

PhMeSiH and also in (1-naphthylphenylmethylsilyl)-benzyl *p*-bromobenzoate (Nyburg, Brook, Pascoe & Szymański, 1972), particularly in the relative shortness of C(13)–C(14). However, the slight distortions from planarity seen in the naphthyl groups of the latter two molecules are not observed here. It can also be seen that the Si and P atoms are not exactly in the plane of their attached phenyl rings.

There are two short intramolecular Pt···H contact distances, one of 2.72 Å to H(7) on the naphthyl group and one of 2.78 Å to H(18) on the phenyl ring of P(2). Since the three large ligands in the coordination plane cause slight distortions from regular square-planar geometry to accommodate them, it is probable that their conformation is decided primarily by intramolecular rather than intermolecular contacts. If this is so, one might expect that the two phenyl rings would be in equivalent positions related by the approximate mirror plane through C(1), Si, Pt and Cl on the underside of the molecule. The fact that they are not may indicate that the rotation of the phenyl ring on P(2) so as to make a shorter contact of the *ortho*-hydrogen with the Pt is due to an attractive interaction, albeit very slight. Similar contacts have been observed in other square-planar complexes of  $d^8$  transition metals (Bennett, Donaldson, Hitchcock & Mason), and in linear Pd<sup>0</sup> bis-phosphine complexes M···H contacts of 2.7 Å are assumed to be essentially bonding (Immirzi & Musco, 1974; Matsumoto, Yoshioka, Nakatsu, Yoshida & Otsuka, 1974). The shape of the naphthyl group is such as to bring H(7) easily into an approximately octahedral site, and the two Pt···H contacts thus complete approximate octahedral coordination of the Pt atom.

All intermolecular contacts out to 4.0 Å have been calculated and there are none significantly shorter than the sum of the van der Waals radii, the shortest non-hydrogen contacts being C···C 3.6 and C···Cl 3.7 Å.

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### Methyl Tri-*O*-acetyl-6-deoxy-6-methylsulphinyl(*S*)- $\alpha$ -D-glucopyranoside

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C<sub>14</sub>H<sub>22</sub>O<sub>9</sub>S, orthorhombic,  $P2_12_1$ ,  $a = 14.559$  (2),  $b = 21.734$  (3),  $c = 5.6928$  (9) Å,  $Z = 4$ . The structure was solved by direct methods with 1599 single-crystal diffractometer data. The sulphur atom is in the *S* configuration. There is no hydrogen bonding in the structure.

#### Experimental

The cell dimensions were obtained from a powder photograph at 20°C, taken in a Guinier–Hägg focusing camera with highly monochromatized Cu  $K\alpha_1$  radiation ( $\lambda = 1.54051$  Å) and KCl ( $a = 6.29194$  Å, Hambling, 1953) as an internal standard. A prismatic crystal was

mounted on a goniometer head with *c* along the spindle axis. Three-dimensional data were collected on a Siemens AED single-crystal diffractometer with graphite-monochromatized Cu  $K\alpha$  radiation and a scintillation detector. The 1716 available independent data with  $\theta < 63^\circ$  were collected with  $\theta$ – $2\theta$  scans of  $2^\circ$  scan width; stationary background measurements were

taken on each side. The 1599 data with  $\sigma(I_{\text{net}})/I_{\text{net}} < 0.25$  were used in the subsequent calculations. The calculations of  $\sigma(I_{\text{net}})$  were based on conventional counter statistics. Lorentz and polarization factors were applied, but not absorption corrections. The crystal structure was solved by a computerized application of direct methods (Norrestam, 1972). Anomalous disper-

sion corrections for sulphur and successive cycles of anisotropic full-matrix least-squares refinement gave  $R$  values of 0.056 for the included data, and 0.063 for the 1716 measured data.\* The positional parameters

\* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31603 (3 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 1. *Fractional atomic coordinates of the nonhydrogen atoms*

The estimated standard deviations are given in parentheses.

	<i>x</i>	<i>y</i>	<i>z</i>
S	0.92439 (10)	0.51537 (6)	0.9055 (3)
O(1)	1.14195 (26)	0.45793 (16)	0.4009 (10)
O(2)	1.22276 (19)	0.34379 (15)	0.3532 (6)
O(22)	1.32449 (25)	0.33423 (24)	0.6444 (8)
O(3)	1.06016 (23)	0.26951 (12)	0.3810 (5)
O(32)	1.05673 (38)	0.27327 (18)	-0.0111 (6)
O(4)	0.90067 (18)	0.35125 (14)	0.3799 (6)
O(42)	0.80560 (25)	0.31304 (20)	0.6517 (7)
O(5)	1.07125 (21)	0.42891 (15)	0.7517 (6)
O(S)	0.89366 (34)	0.55097 (18)	0.6988 (9)
C(1)	1.14718 (33)	0.42113 (27)	0.5960 (14)
C(2)	1.15036 (30)	0.35382 (21)	0.5228 (9)
C(21)	1.30821 (33)	0.33505 (23)	0.4384 (10)
C(22)	1.37678 (41)	0.32738 (31)	0.2469 (13)
C(3)	1.06150 (30)	0.33681 (17)	0.4009 (8)
C(31)	1.05987 (32)	0.24436 (20)	0.1656 (8)
C(32)	1.06200 (44)	0.17539 (23)	0.1854 (11)
C(4)	0.97729 (28)	0.35462 (20)	0.5423 (8)
C(41)	0.81976 (31)	0.32679 (22)	0.4530 (10)
C(42)	0.75366 (39)	0.32090 (31)	0.2539 (12)
C(5)	0.98425 (27)	0.42070 (20)	0.6376 (9)
C(6)	0.91401 (33)	0.43467 (21)	0.8268 (10)
C(7)	1.14296 (66)	0.52334 (30)	0.4581 (28)
C(S)	0.83567 (45)	0.51644 (31)	1.1200 (12)

Table 3. *Hydrogen fractional atomic coordinates and assigned temperature factors*

The estimated standard deviations are given in parentheses.

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i>
H(1)	1.201 (3)	0.429 (2)	0.734 (10)	4.73
H(2)	1.160 (3)	0.328 (2)	0.660 (9)	3.41
H(3)	1.056 (3)	0.354 (2)	0.255 (8)	3.01
H(4)	0.963 (3)	0.324 (2)	0.674 (8)	2.95
H(5)	0.977 (3)	0.452 (2)	0.499 (8)	2.87
H(6)	0.932 (3)	0.411 (2)	0.975 (8)	3.50
H(6')	0.852 (3)	0.429 (2)	0.771 (9)	3.50
H(7)	1.205 (4)	0.525 (3)	0.529 (13)	7.07
H(7')	1.139 (5)	0.545 (3)	0.276 (13)	7.07
H(7'')	1.093 (4)	0.540 (3)	0.580 (12)	7.07
H(22)	1.353 (4)	0.338 (2)	0.073 (10)	4.68
H(22')	1.392 (3)	0.270 (2)	0.213 (10)	4.68
H(22'')	1.421 (4)	0.316 (3)	0.301 (10)	4.68
H(32)	1.116 (3)	0.163 (2)	0.216 (10)	4.35
H(32')	1.008 (3)	0.162 (2)	0.297 (10)	4.35
H(32'')	1.074 (4)	0.154 (2)	0.021 (9)	4.35
H(42)	0.730 (3)	0.357 (2)	0.220 (10)	3.81
H(42')	0.783 (3)	0.302 (2)	0.144 (10)	3.81
H(42'')	0.694 (3)	0.293 (2)	0.298 (9)	3.81
H(S)	0.809 (4)	0.567 (3)	1.115 (11)	5.98
H(S')	0.769 (4)	0.498 (3)	1.047 (11)	5.98
H(S'')	0.851 (4)	0.487 (3)	1.280 (11)	5.98

Table 2. *Anisotropic thermal parameters of the nonhydrogen atoms*

The estimated standard deviations are given in parentheses. The temperature factor expression used is

$$\exp [-(h^2\beta_{11} + k^2\beta_{22} + l^2\beta_{33} + hk\beta_{12} + hl\beta_{13} + kl\beta_{23})].$$

	$\beta_{11}$ ( $\times 10^5$ )	$\beta_{22}$ ( $\times 10^5$ )	$\beta_{33}$ ( $\times 10^4$ )	$\beta_{12}$ ( $\times 10^5$ )	$\beta_{13}$ ( $\times 10^4$ )	$\beta_{23}$ ( $\times 10^4$ )
S	539 (7)	199 (3)	417 (6)	29 (8)	78 (4)	-59 (2)
O(1)	653 (23)	176 (8)	753 (24)	-176 (22)	200 (15)	0 (8)
O(2)	335 (15)	247 (8)	270 (12)	152 (18)	6 (7)	-20 (6)
O(22)	385 (18)	546 (17)	364 (17)	173 (28)	-30 (9)	-47 (9)
O(3)	503 (17)	142 (6)	214 (10)	71 (18)	-16 (9)	-9 (4)
O(32)	1117 (35)	294 (10)	221 (12)	38 (34)	-50 (12)	-7 (6)
O(4)	303 (14)	219 (7)	215 (10)	-8 (16)	-19 (7)	23 (5)
O(42)	499 (20)	353 (11)	281 (14)	-199 (24)	30 (9)	21 (7)
O(5)	315 (14)	254 (8)	378 (13)	21 (19)	-15 (9)	-77 (6)
O(S)	1213 (35)	223 (9)	507 (19)	261 (30)	134 (15)	59 (7)
C(1)	294 (21)	278 (14)	586 (31)	-18 (29)	35 (16)	-121 (13)
C(2)	314 (21)	194 (11)	287 (18)	79 (25)	23 (11)	-24 (8)
C(21)	360 (23)	224 (12)	299 (20)	135 (27)	-16 (12)	-11 (8)
C(22)	476 (32)	329 (18)	402 (25)	284 (37)	57 (16)	27 (11)
C(3)	340 (19)	137 (8)	219 (14)	58 (23)	-19 (11)	1 (6)
C(31)	366 (21)	202 (10)	255 (16)	-20 (27)	-8 (11)	-31 (7)
C(32)	525 (28)	195 (11)	426 (23)	108 (33)	-47 (16)	-53 (9)
C(4)	314 (20)	170 (10)	206 (15)	87 (23)	-25 (9)	-15 (7)
C(41)	344 (22)	176 (11)	299 (20)	-10 (25)	-5 (11)	4 (8)
C(42)	420 (28)	287 (16)	331 (22)	16 (34)	-38 (13)	-17 (10)
C(5)	282 (18)	162 (9)	262 (16)	44 (22)	11 (10)	-19 (8)
C(6)	420 (25)	176 (10)	316 (19)	98 (26)	37 (12)	-27 (8)
C(7)	971 (50)	191 (14)	1687 (100)	-219 (49)	504 (44)	-32 (20)
C(S)	725 (37)	332 (17)	378 (24)	188 (42)	93 (17)	-48 (12)

of the hydrogen atoms were refined with each hydrogen given the approximate isotropic temperature factor of the atom to which it is bonded.

Hughes's (1941) weighting scheme was used ( $F_{\min} = 8.0$ ). For sulphur, carbon and oxygen, the atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974), and for hydrogen

from Stewart, Davidson & Simpson (1965). The final structural parameters are listed in Tables 1, 2 and 3. The atomic labels are given in Fig. 1. Intramolecular distances and angles are listed in Table 4.

### Discussion

The pyranose ring has the normal  ${}^4C_1$  conformation (Table 6). The ring torsion angles, ranging from  $46.5^\circ$  to  $67.5^\circ$  (Table 5), lie outside the normal range for strain-free pyranose rings (Jeffrey, 1973). In the ester-free methyl 6-deoxy-6-methylsulphonyl- $\alpha$ -D-glucopyranoside (Lindberg & Kierkegaard, 1971) the ring torsion angles range from  $60$  to  $70^\circ$  (e.s.d.  $2^\circ$ ). The comparison between the molecules indicates that the flattening of the ring in the present compound is caused by the bulky ester groups.

Table 4. *Intramolecular nonhydrogen bond distances and angles*

Estimated standard deviations are given in parentheses.

S—O(S)	1.478 (5) Å	C(1)—C(2)—C(3)	109.5 (4)°
S—C(S)	1.778 (7)	C(2)—C(3)—C(4)	112.7 (4)
S—C(6)	1.817 (5)	C(3)—C(4)—C(5)	111.9 (3)
C(1)—C(2)	1.522 (8)	C(4)—C(5)—O(5)	109.5 (3)
C(2)—C(3)	1.514 (6)	C(5)—O(5)—C(1)	112.9 (4)
C(3)—C(4)	1.517 (6)	O(5)—C(1)—C(2)	107.9 (4)
C(4)—C(5)	1.539 (6)	C(4)—C(5)—C(6)	113.2 (4)
C(5)—C(6)	1.516 (7)	O(5)—C(5)—C(6)	104.4 (4)
C(1)—O(1)	1.371 (9)	C(5)—C(6)—S	108.2 (3)
C(1)—O(5)	1.427 (7)	C(1)—C(2)—O(2)	110.5 (4)
C(2)—O(2)	1.446 (6)	C(3)—C(2)—O(2)	106.3 (4)
C(3)—O(3)	1.467 (5)	C(2)—C(3)—O(3)	106.9 (3)
C(4)—O(4)	1.451 (5)	C(4)—C(3)—O(3)	106.5 (3)
C(5)—O(5)	1.435 (5)	C(2)—C(1)—O(1)	109.9 (6)
C(7)—O(1)	1.458 (8)	O(5)—C(1)—O(1)	113.0 (4)
C(21)—O(2)	1.349 (6)	C(3)—C(4)—O(4)	105.7 (4)
C(31)—O(3)	1.343 (5)	C(5)—C(4)—O(4)	108.8 (3)
C(41)—O(4)	1.358 (5)	C(6)—S—C(S)	97.0 (3)
C(21)—O(22)	1.197 (7)	C(6)—S—O(S)	106.5 (3)
C(31)—O(32)	1.187 (6)	C(S)—S—O(S)	108.7 (3)
C(41)—O(42)	1.188 (7)	C(1)—O(1)—C(7)	112.8 (8)
C(21)—C(22)	1.487 (9)	C(2)—O(2)—C(21)	117.0 (4)
C(31)—C(32)	1.504 (7)	C(3)—O(3)—C(31)	118.4 (3)
C(41)—C(42)	1.492 (8)	C(4)—O(4)—C(41)	119.4 (4)
		O(2)—C(21)—C(22)	111.8 (5)
		O(3)—C(31)—C(32)	109.7 (4)
		O(4)—C(41)—C(42)	111.1 (5)
		O(2)—C(21)—O(22)	122.5 (5)
		O(3)—C(31)—O(32)	124.0 (4)
		O(4)—C(41)—O(42)	122.8 (5)
		C(22)—C(21)—O(22)	125.7 (5)
		C(32)—C(31)—O(32)	126.3 (5)
		C(42)—C(41)—O(42)	126.1 (5)

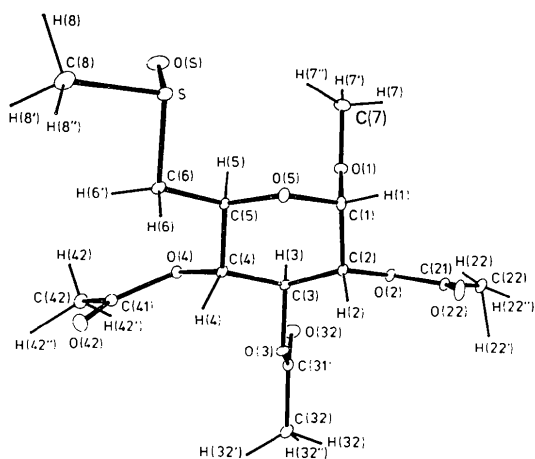


Fig. 1. Thermal-ellipsoid plot, with atom labels, of methyl tri-O-acetyl-6-deoxy-6-methylsulphonyl- $\alpha$ -D-glucopyranoside.

Table 5. *Torsion angles*

O(5)—C(1)—C(2)—C(3)	59.8 (5)°
C(1)—C(2)—C(3)—C(4)	-51.3 (5)
C(2)—C(3)—C(4)—C(5)	46.5 (5)
C(3)—C(4)—C(5)—O(5)	-49.7 (5)
C(4)—C(5)—O(5)—C(1)	62.0 (5)
C(5)—O(5)—C(1)—C(2)	-67.5 (5)
C(1)—O(5)—C(5)—C(6)	-176.6 (5)
C(3)—C(4)—C(5)—C(6)	-165.7 (5)
C(5)—O(5)—C(1)—O(1)	54.3 (6)
C(3)—C(2)—C(1)—O(1)	-63.9 (6)
O(5)—C(1)—O(1)—C(7)	62.1 (7)
C(2)—C(1)—O(1)—C(7)	-177.3 (8)
O(5)—C(5)—C(6)—S	65.4 (4)
C(4)—C(5)—C(6)—S	-175.6 (5)
C(5)—C(6)—S—O(S)	67.7 (4)
C(5)—C(6)—S—C(S)	179.6 (4)
O(5)—C(1)—C(2)—O(2)	176.5 (6)
C(4)—C(3)—C(2)—O(2)	-170.6 (5)
C(1)—C(2)—C(3)—O(3)	-167.9 (5)
C(5)—C(4)—C(3)—O(3)	163.4 (5)
O(5)—C(5)—C(4)—O(4)	-166.1 (5)
C(2)—C(3)—C(4)—O(4)	164.8 (5)
C(6)—C(5)—C(4)—O(4)	77.9 (4)
O(1)—C(1)—C(2)—O(2)	52.9 (5)
O(2)—C(2)—C(3)—O(3)	72.7 (4)
O(3)—C(3)—C(4)—O(4)	-78.3 (4)
C(1)—C(2)—O(2)—C(21)	83.6 (5)
C(3)—C(2)—O(2)—C(21)	-157.7 (5)
C(2)—C(3)—O(3)—C(31)	-117.0 (5)
C(4)—C(3)—O(3)—C(31)	122.3 (5)
C(3)—C(4)—O(4)—C(41)	138.8 (5)
C(5)—C(4)—O(4)—C(41)	-100.9 (5)
C(2)—O(2)—C(21)—C(22)	-178.1 (6)
C(3)—O(3)—C(31)—C(32)	177.9 (5)
C(4)—O(4)—C(41)—C(42)	-173.5 (6)

The average C—C and C—O bond lengths [except the anomeric C(1)—O(1)], 1.522 and 1.457 Å respectively, are normal for pyranoses (Berman, Chu & Jeffrey, 1967). The torsion angle O(5)—C(1)—O(1)—C(7),  $62.1^\circ$ , is in good agreement with predictions made by Jeffrey, Pople & Radom (1972). However, the shortening of the C(1)—O(1) bond, to 1.371 (9) Å, is more pronounced and the difference between the C(5)—O(5) and C(1)—O(5) bond lengths is smaller than predicted values.

Table 6. *Least-squares planes*

The 'best' planes are defined by  $Ax + By + Cz + D = 0$ , where  $x$ ,  $y$  and  $z$  are in Å units along the axes  $a$ ,  $b$  and  $c$ .

Atoms in 'best' plane	Atoms out of 'best' plane	Distance from 'best' plane (Å)	Constants	
O(2)		0.0010	0.154	A
C(21)		-0.0031	0.988	B
C(22)		0.0009	-0.013	C
O(22)		0.0013	-10.093	D
	C(2)	0.0415		
O(3)		-0.0021	0.999	A
C(31)		0.0067	0.024	B
C(32)		-0.0019	-0.022	C
O(32)		-0.0027	-15.526	D
	C(3)	0.0502		
O(4)		0.0012	-0.346	A
C(41)		-0.0038	0.920	B
C(42)		0.0011	0.185	C
O(42)		0.0015	-2.884	D
	C(4)	-0.1474		
C(2)		0.026	0.22	A
C(3)		-0.025	0.68	B
C(5)		0.026	-0.70	C
O(5)		-0.028	-6.81	D
	C(1)	0.720		
	C(4)	-0.594		

As the absolute configuration of the pyranose part of the molecule is known, it could be established that the sulphur atom has the *S* configuration. The geometry of the sulphoxide group is similar to that found for related structures (Dahlén, 1973). The torsion angle O(5)-C(5)-C(6)-S is 65.4° (*gauche-trans*). According to Sundaralingam (1968) this is the preferred conformation when oxygen is present instead of sulphur. The geometry of the acetate groups is in good agreement with previously found values of bond lengths, angles and planarity (Leung & Marchessault, 1974). The three ester groups attached to the ring have, as observed

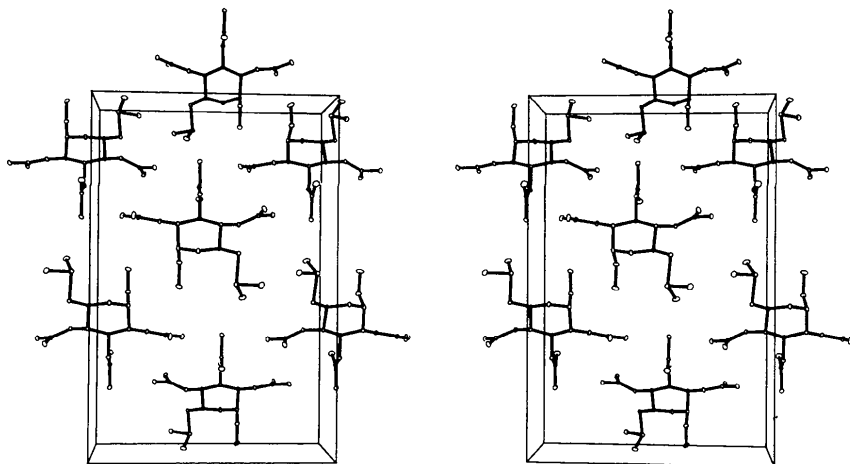
Table 7. *Semi-torsion angles and distances between the ester carbonyl oxygens and the ring hydrogen atoms*

H(2)-[C(2)-O(2)-C(21)]-O(22)	-34 (3)°
H(3)-[C(3)-O(3)-C(31)]-O(32)	3 (3)
H(4)-[C(4)-O(4)-C(41)]-O(42)	24 (3)
H(2)···O(22)	2.40 (4) Å
H(3)···O(32)	2.31 (5)
H(4)···O(42)	2.31 (4)

earlier (Mathieson, 1965; Leung & Marchessault, 1974), a preference for the eclipsed conformation of the carbonyl oxygen to the ring hydrogen (Table 7).

Table 8. *Non-bonded intermolecular contacts less than 3.8 Å involving nonhydrogen atoms and less than 2.8 Å for hydrogen-nonhydrogen contacts*

Contact	Distance (Å)	Symmetry operations
O(42)···C(21 <i>f</i> )	3.259 (7)	-
O(42)···C(22 <i>f</i> )	3.274 (8)	$x, y, z$
O(32)···C(4 <i>b</i> )	3.306 (6)	$x, y, 1+z$
O(32)···C(2 <i>b</i> )	3.459 (6)	$x, y, -1+z$
O(3)···O(32 <i>a</i> )	3.462 (5)	$\frac{1}{2}+x, \frac{1}{2}-y, -z$
O(S)···C(S <i>b</i> )	3.483 (9)	$\frac{1}{2}+x, \frac{1}{2}-y, 1-z$
O(42)···C(42 <i>a</i> )	3.515 (8)	$-\frac{1}{2}+x, \frac{1}{2}-y, -z$
O(22)···C(22 <i>a</i> )	3.517 (8)	$-\frac{1}{2}+x, \frac{1}{2}-y, 1-z$
O(S)···C(42 <i>g</i> )	3.529 (9)	$\frac{3}{2}-x, 1-y, -z$
O(22)···C(41 <i>d</i> )	3.544 (7)	$\frac{3}{2}-x, 1-y, -1-z$
O(22)···C(42 <i>d</i> )	3.573 (8)	
C(21)···C(41 <i>d</i> )	3.575 (9)	
O(22)···C(32 <i>d</i> )	3.597 (8)	
O(2)···O(42 <i>d</i> )	3.616 (6)	
O(32)···C(3 <i>b</i> )	3.622 (6)	
O(22)···O(42 <i>d</i> )	3.628 (7)	
O(4)···C(6 <i>b</i> )	3.638 (6)	
O(32)···O(5 <i>b</i> )	3.649 (5)	O(42)···H(2 <i>f</i> ) 2.33 (5) Å
C(22)···C(32 <i>c</i> )	3.651 (9)	O(22)···H(22 <i>f</i> ) 2.48 (6)
O(32)···C(22 <i>e</i> )	3.668 (8)	O(32)···H(4 <i>b</i> ) 2.51 (4)
O(S)···C(S <i>h</i> )	3.674 (9)	O(32)···H(2 <i>b</i> ) 2.68 (5)
O(42)···C(32 <i>f</i> )	3.675 (8)	O(22)···H(32' <i>d</i> ) 2.69 (5)
C(32)···C(42 <i>c</i> )	3.748 (8)	O(S)···H(42 <i>a</i> ) 2.70 (5)
O(32)···C(42 <i>c</i> )	3.784 (8)	O(S)···H(S' <i>h</i> ) 2.74 (6)

Fig. 2. Stereoscopic view of methyl tri-*O*-acetyl-6-deoxy-6-methylsulphinyl(*S*)- $\alpha$ -D-glucopyranoside.

There is no hydrogen bonding in the structure. The molecule has five pendant groups, each of which terminates in a methyl group, representing the extremities of the molecule. In the crystal structure the pendant groups of one molecule have weak non-bonding interactions with other molecules. Among the shorter intermolecular contacts (Table 8) there is a high frequency for the most charged atoms (*e.g.* carbonyl oxygens). This indicates that weak polar-polar interactions are the principal means by which the structure is bound together.

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## The Crystal Structure of *meso*-Di- $\mu$ -hydroxo-bis[bis(ethylenediamine)chromium(III)] Diperchlorate Dichloride Dihydrate

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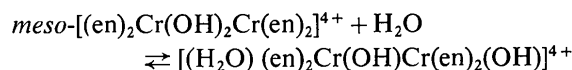
(Received 1 December 1975; accepted 5 January 1976)

$[(\text{en})_2\text{Cr} \begin{array}{c} \text{H} \\ \diagup \text{O} \diagdown \\ \text{O} \end{array} \text{Cr}(\text{en})_2](\text{ClO}_4)_2\text{Cl}_2 \cdot 2\text{H}_2\text{O}$  crystallizes in space group *Pbca* (No. 61) with  $a = 18.773(8)$ ,

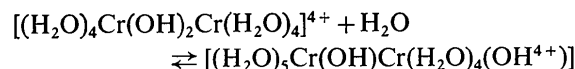
$b = 14.474(5)$ ,  $c = 10.084(7)$  Å,  $Z = 4$ . There is half a formula unit in the asymmetric unit. The structure was solved by Patterson and Fourier techniques and refined by full-matrix least squares to an  $R$  of 0.044 for 2311 diffractometer-collected intensities. As the  $[\text{Cr}(\text{en})_2\text{OH}]_2^{4+}$  ion contains a crystallographic inversion centre, it is the *meso* form and the bridging four-membered ring is planar. The conformation of the complex ion is  ${}^2A_2$  and the Cr–Cr separation 3.059(2) Å.

### Introduction

The present structure analysis was initiated as part of recent studies of the equilibrium between  $\mu$ -hydroxo and di- $\mu$ -hydroxo binuclear complexes of  $\text{Cr}^{\text{III}}$ . The equilibrium



is established unexpectedly fast. Both the forward and reverse rate constants are several orders of magnitude greater than those for water exchange in  $[\text{Cr}(\text{H}_2\text{O})_6]^{3+}$ . The equilibrium constant is 40 times greater than that for the corresponding equilibrium



(Springborg & Toftlund, 1975, 1976).

Although  $[(\text{en})_2\text{Cr}(\text{OH})_2\text{Cr}(\text{en})_2]^{4+}$  was first isolated by Pfeiffer & Stern (1908), the configuration was not established until the present structure analysis. The isomer investigated was the *meso* form. The *rac* form has now been isolated by Springborg & Toftlund (private communication). A structure analysis of  $\mu$ -hydroxo-bis[bis(ethylenediamine)hydroxochromium(III)] perchlorate hydrate is in progress. The crystallographic investigation is intended, *inter alia*, to elucidate the influence of different ligands and of