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## Structure Reports

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## N-Phenyladamantane-1-sulfinamide

Mrityunjay Datta,<sup>a</sup> Alan J Buglass,<sup>a\*</sup> Chang Seop Hong<sup>b</sup> and Jeon Hak Lim<sup>b</sup><sup>a</sup>Department of Chemistry, Korea Advanced Institute of Science and Technology, Daejeon 305-701, Republic of Korea, and <sup>b</sup>Department of Chemistry, Korea University, Seoul 136-701, Republic of Korea

Correspondence e-mail: ajobuglass@kaist.ac.kr

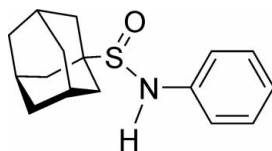
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Key indicators: single-crystal X-ray study;  $T = 293$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å;  $R$  factor = 0.041;  $wR$  factor = 0.126; data-to-parameter ratio = 20.7.

In the racemic title compound,  $\text{C}_{16}\text{H}_{21}\text{NOS}$ , the molecules are packed into polymeric chains in the  $b$ -axis direction and are linked along the  $b$  axis by  $\text{N}-\text{H}\cdots\text{O}$  and  $\text{C}-\text{H}\cdots\text{O}$  hydrogen bonds.

## Related literature

For literature on  $N$ -alkylalkanesulfinamides, see: Sato *et al.* (1975), Schuckmann *et al.* (1978); Ferreira *et al.* (2005). For related literature on cyclic  $N$ -arylalkanesulfinamides (sultims), see: Schulze *et al.* (2005). For the synthesis, see: Stretter *et al.* (1969). For related literature, see: Han *et al.* (2002); Weix & Ellman (2003).



## Experimental

## Crystal data

 $\text{C}_{16}\text{H}_{21}\text{NOS}$  $M_r = 275.40$ Monoclinic,  $P2_1/c$  $a = 11.6614$  (2) Å $b = 14.5582$  (3) Å $c = 9.0632$  (2) Å $\beta = 109.7770$  (10)° $V = 1447.90$  (5) Å<sup>3</sup> $Z = 4$ Mo  $K\alpha$  radiation $\mu = 0.22$  mm<sup>-1</sup> $T = 293$  (2) K $0.12 \times 0.08 \times 0.06$  mm

## Data collection

Bruker APEXII diffractometer  
Absorption correction: multi-scan  
(*SADABS*; Sheldrick, 1996)  
 $T_{\min} = 0.975$ ,  $T_{\max} = 0.987$ 14147 measured reflections  
3563 independent reflections  
2623 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.029$ 

## Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.041$  $wR(F^2) = 0.126$  $S = 1.07$ 

3563 reflections

172 parameters

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 0.28$  e Å<sup>-3</sup> $\Delta\rho_{\text{min}} = -0.30$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1}\cdots\text{O1}^{\dagger}$	0.86	2.17	2.988 (2)	160
$\text{C10}-\text{H10A}\cdots\text{O1}^{\dagger}$	0.97	2.35	3.305 (2)	168

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

Data collection: *APEX2* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *DIAMOND* (Brandenburg, 1998); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LX2059).

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**supplementary materials**

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## ***N*-Phenyladamantane-1-sulfinamide**

**M. Datta, A. J. Buglass, C. S. Hong and J. H. Lim**

### **Comment**

The title compound (I) was prepared from aniline and 1-adamantanesulfinyl chloride, which was itself prepared from adamantane and thionyl chloride in the presence of anhydrous AlCl<sub>3</sub> (Stretter *et al.*, 1969).

The molecular structure of (I) (Fig. 1) resembles those of *N*-alkylsulfonamides, except that the *N*-(aryl)C bond (1.409 Å) is considerably shorter than typical *N*-(alkyl)C bonds in *N*-alkylsulfonamides (1.470–1.530 Å) (Sato *et al.*, 1975; Schuckmann *et al.*, 1978; Ferreira *et al.*, 2005). The short bond suggests significant delocalization of electrons over the nitrogen atom and the benzene ring. This can be interpreted as indicating considerable contributions to the overall structure of (I) from resonance structures such as those in Fig. 2. The molecules of (I) (with alternating (*R*) and (*S*) configurations) are packed in a chain along the *b* axis (Fig. 3). The crystal packing (Fig. 3) is stabilized by intermolecular N—H···O and C—H···O hydrogen bonds (Fig. 3 and Table 1; symmetry code as in Fig. 3). Interest in sulfonamides lies mainly in their performance as chiral building blocks in organic synthesis (Han *et al.*, 2002; Weix and Ellman, 2003).

### **Experimental**

Compound (I) was prepared by the method of Stretter *et al.* (1969), using aniline (424 mg, 4.56 mmol), 1-adamantanesulfinyl chloride (500 mg, 2.28 mmol) and anhydrous diethyl ether (30 ml). Column chromatography (silica gel, ethyl acetate-dichloromethane, 1:9) gave (I) as white crystals (610 mg, 97%) mp 427–428 K. Literature mp was 428 K (Stretter *et al.*, 1969). Single crystals suitable for X-ray analysis were obtained by evaporation of a solution of the title compound (I) in dichloromethane at room temperature. Spectroscopic analysis: FTIR (KBr) (cm<sup>-1</sup>) 3179, 2908, 2851, 1595, 1488, 1450, 1285, 1228, 1175, 1063, 1034, 877. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>, p.p.m. with respect to TMS) 7.26–7.22 (m, 2H), 7.01–6.97 (m, 3H), 5.43 (bs, 1H), 2.18 (s, 3H), 1.92 (dd, *J* = 11.8, 22.8 Hz, 6H), 1.74 (dd, *J* = 12.2, 23.4 Hz, 6H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>, p.p.m. with respect to TMS) 142.4, 129.2, 122.4, 117.9, 58.2, 36.3, 34.6, 28.5. EIMS *m/z* (%) 276 (MH<sup>+</sup>, 39), 275 (*M*<sup>+</sup>, 85), 259 (*M*<sup>+</sup>-16, 16), 228 (18), 227 (*M*<sup>+</sup>-SO, 75), 136 (59), 135 (*M*<sup>+</sup>-PhNHSO, 100), 107 (28), 93 (MH<sup>+</sup>-adamantanesulfinyl, 66), 79 (61).

### **Refinement**

H atoms were located on a difference Fourier map geometrically and refined using a riding model with N—H = 0.86 Å, C—H = 0.93–0.98 Å and with *U*<sub>iso</sub>(H) = 1.2 times *U*<sub>eq</sub>(C, N).

## Figures

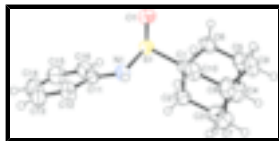


Fig. 1. Molecular structure of (I), with atom labels and 50% probability displacement ellipsoids for non-H atoms.



Fig. 2. Resonance structures for (I).

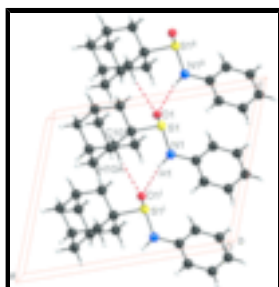


Fig. 3. The packing of (I), viewed down the *b* axis, showing hydrogen bonding [symmetry code: (i) *x*, *-y* + 1/2, *z* - 1/2; (ii) *x*, *-y* + 1/2, *z* + 1/2].

## *N*-Phenyladamantane-1-sulfinamide

### Crystal data

$C_{16}H_{21}NO$

$M_r = 275.40$

Monoclinic,  $P2_1/c$

Hall symbol:  $-P\ 2ybc$

$a = 11.6614\ (2)\ \text{\AA}$

$b = 14.5582\ (3)\ \text{\AA}$

$c = 9.0632\ (2)\ \text{\AA}$

$\beta = 109.7770\ (10)^\circ$

$V = 1447.90\ (5)\ \text{\AA}^3$

$Z = 4$

$F_{000} = 592$

$D_x = 1.263\ \text{Mg m}^{-3}$

Mo  $K\alpha$  radiation

$\lambda = 0.71073\ \text{\AA}$

Cell parameters from 4542 reflections

$\theta = 2.3\text{--}18.3^\circ$

$\mu = 0.22\ \text{mm}^{-1}$

$T = 293\ (2)\ \text{K}$

Block, white

$0.12 \times 0.08 \times 0.06\ \text{mm}$

### Data collection

Bruker APEXII  
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 293\ (2)\ \text{K}$

$\omega$  scans

Absorption correction: multi-scan  
(SADABS; Sheldrick, 1996)

$T_{\min} = 0.975$ ,  $T_{\max} = 0.987$

14147 measured reflections

3563 independent reflections

2623 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.030$

$\theta_{\text{max}} = 28.3^\circ$

$\theta_{\text{min}} = 2.3^\circ$

$h = -15 \rightarrow 15$

$k = -18 \rightarrow 19$

$l = -12 \rightarrow 12$

Refinement

Refinement on $F^2$	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.041$	H-atom parameters constrained
$wR(F^2) = 0.126$	$w = 1/[\sigma^2(F_o^2) + (0.0675P)^2 + 0.1762P]$
$S = 1.07$	where $P = (F_o^2 + 2F_c^2)/3$
3563 reflections	$(\Delta/\sigma)_{\max} = 0.001$
172 parameters	$\Delta\rho_{\max} = 0.28 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\min} = -0.30 \text{ e } \text{\AA}^{-3}$
	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  $F^2 > \sigma(F^2)$  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 ( $\text{\AA}^2$ )

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.58983 (13)	0.39058 (9)	0.82150 (16)	0.0364 (3)
C2	0.56242 (15)	0.48043 (11)	0.7296 (2)	0.0484 (4)
H2A	0.5187	0.5217	0.7757	0.058*
H2B	0.5117	0.4685	0.6219	0.058*
C3	0.68250 (16)	0.52473 (12)	0.7334 (2)	0.0580 (5)
H3	0.6652	0.5824	0.6741	0.070*
C4	0.76125 (18)	0.54460 (12)	0.9024 (3)	0.0638 (5)
H4A	0.8368	0.5736	0.9047	0.077*
H4B	0.7188	0.5863	0.9496	0.077*
C5	0.78874 (16)	0.45500 (12)	0.9943 (2)	0.0535 (4)
H5	0.8397	0.4677	1.1029	0.064*
C6	0.66905 (15)	0.41030 (12)	0.99233 (18)	0.0481 (4)
H6A	0.6261	0.4511	1.0403	0.058*
H6B	0.6861	0.3535	1.0517	0.058*
C7	0.74864 (17)	0.45997 (14)	0.6590 (2)	0.0619 (5)
H7A	0.8239	0.4881	0.6583	0.074*
H7B	0.6983	0.4476	0.5514	0.074*
C8	0.77682 (15)	0.37019 (13)	0.7515 (2)	0.0536 (4)

## supplementary materials

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H8	0.8203	0.3288	0.7034	0.064*
C9	0.85636 (16)	0.38968 (13)	0.9200 (2)	0.0579 (4)
H9A	0.9326	0.4173	0.9220	0.069*
H9B	0.8748	0.3327	0.9789	0.069*
C10	0.65764 (15)	0.32443 (11)	0.7496 (2)	0.0473 (4)
H10A	0.6077	0.3099	0.6427	0.057*
H10B	0.6754	0.2678	0.8094	0.057*
S1	0.44633 (4)	0.34441 (3)	0.82885 (4)	0.04623 (15)
O1	0.47547 (12)	0.25339 (9)	0.90826 (15)	0.0702 (4)
N1	0.37398 (12)	0.32958 (9)	0.63941 (15)	0.0463 (3)
H1	0.4153	0.3178	0.5790	0.056*
C11	0.24605 (13)	0.33580 (9)	0.57455 (18)	0.0390 (3)
C12	0.18648 (15)	0.29240 (11)	0.4345 (2)	0.0499 (4)
H12	0.2305	0.2573	0.3866	0.060*
C13	0.06152 (17)	0.30068 (14)	0.3647 (2)	0.0657 (5)
H13	0.0223	0.2726	0.2687	0.079*
C14	-0.00505 (17)	0.35041 (14)	0.4370 (3)	0.0686 (6)
H14	-0.0893	0.3546	0.3917	0.082*
C15	0.05395 (17)	0.39325 (14)	0.5752 (3)	0.0654 (5)
H15	0.0094	0.4275	0.6235	0.078*
C16	0.17819 (16)	0.38689 (13)	0.6449 (2)	0.0542 (4)
H16	0.2169	0.4169	0.7393	0.065*

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C1	0.0339 (7)	0.0456 (7)	0.0304 (7)	0.0010 (6)	0.0117 (6)	0.0018 (6)
C2	0.0400 (8)	0.0553 (9)	0.0519 (9)	0.0083 (7)	0.0180 (7)	0.0142 (7)
C3	0.0517 (10)	0.0570 (10)	0.0698 (12)	0.0027 (8)	0.0264 (9)	0.0205 (9)
C4	0.0553 (11)	0.0540 (10)	0.0831 (14)	-0.0106 (8)	0.0248 (10)	-0.0099 (9)
C5	0.0447 (9)	0.0632 (10)	0.0455 (9)	-0.0046 (7)	0.0060 (8)	-0.0122 (8)
C6	0.0499 (10)	0.0593 (9)	0.0335 (8)	-0.0017 (7)	0.0121 (7)	-0.0036 (7)
C7	0.0453 (10)	0.0912 (13)	0.0554 (10)	-0.0031 (9)	0.0252 (9)	0.0080 (9)
C8	0.0359 (9)	0.0692 (10)	0.0574 (10)	0.0059 (7)	0.0180 (8)	-0.0127 (8)
C9	0.0376 (9)	0.0674 (11)	0.0607 (11)	0.0053 (8)	0.0061 (8)	-0.0033 (9)
C10	0.0412 (9)	0.0542 (9)	0.0446 (8)	0.0038 (7)	0.0121 (7)	-0.0094 (7)
S1	0.0400 (2)	0.0676 (3)	0.0334 (2)	-0.00510 (17)	0.01537 (18)	0.00352 (16)
O1	0.0590 (8)	0.0870 (9)	0.0588 (8)	-0.0153 (7)	0.0123 (7)	0.0304 (7)
N1	0.0355 (7)	0.0683 (9)	0.0359 (7)	0.0016 (6)	0.0133 (6)	-0.0037 (6)
C11	0.0358 (8)	0.0410 (7)	0.0413 (8)	0.0004 (6)	0.0142 (7)	0.0068 (6)
C12	0.0423 (9)	0.0498 (9)	0.0552 (10)	-0.0023 (7)	0.0131 (8)	-0.0067 (7)
C13	0.0449 (10)	0.0674 (11)	0.0719 (13)	-0.0087 (9)	0.0031 (9)	-0.0068 (10)
C14	0.0361 (10)	0.0729 (12)	0.0899 (16)	0.0032 (8)	0.0122 (11)	0.0168 (11)
C15	0.0509 (11)	0.0761 (12)	0.0751 (14)	0.0205 (9)	0.0292 (10)	0.0158 (10)
C16	0.0498 (10)	0.0646 (10)	0.0500 (10)	0.0118 (8)	0.0192 (8)	0.0012 (8)

### Geometric parameters ( $\text{\AA}$ , $^\circ$ )

C1—C10	1.525 (2)	C8—C10	1.536 (2)
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C1—C2	1.526 (2)	C8—H8	0.980
C1—C6	1.538 (2)	C9—H9A	0.970
C1—S1	1.825 (2)	C9—H9B	0.970
C2—C3	1.531 (2)	C10—H10A	0.970
C2—H2A	0.970	C10—H10B	0.970
C2—H2B	0.970	S1—O1	1.491 (1)
C3—C7	1.513 (3)	S1—N1	1.651 (1)
C3—C4	1.523 (3)	N1—C11	1.409 (2)
C3—H3	0.980	N1—H1	0.860
C4—C5	1.522 (3)	C11—C12	1.377 (2)
C4—H4A	0.970	C11—C16	1.388 (2)
C4—H4B	0.970	C12—C13	1.384 (2)
C5—C9	1.530 (2)	C12—H12	0.930
C5—C6	1.535 (2)	C13—C14	1.378 (3)
C5—H5	0.980	C13—H13	0.930
C6—H6A	0.970	C14—C15	1.360 (3)
C6—H6B	0.970	C14—H14	0.930
C7—C8	1.527 (3)	C15—C16	1.374 (3)
C7—H7A	0.970	C15—H15	0.930
C7—H7B	0.970	C16—H16	0.930
C8—C9	1.521 (2)		
C10—C1—C2	110.6 (1)	C9—C8—C7	109.7 (2)
C10—C1—C6	109.0 (1)	C9—C8—C10	109.5 (1)
C2—C1—C6	109.5 (1)	C7—C8—C10	109.8 (2)
C10—C1—S1	113.4 (1)	C9—C8—H8	109.3
C2—C1—S1	108.1 (1)	C7—C8—H8	109.3
C6—C1—S1	106.1 (1)	C10—C8—H8	109.3
C1—C2—C3	109.1 (1)	C8—C9—C5	109.2 (1)
C1—C2—H2A	109.9	C8—C9—H9A	109.9
C3—C2—H2A	109.9	C5—C9—H9A	109.9
C1—C2—H2B	109.9	C8—C9—H9B	109.9
C3—C2—H2B	109.9	C5—C9—H9B	109.9
H2A—C2—H2B	108.3	H9A—C9—H9B	108.3
C7—C3—C4	110.0 (2)	C1—C10—C8	108.6 (1)
C7—C3—C2	109.1 (2)	C1—C10—H10A	110.0
C4—C3—C2	109.8 (1)	C8—C10—H10A	110.0
C7—C3—H3	109.3	C1—C10—H10B	110.0
C4—C3—H3	109.3	C8—C10—H10B	110.0
C2—C3—H3	109.3	H10A—C10—H10B	108.3
C5—C4—C3	109.4 (1)	O1—S1—N1	109.79 (8)
C5—C4—H4A	109.8	O1—S1—C1	106.43 (7)
C3—C4—H4A	109.8	N1—S1—C1	99.34 (6)
C5—C4—H4B	109.8	C11—N1—S1	121.5 (1)
C3—C4—H4B	109.8	C11—N1—H1	119.3
H4A—C4—H4B	108.3	S1—N1—H1	119.3
C4—C5—C9	109.6 (2)	C12—C11—C16	118.7 (2)
C4—C5—C6	109.6 (1)	C12—C11—N1	119.2 (1)
C9—C5—C6	109.5 (1)	C16—C11—N1	122.0 (1)
C4—C5—H5	109.4	C11—C12—C13	120.4 (2)

## supplementary materials

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C9—C5—H5	109.4	C11—C12—H12	119.8
C6—C5—H5	109.4	C13—C12—H12	119.8
C5—C6—C1	109.0 (1)	C14—C13—C12	120.3 (2)
C5—C6—H6A	109.9	C14—C13—H13	119.9
C1—C6—H6A	109.9	C12—C13—H13	119.9
C5—C6—H6B	109.9	C15—C14—C13	119.2 (2)
C1—C6—H6B	109.9	C15—C14—H14	120.4
H6A—C6—H6B	108.3	C13—C14—H14	120.4
C3—C7—C8	109.6 (1)	C14—C15—C16	121.3 (2)
C3—C7—H7A	109.7	C14—C15—H15	119.4
C8—C7—H7A	109.7	C16—C15—H15	119.4
C3—C7—H7B	109.7	C15—C16—C11	120.1 (2)
C8—C7—H7B	109.7	C15—C16—H16	120.0
H7A—C7—H7B	108.2	C11—C16—H16	120.0

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$N1-H1\cdots O1^i$	0.86	2.17	2.988 (2)	160
$C10-H10A\cdots O1^i$	0.97	2.35	3.305 (2)	168

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



Fig. 1

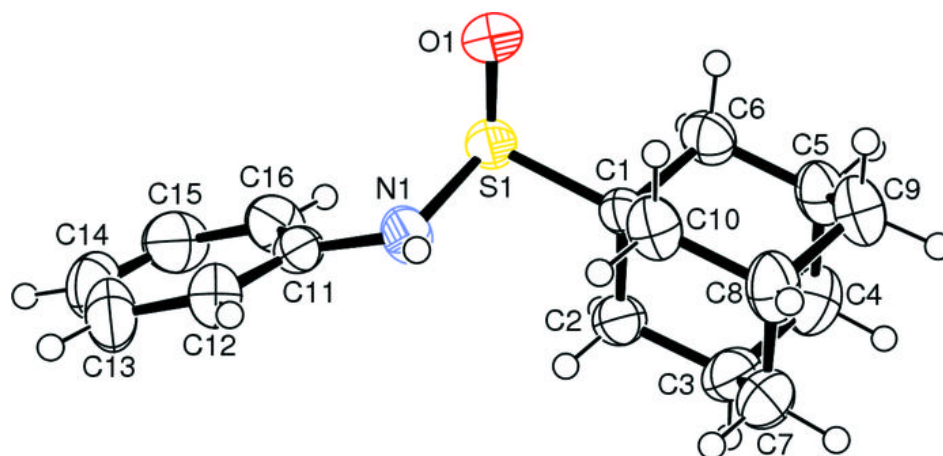


Fig. 2

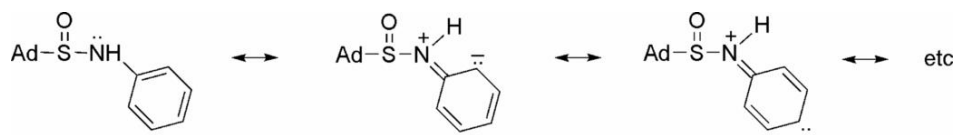


Fig. 3

