The Crystal Structure of Methyl-6-deoxy-6-methylsulphinyl-α-D-glucopyranoside

BÖRJE LINDBERG and
PEDER KIERKEGAARD

Institute for Inorganic and Physical Chemistry, University of Stockholm, Box 6801, S-113 86 Stockholm, Sweden

The crystal structure of methyl-6-deoxy-6-methylsulphinyl-α-D-glucopyranoside, $C_8H_{16}O_6S$, has been determined from three-dimensional single-crystal X-ray film data. The coordinates of the nonhydrogen atoms derived from Patterson and Fourier syntheses were refined by least-squares techniques with isotropic temperature factors.

The following data were obtained for the trigonal structure:

Unit cell dimensions (from a Guinier-Hägg powder photograph taken with $CuK\alpha_1$ radiation at 25°C): $a = 12.2364 \pm 2$ (Å), $c = 6.3260 \pm 2$ (Å).

Cell content: 3 $C_8H_{16}O_6S$ (observed density 1.42 g/cm³, calculated density 1.46 g/cm³). Space group: $P3_2$ (No. 145).

Arrangements of atoms (*cf.* Table 1): All atoms in point position 3(*a*): (x,y,z); $(\bar{y}, x-y, \frac{2}{3}+z)$; $(y-x, \bar{x}, \frac{1}{3}+z)$.

The discrepancy factor, R, is 10.0 % (calculated for all 356 reflections, $hk0-hk3$, registered with $CuK\alpha$ radiation).

Table 2. Bond lengths and angles.

C(1)−C(2)	1.55	C(1)−C(2)−C(3)	109
C(2)−C(3)	1.53	C(2)−C(3)−C(4)	108
C(3)−C(4)	1.54	C(3)−C(4)−C(5)	104
C(4)−C(5)	1.54	C(4)−C(5)−O(5)	110
C(5)−O(5)	1.42	C(5)−O(5)−C(1)	111
C(5)−C(6)	1.56	O(5)−C(1)−C(2)	106
C(1)−O(5)	1.42	O(5)−C(1)−O(1)	116
C(1)−O(1)	1.40	C(1)−O(1)−C(7)	107
O(1)−C(7)	1.46	O(1)−C(1)−C(2)	109
C(2)−O(2)	1.47	C(1)−C(2)−O(2)	110
C(3)−O(3)	1.45	O(2)−C(2)−C(3)	107
C(4)−O(4)	1.46	C(2)−C(3)−O(3)	107
S −C(6)	1.82	O(3)−C(3)−C(4)	109
S −C(8)	1.72	C(3)−C(4)−O(4)	107
S −O(6)	1.54	O(4)−C(4)−C(5)	110
		C(4)−C(5)−C(6)	107
		C(6)−C(5)−O(5)	101
		C(5)−C(6)−S	111
		C(6)−S −C(8)	101
		C(6)−S −O(6)	106
		C(8)−S −O(6)	111

The crystals are built up of $C_8H_{16}O_6S$ molecules (*cf.* Fig. 1) with the absolute configuration found to be S[1] which is in agreement with the conclusions by Lindberg and Lundström[2] from ORD data. Some intermolecular $O(H)−O$ contacts likely to correspond to hydrogen bonds are present in the structure so the molecules are held together in addition to van der Waals interactions by hydrogen bonds.

Table 2 shows bond lengths ($\sigma \approx \pm 0.03$ Å) and bond angles ($\sigma \approx \pm 2°$), uncorrected

Table 1. Atomic parameters and standard deviations obtained by full matrix least-squares refinement with isotropic temperature factors.

Atom	x		y		z		B	
S	0.7205	± 6	0.8600	± 6	2/3 [a]		5.5	±0.2
O(1)	0.5154	16	0.6407	15	0.2631	±34	5.3	0.4
O(2)	0.4078	16	0.3820	17	0.3200	33	5.9	0.4
O(3)	0.6100	14	0.3567	13	0.1814	30	4.3	0.3
O(4)	0.8148	16	0.5742	15	0.9840	34	5.4	0.6
O(5)	0.5411	13	0.6181	13	0.8942	30	3.9	0.3
O(6)	0.7813	16	0.9404	17	0.8653	35	5.8	0.4
C(1)	0.4708	20	0.5645	20	0.0827	44	3.8	0.5
C(2)	0.4747	21	0.4420	21	0.1234	50	4.8	0.5
C(3)	0.6115	19	0.4741	21	0.1517	43	4.3	0.5
C(4)	0.6833	21	0.5368	21	0.9465	47	4.3	0.5
C(5)	0.6699	20	0.6551	20	0.9249	48	4.0	0.5
C(6)	0.7264	24	0.7154	25	0.7050	54	5.9	0.7
C(7)	0.4971	24	0.7485	24	0.2290	47	5.4	0.6
C(8)	0.8191	26	0.9232	25	0.4519	50	6.1	0.6

[a] Arbitrarily set to 2/3.

for thermal motion, and their standard deviations.

A schematic drawing showing a projection of the structure along the c axis is given in Fig. 1.
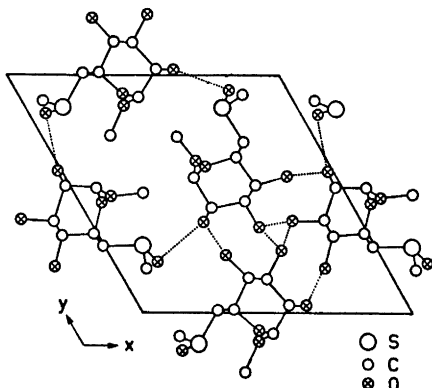

Fig. 1. The structure of methyl-6-deoxy-6-methylsulfinyl-α-D-glucopyranoside. Schematic drawing showing the xy projection. Dashed lines indicate probable system of hydrogen bonds.

Full details of this structure investigation and a discussion of the results will be given elsewhere.

1. Cahn, R. S., Ingold, C. K. and Prelog, V. *Experientia* **12** (1956) 81.
2. Lindberg, B. and Lundström, H. *Acta Chem. Scand.* **22** (1968) 1861.

The Valence Electron Density Distribution of Strained Single Bonds in the Iterative Extended Hückel Approach

V. Tricyclo[1.1.0.0²,⁴]butane (Tetrahedrane)

OLLE MÅRTENSSON

Quantum Chemistry Group,* Uppsala University, Box 518, S-751 20 Uppsala, Sweden

Tricyclo[1.1.0 ²,⁴]butane (tetrahedrane) (Fig. 1) is considered to form the most strained system of the hydrocarbons built up from three-membered rings. One would also expect this, since the angular geometry of tetrahedrane is completely determined by the high symmetry and there are no angular degrees of freedom available for a minimizing of the energy. By contrast, bicyclo[1.0.1]-butane, for instance, has several angular parameters available for this purpose. The bridgehead $C-C-H$ bond is here by Haller and Srinivasan[1] estimated to have a classical valence angle
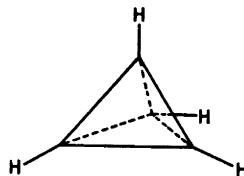

Fig. 1. The valence skeleton of tricyclobutane (tetrahedrane).

of 163° as compared to 144°44′ for the $C-C-H$ bond in tetrahedrane, a value fixed by symmetry. The angle between the triangular planes in bicyclobutane is estimated to be 126° as compared to the symmetry-fixed value of 70°32′ in tetrahedrane. We have also the possibility of varying the conformation of the methylene groups in bicyclobutane, but this is probably of minor importance in this context.