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## (2S)-Ethyl 2-[(S<sub>5</sub>)-benzylsulfinylamino]-3,3-dimethylbutanoate

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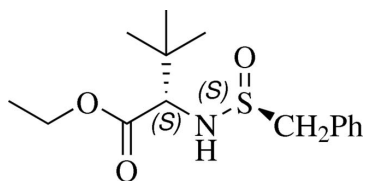
Received 30 July 2008; accepted 8 September 2008

Key indicators: single-crystal X-ray study;  $T = 293$  K; mean  $\sigma(\text{C}-\text{C}) = 0.006$  Å;  $R$  factor = 0.055;  $wR$  factor = 0.134; data-to-parameter ratio = 17.0.

The title compound,  $\text{C}_{15}\text{H}_{23}\text{NO}_3\text{S}$ , is an unexpected 1,3-migration product in the addition of benzylzinc bromide to *N*-*tert*-butanesulfinyl iminoacetate. In the crystal structure, molecules are linked by  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bonds and weak  $\text{C}-\text{H}\cdots\text{O}$  hydrogen bonds.

### Related literature

For general background, see: Ellman *et al.* (2002); Lin *et al.* (2008); Daniel & Stockman (2006). For the synthesis of the titled compound, see: Sun *et al.* (2008).



### Experimental

#### Crystal data

 $\text{C}_{15}\text{H}_{23}\text{NO}_3\text{S}$ 
 $M_r = 297.40$ 

 Monoclinic,  $P2_1$ 
 $a = 11.166$  (2) Å

 $b = 7.1917$  (14) Å

 $c = 11.460$  (2) Å

 $\beta = 115.473$  (3)°

 $V = 830.8$  (3) Å<sup>3</sup>
 $Z = 2$ 

 Mo  $K\alpha$  radiation

 $\mu = 0.20$  mm<sup>-1</sup>
 $T = 293$  (2) K

 $0.49 \times 0.41 \times 0.17$  mm

#### Data collection

Bruker SMART APEX CCD area-detector diffractometer

Absorption correction: multi-scan (SADABS; Bruker, 2001)

 $T_{\min} = 0.908$ ,  $T_{\max} = 0.967$ 

4782 measured reflections

3221 independent reflections

 2661 reflections with  $I > 2\sigma(I)$ 
 $R_{\text{int}} = 0.120$ 

#### Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.054$ 
 $wR(F^2) = 0.134$ 
 $S = 0.97$ 

3221 reflections

189 parameters

2 restraints

H atoms treated by a mixture of independent and constrained refinement

 $\Delta\rho_{\text{max}} = 0.43$  e Å<sup>-3</sup>
 $\Delta\rho_{\text{min}} = -0.23$  e Å<sup>-3</sup>

Absolute structure: Flack (1983), 1295 Friedel pairs

 Flack parameter:  $-0.09$  (11)

**Table 1**

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C}7-\text{H}7\text{B}\cdots\text{O}1$	0.96	2.61	3.234 (5)	123
$\text{C}9-\text{H}9\text{B}\cdots\text{O}3^i$	0.97	2.48	3.296 (5)	142
$\text{N}1-\text{H}1\text{A}\cdots\text{O}3^i$	0.859 (17)	2.13 (2)	2.932 (3)	156 (3)

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

Data collection: SMART (Bruker, 2001); cell refinement: SAINT (Bruker, 2001); 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: ZL2133).

### References

- Bruker (2001). SAINT, SMART and SADABS. Bruker AXS Inc., Madison, Wisconsin, USA.
- Daniel, M. & Stockman, R. A. (2006). *Tetrahedron*, **62**, 8869–89085.
- Ellman, J. A., Owens, T. D. & Tang, T. P. (2002). *Acc. Chem. Res.* **35**, 984–995.
- Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.
- Lin, G.-Q., Xu, M.-H., Zhong, Y.-W. & Sun, X.-W. (2008). *Acc. Chem. Res.* **41**, 831–840.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Sun, X., Zheng, W. & Wei, B.-G. (2008). *Tetrahedron Lett.* **49**, 6195–6197.

**supplementary materials**

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## (2*S*)-Ethyl 2-[(*S*<sub>S</sub>)-benzylsulfinylamino]-3,3-dimethylbutanoate

W. Zheng, X. Sun, J. Sun and B.-G. Wei

### Comment

*N*-*tert*-Butanesulfinylamide has received considerable attention in the auxiliary-aided asymmetric synthesis of a broad range of chiral amines (Ellman *et al.*, 2002; Stockman *et al.*, 2006; Lin *et al.*, 2008). In our research on the asymmetric addition of organozinc reagents to chiral *N*-*tert*-butanesulfinyl iminoacetates, an unexpected rearrangement product was obtained instead of the desired nucleophilic addition product. The structure of the compound obtained by 1,3-migration of the *tert*-butyl group was determined to be (2*S*)-ethyl 3,3-dimethyl-2-[(*S*<sub>S</sub>)-benzylsulfinylamino]butanoate. The reaction sequence (Sun *et al.*, 2008) is briefly shown in Fig. 4. The absolute configuration at the sulfur atom (as determined by the Flack parameter) is *S* as in the starting material. The new chiral center at C1 also exhibits an *S*-configuration. We believe this unusual rearrangement reaction could be developed to be a novel and convenient approach to prepare *tert*-leucine.

The crystal packing in the title compound is stabilized by an intramolecular hydrogen interaction (C7—H7B···O1) and by two intermolecular hydrogen bonds (N1—H1A···O3<sup>1</sup> and C9—H9B···O3<sup>1</sup>, symmetry operator: (i) -x, y-1/2, -z+1) which lead to the formation of an one-dimensional hydrogen bonded chain along the b axis as shown in Fig. 3.

### Experimental

To a solution of ethyl *N*-(*tert*-butanesulfinyl)iminoacetate (1 mmol) and Ni(acac)<sub>2</sub> (10 mol%) in anhydrous THF (10 ml) was added freshly prepared benzylzinc bromide (2.5 ml, 1 *M* in THF) at 195 K under an argon atmosphere. Then the mixture was allowed to warm to room temperature. After stirring for another 6 h, the reaction was quenched with saturated aqueous NH<sub>4</sub>Cl (4 ml). The mixture was extracted with EtOAc (10 ml) twice. The combined organic phases were washed with brine and dried with anhydrous Na<sub>2</sub>SO<sub>4</sub>. After concentrating under reduced pressure, the residue was purified by silica gel chromatography to give the title compound (yield: 47%). Suitable crystals were obtained by recrystallization from acetone (m.p. 421–423 K). [α]<sub>D</sub><sup>25</sup> 132.2 (c = 0.60, CHCl<sub>3</sub>). <sup>1</sup>H NMR (δ, CDCl<sub>3</sub>) 7.31–7.42 (*m*, 5H), 4.33 (*d*, *J* = 9.0, 1H), 4.12–4.20 (*m*, 2H), 4.03 (*s*, 2H), 3.48 (*d*, *J* = 9.0, 1H), 1.24 (*t*, *J* = 7.0, 3H), 0.83 (*s*, 9H). HRMS for (C<sub>15</sub>H<sub>23</sub>NO<sub>3</sub>S) found 289.1469, Calcd 289.1477.

### Refinement

Hydrogen atoms bonded to carbon were generated geometrically (C—H = 0.93, 0.98, 0.97 or 0.96 Å for phenyl, tertiary, methylene or methyl H atoms respectively) and refined in the riding model approximation. The hydrogen atom bound to the N atom was located from a difference density Fourier map, was refined isotropically and the N—H distance was restrained to 0.86 (2) Å. The displacement parameters of methyl H atoms were set to 1.5 times *U*<sub>eq</sub> of the equivalent isotropic displacement parameters of their parent atoms, while those of other H atoms bound to C were set to 1.2 times *U*<sub>eq</sub>.

## Figures

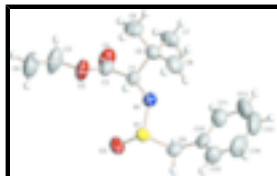


Fig. 1. Plot of  $C_{15}H_{23}NO_3S$  with 50% probability levels. H atoms are drawn as spheres of arbitrary radius.

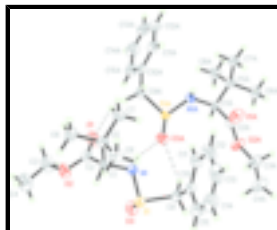


Fig. 2. Hydrogen bonding in the title compound. Symmetry code: (i)  $-x, y-1/2, -z+1$ .

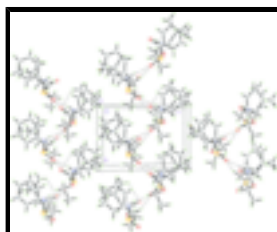


Fig. 3. Molecular packing plot showing the one-dimensional polymeric chains of the title compound. Hydrogen bond interactions are shown as dashed lines.



Fig. 4. Reaction sequence.

## (2S)-Ethyl 2-[(S)-benzylsulfinylamino]-3,3-dimethylbutanoate

### Crystal data

$C_{15}H_{23}NO_3S$

$M_r = 297.40$

Monoclinic,  $P2_1$

Hall symbol: P 2yb

$a = 11.166$  (2) Å

$b = 7.1917$  (14) Å

$c = 11.460$  (2) Å

$\beta = 115.473$  (3)°

$V = 830.8$  (3) Å<sup>3</sup>

$Z = 2$

$F_{000} = 320$

$D_x = 1.189$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation

$\lambda = 0.71073$  Å

Cell parameters from 1845 reflections

$\theta = 3.4$ – $23.9$ °

$\mu = 0.20$  mm<sup>-1</sup>

$T = 293$  (2) K

Prismatic, colorless

$0.49 \times 0.41 \times 0.17$  mm

### Data collection

Bruker SMART APEX CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 293$ (2) K

3221 independent reflections

2661 reflections with  $I > 2\sigma(I)$

$R_{int} = 0.120$

$\theta_{max} = 27.0$ °

$\varphi$ and $\omega$ scans	$\theta_{\min} = 2.0^\circ$
Absorption correction: multi-scan (SADABS; Bruker, 2001)	$h = -11 \rightarrow 14$
$T_{\min} = 0.908$ , $T_{\max} = 0.967$	$k = -8 \rightarrow 9$
4782 measured reflections	$l = -14 \rightarrow 13$

### Refinement

Refinement on $F^2$	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.055$	$w = 1/[\sigma^2(F_o^2) + (0.0662P)^2]$
$wR(F^2) = 0.134$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.97$	$(\Delta/\sigma)_{\max} = 0.001$
3221 reflections	$\Delta\rho_{\max} = 0.43 \text{ e } \text{\AA}^{-3}$
189 parameters	$\Delta\rho_{\min} = -0.23 \text{ e } \text{\AA}^{-3}$
2 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983), 1295 Friedel pairs
Secondary atom site location: difference Fourier map	Flack parameter: $-0.09$ (11)

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

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.04167 (7)	0.26178 (12)	0.67424 (6)	0.0442 (2)
O1	0.3061 (3)	0.0339 (4)	0.5596 (3)	0.0741 (8)
O2	0.4124 (3)	0.2630 (5)	0.6946 (2)	0.0696 (7)
O3	0.0313 (3)	0.3869 (4)	0.5670 (2)	0.0665 (7)
N1	0.1252 (2)	0.0717 (4)	0.6804 (2)	0.0420 (6)
C1	0.2690 (3)	0.0814 (4)	0.7500 (3)	0.0397 (6)
H1	0.2901	0.1873	0.8094	0.048*
C2	0.3305 (3)	0.1206 (5)	0.6568 (3)	0.0501 (8)
C3	0.4801 (5)	0.3093 (8)	0.6131 (5)	0.0923 (17)
H3A	0.5348	0.2058	0.6108	0.111*

## supplementary materials

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H3B	0.4155	0.3353	0.5255	0.111*
C4	0.5613 (6)	0.4705 (9)	0.6677 (5)	0.108 (2)
H4A	0.5056	0.5775	0.6547	0.162*
H4B	0.6189	0.4900	0.6263	0.162*
H4C	0.6136	0.4516	0.7586	0.162*
C5	0.3281 (3)	-0.0954 (5)	0.8332 (3)	0.0478 (7)
C6	0.4791 (4)	-0.0865 (6)	0.8887 (4)	0.0681 (10)
H6A	0.5167	-0.1912	0.9445	0.102*
H6B	0.5099	0.0264	0.9371	0.102*
H6C	0.5057	-0.0892	0.8194	0.102*
C7	0.2792 (4)	-0.2720 (5)	0.7539 (4)	0.0649 (10)
H7A	0.3185	-0.3781	0.8076	0.097*
H7B	0.3039	-0.2702	0.6833	0.097*
H7C	0.1844	-0.2791	0.7206	0.097*
C8	0.2833 (4)	-0.0948 (6)	0.9414 (3)	0.0653 (10)
H8A	0.1882	-0.0909	0.9048	0.098*
H8B	0.3191	0.0124	0.9952	0.098*
H8C	0.3145	-0.2055	0.9924	0.098*
C9	-0.1205 (3)	0.1504 (5)	0.6172 (3)	0.0513 (8)
H9A	-0.1880	0.2455	0.5975	0.062*
H9B	-0.1384	0.0834	0.5380	0.062*
C10	-0.1293 (3)	0.0198 (5)	0.7130 (3)	0.0494 (8)
C11	-0.1140 (4)	-0.1679 (6)	0.7036 (4)	0.0664 (10)
H11	-0.1014	-0.2145	0.6340	0.080*
C12	-0.1169 (5)	-0.2890 (6)	0.7960 (5)	0.0895 (15)
H12	-0.1062	-0.4161	0.7885	0.107*
C13	-0.1357 (5)	-0.2210 (10)	0.8991 (5)	0.0940 (15)
H13	-0.1374	-0.3023	0.9615	0.113*
C14	-0.1515 (5)	-0.0390 (8)	0.9098 (5)	0.0903 (16)
H14	-0.1651	0.0056	0.9794	0.108*
C15	-0.1479 (4)	0.0835 (6)	0.8193 (4)	0.0702 (11)
H15	-0.1579	0.2101	0.8289	0.084*
H1A	0.102 (3)	0.001 (3)	0.614 (2)	0.049 (7)*

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
S1	0.0470 (4)	0.0432 (4)	0.0420 (3)	0.0052 (4)	0.0189 (3)	0.0057 (4)
O1	0.0850 (19)	0.095 (2)	0.0564 (14)	-0.0256 (17)	0.0441 (13)	-0.0240 (15)
O2	0.0750 (16)	0.0817 (16)	0.0677 (13)	-0.0294 (18)	0.0455 (12)	-0.0137 (18)
O3	0.0640 (16)	0.0639 (16)	0.0641 (15)	0.0019 (13)	0.0206 (12)	0.0255 (13)
N1	0.0404 (14)	0.0454 (15)	0.0379 (12)	-0.0008 (11)	0.0146 (11)	-0.0041 (11)
C1	0.0383 (15)	0.0425 (17)	0.0387 (14)	-0.0046 (13)	0.0168 (12)	-0.0055 (13)
C2	0.0424 (17)	0.060 (2)	0.0489 (18)	-0.0037 (16)	0.0205 (14)	-0.0013 (16)
C3	0.098 (4)	0.119 (5)	0.086 (3)	-0.040 (3)	0.065 (3)	-0.019 (3)
C4	0.105 (4)	0.132 (5)	0.112 (4)	-0.035 (4)	0.070 (3)	0.001 (3)
C5	0.0499 (18)	0.0452 (17)	0.0415 (16)	0.0018 (15)	0.0132 (13)	-0.0001 (14)
C6	0.048 (2)	0.073 (2)	0.064 (2)	0.0088 (19)	0.0065 (17)	0.002 (2)

C7	0.067 (2)	0.048 (3)	0.067 (2)	0.0015 (18)	0.0159 (17)	-0.0079 (17)
C8	0.080 (3)	0.067 (2)	0.0490 (19)	0.004 (2)	0.0276 (18)	0.0121 (18)
C9	0.0421 (17)	0.065 (2)	0.0451 (16)	0.0087 (16)	0.0174 (14)	0.0066 (15)
C10	0.0369 (17)	0.059 (2)	0.0522 (18)	-0.0030 (14)	0.0190 (14)	0.0005 (15)
C11	0.062 (2)	0.070 (3)	0.064 (2)	-0.0145 (19)	0.0243 (18)	-0.0035 (18)
C12	0.092 (4)	0.058 (3)	0.113 (4)	-0.018 (2)	0.038 (3)	0.011 (2)
C13	0.086 (3)	0.108 (4)	0.094 (3)	-0.021 (4)	0.044 (2)	0.033 (4)
C14	0.095 (4)	0.121 (5)	0.077 (3)	-0.006 (3)	0.057 (3)	0.012 (3)
C15	0.074 (3)	0.075 (3)	0.074 (2)	-0.001 (2)	0.043 (2)	-0.002 (2)

*Geometric parameters (Å, °)*

S1—O3	1.487 (2)	C6—H6C	0.9600
S1—N1	1.639 (3)	C7—H7A	0.9600
S1—C9	1.824 (4)	C7—H7B	0.9600
O1—C2	1.201 (4)	C7—H7C	0.9600
O2—C2	1.316 (4)	C8—H8A	0.9600
O2—C3	1.471 (4)	C8—H8B	0.9600
N1—C1	1.455 (4)	C8—H8C	0.9600
N1—H1A	0.859 (17)	C9—C10	1.480 (5)
C1—C2	1.523 (4)	C9—H9A	0.9700
C1—C5	1.555 (4)	C9—H9B	0.9700
C1—H1	0.9800	C10—C11	1.371 (5)
C3—C4	1.439 (7)	C10—C15	1.398 (5)
C3—H3A	0.9700	C11—C12	1.382 (6)
C3—H3B	0.9700	C11—H11	0.9300
C4—H4A	0.9600	C12—C13	1.375 (7)
C4—H4B	0.9600	C12—H12	0.9300
C4—H4C	0.9600	C13—C14	1.334 (8)
C5—C7	1.520 (5)	C13—H13	0.9300
C5—C8	1.524 (5)	C14—C15	1.374 (6)
C5—C6	1.526 (5)	C14—H14	0.9300
C6—H6A	0.9600	C15—H15	0.9300
C6—H6B	0.9600		
O3—S1—N1	112.37 (15)	H6A—C6—H6C	109.5
O3—S1—C9	104.90 (15)	H6B—C6—H6C	109.5
N1—S1—C9	96.19 (15)	C5—C7—H7A	109.5
C2—O2—C3	116.2 (3)	C5—C7—H7B	109.5
C1—N1—S1	117.2 (2)	H7A—C7—H7B	109.5
C1—N1—H1A	111.1 (19)	C5—C7—H7C	109.5
S1—N1—H1A	120.2 (19)	H7A—C7—H7C	109.5
N1—C1—C2	110.4 (2)	H7B—C7—H7C	109.5
N1—C1—C5	111.9 (2)	C5—C8—H8A	109.5
C2—C1—C5	112.4 (3)	C5—C8—H8B	109.5
N1—C1—H1	107.3	H8A—C8—H8B	109.5
C2—C1—H1	107.3	C5—C8—H8C	109.5
C5—C1—H1	107.3	H8A—C8—H8C	109.5
O1—C2—O2	123.9 (3)	H8B—C8—H8C	109.5
O1—C2—C1	124.3 (3)	C10—C9—S1	112.7 (2)

## supplementary materials

O2—C2—C1	111.8 (3)	C10—C9—H9A	109.1
C4—C3—O2	107.8 (4)	S1—C9—H9A	109.1
C4—C3—H3A	110.1	C10—C9—H9B	109.1
O2—C3—H3A	110.1	S1—C9—H9B	109.1
C4—C3—H3B	110.1	H9A—C9—H9B	107.8
O2—C3—H3B	110.1	C11—C10—C15	117.5 (4)
H3A—C3—H3B	108.5	C11—C10—C9	121.1 (3)
C3—C4—H4A	109.5	C15—C10—C9	121.4 (3)
C3—C4—H4B	109.5	C10—C11—C12	121.0 (4)
H4A—C4—H4B	109.5	C10—C11—H11	119.5
C3—C4—H4C	109.5	C12—C11—H11	119.5
H4A—C4—H4C	109.5	C13—C12—C11	119.8 (4)
H4B—C4—H4C	109.5	C13—C12—H12	120.1
C7—C5—C8	109.2 (3)	C11—C12—H12	120.1
C7—C5—C6	109.4 (3)	C14—C13—C12	120.2 (4)
C8—C5—C6	110.6 (3)	C14—C13—H13	119.9
C7—C5—C1	111.6 (2)	C12—C13—H13	119.9
C8—C5—C1	107.2 (3)	C13—C14—C15	120.8 (5)
C6—C5—C1	108.8 (3)	C13—C14—H14	119.6
C5—C6—H6A	109.5	C15—C14—H14	119.6
C5—C6—H6B	109.5	C14—C15—C10	120.8 (4)
H6A—C6—H6B	109.5	C14—C15—H15	119.6
C5—C6—H6C	109.5	C10—C15—H15	119.6
O3—S1—N1—C1	84.8 (2)	N1—C1—C5—C6	172.6 (3)
C9—S1—N1—C1	-166.3 (2)	C2—C1—C5—C6	47.8 (3)
S1—N1—C1—C2	-95.2 (3)	O3—S1—C9—C10	-179.6 (3)
S1—N1—C1—C5	138.8 (2)	N1—S1—C9—C10	65.2 (3)
C3—O2—C2—O1	-2.6 (6)	S1—C9—C10—C11	-99.0 (4)
C3—O2—C2—C1	178.3 (4)	S1—C9—C10—C15	78.2 (4)
N1—C1—C2—O1	-51.2 (4)	C15—C10—C11—C12	0.1 (6)
C5—C1—C2—O1	74.6 (4)	C9—C10—C11—C12	177.4 (4)
N1—C1—C2—O2	128.0 (3)	C10—C11—C12—C13	0.1 (7)
C5—C1—C2—O2	-106.3 (3)	C11—C12—C13—C14	0.2 (8)
C2—O2—C3—C4	178.3 (4)	C12—C13—C14—C15	-0.7 (9)
N1—C1—C5—C7	51.8 (3)	C13—C14—C15—C10	0.9 (7)
C2—C1—C5—C7	-73.1 (4)	C11—C10—C15—C14	-0.5 (6)
N1—C1—C5—C8	-67.8 (3)	C9—C10—C15—C14	-177.8 (4)
C2—C1—C5—C8	167.4 (3)		

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C7—H7B $\cdots$ O1	0.96	2.61	3.234 (5)	123
C9—H9B $\cdots$ O3 <sup>i</sup>	0.97	2.48	3.296 (5)	142
N1—H1A $\cdots$ O3 <sup>i</sup>	0.859 (17)	2.13 (2)	2.932 (3)	156 (3)

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

Fig. 1

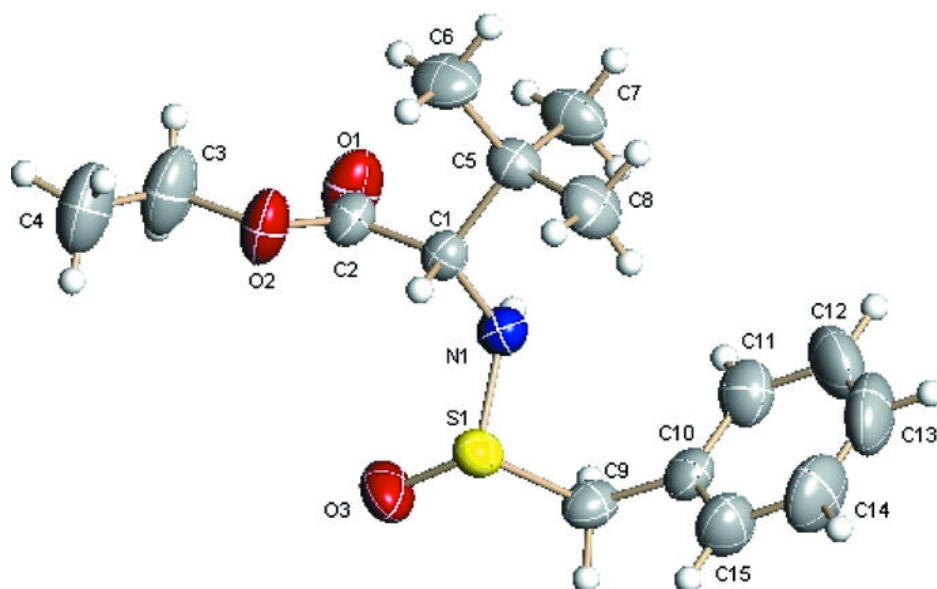


Fig. 2

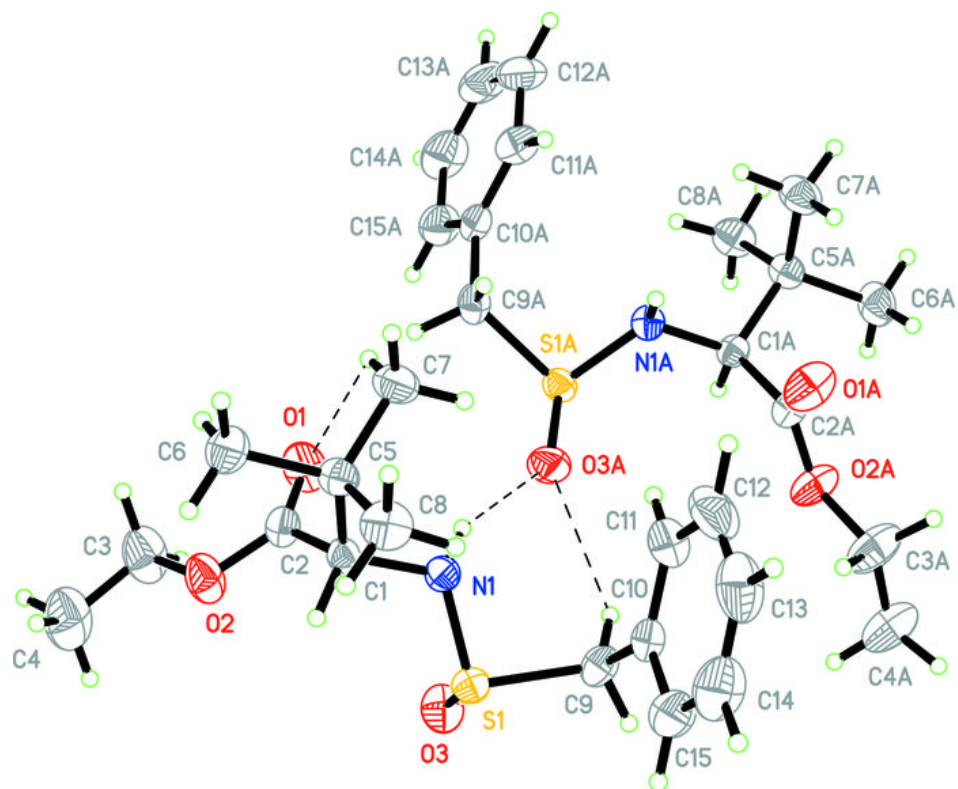


Fig. 3

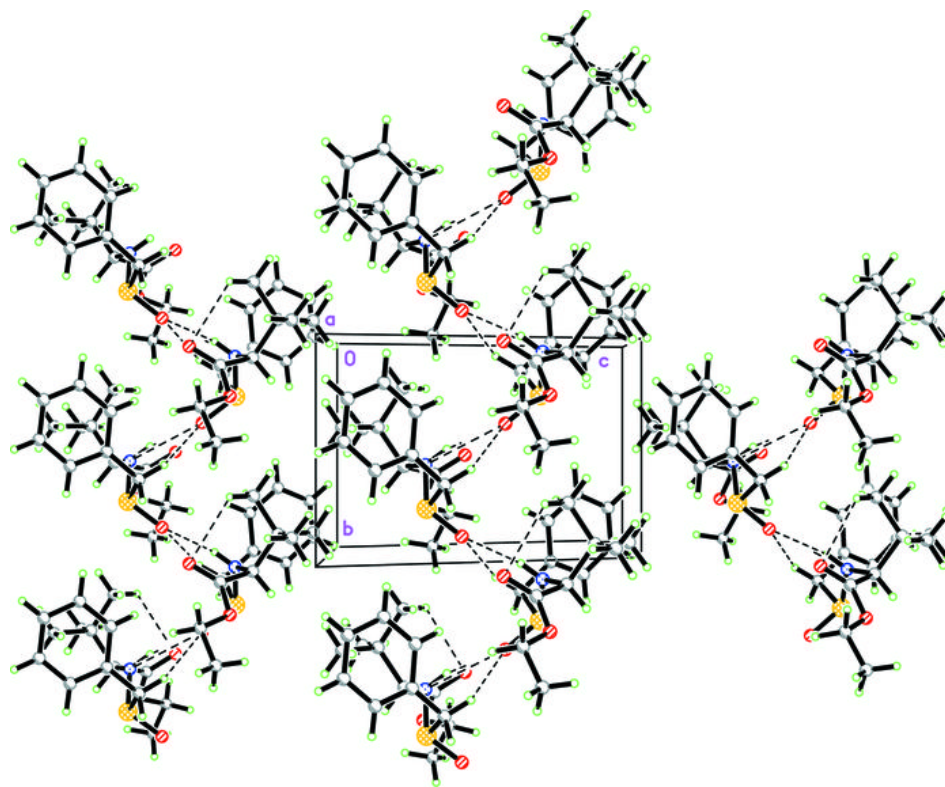


Fig. 4

