O(24)	0.5769 (3)	0.0753 (2)	0.1765 (1)	0.068 (1)
N(25)	0.4719 (3)	0.1497 (2)	0.3240 (1)	0.044 (1)
C(26)	0.5938 (3)	0.1142 (2)	0.3850 (2)	0.038 (1)
C(27)	0.6982 (3)	0.2160 (3)	0.4721 (2)	0.047 (1)
C(28)	0.8042 (3)	0.1816 (3)	0.5362 (2)	0.058 (1)
C(29)	0.8050 (4)	0.0471 (3)	0.5142 (2)	0.063 (1)
C(30)	0.7025 (4)	−0.0546 (3)	0.4266 (2)	0.061 (1)
C(31)	0.5965 (3)	−0.0211 (3)	0.3615 (2)	0.051 (1)
H(N19)	0.0774	0.1860	0.1156	0.080
H(N22)	0.2358	0.1603	0.2150	0.080
H(N25)	0.4099	0.1987	0.3575	0.080

Table 2. *Selected geometric parameters (Å, °)*

C(1)—N(7)	1.408 (3)	N(19)—C(20)	1.342 (3)
N(7)—C(8)	1.357 (3)	C(20)—O(21)	1.216 (3)
C(8)—O(9)	1.233 (3)	C(20)—N(22)	1.402 (3)
C(8)—N(10)	1.366 (3)	N(22)—C(23)	1.349 (3)
N(10)—C(11)	1.286 (3)	C(23)—O(24)	1.325 (3)
C(11)—N(12)	1.167 (3)	C(23)—N(25)	1.324 (3)
C(13)—N(19)	1.414 (3)	N(25)—C(26)	1.431 (3)
C(1)—N(7)—C(8)	127.6 (2)	N(19)—C(20)—N(22)	110.6 (2)
N(7)—C(8)—O(9)	123.4 (2)	O(21)—C(20)—N(22)	122.9 (2)
N(7)—C(8)—N(10)	111.9 (2)	C(20)—N(22)—C(23)	126.8 (2)
O(9)—C(8)—N(10)	124.7 (2)	N(22)—C(23)—O(24)	120.8 (2)
C(8)—N(10)—C(11)	116.4 (2)	N(22)—C(23)—N(25)	116.7 (2)
N(10)—C(11)—N(12)	175.0 (3)	O(24)—C(23)—N(25)	122.5 (2)
C(13)—N(19)—C(20)	128.7 (2)	C(23)—N(25)—C(26)	125.8 (2)
N(19)—C(20)—O(21)	126.6 (2)		

Table 3. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	*D*···*A*	*D*—H···*A*
N(19)—H(N19)···N(12)	0.866 (2)	2.889 (3)	170.7 (2)
N(22)—H(N22)···N(12)	0.823 (2)	2.942 (3)	156.1 (1)
N(25)—H(N25)···O(9)	0.868 (2)	2.814 (3)	165.5 (1)

H atoms bonded to N atoms were located from a difference Fourier map, while those bonded to C atoms were introduced in calculated positions and refined using a riding model with fixed isotropic *U*'s.

Data collection: *P3/V Data Collection Program* (Siemens, 1989). Cell refinement: *P3/V Data Collection Program.* Data reduction: *SHELXTL-Plus* (Sheldrick, 1990). Program(s) used to solve structure: *SHELXTL-Plus.* Program(s) used to refine structure: *SHELXTL-Plus.* Molecular graphics: *SHELXTL-Plus.* Software used to prepare material for publication: *SHELXTL-Plus.*

The project was supported by the National Natural Science Foundation of China.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: TA1026). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Gilli, G., Bertolasi, V., Bellucci, F., Fabrizio, B. & Ferretti, V. (1986). *J. Am. Chem. Soc.* **108**, 2420–2424.

Sheldrick, G. M. (1990). *SHELXTL-Plus. Structure Determination Software Programs.* Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Siemens (1989). *P3/V Data Collection Program.* Version 4.11. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Yang, Q.-C., Huang, D.-M., Chen H.-Y. & Tang, Y.-Q. (1995). *Acta Cryst.* **C51**, 1412–1414.

Acta Cryst. (1996). **C52**, 472–474

N-[2-(1-3*H*-Indenyl)ethyl]-*p*-toluenesulfonamide

GRAEME J. GAINSFORD* AND CORNELIS LENSINK

The New Zealand Institute for Industrial Research and Development, Materials Chemistry, PO Box 31-310, Lower Hutt, New Zealand. E-mail: g.gainsford@irl.cri.nz

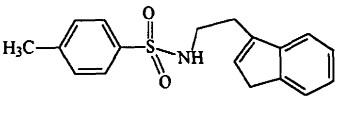
(*Received 23 May 1995; accepted 19 September 1995*)

Abstract

Lithium indene reacts with *N*-*p*-toluenesulfonylaziridene to yield *N*-[2-(1-3*H*-indenyl)ethyl-*p*-toluenesulfonamide, $C_{18}H_{19}NO_2S$. The structure consists of independent molecules with the indene group substituted at the expected 1 position of the five-membered ring. The sulfonamide moiety has a planar arrangement about the N atom.

Comment

The current interest in bidentate cyclopentadiene alkoxides and amides as ligands (Hughes, Meetsma & Teuben, 1993) for early transition metals prompted us to investigate the synthesis of molecules of this type *via* the reaction of cyclopentadienyls with aziridenes. The title compound, (I), was prepared by the reaction of the indenyl anion with *N*-*p*-toluenesulfonylaziridene. An analogous reaction has been described for the substitution of epoxides with fluorenyl or cyclopentadienyl to yield cyclopentadiene alkoxides (Rieger, Steimann & Fawzi, 1992).



(I)

The structure of (I) (Fig. 1, Table 1) shows it to be a 1,2-disubstituted ethane with a regular indenyl moiety substituted at the C atom in position 1 of the five-membered ring and having the double bond in the 1 position [C1=C2 1.340 (4) Å], as expected from NMR evidence. The sulfonamido moiety displays a basically planar N atom, with the bound H atom 0.25 (3) Å from the plane through atoms S1, N1, C10; the closest intermolecular contact is 2.23 Å for O2···H1(N1). Only C2 is significantly out of the indenyl plane [0.023 (4) Å]; there is an angle of 40.2 (1)° between this and the plane of the toluene moiety. There are no significant deviations from expected bond lengths and angles in the molecule (*International Tables for Crystallography*, 1992, Vol. C, Table 9.5.1.1).

Acta Crystallographica Section C
ISSN 0108-2701 ©1996

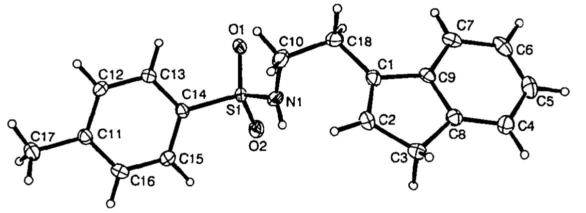


Fig. 1. View of $C_{18}H_{19}NO_2S$ showing the labelling of the non-H atoms. Displacement ellipsoids are shown at 50% probability levels and H atoms are drawn as small circles of arbitrary radii.

Experimental

Freshly distilled indene (3.74 g, 32.6 mmol) was dissolved in 100 ml of dry tetrahydrofuran and cooled to 195 K. *N*-Butyllithium (13 ml, 1.6 *M*) was added *via* syringe. The reaction mixture was allowed to warm to 273 K, subsequently cooled to 233 K and *N*-*p*-toluenesulfonylaziridine (4.0 g, 20.3 mmol) was added as a solid in one portion. The reaction mixture was allowed to warm slowly to room temperature and stirred for 17 h. Saturated NH_4Cl (100 ml) was added to the reaction mixture followed by 50 ml of ethyl acetate. The organic layer was separated from the aqueous layer, washed with brine and dried on Na_2SO_4. The solvent was removed to yield the title compound as off-white (light brown) crystals (3.06 g, 9.8 mmol). ^{13}C and ^1H NMR spectra are in agreement with the structure.

Crystal data

$C_{18}H_{19}NO_2S$
$M_r = 313.40$
Monoclinic
$P2_1/n$
$a = 12.681\ (7)$ Å
$b = 7.261\ (4)$ Å
$c = 16.654\ (9)$ Å
$\beta = 93.65\ (2)°$
$V = 1530.3\ (15)$ Å^3
$Z = 4$
$D_x = 1.360$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 17 reflections
$\theta = 4.9–14.02°$
$\mu = 0.218$ mm^{-1}
$T = 130\ (2)$ K
Needle
0.82 × 0.18 × 0.08 mm
Light brown

Data collection

Siemens/Nicolet *P*3 four-circle diffractometer
ω scans
Absorption correction: ψ scan (*XPREP*; Siemens, 1990)
$T_{min} = 0.736$, $T_{max} = 0.768$
3216 measured reflections
2693 independent reflections

1985 observed reflections $[I > 2\sigma(I)]$
$R_{int} = 0.0272$
$\theta_{max} = 24.98°$
$h = -15 \rightarrow 2$
$k = 0 \rightarrow 8$
$l = -19 \rightarrow 19$
3 standard reflections monitored every 97 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0454$
$wR(F^2) = 0.1053$
$S = 1.044$
2693 reflections
204 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o^2) + (0.0426P)^2 + 0.7882P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = -0.003$
$\Delta\rho_{max} = 0.219$ e Å^{-3}
$\Delta\rho_{min} = -0.330$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
S1	0.16469 (5)	0.02025 (9)	0.06071 (4)	0.0183 (2)
O1	0.27669 (14)	0.0448 (3)	0.06146 (10)	0.0240 (4)
O2	0.09597 (15)	0.1610 (3)	0.02682 (10)	0.0237 (4)
N1	0.1364 (2)	−0.1644 (4)	0.01147 (14)	0.0242 (6)
C1	0.1484 (2)	−0.4192 (3)	−0.13121 (15)	0.0178 (6)
C2	0.0547 (2)	−0.4894 (4)	−0.11407 (15)	0.0206 (6)
C3	−0.0152 (2)	−0.5227 (4)	−0.18816 (15)	0.0218 (6)
C4	0.0333 (2)	−0.4670 (4)	−0.3367 (2)	0.0243 (6)
C5	0.1127 (2)	−0.4071 (4)	−0.3850 (2)	0.0270 (7)
C6	0.2085 (2)	−0.3462 (4)	−0.3506 (2)	0.0269 (7)
C7	0.2282 (2)	−0.3426 (4)	−0.2677 (2)	0.0214 (6)
C8	0.0533 (2)	−0.4665 (4)	−0.25422 (15)	0.0184 (6)
C9	0.1500 (2)	−0.4041 (3)	−0.21945 (15)	0.0175 (6)
C10	0.2065 (2)	−0.3251 (4)	0.0112 (2)	0.0257 (7)
C11	0.0820 (2)	−0.0526 (4)	0.32097 (15)	0.0184 (6)
C12	0.1821 (2)	−0.0986 (4)	0.29725 (15)	0.0207 (6)
C13	0.2081 (2)	−0.0776 (4)	0.21806 (15)	0.0201 (6)
C14	0.1327 (2)	−0.0087 (3)	0.16171 (14)	0.0153 (5)
C15	0.0323 (2)	0.0365 (4)	0.18421 (15)	0.0201 (6)
C16	0.0081 (2)	0.0148 (4)	0.2631 (2)	0.0206 (6)
C17	0.0547 (2)	−0.0717 (4)	0.40725 (15)	0.0242 (6)
C18	0.2393 (2)	−0.3661 (4)	−0.0738 (2)	0.0240 (6)

Table 2. *Selected geometric parameters (Å, °)*

S1—O1	1.431 (2)	C3—C8	1.501 (3)
S1—O2	1.435 (2)	C4—C8	1.380 (4)
S1—N1	1.600 (3)	C4—C5	1.398 (4)
S1—C14	1.768 (3)	C5—C6	1.381 (4)
N1—C10	1.467 (4)	C6—C7	1.388 (4)
C1—C2	1.340 (4)	C7—C9	1.389 (4)
C1—C9	1.475 (3)	C8—C9	1.398 (4)
C1—C18	1.500 (4)	C10—C18	1.530 (4)
C2—C3	1.493 (4)	C11—C17	1.506 (3)
O1—S1—O2	119.49 (12)	C5—C6—C7	121.1 (3)
O1—S1—N1	107.40 (13)	C6—C7—C9	118.6 (3)
O2—S1—N1	106.52 (12)	C4—C8—C9	120.9 (2)
O1—S1—C14	107.11 (11)	C4—C8—C3	130.6 (2)
O2—S1—C14	106.77 (11)	C9—C8—C3	108.5 (2)
N1—S1—C14	109.30 (13)	C7—C9—C8	120.3 (2)
C10—N1—S1	123.5 (2)	C7—C9—C1	131.0 (2)
C2—C1—C9	108.0 (2)	C8—C9—C1	108.7 (2)
C2—C1—C18	128.2 (2)	N1—C10—C18	111.0 (2)
C9—C1—C18	123.8 (2)	C16—C11—C17	120.5 (2)
C1—C2—C3	111.9 (2)	C12—C11—C17	121.2 (2)
C2—C3—C8	102.7 (2)	C15—C14—S1	119.9 (2)
C8—C4—C5	118.6 (3)	C13—C14—S1	119.7 (2)
C6—C5—C4	120.5 (2)	C1—C18—C10	113.3 (2)
O1—S1—N1—C10	33.0 (3)	O1—S1—C14—C13	−25.9 (2)
O2—S1—N1—C10	162.1 (2)	O2—S1—C14—C13	−155.0 (2)
C14—S1—N1—C10	−82.9 (2)	N1—S1—C14—C13	90.2 (2)
C9—C1—C2—C3	−1.5 (3)	C13—C14—C15—C16	0.8 (4)
S1—N1—C10—C18	−118.7 (2)	C17—C11—C16—C15	178.7 (2)
O1—S1—C14—C15	154.5 (2)	C2—C1—C18—C10	−23.0 (4)
O2—S1—C14—C15	25.4 (2)	C9—C1—C18—C10	158.3 (2)
N1—S1—C14—C15	−89.4 (2)	N1—C10—C18—C1	−63.9 (3)

Data collection: *XSCANS* (Siemens, 1994). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *SHELXL93 CIFTAB*.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, torsion angles and complete geometry have been deposited with the IUCr (Reference: KH1057). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Hughes, K. H., Meetsma, A. & Teuben, J. H. (1993). *Organometallics*, **12**, 1936.

Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Rieger, B., Steimann, M. & Fawzi, R. (1992). *Chem. Ber.* **125**, 2373–2377.

Sheldrick, G. M. (1985). *SHELXS86. Program for the Solution of Crystal Structures*. University of Göttingen, Germany.

Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.

Siemens (1990). *XPREP*. Version 4.1 for MS-DOS. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Siemens (1994). *XSCANS User's Manual*. Version 2.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Acta Cryst. (1996). C**52**, 474–477

Dupeyredioxyl (1,3,5,7-Tetramethyl-2,6-diazaadamantane-*N*,*N*′-dioxyl)

Yves Dromzee,[a] Robert Chiarelli,[b] Serge Gambarelli[b] and André Rassat[b]

[a]*Chimie des Métaux de Transition, Université Paris 6, BP42 F-75252 Paris CEDEX 05, France, and* [b]*Département de Chimie de l'Ecole Normale Supérieure, 24 rue Lhomond, F-75231, Paris CEDEX 05, France*

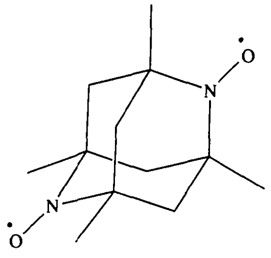
(*Received 7 November 1994; accepted 21 June 1995*)

Abstract

The title compound, $C_{12}H_{20}N_2O_2$, exhibits two crystalline forms (α monoclinic and β orthorhombic) in which the diazaadamantane skeleton has the same structure but the packing is different. Both are obtained by crystallization in diethyl ether. The monoclinic form has been reported to possess a ferromagnetic transition.

Comment

The title compound, (I), has been synthesized (Chiarelli & Rassat, 1993) and it has been shown that the monoclinic form undergoes a ferromagnetic transition at 1.48 K (Chiarelli & Rassat, 1991; Chiarelli, Rassat & Rey, 1992; Fluekiger, Weber, Chiarelli, Rassat & Ellinger, 1993; Chiarelli, Novak, Rassat & Tholence, 1993; Chiarelli, Rassat, Dromzee, Jeannin, Novak & Tholence, 1993). It is one of the purely organic materials which exhibits a well characterized ferromagnetic transition (see Miller & Epstein, 1994). Above the transition temperature, crystals of the α form show a spontaneous magnetization and absence of hysteresis. A simplified (partial) crystal structure of the α form has been published (Chiarelli, Novak, Rassat & Tholence, 1993). Complementary information about the α form and a full report of the structure of the β form are presented here.



(I)

By evaporation of a diethyl ether solution of (I) at room temperature, it is possible to obtain two different crystalline forms in the same crop, *i.e.* short parallelepipeds (α form) and needle-like crystals (β form). The α form is monoclinic and the β form is orthorhombic. We have not been able to induce a selectivity towards one type of crystallization.

Atomic coordinates are given in Tables 1 and 4 for the monoclinic and orthorhombic forms, respectively. Selected bond distances and bond angles are given in

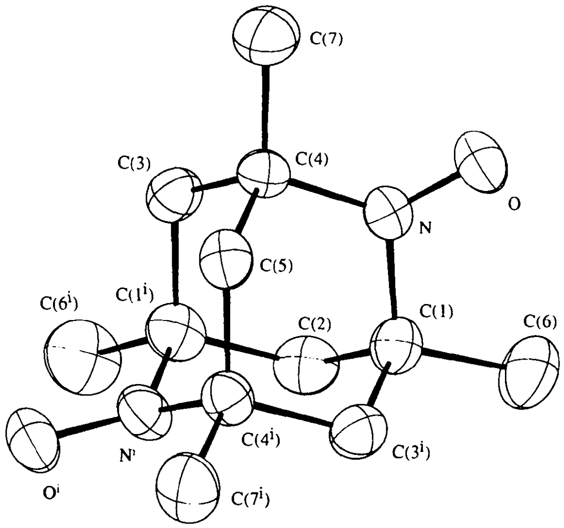


Fig. 1. The molecular structure of $C_{12}H_{20}N_2O_2$ (α form; monoclinic) showing the numbering scheme employed and 50% probability displacement ellipsoids. H atoms are omitted for clarity. [Symmetry code: (i) $1-x, y, \frac{1}{2}-z$.]