

8-(*N*-Methyl-*N*-tosylamino)bicyclo[4.2.0]octane-7-spiro-2'-oxacyclopropane

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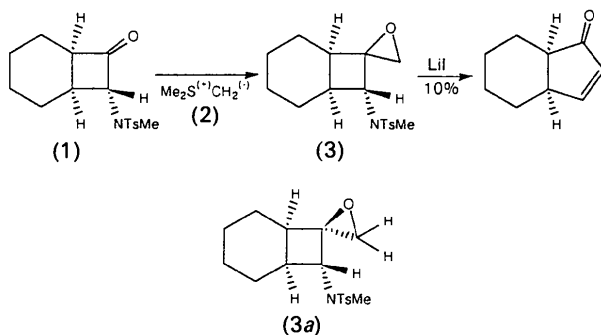
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(Received 7 April 1992; accepted 29 July 1992)

**Abstract.** C<sub>17</sub>H<sub>23</sub>NO<sub>3</sub>S, *M<sub>r</sub>* = 321.4, monoclinic, *P*2<sub>1</sub>/*c*, *a* = 8.724 (3), *b* = 12.009 (3), *c* = 15.780 (4) Å, β = 91.81 (3)°, *V* = 1652.4 (9) Å<sup>3</sup>, *Z* = 4, *D<sub>x</sub>* = 1.29 g cm<sup>-3</sup>, λ(Mo *Kα*) = 0.71069 Å, μ = 2.08 cm<sup>-1</sup>, *F*(000) = 688, *T* = 291 K, *R* = 0.071 for 1671 observed reflections. In the four-membered ring the nitrogen substituent is *exo*; the ring is far from planar, with a dihedral angle about one diagonal of 32 (1)°.

**Experimental.** During development of an original two-step sequence leading to cyclopentenones, we studied the homologation of the cyclobutanone (1) with the sulfonium ylide (2) (Corey & Chaykovsky, 1965). During the course of this investigation, we were able to isolate two diastereoisomeric forms of the oxaspirohexane (3). The configuration of the major isomer (3*a*) was established by this study, thus showing that it is the convex face of the cyclobutanone (1) which is more accessible to attack by the sulfonium ylide (2).



A parallelepiped crystal with dimensions 0.43 × 0.12 × 0.05 mm was used for data collection. Lattice parameters were refined using 30 reflections in the range 6 ≤ 2θ ≤ 30°. Using a Huber four-circle diffractometer with graphite-monochromated Mo *Kα* radiation and ω scan technique, 3791 independent

Table 1. Atomic coordinates (× 10<sup>4</sup>) and equivalent isotropic temperature factors (Å<sup>2</sup> × 10<sup>3</sup>)
$$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* a_i a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U<sub>eq</sub></i>
C1	13867 (6)	9545 (4)	8395 (4)	42 (2)
C2	14540 (6)	9233 (5)	7547 (4)	53 (2)
C3	14517 (7)	7987 (5)	7374 (4)	68 (3)
C4	15252 (8)	7355 (6)	8129 (5)	82 (3)
C5	14263 (8)	7505 (6)	8910 (4)	73 (3)
C6	14151 (6)	8747 (5)	9176 (4)	57 (2)
C7	12551 (6)	9066 (5)	9448 (4)	50 (2)
C8	12131 (6)	9393 (4)	8531 (3)	38 (2)
N9	11006 (5)	10298 (4)	8392 (3)	44 (2)
C10	11506 (7)	11449 (5)	8573 (4)	63 (3)
S11	9668 (2)	10129 (1)	7645 (1)	49 (1)
O12	9126 (4)	9003 (4)	7718 (3)	61 (2)
O13	8628 (4)	11046 (4)	7718 (3)	69 (2)
C14	10574 (5)	10266 (5)	6658 (3)	41 (2)
C15	11127 (6)	9322 (5)	6261 (4)	47 (2)
C16	11927 (7)	9441 (5)	5527 (4)	53 (2)
C17	12175 (6)	10476 (6)	5166 (4)	53 (2)
C18	11573 (7)	11414 (6)	5567 (4)	60 (3)
C19	10774 (7)	11306 (5)	6311 (4)	52 (2)
C20	13062 (7)	10596 (6)	4361 (4)	71 (3)
O21	11598 (5)	8441 (4)	9986 (3)	75 (2)
C22	12084 (8)	9555 (6)	10243 (4)	70 (3)

reflections were measured with sinθ/λ ≤ 0.65 Å<sup>-1</sup> (−11 ≤ *h* ≤ 11, 0 ≤ *k* ≤ 15, 0 ≤ *l* ≤ 20), of which 1671 had *I* ≥ 2.5σ(*I*). A standard reflection (206) was checked every 50 reflections and showed no significant deviation. *L<sub>p</sub>* correction applied. No correction for absorption. The structure was solved by direct methods using *SHELXS86* (Sheldrick, 1985). H atoms were placed in computed positions. Anisotropic least-squares refinement (*SHELX76*; Sheldrick, 1976) was based on *F*; H atoms were treated isotropically with a common refined temperature factor (*U* = 0.107 Å<sup>2</sup>). *R* = 0.071, *wR* = 0.061 [*w* = 1/(σ<sup>2</sup> + 0.00006*F*<sup>2</sup>)], *S* = 2.46 for 1671 observed reflections and 200 parameters. Final maximum shift to e.s.d. ratio was 0.03. Maximum and minimum heights in the final difference Fourier synthesis were 0.38 and −0.36 e Å<sup>-3</sup>, respectively. Atomic scattering factors were obtained from *International Tables for X-ray Crystallography* (1974, Vol. IV).

Table 2. Bond distances (Å) and angles (°)

C2—C1	1.525 (8)	C6—C1	1.574 (7)
C8—C1	1.548 (6)	C3—C2	1.521 (8)
C4—C3	1.537 (8)	C5—C4	1.537 (8)
C6—C5	1.554 (8)	C7—C6	1.522 (7)
C8—C7	1.533 (7)	O21—C7	1.421 (6)
C22—C7	1.455 (8)	N9—C8	1.476 (6)
C10—N9	1.474 (7)	S11—N9	1.646 (4)
C19—C14	1.378 (7)	O12—S11	1.438 (4)
C17—C16	1.388 (8)	O13—S11	1.434 (4)
C17—C18	1.402 (8)	C14—S11	1.776 (5)
C20—C17	1.515 (8)	C15—C14	1.389 (7)
C22—O21	1.457 (8)	C16—C15	1.378 (8)
		C19—C18	1.391 (8)
C6—C1—C2	118.9 (5)	C8—C1—C2	119.9 (5)
C8—C1—C6	87.2 (4)	C3—C2—C1	113.3 (5)
C4—C3—C2	110.1 (6)	C5—C4—C3	109.5 (5)
C6—C5—C4	111.8 (5)	C5—C6—C1	112.5 (5)
C7—C6—C1	86.8 (4)	C7—C6—C5	112.6 (5)
C8—C7—C6	89.6 (4)	O21—C7—C6	126.5 (5)
O21—C7—C8	124.9 (5)	C22—C7—C6	129.0 (6)
C22—C7—C8	130.2 (5)	C22—C7—O21	60.9 (4)
C7—C8—C1	87.4 (4)	N9—C8—C1	122.8 (4)
N9—C8—C7	117.8 (4)	C10—N9—C8	118.1 (4)
S11—N9—C8	117.9 (3)	S11—N9—C10	116.9 (4)
O12—S11—N9	106.6 (2)	O13—S11—N9	106.5 (2)
O13—S11—O12	120.4 (3)	C14—S11—N9	107.0 (2)
C14—S11—O12	108.3 (3)	C14—S11—O13	107.5 (3)
C15—C14—S11	119.4 (5)	C19—C14—S11	119.9 (4)
C19—C14—C15	120.7 (5)	C16—C15—C14	119.1 (6)
C17—C16—C15	121.9 (6)	C18—C17—C16	117.9 (6)
C20—C17—C16	121.4 (6)	C20—C17—C18	120.7 (6)
C19—C18—C17	120.8 (6)	C18—C19—C14	119.6 (5)
C22—O21—C7	60.7 (4)	O21—C22—C7	58.4 (4)

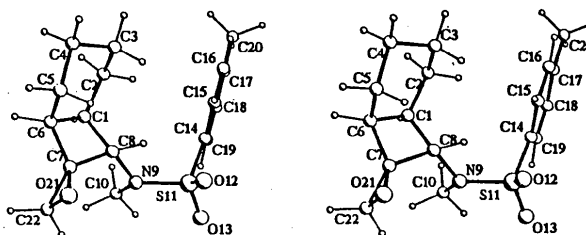


Fig. 1. Stereoscopic view of the molecule with atom numbering.

**Related literature.** In a preceding paper we determined the structure of 8-(*N*-methyl-*N*-*p*-tolylsulfonfylamino)bicyclo[4.2.0]octan-7-one (Tinant, Declercq & Gobeaux, 1990). In this compound the configuration at C8 is also *exo* and the puckering in the cyclobutane ring is 31 (1) and 29 (1)° in the two independent molecules, respectively.

JFD and JPD thank the Fonds de Développement Scientifique Catholic University of Louvain for its financial support.

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The atomic parameters are given in Table 1.\* Fig. 1 is a stereoscopic view of the molecule showing the numbering of the atoms (*PLUTO*; Motherwell & Clegg, 1978). Bond distances and angles are given in Table 2.

\* Lists of structure factors, anisotropic thermal parameters, bond distances and angles, and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55612 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HA1011]

*Acta Cryst.* (1993). **C49**, 562–565

## Structure of the *p*-Nitrobenzoate Ester of 3-*endo*-(*p*-Methoxybenzyl)isoborneol

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(Received 28 February 1992; accepted 21 July 1992)

**Abstract.** (1*R*,2*R*,3*S*,4*R*)-3-[(4-Methoxyphenyl)methyl]-1,7,7-trimethylbicyclo[2.2.1]hept-2-yl 4-nitro-

benzoate, C<sub>25</sub>H<sub>29</sub>NO<sub>5</sub>, *M<sub>r</sub>* = 423.5, orthorhombic, *P*2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>, *a* = 12.104 (3), *b* = 26.712 (5), *c* = 7.138 (1) Å, *V* = 2308.0 (8) Å<sup>3</sup>, *Z* = 4, *D<sub>x</sub>* = 1.22 g cm<sup>-3</sup>, λ(Mo *K*α) = 0.71073 Å, μ = 0.8 cm<sup>-1</sup>,

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