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4-Amino-12-methylsulfonyloxy-[2.2]-paracyclophane

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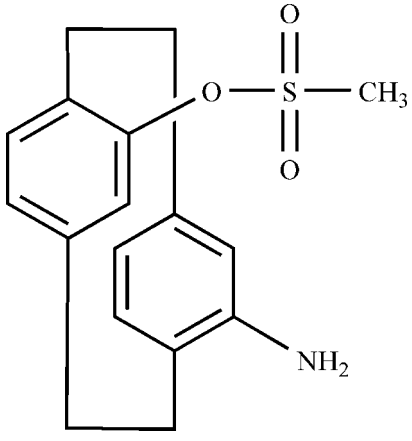
Key indicators: single-crystal X-ray study; T = 273 K; mean σ(C–C) = 0.004 Å; R factor = 0.038; wR factor = 0.084; data-to-parameter ratio = 13.4.

The title compound, $C_{17}H_{19}NO_3S$, was synthesized from 4-benzhydrylideneamino-12-hydroxy-[2.2]paracyclophane and methanesulfonyl chloride. In the molecule, the distance between the centroids of two aromatic rings is 2.960 (5) Å. In the crystal, weak N—H···O and C—H···O hydrogen bonds link the molecules into layers parallel to the *ac* plane.

Related literature

For background to [2.2]paracyclophane, see: Cram *et al.* (1959); Liebman & Greenberg (1976); Dyson *et al.* (1998). For its synthesis and applications in catalysis, see: Hou *et al.* (2000); Duan *et al.* (2008, 2012). For a related structure, see: Ma *et al.* (2012).



Experimental

Crystal data

$C_{17}H_{19}NO_3S$
M_r = 317.39
Orthorhombic, $P2_12_12_1$
a = 8.017 (7) Å
b = 11.734 (9) Å
c = 16.131 (13) Å
V = 1517 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.23 mm^{-1}
T = 273 K
0.13 × 0.12 × 0.10 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2007)
T_{min} = 0.971, T_{max} = 0.978
7769 measured reflections
2676 independent reflections
2266 reflections with $I > 2\sigma(I)$
R_{int} = 0.037

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.084
S = 1.04
2676 reflections
200 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}
Absolute structure: Flack (1983), 1122 Friedel pairs
Absolute structure parameter: 0.02 (9)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1B···O2^{i}	0.86	2.43	3.262 (3)	163
C10—H10B···O1^{ii}	0.97	2.52	3.390 (4)	149

Symmetry codes: (i) $-x+\frac{5}{2}, -y+1, z-\frac{1}{2}$; (ii) $x-1, y, z$.

Data collection: *SMART* (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: CV5436).

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supplementary materials

Acta Cryst. (2014). E**70**, o18 [doi:10.1107/S1600536813032595]

4-Amino-12-methylsulfonyloxy-[2.2]paracyclophane

Xiangchao Meng, Wenzeng Duan and Yinfeng Han

1. Comment

Since the first synthesis of [2.2]paracyclophane (Cram, 1959), its structure atrracted considerable interest (Liebman *et al.*, 1976; Dyson *et al.*, 1998). [2.2]Paracyclophane needs only one substituent to become planar chiral, so, there has been notable progress with regard to the synthesis of new derivatives and their applications in asymmetric catalysis(Hou *et al.*, 2000; Duan *et al.*, 2008).

In the title compound (Fig. 1), all bond lengths and angles are normal and in agreement with those observed in the related structure (Ma *et al.*, 2012). In the molecule, the distance between the centroids of two aromatic rings is 2.960 (5) Å. The crystal packing exhibits weak intermolecular N—H···O and C—H···O hydrogen bonds (Table 1), which link the molecules into layers parallel to the *ac* plane.

2. Experimental

The title compound was prepared by the method reported by Duan *et al.* (2012). The crystals were obtained by recrystallization from hexane and ethyl acetate.

3. Refinement

All the H atoms were located in difference maps, but placed in idealized positions (N—H 0.86 Å, C—H 0.93–0.97 Å), and refined as riding, with with U_{iso}(H) = 1.2–1.5 U_{eq} of the parent atom.

Computing details

Data collection: *SMART* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT* (Bruker, 2007); 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* (Sheldrick, 2008).

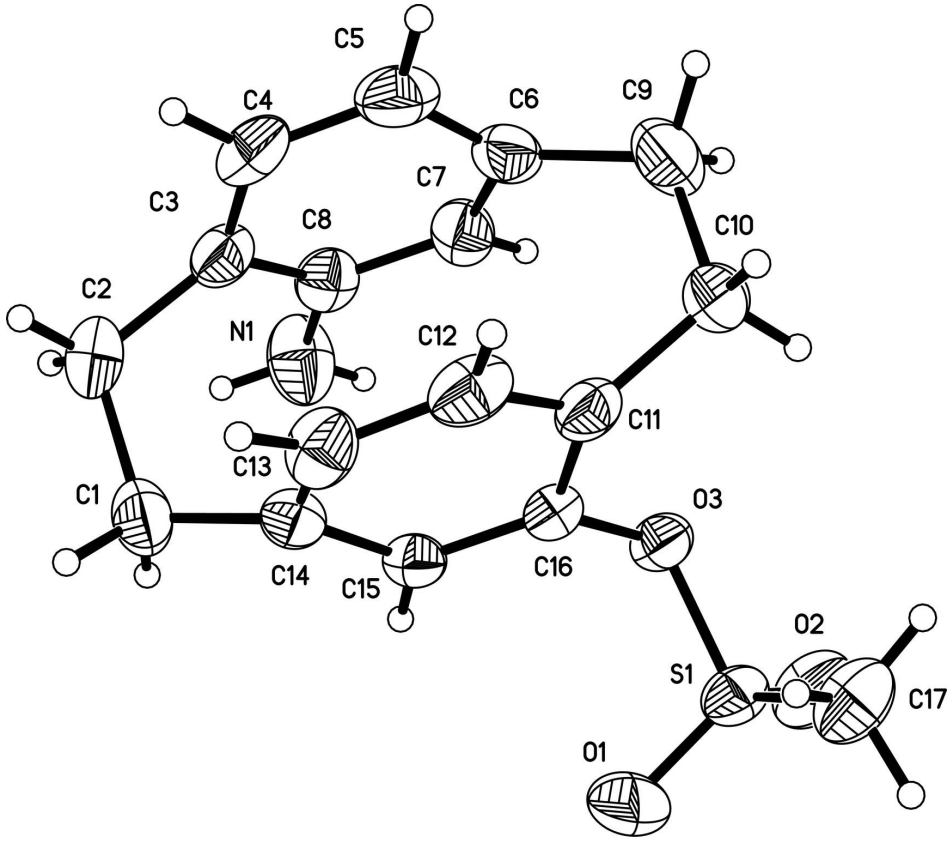

Figure 1

The molecular structure of the title compound showing the atom-numbering scheme and 50% probability displacement ellipsoids.

4-Amino-12-methylsulfonyloxy-[2.2]paracyclophane

Crystal data

$C_{17}H_{19}NO_3S$
$M_r = 317.39$
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a = 8.017$ (7) Å
$b = 11.734$ (9) Å
$c = 16.131$ (13) Å
$V = 1517$ (2) Å^3
$Z = 4$
$F(000) = 672$
$D_x = 1.389$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2210 reflections
$\theta = 2.8$–$23.1°$
$\mu = 0.23$ mm^{-1}
$T = 273$ K
Block, colourless
0.13 × 0.12 × 0.10 mm

Data collection

Bruker SMART CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2007)
$T_{min} = 0.971$, $T_{max} = 0.978$
7769 measured reflections
2676 independent reflections
2266 reflections with $I > 2\sigma(I)$
$R_{int} = 0.037$
$\theta_{max} = 25.0°$, $\theta_{min} = 2.2°$
$h = -9 \rightarrow 9$
$k = -13 \rightarrow 13$
$l = -11 \rightarrow 19$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.084$
$S = 1.04$
2676 reflections
200 parameters
0 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
$w = 1/[\sigma^2(F_o^2) + (0.0436P)^2 + 0.0218P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.15$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ e Å^{-3}
Absolute structure: Flack (1983), 1122 Friedel pairs
Absolute structure parameter: 0.02 (9)

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	1.0276 (4)	0.2690 (2)	0.10319 (15)	0.0464 (7)
H1C	1.0358	0.1905	0.0849	0.056*
H1D	1.1386	0.3019	0.1019	0.056*
C2	0.9100 (3)	0.3373 (2)	0.04181 (16)	0.0484 (7)
H2A	0.9781	0.3785	0.0022	0.058*
H2B	0.8411	0.2839	0.0113	0.058*
C3	0.7987 (3)	0.4208 (2)	0.08747 (14)	0.0386 (6)
C4	0.6334 (4)	0.3938 (2)	0.10371 (16)	0.0478 (7)
H4	0.5785	0.3439	0.0682	0.057*
C5	0.5469 (3)	0.4382 (2)	0.17062 (18)	0.0492 (7)
H5	0.4357	0.4188	0.1794	0.059*
C6	0.6281 (3)	0.5120 (2)	0.22456 (16)	0.0412 (6)
C7	0.7798 (3)	0.5569 (2)	0.19893 (15)	0.0402 (6)
H7	0.8257	0.6175	0.2283	0.048*
C8	0.8654 (3)	0.5139 (2)	0.13052 (15)	0.0376 (6)
C9	0.5778 (4)	0.5230 (3)	0.31434 (17)	0.0551 (8)
H9A	0.4571	0.5206	0.3180	0.066*
H9B	0.6140	0.5967	0.3348	0.066*
C10	0.6529 (3)	0.4269 (2)	0.37183 (16)	0.0491 (7)
H10A	0.7088	0.4625	0.4185	0.059*
H10B	0.5624	0.3808	0.3935	0.059*
C11	0.7741 (3)	0.3510 (2)	0.32766 (15)	0.0359 (6)
C12	0.7184 (3)	0.2616 (2)	0.27724 (15)	0.0425 (7)
H12	0.6159	0.2280	0.2886	0.051*

C13	0.8110 (3)	0.2222 (2)	0.21135 (16)	0.0432 (7)
H13	0.7716	0.1612	0.1802	0.052*
C14	0.9620 (3)	0.27203 (19)	0.19075 (14)	0.0358 (6)
C15	1.0336 (3)	0.34300 (19)	0.24992 (15)	0.0336 (6)
H15	1.1437	0.3666	0.2440	0.040*
C16	0.9421 (3)	0.37825 (19)	0.31712 (14)	0.0304 (6)
C17	1.0148 (4)	0.3455 (2)	0.51017 (17)	0.0548 (8)
H17A	0.9649	0.2812	0.4830	0.082*
H17B	0.9287	0.3953	0.5303	0.082*
H17C	1.0816	0.3196	0.5558	0.082*
N1	1.0226 (3)	0.5546 (2)	0.11279 (15)	0.0610 (7)
H1A	1.0668	0.6060	0.1438	0.073*
H1B	1.0764	0.5286	0.0707	0.073*
O1	1.2545 (2)	0.34091 (17)	0.40407 (12)	0.0602 (6)
O2	1.2004 (2)	0.52194 (15)	0.47560 (11)	0.0553 (5)
O3	1.0125 (2)	0.46195 (13)	0.37118 (10)	0.0355 (4)
S1	1.14007 (8)	0.41905 (5)	0.43999 (4)	0.03807 (18)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0554 (18)	0.0443 (15)	0.0395 (15)	0.0054 (14)	0.0040 (13)	−0.0094 (12)
C2	0.059 (2)	0.0540 (16)	0.0325 (15)	−0.0026 (14)	0.0012 (12)	−0.0085 (12)
C3	0.0400 (15)	0.0459 (14)	0.0298 (13)	−0.0008 (13)	−0.0051 (10)	0.0041 (11)
C4	0.0484 (17)	0.0558 (17)	0.0393 (15)	−0.0035 (15)	−0.0172 (14)	−0.0016 (12)
C5	0.0306 (15)	0.0631 (18)	0.0538 (17)	0.0011 (14)	−0.0059 (13)	0.0068 (14)
C6	0.0382 (16)	0.0432 (15)	0.0421 (15)	0.0115 (13)	0.0007 (13)	0.0056 (11)
C7	0.0513 (17)	0.0289 (13)	0.0404 (15)	0.0050 (12)	−0.0003 (12)	−0.0003 (10)
C8	0.0431 (15)	0.0360 (13)	0.0338 (13)	−0.0012 (12)	0.0014 (12)	0.0090 (10)
C9	0.056 (2)	0.0561 (17)	0.0530 (18)	0.0177 (16)	0.0161 (14)	0.0021 (14)
C10	0.0341 (14)	0.0767 (19)	0.0365 (14)	0.0078 (15)	0.0061 (12)	0.0012 (14)
C11	0.0330 (15)	0.0466 (15)	0.0281 (13)	−0.0016 (12)	−0.0026 (11)	0.0087 (11)
C12	0.0351 (15)	0.0496 (16)	0.0429 (16)	−0.0107 (13)	−0.0067 (12)	0.0127 (12)
C13	0.0544 (19)	0.0347 (14)	0.0404 (15)	−0.0060 (13)	−0.0051 (13)	−0.0017 (11)
C14	0.0388 (16)	0.0315 (12)	0.0372 (14)	0.0068 (12)	−0.0027 (11)	0.0004 (11)
C15	0.0270 (14)	0.0386 (13)	0.0353 (13)	0.0041 (11)	−0.0025 (11)	0.0011 (11)
C16	0.0300 (14)	0.0328 (12)	0.0283 (13)	0.0000 (11)	−0.0044 (11)	0.0015 (10)
C17	0.064 (2)	0.0572 (17)	0.0435 (17)	−0.0100 (16)	−0.0063 (14)	0.0125 (13)
N1	0.0609 (17)	0.0645 (16)	0.0575 (15)	−0.0188 (14)	0.0172 (13)	−0.0116 (12)
O1	0.0347 (11)	0.0791 (13)	0.0669 (14)	0.0165 (11)	−0.0073 (10)	−0.0073 (10)
O2	0.0593 (13)	0.0522 (11)	0.0543 (12)	−0.0180 (10)	−0.0204 (9)	−0.0005 (9)
O3	0.0377 (10)	0.0345 (8)	0.0344 (9)	0.0015 (8)	−0.0078 (8)	−0.0002 (7)
S1	0.0327 (3)	0.0438 (3)	0.0377 (3)	−0.0030 (3)	−0.0080 (3)	0.0024 (3)

Geometric parameters (Å, º)

C1—C14	1.508 (4)	C10—H10A	0.9700
C1—C2	1.585 (4)	C10—H10B	0.9700
C1—H1C	0.9700	C11—C16	1.394 (3)
C1—H1D	0.9700	C11—C12	1.401 (4)

C2—C3	1.516 (4)	C12—C13	1.377 (4)
C2—H2A	0.9700	C12—H12	0.9300
C2—H2B	0.9700	C13—C14	1.384 (4)
C3—C4	1.387 (4)	C13—H13	0.9300
C3—C8	1.400 (3)	C14—C15	1.391 (3)
C4—C5	1.385 (4)	C15—C16	1.373 (3)
C4—H4	0.9300	C15—H15	0.9300
C5—C6	1.390 (4)	C16—O3	1.430 (3)
C5—H5	0.9300	C17—S1	1.742 (3)
C6—C7	1.389 (4)	C17—H17A	0.9600
C6—C9	1.509 (4)	C17—H17B	0.9600
C7—C8	1.394 (4)	C17—H17C	0.9600
C7—H7	0.9300	N1—H1A	0.8600
C8—N1	1.377 (3)	N1—H1B	0.8600
C9—C10	1.579 (4)	O1—S1	1.421 (2)
C9—H9A	0.9700	O2—S1	1.422 (2)
C9—H9B	0.9700	O3—S1	1.5908 (18)
C10—C11	1.499 (4)		

C14—C1—C2	111.5 (2)	C9—C10—H10A	109.0
C14—C1—H1C	109.3	C11—C10—H10B	109.0
C2—C1—H1C	109.3	C9—C10—H10B	109.0
C14—C1—H1D	109.3	H10A—C10—H10B	107.8
C2—C1—H1D	109.3	C16—C11—C12	114.1 (2)
H1C—C1—H1D	108.0	C16—C11—C10	123.2 (2)
C3—C2—C1	111.9 (2)	C12—C11—C10	120.9 (2)
C3—C2—H2A	109.2	C13—C12—C11	121.9 (2)
C1—C2—H2A	109.2	C13—C12—H12	119.1
C3—C2—H2B	109.2	C11—C12—H12	119.1
C1—C2—H2B	109.2	C12—C13—C14	121.0 (2)
H2A—C2—H2B	107.9	C12—C13—H13	119.5
C4—C3—C8	116.7 (2)	C14—C13—H13	119.5
C4—C3—C2	120.4 (2)	C13—C14—C15	116.7 (2)
C8—C3—C2	121.3 (2)	C13—C14—C1	121.3 (2)
C5—C4—C3	122.7 (3)	C15—C14—C1	120.9 (2)
C5—C4—H4	118.7	C16—C15—C14	120.1 (2)
C3—C4—H4	118.7	C16—C15—H15	119.9
C4—C5—C6	119.2 (3)	C14—C15—H15	120.0
C4—C5—H5	120.4	C15—C16—C11	122.9 (2)
C6—C5—H5	120.4	C15—C16—O3	118.5 (2)
C5—C6—C7	117.4 (2)	C11—C16—O3	117.7 (2)
C5—C6—C9	122.0 (3)	S1—C17—H17A	109.5
C7—C6—C9	119.2 (3)	S1—C17—H17B	109.5
C6—C7—C8	122.0 (3)	H17A—C17—H17B	109.5
C6—C7—H7	119.0	S1—C17—H17C	109.5
C8—C7—H7	119.0	H17A—C17—H17C	109.5
N1—C8—C7	119.3 (2)	H17B—C17—H17C	109.5
N1—C8—C3	121.1 (2)	C8—N1—H1A	120.0
C7—C8—C3	119.1 (3)	C8—N1—H1B	120.0

C6—C9—C10	113.6 (2)	H1A—N1—H1B	120.0
C6—C9—H9A	108.8	C16—O3—S1	117.52 (14)
C10—C9—H9A	108.8	O1—S1—O2	119.55 (14)
C6—C9—H9B	108.8	O1—S1—O3	109.55 (12)
C10—C9—H9B	108.8	O2—S1—O3	103.40 (10)
H9A—C9—H9B	107.7	O1—S1—C17	108.54 (14)
C11—C10—C9	113.1 (2)	O2—S1—C17	110.74 (14)
C11—C10—H10A	109.0	O3—S1—C17	103.86 (13)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*B*···O2^{i}	0.86	2.43	3.262 (3)	163
C10—H10*B*···O1^{ii}	0.97	2.52	3.390 (4)	149

Symmetry codes: (i) −*x*+5/2, −*y*+1, *z*−1/2; (ii) *x*−1, *y*, *z*.