3)
C32—N1—C7—C3	-162.3 (3)	C18—C17—C22—C21	1.2 (5)
C4—C3—C7—N1	-64.0 (4)	C2—C17—C22—C21	178.5 (3)
C2—C3—C7—N1	173.9 (3)	C20—C21—C22—C17	-0.1 (6)
C8—C3—C7—N1	56.0 (4)	C4—C5—C24—C25	-179.2 (3)
C16—N3—C8—C9	65.9 (4)	C6—C5—C24—C25	-1.4 (6)
C1—N3—C8—C9	-166.9 (3)	C5—C24—C25—C26	154.6 (4)
C16—N3—C8—C15	-45.2 (4)	C5—C24—C25—C30	-26.8 (7)
C1—N3—C8—C15	82.0 (3)	C30—C25—C26—C27	0.7 (6)
C16—N3—C8—C3	-165.3 (3)	C24—C25—C26—C27	179.4 (4)
C1—N3—C8—C3	-38.1 (3)	C25—C26—C27—C28	-2.0 (6)
C4—C3—C8—N3	-106.3 (3)	C26—C27—C28—C29	2.5 (6)
C7—C3—C8—N3	135.8 (3)	C26—C27—C28—S2	-177.4 (3)
C2—C3—C8—N3	12.8 (3)	C31—S2—C28—C27	-3.5 (4)
C4—C3—C8—C9	17.9 (4)	C31—S2—C28—C29	176.5 (3)
C7—C3—C8—C9	-100.0 (4)	C27—C28—C29—C30	-1.9 (6)
C2—C3—C8—C9	136.9 (3)	S2—C28—C29—C30	178.1 (3)
C4—C3—C8—C15	135.6 (3)	C28—C29—C30—C25	0.6 (6)
C7—C3—C8—C15	17.7 (4)	C26—C25—C30—C29	0.0 (6)
C2—C3—C8—C15	-105.3 (3)	C24—C25—C30—C29	-178.6 (4)
N3—C8—C9—C10	61.9 (5)	C6—N1—C32—C33	-70.4 (4)
C15—C8—C9—C10	178.8 (4)	C7—N1—C32—C33	169.3 (3)
C3—C8—C9—C10	-57.4 (5)	N1—C32—C33—C34	157.8 (3)
N3—C8—C9—C14	-114.0 (3)	N1—C32—C33—C38	-26.7 (5)

## supplementary materials

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C15—C8—C9—C14	2.9 (4)	C38—C33—C34—C35	-2.0 (6)
C3—C8—C9—C14	126.7 (3)	C32—C33—C34—C35	173.7 (4)
C14—C9—C10—C11	-1.5 (5)	C33—C34—C35—C36	0.3 (6)
C8—C9—C10—C11	-177.2 (4)	C34—C35—C36—C37	1.4 (6)
C9—C10—C11—C12	-0.6 (6)	C35—C36—C37—C38	-1.3 (6)
C10—C11—C12—C13	2.3 (6)	C36—C37—C38—C33	-0.4 (6)
C11—C12—C13—C14	-1.7 (5)	C34—C33—C38—C37	2.1 (6)
C12—C13—C14—C9	-0.5 (6)	C32—C33—C38—C37	-173.4 (4)
C12—C13—C14—N2	178.4 (3)	C11—C39—C12—C39'	61 (2)
C10—C9—C14—C13	2.1 (5)	C11'—C39'—C12—C39	-58.0 (14)
C8—C9—C14—C13	178.7 (3)		

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

<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>
C1—H1A $\cdots$ O2	0.97	2.53	3.149 (5)	122
C7—H7B $\cdots$ O2	0.97	2.37	2.971 (5)	119
C10—H10 $\cdots$ O1	0.93	2.57	3.220 (5)	127
C39—H39A $\cdots$ N3	0.97	2.44	3.405 (10)	175
C39—H39B $\cdots$ O1	0.97	2.53	3.140 (7)	121

Fig. 1

