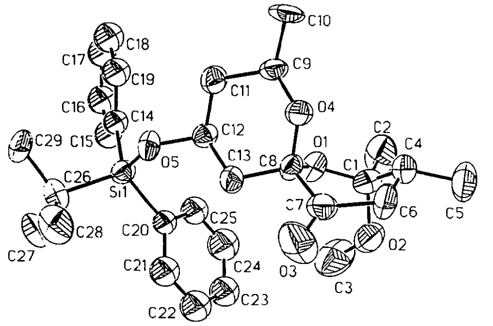

Fig. 1. A perspective view of the spiroketal with ellipsoids at 50% probability.

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(*R*)-(1-Methyloxiranyl)methyl 4-Nitrobenzenesulfonate

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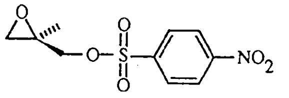
Abstract. $C_{10}H_{11}NO_6S$, $M_r = 273.3$, monoclinic, $P2_1$, $a = 10.2612$ (8), $b = 8.1670$ (8), $c = 15.2974$ (12) Å, $\beta = 108.885$ (6)°, $V = 1213.0$ (2) Å^3, $Z = 4$, $D_x = 1.496$ g cm^{-3}, λ(Cu $K\alpha$) = 1.54184 Å, $\mu = 25.39$ cm^{-1}, $F(000) = 568$, $T = 297$ K, $R = 0.046$ for 4453 observations having $I > 3\sigma(I)$ (of 4798 unique data). The two independent molecules in the asymmetric unit differ conformationally, principally in torsion angle differences about the two bonds to the CH_2 group that link the oxirane three-membered ring to the 4-nitrobenzenesulfonate group. In molecule (*A*), the C(ring)—C—C—O torsion angle is −23.0 (4)°, while in molecule (*B*) that torsion angle is −146.8 (3)°. The S—O—C—C torsion angle in molecule (*A*) is 133.2 (2)°, while in molecule (*B*), it is −143.9 (2)°. The C—S—O—C torsion angles are −80.9 (2)° in molecule (*A*) and 76.5 (2)° in molecule (*B*). The molecules pack with a pseudocenter near $\frac{1}{2}, \frac{1}{2}, \frac{3}{4}$ as a pseudoracemate. Only the position of the epoxy O atom greatly deviates from a centrosymmetric motif.

Experimental. Pale yellow prisms of (1), m.p. 313.5–314.5 K, were isolated by two successive recrystallizations from ethanol of the crude product prepared by the reaction of (*S*)-(−)-2-methylglycidol with 4-nitrobenzenesulfonyl chloride in dichloromethane at 273 K. Crystal size 0.28 × 0.35 × 0.45 mm, mounted in capillary in random orientation on an Enraf–Nonius CAD-4 diffractometer equipped with a graphite monochromator, Cu $K\alpha$ radiation. Cell dimensions from setting angles of 25 reflections having $12 < \theta < 30°$. The space group was determined from systematic absences $0k0$ with k odd and the known chirality of the compound.



(1)

A hemisphere of data having $4 < 2\theta < 150°$, $0 \le h \le 12$, $-10 \le k \le 10$, $-19 \le l \le 19$ was measured using ω–2θ scans designed for $I = 25\sigma(I)$, subject to maximum scan time of 90 s, scan rates varied 0.66–3.30° min^{-1}. Data corrected for background, Lorentz and polarization effects. Intensities of standard reflections 300, 020, 004 varied only randomly, and no decay correction was applied. Absorption corrections were based on ψ scans, and relative transmission coefficients ranged from 0.8982 to 0.9975 with an average value of 0.9593. The extinction coefficient was refined in the least squares to $g = 2.9$ (2) × 10^{-6} where the correction factor $(1 + gI_c)^{-1}$ was applied to F_c. A total of 5090 data was measured. $R_{\text{int}} = 0.020$ for averaging redundant data. The structure was solved by direct methods, using *RANTAN* (Yao, 1981), and was refined by weighted full-matrix least squares; non-H atoms refined aniso-

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Table 1. *Coordinates and equivalent isotropic thermal parameters* (Å^2)

$$B_{eq} = (4/3)[a^2\beta_{11} + b^2\beta_{22} + c^2\beta_{33} + (2ab\cos\gamma)\beta_{12} + (2ac\cos\beta)\beta_{13} + (2bc\cos\alpha)\beta_{23}].$$

	x	y	z	B_{eq}
S1*A*	0.79270 (8)	1	0.58911 (5)	4.54 (2)
O1*A*	0.9090 (2)	1.0810 (3)	0.6509 (2)	5.60 (6)
O2*A*	0.7002 (3)	1.0858 (4)	0.5139 (2)	6.27 (6)
O3*A*	0.8392 (2)	0.8491 (3)	0.5427 (1)	4.68 (5)
O4*A*	0.3645 (3)	0.6252 (6)	0.7652 (2)	10.8 (1)
O5*A*	0.5315 (3)	0.6566 (6)	0.8877 (2)	8.9 (1)
O6*A*	0.8626 (3)	0.5187 (4)	0.4941 (2)	5.86 (6)
N1*A*	0.4789 (3)	0.6718 (4)	0.8055 (2)	5.42 (7)
C1*A*	0.6980 (3)	0.9083 (4)	0.6542 (2)	3.78 (6)
C2*A*	0.5643 (3)	0.8595 (5)	0.6101 (2)	4.76 (7)
C3*A*	0.4917 (3)	0.7816 (5)	0.6593 (2)	5.02 (8)
C4*A*	0.5551 (3)	0.7567 (4)	0.7521 (2)	4.19 (6)
C5*A*	0.6885 (3)	0.8061 (5)	0.7980 (2)	4.83 (7)
C6*A*	0.7607 (3)	0.8817 (5)	0.7482 (2)	4.70 (7)
C7*A*	0.9593 (3)	0.7545 (4)	0.5968 (2)	4.77 (7)
C8*A*	0.9281 (3)	0.5757 (4)	0.5865 (2)	4.22 (6)
C9*A*	0.7890 (4)	0.5123 (5)	0.5602 (3)	6.14 (9)
C10*A*	1.0477 (4)	0.4721 (6)	0.6422 (3)	6.6 (1)
S1*B*	0.22028 (7)	−0.0432 (1)	0.89461 (5)	3.83 (1)
O1*B*	0.1058 (2)	−0.1184 (3)	0.8281 (2)	4.90 (5)
O2*B*	0.3070 (2)	−0.1355 (3)	0.9692 (2)	5.26 (5)
O3*B*	0.1709 (2)	0.1044 (3)	0.9421 (1)	3.94 (4)
O4*B*	0.6706 (3)	0.3531 (6)	0.7474 (2)	10.07 (9)
O5*B*	0.5167 (3)	0.3128 (6)	0.6194 (2)	9.1 (1)
O6*B*	0.0550 (3)	0.4090 (4)	1.0042 (2)	6.17 (6)
N1*B*	0.5601 (3)	0.3022 (4)	0.7021 (2)	5.32 (7)
C1*B*	0.3214 (3)	0.0551 (4)	0.8366 (2)	3.49 (5)
C2*B*	0.4518 (3)	0.1104 (5)	0.8873 (2)	4.35 (7)
C3*B*	0.5298 (3)	0.1897 (5)	0.8434 (2)	4.66 (7)
C4*B*	0.4763 (3)	0.2154 (4)	0.7491 (2)	4.10 (6)
C5*B*	0.3468 (3)	0.1622 (4)	0.6977 (2)	4.43 (7)
C6*B*	0.2681 (3)	0.0806 (4)	0.7415 (2)	4.26 (6)
C7*B*	0.0611 (3)	0.2106 (4)	0.8863 (2)	4.15 (6)
C8*B*	0.0935 (3)	0.3803 (4)	0.9227 (2)	4.05 (6)
C9*B*	−0.0221 (4)	0.4858 (5)	0.9182 (3)	5.94 (9)
C10*B*	0.2297 (4)	0.4476 (5)	0.9269 (3)	5.90 (9)

Table 2. *Bond distances* (Å), *angles* (°) *and selected torsion angles* (°) *for molecules* (*A*) *and* (*B*)

	(*A*)	(*B*)
S1—O1	1.423 (2)	1.421 (2)
S1—O2	1.418 (2)	1.415 (2)
S1—O3	1.572 (3)	1.573 (2)
S1—C1	1.766 (3)	1.762 (3)
O3—C7	1.462 (4)	1.458 (3)
O4—N1	1.198 (4)	1.196 (4)
O5—N1	1.203 (4)	1.200 (4)
O6—C8	1.432 (4)	1.443 (4)
O6—C9	1.445 (6)	1.441 (4)
N1—C4	1.474 (5)	1.470 (5)
C1—C2	1.378 (4)	1.388 (4)
C1—C6	1.390 (4)	1.394 (4)
C2—C3	1.374 (5)	1.364 (5)
C3—C4	1.371 (4)	1.382 (4)
C4—C5	1.381 (4)	1.377 (4)
C5—C6	1.370 (5)	1.376 (5)
C7—C8	1.492 (5)	1.491 (5)
C8—C9	1.447 (5)	1.449 (5)
C8—C10	1.507 (5)	1.484 (5)
O1—S1—O2	120.4 (1)	120.4 (1)
O1—S1—O3	110.6 (1)	110.3 (1)
O1—S1—C1	108.6 (1)	108.7 (1)
O2—S1—O3	104.0 (1)	104.4 (1)
O2—S1—C1	108.8 (2)	109.3 (1)
O3—S1—C1	103.0 (1)	102.3 (1)
S1—O3—C7	118.8 (2)	119.3 (2)
C8—O6—C9	60.4 (2)	60.3 (2)
O4—N1—O5	122.6 (4)	123.0 (4)
O4—N1—C4	118.1 (3)	118.6 (3)
O5—N1—C4	119.2 (3)	118.4 (3)
S1—C1—C2	119.3 (2)	119.0 (2)
S1—C1—C6	119.7 (2)	119.9 (2)
C2—C1—C6	120.9 (3)	121.1 (3)
C1—C2—C3	119.8 (3)	119.5 (3)
C2—C3—C4	118.4 (3)	119.2 (3)
N1—C4—C3	119.0 (3)	119.1 (2)
N1—C4—C5	118.1 (3)	118.8 (3)
C3—C4—C5	122.9 (3)	122.2 (3)
C4—C5—C6	118.3 (3)	118.9 (3)
C1—C6—C5	119.6 (3)	119.1 (2)
O3—C7—C8	110.2 (2)	107.3 (2)
O6—C8—C7	116.0 (3)	112.6 (3)
O6—C8—C9	60.2 (2)	59.8 (2)
O6—C8—C10	114.3 (3)	114.8 (3)
C7—C8—C9	122.5 (3)	117.1 (3)
C7—C8—C10	112.5 (3)	117.0 (3)
C9—C8—C10	120.4 (3)	121.8 (3)
O6—C9—C8	59.4 (2)	59.9 (2)
C1—S1—O3—C7	−80.9 (2)	76.3 (2)
O3—S1—C1—C2	−79.3 (3)	75.4 (3)
S1—O3—C7—C8	133.2 (2)	−143.9 (2)
O4—N1—C4—C3	1.2 (5)	−2.5 (5)
O5—N1—C4—C3	−176.4 (4)	174.4 (4)
O3—C7—C8—O6	46.8 (4)	−80.4 (3)
O3—C7—C8—C9	−23.0 (4)	−146.8 (3)
O3—C7—C8—C10	−179.0 (3)	55.8 (4)

tropically; H atoms located by ΔF and refined isotropically.

The function minimized was $\sum w(|F_o| - |F_c|)^2$ and weights were assigned as $w = 4F_o^2\mathrm{Lp}[S^2(C + R^2B) + (0.02F_o^2)^2]^{-1}$, where S = scan rate, C = total integrated peak count, R = scan time/background counting time, B = total background count, Lp = Lorentz–polarization factor, using Enraf–Nonius *SDP* (Frenz & Okaya, 1980), scattering factors of Cromer & Waber (1974), anomalous coefficients of Cromer (1974). Of 4798 unique data, 4453 reflections having $I > 3\sigma(I)$ were used in the refinement. Final $R = 0.046$ (0.050 for all data), $wR = 0.064$, $S = 3.438$ for 413 variables. Maximum shift 0.10σ in the final cycle; maximum residual density 0.38, minimum -0.32 e Å^{-3}. The mirror-image (*S*)-isomer was refined under identical conditions, yielding $R = 0.056$, $wR = 0.080$, $S = 4.307$, thus the (*R*)-isomer is confirmed. Table 1 presents the final coordinates* and equivalent isotropic thermal parameters, and Table 2 presents bond distances, angles and selected torsion angles. Fig. 1 illustrates the two independent molecules and the numbering scheme, and Fig. 2 shows the unit cell.

Related literature. Structure of *p*-nitrophenylsulfonic acid–(7-*exo*-isopropenylbicyclo[4.1.0]hept-7-*endo*-yl) ester: Seebach, Dammann, Lindner & Kitschke (1979). Preparation of glycidyl arenesulfonate: Klunder, Onami & Sharpless (1989).

The National Institutes of Health supported this work through grant GM42016.

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* Tables of H-atom coordinates, bond distances and angles involving H atoms, torsion angles, anisotropic thermal parameters, least-squares planes, and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54657 (33 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: ST0535]

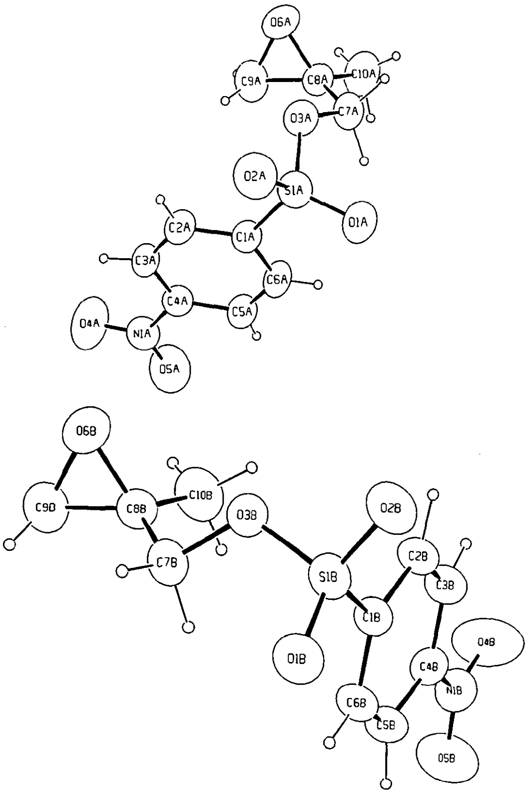

Fig. 1. Numbering scheme and thermal ellipsoids drawn at the 40% probability level for the two independent molecules. H atoms are drawn as circles of arbitrary radius.

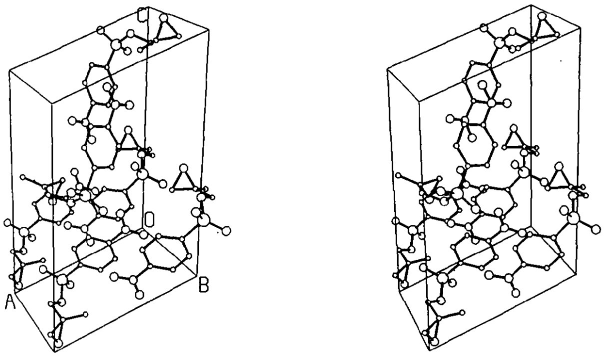

Fig. 2. Stereoview of the unit cell, illustrating the pseudoracemic nature of the packing.

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Structure of 1-Acetylindole-2,3-dione

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Abstract. $C_{10}H_7NO_3$, $M_r = 189{\cdot}17$, monoclinic, $P2_1/n$, $a = 8{\cdot}892\,(2)$, $b = 5{\cdot}108\,(2)$, $c = 18{\cdot}573\,(3)$ Å, $\beta = 98{\cdot}22\,(2)^\circ$, $V = 834{\cdot}8\,(7)$ Å^3, $Z = 4$, $D_x = 1{\cdot}505$ g cm^{-3}, $\lambda(\text{Mo }K\alpha) = 0{\cdot}71073$ Å, $\mu = 1{\cdot}06$ cm^{-1}, $F(000) = 392$, $T = 298$ K, $R = 0{\cdot}038$ for 1488 observed reflections. The heterocyclic ring is essentially planar making a dihedral angle of 7·9 (6)° with the acetyl moiety. The C(2)—C(3) distance is 1·538 (2) Å.

Experimental. Crystals of the title compound were obtained from acetic anhydride at 298 K. The data collection and refinement parameters are summarized in Table 1.

The structure was solved using standard direct methods and difference Fourier techniques. In final cycles of full-matrix least-squares refinement all non-H atoms were treated anisotropically, the H atoms were refined isotropically.

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