

$V = 2492.9 (8) \text{ \AA}^3$
 $Z = 8$
 $D_x = 1.206 \text{ Mg m}^{-3}$

Parallelepiped
 $0.26 \times 0.24 \times 0.19 \text{ mm}$
 Colourless

Data collection

Siemens R3m/V diffractometer
 $R_{\text{int}} = 0$
 $\theta_{\text{max}} = 25^\circ$
 $h = 0 \rightarrow 19$
 Absorption correction: none
 $k = 0 \rightarrow 20$
 $l = -10 \rightarrow 0$
 1273 measured reflections
 1270 independent reflections
 814 observed reflections
 $[F > 2.5\sigma(F)]$
 3 standard reflections
 frequency: 97 min
 intensity variation: 5%

Refinement

Refinement on F
 Final $R = 0.0520$
 $wR = 0.0586$
 $S = 1.452$
 814 reflections
 71 parameters
 Calculated weights,
 $w = 1/[\sigma^2(F) + 0.0005F^2]$
 $(\Delta/\sigma)_{\text{max}} = 0.0021$
 $\Delta\rho_{\text{max}} = 0.24 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.27 \text{ e \AA}^{-3}$
 Extinction correction: Larson (1967)
 Extinction coefficient: 0.0004 (2)
 Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B)

Data collection: Siemens P3VAX version 3.4. Data reduction: REFRED (Reibenspies, 1992). Program(s) used to solve structure: SHELXTL-Plus (Sheldrick, 1990). Program(s) used to refine structure: SHELXTL-Plus. Software used to prepare material for publication: SHELXTL-Plus.

The title compound was obtained from the Aldrich Chemical Co. and recrystallized from water. A suitable crystal was chosen and mounted on a glass fiber with epoxy cement at room temperature. Inspection of axial photographs about each of the three axes confirmed axis length and Laue symmetry. ω scans of several intense reflections indicated acceptable crystal quality.

Table 1. Fractional atomic coordinates and equivalent isotropic thermal parameters (\AA^2)

$$U_{\text{eq}} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j$$

	x	y	z	U_{eq}
O(1)	0.5000	0.2500	-0.0269 (3)	0.049 (1)
O(2)	0.5000	0.1484 (2)	0.2500	0.058 (1)
O(3)	0.1268 (1)	0.2500	0.2500	0.041 (1)
N(1)	0.3702 (1)	0.0230 (1)	0.1368 (2)	0.036 (1)
N(2)	0.2220 (1)	0.1240 (1)	0.1367 (2)	0.034 (1)
C(1)	0.3335 (2)	-0.1024 (2)	0.0152 (3)	0.036 (1)
C(2)	0.4010 (2)	-0.0513 (2)	0.0753 (3)	0.039 (1)
C(3)	0.3534 (2)	0.0826 (2)	0.0211 (3)	0.038 (1)
C(4)	0.3014 (2)	0.1491 (2)	0.0840 (3)	0.038 (1)

Table 2. Geometric parameters (\AA , $^\circ$)

N(1)—C(2)	1.460 (3)	C(1)—C(2)	1.513 (4)
N(1)—C(3)	1.465 (3)	C(3)—C(4)	1.523 (4)
N(2)—C(4)	1.464 (3)	C(1)—N(2) ⁱ	1.466 (3)
N(2)—C(1) ⁱ	1.466 (3)		
C(2)—N(1)—C(3)	113.2 (2)	N(1)—C(2)—C(1)	111.1 (2)
C(4)—N(2)—C(1) ⁱ	113.9 (2)	N(1)—C(3)—C(4)	110.9 (2)
C(2)—C(1)—N(2) ⁱ	110.5 (2)	N(2)—C(4)—C(3)	114.7 (2)

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

The 2θ scan width was $2.0^\circ + K\alpha$ separation, with a variable θ scan rate of $1.5\text{--}14.6^\circ \text{ min}^{-1}$. Background measurements were made by stationary crystal and stationary counter techniques at the beginning and end of each reflection for half the total scan time. Intensities were corrected by a peak-profiling method (Diamond, 1969). Carbon-bound H atoms were placed in idealized positions [$\text{C—H} = 0.96 \text{ \AA}$, $U(\text{H}) = 0.08 \text{ \AA}^2(\text{fixed})$]. Nitrogen-bound and oxygen-bound H atoms were located in a difference Fourier map. The nitrogen-bound H atoms were set to an idealized bond distance [$\text{N—H} = 0.90 \text{ \AA}$, $U(\text{H}) = 0.08 \text{ \AA}^2(\text{fixed})$]; the distances of the oxygen-bound H atoms were not modified [$U(\text{H}) = 0.08 \text{ \AA}^2(\text{fixed})$]. The H-atom parameters were not refined.

The X-ray diffraction and crystallographic computing system was purchased from funds provided by the National Science Foundation (USA), grant CHE-8513273.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55237 (8 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: ST1013]

References

- Cram, D. J. & Trueblood, K. N. (1981). *Top. Curr. Chem.* **98**, 43–106.
 Dale, J. (1980). *Isr. J. Chem.* **20**, 3–11.
 Diamond, R. (1969). *Acta Cryst.* **A25**, 43–55.
 Hancock, R. D. & Martell, A. E. (1989). *Chem. Rev.* **89**, 1875–1914.
 Larson, A. C. (1967). *Acta Cryst.* **23**, 664–665.
 Reibenspies, J. H. (1992). REFRED. Program for data reduction for Siemens R3m/V diffractometer. Texas A&M Univ., College Station, Texas, USA.
 Reibenspies, J. H. & Anderson, O. P. (1990). *Acta Cryst.* **C46**, 163–165.
 Sheldrick, G. M. (1990). SHELXTL-Plus. Revision 4.11V. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
 Weber, E. & Vogtle, F. (1981). *Top. Curr. Chem.* **98**, 1–41.

Acta Cryst. (1992). **C48**, 1718–1720

1-Benzenesulfonyl-3-methyl-2-oxo-2,3-dihydroindol-3-yl Acetate

DAVID A. GROSSIE AND DANIEL M. KETCHA

Department of Chemistry, Wright State University,
 Dayton, Ohio 45435, USA

(Received 15 April 1992; accepted 12 June 1992)

Abstract

The title compound was synthesized via a novel oxidation employing manganese(III) acetate. All bond distances and angles are similar to those found in previously reported oxindole-ring systems. In the title compound,

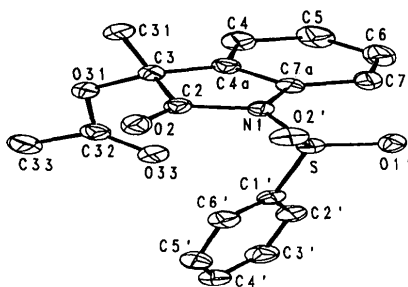
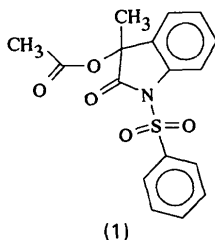


Fig. 1. Molecular structure of 1-benzensulfonyl-3-methyl-2-oxo-2,3-dihydroindol-3-yl acetate. Non-H atoms are shown at 30% of thermal probability. H atoms have been omitted for clarity.

the five-membered ring of the oxindole takes on an envelope conformation, with the lone sp^3 C atom, C3, deviating from planarity by 0.152(4) Å. The angle between the planes of the oxindole benzene ring and the planar portion of the oxindole five-membered ring (C4a, N1, C2, C7a) is 0.8(1)°.

Comment

The title compound (1) was prepared from 1-benzensulfonyl-3-methylindole using manganese(III) acetate by an unusual process which involved an initial C2 acetoxylation followed by hydrolysis and isomerization to afford an oxindole. Subsequent *in situ* acetoxylation of the C3 benzylic position produced the title compound. A similar oxidation of indole-3-propionic acid to an oxindole using thallium(III) nitrate has been observed by Ohnuma, Kimura & Ban (1981) and Ohnuma, Kasuya, Kimura & Ban (1982); in this case, intramolecular cyclization of the carboxylic acid function occurred to afford a spiro- γ -lactone.



Experimental

Crystal data

$C_{17}H_{15}NO_5S$
 $M_r = 345.37$
 Monoclinic
 $P2_1/n$
 $a = 13.452$ (2) Å
 $b = 7.952$ (1) Å
 $c = 15.671$ (1) Å
 $\beta = 106.97$ (1)°
 $V = 1603.3$ (3) Å³
 $Z = 4$
 $D_x = 1.431$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71069$ Å
 Cell parameters from 25 reflections
 $\theta = 17.7$ – 24.1 °
 $\mu = 2.3$ cm⁻¹
 $T = 293$ K
 Rectangular
 $0.57 \times 0.40 \times 0.38$ mm
 Colorless

Data collection

Enraf-Nonius CAD-4 diffractometer
 $\omega/2\theta$ scans
 Absorption correction: analytical
 $T_{\min} = 0.8956$, $T_{\max} = 0.9279$
 3201 measured reflections
 2837 independent reflections
 2321 observed reflections
 $[I_{\text{net}} > 3.0\sigma(I_{\text{net}})]$

$R_{\text{int}} = 0.011$
 $\theta_{\text{max}} = 27.32$ °
 $h = 0 \rightarrow 16$
 $k = 0 \rightarrow 9$
 $l = -18 \rightarrow 17$
 3 standard reflections
 frequency: 120 min
 intensity variation: 1.6%

Refinement

Refinement on F
 Final $R = 0.041$
 $wR = 0.040$
 $S = 1.015$
 2321 reflections
 217 parameters
 H-atom parameters not refined
 Calculated weights; Regina weighting scheme (Robertson & Wang, 1990)

$(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.328$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.35$ e Å⁻³
 Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B)

Data reduction: *Xtal3.0 DIFDAT*, *ABSORB*, *SORTRF* and *AD-DREF* (Hall & Stewart, 1990). Program(s) used to solve structure: *Xtal3.0 GENTAN*. Program(s) used to refine structure: *Xtal3.0 CRYLSQ*. Molecular graphics: *Xtal3.0 PIG* and *ORTEP*. Software used to prepare material for publication: *Xtal3.0 BONDLA* and *CIFIO*.

Table 1. Fractional atomic coordinates and equivalent isotropic thermal parameters (Å²)

$$U_{\text{eq}} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	U_{eq}
N1	0.6963 (1)	0.8456 (2)	0.5032 (1)	0.0342 (8)
C2	0.7964 (2)	0.8548 (3)	0.4933 (1)	0.037 (1)
O2	0.8178 (1)	0.8413 (3)	0.4246 (1)	0.0520 (9)
C3	0.8717 (2)	0.8940 (3)	0.5856 (2)	0.040 (1)
C4a	0.8048 (2)	0.8731 (3)	0.6462 (1)	0.039 (1)
C4	0.8313 (2)	0.8859 (4)	0.7377 (2)	0.055 (1)
C5	0.7534 (3)	0.8759 (5)	0.7788 (2)	0.064 (2)
C6	0.6513 (2)	0.8563 (4)	0.7287 (2)	0.060 (2)
C7	0.6232 (2)	0.8442 (3)	0.6362 (2)	0.046 (1)
C7a	0.7021 (2)	0.8507 (3)	0.5964 (1)	0.0351 (9)
S	0.59464 (4)	0.78598 (7)	0.41727 (4)	0.0352 (3)
O1'	0.5088 (1)	0.7726 (2)	0.4523 (1)	0.0492 (8)
O2'	0.5896 (1)	0.8982 (2)	0.3460 (1)	0.0519 (9)
C1'	0.6291 (2)	0.5854 (3)	0.3888 (1)	0.0355 (9)
C2'	0.6292 (2)	0.4532 (3)	0.4461 (2)	0.044 (1)
C3'	0.6543 (2)	0.2955 (4)	0.4228 (2)	0.055 (1)
C4'	0.6779 (2)	0.2707 (4)	0.3439 (2)	0.059 (1)
C5'	0.6768 (2)	0.4025 (4)	0.2873 (2)	0.057 (1)
C6'	0.6525 (2)	0.5634 (3)	0.3095 (2)	0.046 (1)
C31	0.9137 (2)	1.0717 (3)	0.5859 (2)	0.053 (1)
O31	0.9624 (1)	0.7869 (2)	0.6051 (1)	0.0448 (8)
C32	0.9450 (2)	0.6190 (3)	0.5962 (2)	0.042 (1)
O33	0.8594 (1)	0.5610 (2)	0.5688 (1)	0.0518 (9)
C33	1.0438 (2)	0.5238 (4)	0.6230 (2)	0.058 (1)

Table 2. Geometric parameters (Å, °)

N1—C2	1.402 (3)	C7—C7a	1.380 (4)
N1—C7a	1.439 (3)	S—O1'	1.420 (2)
N1—S	1.684 (2)	S—O2'	1.416 (2)
C2—O2	1.196 (3)	S—C1'	1.755 (2)
C2—C3	1.536 (3)	C1'—C2'	1.382 (3)
C3—C4a	1.497 (4)	C1'—C6'	1.378 (4)
C3—C31	1.521 (4)	C2'—C3'	1.375 (4)
C3—O31	1.445 (3)	C3'—C4'	1.378 (5)
C4a—C4	1.376 (3)	C4'—C5'	1.370 (4)
C4a—C7a	1.386 (3)	C5'—C6'	1.389 (4)
C4—C5	1.384 (5)	O31—C32	1.356 (3)
C5—C6	1.377 (4)	C32—O33	1.198 (3)
C6—C7	1.391 (4)	C32—C33	1.480 (4)
C2—N1—C7a	110.0 (2)	N1—C7a—C7	129.5 (2)
C2—N1—S	120.5 (2)	C4a—C7a—C7	121.4 (2)
C7a—N1—S	127.6 (2)	N1—S—O1'	105.9 (1)
N1—C2—O2	125.9 (2)	N1—S—O2'	107.1 (1)
N1—C2—C3	107.3 (2)	N1—S—C1'	104.25 (9)
O2—C2—C3	126.8 (2)	O1'—S—O2'	119.7 (1)
C2—C3—C4a	102.8 (2)	O1'—S—C1'	109.6 (1)
C2—C3—C31	109.5 (2)	O2'—S—C1'	109.1 (1)
C2—C3—O31	111.3 (2)	S—C1'—C2'	118.4 (2)
C4a—C3—C31	113.1 (2)	S—C1'—C6'	119.4 (2)
C4a—C3—O31	115.2 (2)	C2'—C1'—C6'	122.1 (2)
C31—C3—O31	104.9 (2)	C1'—C2'—C3'	118.4 (3)
C3—C4a—C4	129.2 (2)	C2'—C3'—C4'	120.5 (3)
C3—C4a—C7a	110.0 (2)	C3'—C4'—C5'	120.6 (3)
C4—C4a—C7a	120.6 (2)	C4'—C5'—C6'	120.1 (3)
C4a—C4—C5	118.6 (2)	C1'—C6'—C5'	118.3 (2)
C4—C5—C6	120.4 (3)	C3—O31—C32	116.7 (2)
C5—C6—C7	121.7 (3)	O31—C32—O33	122.3 (2)
C6—C7—C7a	117.2 (2)	O31—C32—C33	111.2 (2)
N1—C7a—C4a	109.0 (2)	O33—C32—C33	126.5 (2)

H atoms were additively constrained to attached atoms and thermal parameters were not refined. *Xtal/PC3.0* (Hall & Stewart, 1990; Grossie, 1990) was used in the course of the investigation.

We thank the Polymer Branch of the Materials Directorate, Wright Laboratory, Wright-Patterson Air Force Base, for diffractometer time.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry, together with least-squares-planes data, have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55221 (26 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: ST1011]

References

- Grossie, D. A. (1990). *Acta Cryst.* A46, C-39.
Hall, S. R. & Stewart, J. M. (1990). Editors. *Xtal3.0 Reference Manual*. Univs. of Western Australia, Australia, and Maryland, USA.
Ohnuma, T., Kasuya, H., Kimura, Y. & Ban, Y. (1982). *Heterocycles*, 17, 377-380.
Ohnuma, T., Kimura, Y. & Ban, Y. (1981). *Tetrahedron Lett.* 22, 4969-4972.
Robertson, B. E. & Wang, H. (1990). In *Xtal3.0 Reference Manual*, edited by S. R. Hall & J. M. Stewart. Univs. of Western Australia, Australia, and Maryland, USA.

Acta Cryst. (1992). C48, 1720-1722

Structure du 2 α ,3 α ,7 α ,13 α -Diépoxyhimachalane

A. CHIARONI ET C. RICHE

Institut de Chimie des Substances Naturelles, CNRS, 91198 Gif sur Yvette CEDEX, France

A. BENHARREF ET A. CHEKROUN

Département de Chimie, Faculté des Sciences Semlalia, Université Cadi Ayyad, BP S15, Marrakech, Maroc

J.-P. LAVERGNE

Laboratoire de Synthèse et d'Etudes Physicochimiques, d'Aminoacides et de Peptides, Université de Montpellier II, Sciences et Techniques du Languedoc, UA 468, Place Eugène Bataillon, 34095 Montpellier CEDEX 5, France

(Reçu le 4 février 1992, accepté le 11 mai 1992)

Abstract

The stereochemistry of the major isomer resulting from the epoxidation of α -cis-himachalene has been established. Hence, the configurations of the three resulting derivatives have been deduced. The seven-membered ring is chair shaped while the six-membered ring adopts a half-chair conformation.

Commentaire

La littérature ne rapporte aucune réaction d'oxydation de l' α -cis-himachalène, composant de l'huile essentielle du cèdre de l'Atlas (*Cedrus Atlantica*), alors que son isomère le β -himachalène a été largement étudié (Joseph & Dev, 1968a; Narula & Dev, 1977; Shankaranaryan, Krishnappa & Dev, 1977; Shankaranaryan, Bishrya & Dev, 1977; Chiaroni, Païs, Riche, Benharref, Chekroun & Lavergne, 1991; Benharref, Chekroun & Lavergne, 1991). Nous avons réalisé l'époxidation de ce sesquiterpène et obtenu des composés dont la structure a été déterminée par l'utilisation de la RMN 360 MHz à deux dimensions. Une analyse cristallographique a été cependant nécessaire pour déterminer la stéréochimie de ces époxydes. Comme le montre le schéma, l'action de l'acide *m*-chloroperbenzoïque (*m*-CPBA) sur (1), en quantité stoechiométrique, conduit avec un rendement quantitatif au seul composé: 2 α ,3 α -époxyhimachal-7-13-ène (2). En présence de deux équivalents de *m*-CPBA, l' α -cis-himachalène (1) donne un mélange de deux époxydes (3) (85%) et (4) (15%). Le même résultat est obtenu par l'action d'une quantité stoechiométrique du