

Les distances et angles peuvent servir de référence pour toute molécule possédant un noyau dioxapyrène, la structure de cette molécule n'ayant, à notre connaissance, jamais été déterminée. Elles sont différentes de celles du pyrène (Hazell, Larsen & Lehmann, 1972), en ce qui concerne le cycle naphthalène central qu'ils ont en commun.

Le caractère éthylénique renforcé des doubles liaisons hétérocycliques [C(2)—C(3), C'(2)—C'(3)] est en excellent accord avec les paramètres RMN ^1H et ^{13}C , qui concernent, entre autre, le déplacement anormalement élevé (*ca* 2 p.p.m.) des protons situés en α et β des hétéroatomes vers les champs forts [hydrogènes H_2 et H_3 portés par atomes C(2) et C(3)].

La plupart des angles de valence s'écartent notablement de 120° , contrairement à ce qui est observé pour le pyrène. C'est le cas notamment des angles C(3)—C(4)—C(5) proches de 115° et de l'angle O(1)—C(2)—C(3) égal à 125° . La molécule est parfaitement plane comme pour le pyrène, les angles de torsion étant tous proches de 180° .

Le recouvrement des molécules en x , y , z et x , $1 + y$, z dont les plans moyens sont à 3,50 Å est semblable à celui habituellement observé pour des molécules polyaromatiques: triphénylène (Cotrait, Marsau, Destrade & Malthete, 1979), benzpérylène (Trotter, 1958), coronène (Robertson & White, 1947), *etc.*

La cohésion cristalline est essentiellement assurée par les forces de stacking suivant la direction $0y$ et

des interactions de van der Waals entre molécules voisines, suivant les deux autres directions.

Les charges électriques ponctuelles partielles, exprimées en électrons, ont été calculées grâce au logiciel *MOPAC* (Stewart, 1988, 1989) et sont données dans le Tableau 1 pour les atomes C et O; ils sont inférieurs à 0,01 e pour les hydrogènes.

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Structure of 2-(Fluoromethyl)-2-[(*p*-tolylsulfinyl)methyl]oxirane

BY GIANCARLO FABRIZI* AND WALTER FEDELI

Dipartimento di Chimica, Ingegneria Chimica e Materiali, Università dell'Aquila, I-67100 L'Aquila, Italy

AND DORIANO LAMBA

Istituto di Strutturistica Chimica Giordano Giacomello, CNR, Area della Ricerca di Roma, CP No. 10, I-00016 Monterotondo Stazione, Roma, Italy

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Abstract. $\text{C}_{11}\text{H}_{13}\text{FO}_2\text{S}$, $M_r = 228.28$, orthorhombic, $P2_12_12_1$, $a = 4.762$ (1), $b = 11.196$ (4), $c = 21.421$ (7) Å, $V = 1142.1$ Å³, $Z = 4$, $D_x = 1.328$ Mg m⁻³, $\lambda(\text{Cu } K\alpha) = 1.54184$ Å, $\mu = 2.43$ mm⁻¹, $F(000) = 480$, $T = 298$ K, $R = 0.061$ for 794 reflections with $F_o \geq 4\sigma(F_o)$. The absolute con-

figuration of the new chiral centre C(2) is *R*. The conformation around the central S(1)—C(21) bond is *transoid* with the torsion angle C(2)—C(21)—S(1)—C(22), defining the orientation of the phenyl ring and the fluoromethyloxirane moiety, -178.3 (5)°.

Experimental. As part of a program directed toward the development of an asymmetric approach to

* Author to whom correspondence should be addressed.

Table 1. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement coefficients ($\text{\AA}^2 \times 10^3$)

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

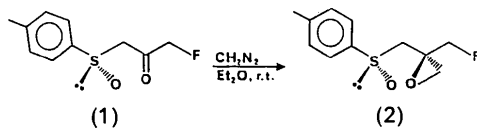
	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
S(1)	-61 (4)	-3487 (2)	-2760 (1)	63 (1)
F(1)	-3450 (15)	-4742 (5)	-4455 (3)	118 (3)
O(1)	-2139 (11)	-2144 (5)	-4393 (2)	66 (2)
O(2)	820 (15)	-4737 (5)	-2871 (3)	93 (2)
C(2)	-1183 (16)	-3113 (5)	-3995 (3)	55 (2)
C(3)	655 (16)	-2116 (7)	-4142 (3)	73 (3)
C(20)	-819 (20)	-4252 (7)	-4324 (4)	79 (3)
C(21)	-2607 (14)	-3142 (6)	-3355 (3)	58 (2)
C(22)	-2477 (14)	-3483 (5)	-2119 (3)	54 (2)
C(23)	-3493 (18)	-4531 (6)	-1895 (3)	66 (3)
C(24)	-5379 (18)	-4492 (7)	-1385 (3)	72 (3)
C(25)	-6131 (16)	-3446 (7)	-1121 (3)	64 (2)
C(26)	-5106 (20)	-2393 (6)	-1356 (3)	73 (3)
C(27)	-3224 (17)	-2396 (6)	-1852 (3)	64 (2)
C(30)	-8137 (20)	-3427 (8)	-570 (4)	95 (3)

Table 2. Bond lengths (\AA), valence angles ($^\circ$) and geometrical parameters of selected intermolecular interactions (\AA , $^\circ$)

S(1)—O(2)	1.481 (6)	S(1)—C(21)	1.801 (7)	
S(1)—C(22)	1.791 (6)	F(1)—C(20)	1.396 (12)	
O(1)—C(2)	1.453 (8)	O(1)—C(3)	1.435 (9)	
C(2)—C(3)	1.453 (10)	C(2)—C(20)	1.467 (10)	
C(2)—C(21)	1.531 (9)	C(22)—C(23)	1.356 (9)	
C(22)—C(27)	1.391 (9)	C(23)—C(24)	1.415 (11)	
C(24)—C(25)	1.349 (11)	C(25)—C(26)	1.371 (11)	
C(25)—C(30)	1.519 (11)	C(26)—C(27)	1.390 (11)	
O(2)—S(1)—C(21)	106.2 (3)	O(2)—S(1)—C(22)	107.9 (3)	
C(21)—S(1)—C(22)	96.3 (3)	C(2)—O(1)—C(3)	60.4 (5)	
O(1)—C(2)—C(3)	59.2 (4)	O(1)—C(2)—C(20)	113.9 (6)	
C(3)—C(2)—C(20)	119.5 (7)	O(1)—C(2)—C(21)	113.7 (5)	
C(3)—C(2)—C(21)	118.5 (6)	C(20)—C(2)—C(21)	117.7 (6)	
O(1)—C(3)—C(2)	60.4 (5)	F(1)—C(20)—C(2)	109.4 (7)	
S(1)—C(21)—C(2)	109.9 (5)	S(1)—C(22)—C(23)	119.8 (5)	
S(1)—C(22)—C(27)	118.8 (5)	C(23)—C(22)—C(27)	121.4 (6)	
C(22)—C(23)—C(24)	118.2 (6)	C(23)—C(24)—C(25)	121.3 (7)	
C(24)—C(25)—C(26)	119.8 (7)	C(24)—C(25)—C(30)	120.4 (7)	
C(26)—C(25)—C(30)	119.8 (7)	C(25)—C(26)—C(27)	120.5 (7)	
C(22)—C(27)—C(26)	118.8 (6)			
<i>D</i> — <i>H</i> ... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> — <i>H</i>	<i>H</i> ... <i>A</i>	<i>D</i> — <i>H</i> ... <i>A</i>
C(27)—H(27A)...O(2)	3.244 (9)	0.960 (11)	2.305 (9)	165.8 (7)
C(26)—H(26A)...F(1 ^b)	3.506 (9)	0.960 (11)	2.562 (9)	167.8 (7)

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

fluoro-substituted organic molecules (Bravo & Resnati, 1990; Cavicchio, Marchetti, Arnone, Bravo & Viani, 1990), an efficient transfer of chirality from S to the central C atom was observed when (*S*)-(–)-1-fluoro-3-(*p*-tolylsulfinyl)propan-2-one (1) was reacted with diazomethane (Cavicchio, Bravo, Marchetti & Zappalá, 1991). Here we report on the crystal and molecular structure of 2-(fluoromethyl)-2-[(*p*-tolylsulfinyl)methyl]oxirane (2), obtained in good yield and with high diastereomeric excess, in order to determine the absolute configuration of the new chiral centre at C(2).



Transparent prismatic crystals of the title compound were obtained by slow evaporation of an ethyl acetate solution; dimensions $0.48 \times 0.10 \times 0.08$ mm. Siemens *R3m/V* four-circle diffractometer, graphite-monochromated Cu *K* α radiation; $2\theta_{\text{max}} = 130^\circ$ (*hkl* range: *h* 0 to 6, *k* 0 to 13, *l* 0 to 24), ω - 2θ scan. Cell parameters by least squares on 24 reflections ($45 \leq 2\theta \leq 67^\circ$). No significant intensity variation for the three check reflections 200, 002, 021, monitored every 100 reflections. Lorentz and polarization corrections; analytical absorption correction, minimum and maximum transmission factors 0.86 and 0.99; no extinction correction. Total of 1182 reflections, 1116 unique reflections ($R_{\text{int}} = 0.008$), 794 with $F_o \geq 4\sigma(F_o)$. Structure solved by direct methods using *SHELXTL-Plus* (Sheldrick, 1990). H atoms in idealized positions. Anisotropic least-squares refinement using *F*; H atoms isotropic with common refined temperature factor. $w = 1/\sigma^2(F_o) + 0.0077F_o^2$. $R = 0.061$, $wR = 0.083$, $S = 0.86$ and the maximum shift/e.s.d. = 0.55 [*z* of C(30)]. Final Fourier synthesis was featureless with $-0.47 \leq \Delta\rho \leq 0.26 \text{ e \AA}^{-3}$. The absolute configuration *R* at C(2) was established on the basis of the known configuration *S* at S(1). Atomic scattering factors

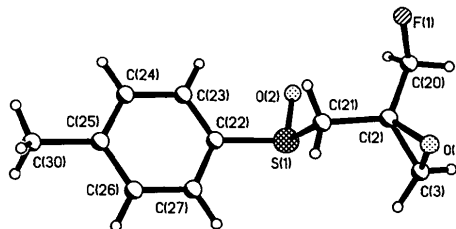
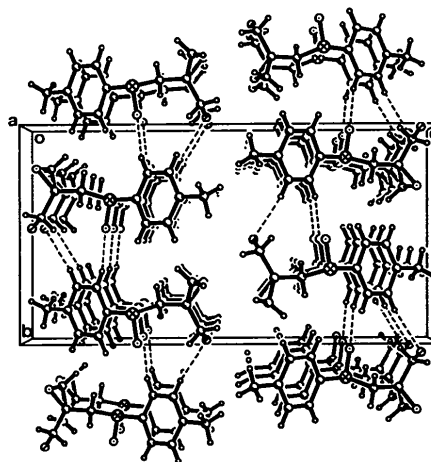


Fig. 1. Structure and conformation of the title compound with numbering of the atoms.

Fig. 2. The crystal packing of the molecules, projected along the *a* axis.

were from *International Tables for X-ray Crystallography* (1974, Vol. IV). The *PARST* program (Nardelli, 1983) was used for molecular geometry calculations. The atomic parameters are given in Table 1.* The bond lengths, valence angles and geometrical parameters of selected intermolecular interactions are given in Table 2. A perspective view of the title compound is given in Fig. 1. The crystal packing along the *a* axis is shown in Fig. 2.

Related literature. The geometry at S(1) conforms to that found for related *p*-tolylsulfinyl derivatives (de la Camp & Hope, 1970; Hua, Badejo, McCann & Takusagawa, 1987). In the epoxidic ring the C(2)—O(1) and C(3)—O(1) bond lengths of 1.453 (8) and 1.435 (9) Å, respectively, are close to the accepted value of 1.446 (14) Å for C(sp³)—O bonds in epoxides with any substitution (Allen, Kennard,

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54857 (7 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: GE0292]

Watson, Brammer, Orpen & Taylor, 1987). In the structure two short contacts, C—H...O and C—H...F, exist, which can be reasonably described as hydrogen bonds (Taylor & Kennard, 1982).

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Structure of 4,6-Diacetylresorcinol

BY M. K. KOKILA, K. A. NIRMALA AND PUTTARAJA

Department of Physics, Bangalore University, Bangalore-560 056, India

AND N. SHAMALA

Department of Physics, Indian Institute of Science, Bangalore-560 012, India

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Abstract. C₁₀H₁₀O₄, *M_r* = 194.19, monoclinic, *P*2₁/*c*, *a* = 7.089 (1), *b* = 11.361 (1), *c* = 11.656 (1) Å, β = 100.45 (3)°, *V* = 922.92 (1) Å³, *Z* = 4, *D_m* = 1.410 (5), *D_x* = 1.397 Mg m⁻³, λ(Cu Kα) = 1.5418 Å, μ(Cu Kα) = 0.89 mm⁻¹, *T* = 300 K, *F*(000) = 408, final *R* = 0.057 for 1701 observed reflections. The molecule is almost planar, with O(9) and O(12) of the acetyl groups deviating by 0.074 (1) and 0.071 (2) Å from the mean plane of the benzene ring. The bond lengths and bond angles of the benzene ring are normal. There are intramolecular hydrogen bonds between O(9) and H(14) and between O(12) and H(13); there are no intermolecular hydrogen

bonds. The molecules are packed in layers parallel to the *ac* plane and are held together essentially by van der Waals interactions.

Experimental. The title compound is obtained by the reaction of acetic anhydride and resorcinol in the presence of ZnCl₂; crystals suitable for X-ray analysis were grown in benzene solution by slow evaporation; orange crystal 0.3 × 0.4 × 0.2 mm mounted on an Enraf–Nonius CAD-4 diffractometer, ω/2θ scan; cell dimensions from least-squares refinement of 25 reflections in the 2θ range 0 to 55°; density measurements by flotation technique using potassium iodide