# The Crystal Structure and Absolute Configuration of the Complex
## (+)-*trans*-[PtCl{SiMe(1-C₁₀H₇)Ph} (PMe₂Ph)₂]

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(+)-[PtCl{SiMe(1-C₁₀H₇)Ph}(PMe₂Ph)₂] is monoclinic, $P2_1$, with $a = 12\cdot797$ (2), $b = 12\cdot491$ (2), $c = 10\cdot186$ (2) Å, $\beta = 104\cdot49$ (2)°, $Z = 2$. Least-squares refinement on 2876 reflexions has converged at $R = 0\cdot030$. The molecule has the absolute configuration ($S$) which corresponds to that of the ($R$) (+)-Me(1-C₁₀H₇)PhSiH from which it was prepared. The coordination is slightly distorted, square-planar with *trans* phosphine ligands (average Pt–P 2·299 Å). The long Pt–Cl bond of 2·462 Å reflects the high *trans* influence of the silyl ligand.

## Introduction

It has been shown (Eaborn, Kapoor, Tune, Turpin & Walton, 1972) that the title compound (I) can be prepared by treating the optically active silane ($R$) (+)-Me(1-C₁₀H₇)PhSiH (II) with *cis*-[PtCl₂(PMe₂Ph)₂] and that the parent silane (II) regenerated by treating (I) with LiAlH₄ shows little loss of optical activity. The structure of (I) has been determined to confirm that the formation of (I) from (II) occurs with retention of configuration at the Si atom. A preliminary account of this work has been published (Eaborn, Hitchcock, Tune & Walton, 1973).

## Table 1. *Crystallographic data*

C₃₃H₃₇SiP₂ClPt, $M = 754\cdot3$, monoclinic, $a = 12\cdot797$ (2),
$b = 12\cdot491$ (1), $c = 10\cdot186$ (2) Å, $\beta = 104\cdot49$ (2)°,
[$\lambda$ (Mo $K\alpha_1$) = 0·70926 Å], $U = 1576\cdot4$ Å³, $F(000) = 748$, $Z = 2$,
$D_c = 1\cdot59$ g cm⁻³, Mo $K\alpha$ radiation, $\mu = 49\cdot2$ cm⁻¹.
Space group $P2_1$ (No. 4) from systematic absences of $0k0$ for $k$ odd.

## Experimental

Samples of the title compound recrystallized from chloroform/pentane were supplied by D. J. Tune. Crystal data for the compound are given in Table 1. The crystal used was 0·62 × 0·24 × 0·14 mm, elongated along $c$, and bounded by the parallel faces {100}, {110}, {1$\bar{1}$0}, {201} and {20$\bar{1}$}. Preliminary cell dimensions were obtained from Weissenberg and precession films and the systematic absences indicated the space group to be either $P2_1$ or $P2_1/m$. Because of the optical activity of the compound, $P2_1$ was presumed to be correct and was confirmed by the successful structural analysis. The crystal was transferred to a Hilger and Watts Y290 four-circle diffractometer and accurate cell dimensions were derived by least squares from the $2\theta$ values for 12 reflexions. Intensities were collected with Mo $K\alpha$ radiation and a graphite crystal monochromator. Data for the unique quadrant with $\theta < 25°$ were measured by the $\omega/2\theta$ step-scan technique with counting times of

60 s for the peak and 30 s each for background counts at either end of the peak scan. In addition, data for $\theta < 10°$ were repeated, measuring both members of the Friedel pairs ($\pm k$). Lorentz, polarization and, at a later stage, absorption corrections were applied and

## Table 2. *Final atom positions and isotropic thermal parameters* (Å² × 10³)

(a) Heavy atoms (positional parameters are × 10⁴)

| | $x$ | $y$ | $z$ | $U_{iso}$ |
|---|---|---|---|---|
| Pt | 2068·8 (2) | 0 | 2302·0 (3) | – |
| Cl | 195 (2) | −655 (2) | 1684 (3) | – |
| P(1) | 1925 (2) | 196 (3) | 25 (2) | – |
| P(2) | 2200 (2) | −797 (2) | 4380 (2) | – |
| Si | 3627 (2) | 1017 (2) | 2964 (3) | – |
| C(1) | 4885 (7) | 315 (8) | 2848 (9) | 59 (2) |
| C(2) | 3525 (8) | 2308 (8) | 7 (9) | 47 (2) |
| C(3) | 4462 (8) | 2837 (9) | 1885 (10) | 61 (2) |
| C(4) | 4417 (10) | 3838 (11) | 1253 (10) | 78 (3) |
| C(5) | 3447 (10) | 4293 (11) | 669 (12) | 78 (3) |
| C(6) | 2508 (9) | 3796 (10) | 726 (11) | 67 (3) |
| C(7) | 2551 (8) | 2834 (8) | 1357 (10) | 56 (2) |
| C(8) | 3896 (7) | 1593 (8) | 4749 (9) | 52 (2) |
| C(9) | 4921 (9) | 1522 (10) | 5594 (11) | 70 (3) |
| C(10) | 5161 (11) | 1960 (11) | 6943 (13) | 81 (3) |
| C(11) | 4392 (10) | 2438 (12) | 7400 (13) | 83 (3) |
| C(12) | 3324 (9) | 2540 (10) | 6591 (11) | 65 (3) |
| C(13) | 2482 (11) | 3046 (12) | 7062 (13) | 82 (3) |
| C(14) | 1494 (12) | 3089 (13) | 6317 (15) | 93 (4) |
| C(15) | 1217 (10) | 2693 (10) | 4973 (12) | 70 (3) |
| C(16) | 1988 (9) | 2203 (10) | 4463 (12) | 55 (3) |
| C(17) | 3067 (8) | 2115 (8) | 5257 (10) | 55 (2) |
| C(18) | 726 (9) | 848 (10) | −987 (11) | 67 (3) |
| C(19) | 2978 (8) | 734 (9) | −672 (11) | 64 (3) |
| C(20) | 1845 (8) | −1189 (8) | −570 (10) | 55 (2) |
| C(21) | 2768 (9) | −1774 (10) | −379 (12) | 71 (3) |
| C(22) | 2733 (11) | −2849 (11) | −758 (13) | 80 (3) |
| C(23) | 1771 (10) | −3341 (12) | −1289 (13) | 82 (3) |
| C(24) | 865 (10) | −2782 (11) | −1454 (12) | 75 (3) |
| C(25) | 857 (9) | −1691 (10) | −1124 (11) | 67 (3) |
| C(26) | 3425 (10) | −785 (11) | 5727 (12) | 67 (3) |
| C(27) | 1208 (9) | −361 (9) | 5288 (11) | 71 (3) |
| C(28) | 1999 (9) | −2245 (10) | 4130 (12) | 56 (3) |
| C(29) | 1999 (8) | −2720 (9) | 2919 (11) | 61 (2) |
| C(30) | 1955 (10) | −3841 (11) | 2786 (13) | 79 (3) |
| C(31) | 1883 (11) | −4437 (12) | 3865 (14) | 92 (4) |
| C(32) | 1835 (12) | −3984 (13) | 5054 (14) | 93 (4) |
| C(33) | 1876 (10) | −2861 (10) | 5221 (12) | 72 (3) |

Table 2 (cont.)

(b) Hydrogen atoms (positional parameters are × 10³)

| | x | y | z | Bonded to |
|---|---|---|---|---|
| H(1) | 554 | 118 | 524 | C(9) |
| H(2) | 595 | 192 | 753 | C(10) |
| H(3) | 465 | 278 | 843 | C(11) |
| H(4) | 265 | 339 | 805 | C(13) |
| H(5) | 88 | 347 | 669 | C(14) |
| H(6) | 39 | 273 | 436 | C(15) |
| H(7) | 179 | 191 | 346 | C(16) |
| H(8) | 524 | 249 | 233 | C(3) |
| H(9) | 516 | 425 | 126 | C(4) |
| H(10) | 342 | 505 | 10 | C(5) |
| H(11) | 175 | 417 | 30 | C(6) |
| H(12) | 181 | 246 | 142 | C(7) |
| H(13) | 354 | −137 | 6 | C(21) |
| H(14) | 348 | −329 | −64 | C(22) |
| H(15) | 172 | −417 | −156 | C(23) |
| H(16) | 10 | −318 | −184 | C(24) |
| H(17) | 10 | −124 | −129 | C(25) |
| H(18) | 205 | −222 | 208 | C(29) |
| H(19) | 197 | −420 | 183 | C(30) |
| H(20) | 186 | −529 | 376 | C(31) |
| H(21) | 180 | −449 | 588 | C(32) |
| H(22) | 178 | −250 | 614 | C(33) |
| H(23) | 516 | −21 | 366 | C(1) |
| H(24) | 551 | 89 | 284 | C(1) |
| H(25) | 475 | −11 | 192 | C(1) |
| H(26) | 148 | 119 | −100 | C(18) |
| H(27) | 20 | 129 | −53 | C(18) |
| H(28) | 25 | 51 | −189 | C(18) |
| H(29) | 374 | 43 | −11 | C(19) |
| H(30) | 300 | 158 | −59 | C(19) |
| H(31) | 286 | 52 | −169 | C(19) |
| H(32) | 398 | −102 | 517 | C(26) |
| H(33) | 342 | −131 | 653 | C(26) |
| H(34) | 363 | −0 | 616 | C(26) |
| H(35) | 201 | −19 | 577 | C(27) |
| H(36) | 59 | −87 | 543 | C(27) |
| H(37) | 91 | 44 | 520 | C(27) |

equivalent reflexions in the full data set were averaged to give 2876 non-zero independent reflexions. The 10° data set was checked to find the 20 reflexions with the largest Friedel differences ($|F_{hkl}| - |F_{\bar{h}\bar{k}\bar{l}}|$), which were later used to find the absolute configuration.

## Structure solution and refinement

The structure solution and initial refinement were carried out with a data set of 2680 reflexions with $I > 3\sigma(I)$. The positions of all the non-hydrogen atoms were derived by Patterson and Fourier methods in an arbitrary absolute configuration, and refined by full-matrix least squares minimizing $\sum \omega(|F_o| - |F_c|)^2$. The Pt, Cl, P and Si were allowed anisotropic, and the C atoms isotropic

thermal parameters. This refinement with unit weights converged at $R = 0.042$, and a comparison of observed and calculated structure factors for the selected Friedel pairs showed the absolute configuration to be correct. At this stage, a difference map showed peaks at positions near the calculated H atom positions, and these atoms were included at idealized positions with fixed isotropic temperature factors equal to those of the C atoms to which they were bonded. The full data set was then corrected for absorption and a final least-squares refinement with all 2876 non-zero reflexions and empirical weights $\omega$ calculated as $\omega = 1/\{1 + [(|F_o| - 40)/33]^2\}$ and with H atom parameters fixed converged at $R = 0.030$ and $R_\omega = 0.034$. The largest parameter shift was then less than 0.04 of the corresponding e.s.d., and the e.s.d. of an observation of unit weight was 1.54. A final difference synthesis had electron density up to $\pm 0.9$ e Å$^{-3}$ near the heavy atoms and $< \pm 0.4$ e Å$^{-3}$ elsewhere. The atom parameters are listed in Tables 2 and 3.*

Scattering factors were taken from Cromer & Waber (1965) except those for H which were taken from Stewart, Davidson & Simpson (1965). Dispersion corrections for Pt, Cl, P and Si were taken from Cromer (1965). Initial data processing was done with local programs and the structure solution and refinement with the X-RAY 70 program system on the ICL 1906A computer at the Atlas Computer Laboratory.

## Description

Fig. 1 gives the atom numbering and the molecular stereochemistry which shows that the 1-naphthylphenylmethylsilyl ligand is in the absolute configuration (S) which corresponds to that of the (R)(+)-Me(1-C₁₀H₇)PhSiH (Okaya & Ashida, 1966) from which it was prepared. The structural parameters are listed in Table 4. The coordination about Pt is slightly distorted, square-planar with trans phosphine ligands 0.3 Å below the mean coordination plane and the Si and Cl atoms 0.3 Å above the plane (Table 5) as viewed in Fig. 1. The ligands adopt a very symmetrical arrangement with the phosphines eclipsed along the P(1)···P(2) direction, each having a methyl group C(19) and C(26) approximately in the coordination

Table 3. Anisotropic thermal parameters (Å² × 10⁴) with standard deviations in parentheses

Parameters are in the form $\exp[-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + U_{12}hka^*b^* + U_{13}hla^*c^* + U_{23}klb^*c^*)]$.

| | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{12}$ | $U_{13}$ | $U_{23}$ |
|---|---|---|---|---|---|---|
| Pt | 418 (1) | 393 (1) | 410 (1) | −15 (3) | 128 (1) | 21 (2) |
| Cl | 437 (11) | 764 (18) | 624 (14) | −111 (12) | 110 (10) | 91 (13) |
| P(1) | 576 (11) | 468 (22) | 451 (10) | −18 (13) | 172 (8) | 33 (12) |
| P(2) | 553 (13) | 469 (13) | 435 (12) | −46 (12) | 126 (10) | 60 (10) |
| Si | 429 (12) | 419 (13) | 557 (13) | 11 (10) | 152 (10) | −21 (11) |

plane and a phenyl group below the plane. In order to minimize the intramolecular contacts, the silyl group has its bulky substituents above the mean plane, and the phosphines are bent slightly towards the Cl atom to reduce the C(19) and C(26) methyl$\cdots$silicon contacts (Si$\cdots$C 3·6 Å). The Pt–P lengths do not differ significantly from the mean of 2·299 Å which is in good agreement with other $Pt^{II}$ *trans* phosphine distances, *e.g.* 2·300 Å in *trans*-$[Pt(PEt_3)_2Cl_2]$ (Messmer & Amma, 1966), 2·293 Å in *trans*-$\{PtCl(PPhMe_2)_2[CH_2Si(CH_3)_3]\}$ (Jovanović, Manojlović-Muir & Muir, 1974). The Pt–Si length of 2·317 Å and the Pt–Cl bond at 2·462 Å are in good agreement with the corresponding bond lengths of 2·29 and 2·45 Å observed in *trans*-$[PtCl(SiMePh_2)$ $(PMe_2Ph)_2]$ (Harrison, 1968), which illustrates the high *trans* influence of the silyl ligand (McWeeny, Mason & Towl, 1969).

The angles subtended at the Si atom by its substituents are less than the corresponding angles in the parent silane $(1\text{-}C_{10}H_7)PhMeSiH$ (all near to 110°).

Table 4. *Intramolecular distances, bond angles and torsion angles*

(a) Distances (Å)

| | | | |
|---|---|---|---|
| Pt—P(1) | 2·292 (2) | C(10)–C(11) | 1·33 (2) |
| Pt—P(2) | 2·306 (2) | C(11)–C(12) | 1·41 (2) |
| Pt—Cl | 2·462 (2) | C(12)–C(13) | 1·43 (2) |
| Pt—Si | 2·317 (2) | C(13)–C(14) | 1·30 (2) |
| Si—C(1) | 1·863 (8) | C(14)–C(15) | 1·41 (2) |
| Si—C(2) | 1·905 (10) | C(15)–C(16) | 1·37 (2) |
| Si—C(8) | 1·904 (10) | C(16)–C(17) | 1·42 (2) |
| P(1)–C(18) | 1·814 (11) | C(17)–C(18) | 1·45 (2) |
| P(1)–C(19) | 1·803 (12) | C(12)–C(17) | 1·42 (2) |
| P(1)–C(20) | 1·827 (11) | Pt$\cdots$H(7) | 2·72 |
| P(2)–C(26) | 1·807 (11) | Pt$\cdots$H(18) | 2·78 |
| P(2)–C(27) | 1·830 (13) | Phenyl rings | |
| P(2)–C(28) | 1·836 (13) | C–C { max. | 1·41 (2) |
| C(8)–C(9) | 1·38 (1) | min. | 1·35 (2) |
| C(9)–C(10) | 1·44 (2) | average | 1·377 |

(b) Angles (°)

| | | | |
|---|---|---|---|
| P(1)—Pt—P(2) | 160·5 (1) | C(9)—C(8)—C(17) | 118·3 (9) |
| Si——Pt—Cl | 165·6 (1) | C(8)—C(17)—C(12) | 119·7 (8) |
| Si——Pt—P(1) | 94·6 (1) | C(17)–C(12)–C(11) | 118·9 (11) |
| Si——Pt—P(2) | 96·3 (1) | C(12)–C(11)–C(10) | 121·9 (12) |
| Cl——Pt—P(1) | 87·2 (1) | C(11)–C(10)–C(9) | 120·3 (11) |
| Cl——Pt—P(2) | 86·2 (1) | C(10)–C(9)—C(8) | 120·9 (11) |
| C(1)—Si—C(2) | 107·8 (4) | C(17)–C(12)–C(13) | 118·2 (9) |
| C(1)—Si—C(8) | 106·6 (4) | C(12)–C(13)–C(14) | 121·7 (13) |
| C(2)—Si—C(8) | 99·9 | C(13)–C(14)–C(15) | 121·4 (15) |
| C(18)–P(1)–C(19) | 102·6 (5) | C(14)–C(15)–C(16) | 119·6 (11) |
| C(18)–P(1)–C(20) | 105·5 (5) | C(15)–C(16)–C(17) | 120·6 (10) |
| C(19)–P(1)–C(20) | 102·0 (5) | C(16)–C(17)–C(12) | 118·5 (10) |
| C(26)–P(2)–C(27) | 101·2 (6) | Phenyl rings, internal angles | |
| C(26)–P(2)–C(28) | 100·3 (6) | C–C–C { max. | 122·6 (10) |
| C(27)–P(2)–C(28) | 105·8 (6) | min. | 116·4 (9) |
| | | average | 120·00 |

(c) Torsion angles (°)

| | | | |
|---|---|---|---|
| C(2)—Si—Pt–P(1) | 46 | C(26)–P(2)—Pt—Cl | 179 |
| C(8)—Si—Pt–P(2) | −38 | C(27)–P(2)—Pt—Cl | −56 |
| C(1)—Si—Pt–P(1) | −78 | C(7)—C(2)—Si—Pt | 28 |
| C(20)–P(1)–Pt–Cl | −68 | C(17)–C(8)—Si—Pt | −46 |
| C(18)–P(1)–Pt–Cl | 47 | C(21)–C(20)–P(1)–Pt | −80 |
| C(19)–P(1)–Pt–Cl | 178 | C(29)–C(28)–P(2)–Pt | 12 |
| C(28)–P(2)–Pt–Cl | 63 | | |

Table 5. *Least-squares planes*

The equation of a plane is given in the form $pX+qY+rZ=S$ where $X, Y, Z$ are coordinates in Å with respect to the axes $a, b, c^*$. Deviations of atoms from the plane are given in Å; atoms marked † were not included in the calculation of the plane.

(a)    $0·4955X-0·8538Y-0·1600Z=0·6762$
Pt $-0·018$, Cl $-0·333$, P(1) 0·329, P(2) 0·325, Si $-0·303$

(b)    $0·2009X-0·4571Y-0·8664Z=-2·1802$
C(2) 0·009, C(3) 0·001, C(4) $-0·010$, C(5) 0·010, C(6) 0·001, C(7) $-0·010$, Si† $-0·15$

(c)    $0·3029X+0·2525Y-0·9190Z=0·8970$
C(20) 0·004, C(21) $-0·011$, C(22) 0·009, C(23) 0·002, C(24) $-0·010$, C(25) 0·007, P(1)† $-0·114$

(d)    $-0·9503X+0·0524Y-0·3070Z=-2·8015$
C(28) $-0·026$, C(29) 0·016, C(30) 0·004, C(31) $-0·013$, C(32) 0·003, C(33) 0·017, P(2)† $-0·191$

(e)    $0·3249X+0·8782Y-0·3509Z=1·3290$
C(8) 0·003, C(9) $-0·011$, C(10) $-0·009$, C(11) $-0·001$, C(12) 0·014, C(13) 0·017, C(14) $-0·027$, C(15) 0·000, C(16) 0·010, C(17) 0·013, Si† 0·024

C(2) and C(8) make angles of 100°, whereas C(1) makes angles of 107° with C(2) and 108° with C(8), showing that the naphthyl and phenyl rings are squashed slightly together by the contacts with the C(26) and C(19) methyl groups. In the phosphine ligands the angles subtended at P(1) by C(18) and C(20), and at P(2) by C(27) and C(28) are both 106°, whilst the other angles are near 101°. In the naphthyl moiety the pattern of bond lengths is very like that in the parent $(1\text{-}C_{10}H_7)$-
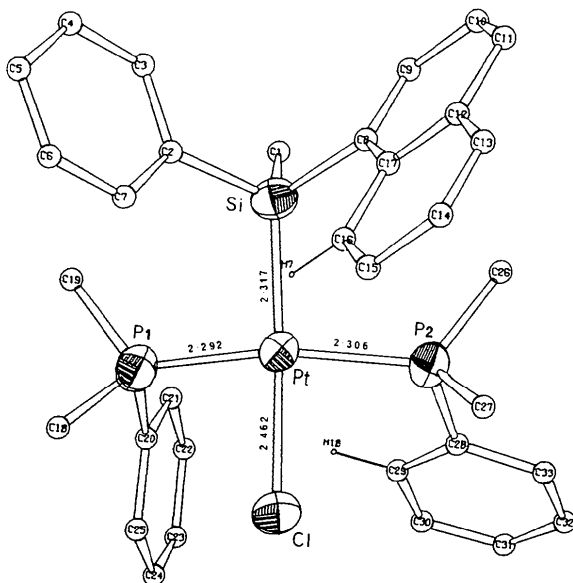


Fig. 1. Perspective view of the molecule showing the atom numbering scheme with 50% thermal vibration ellipsoids for the heavy atoms. Only the two H atoms with short contacts to the Pt atom are shown.

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 $Pd^0$ 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.

### References

BENNETT, M. J., DONALDSON, P. B., HITCHCOCK, P. B. & MASON, R. (1975). Inorg. Chim. Acta, 12, L9–L10.
CROMER, D. T. (1965). Acta Cryst. 18, 17–23.
CROMER, D. T. & WABER, J. T. (1965). Acta Cryst. 18, 104–109.
EABORN, C., HITCHCOCK, P. B., TUNE, D. J. & WALTON, D. R. M. (1973). J. Organomet. Chem. 54, C1–C2.
EABORN, C., KAPOOR, P. N., TUNE, D. J., TURPIN, C. L. & WALTON, D. R. M. (1972). J. Organomet. Chem. 34, 153–154.
HARRISON, P. M. (1968). Ph. D. Thesis, Univ. of Sheffield.
IMMIRZI, A. & MUSCO, A. (1974). Chem. Commun. pp. 415–416.
JOVANOVIĆ, B., MANOJLOVIĆ-MUIR, L. & MUIR, K. (1974). J. Chem. Soc. Dalton, pp. 195–198.
MCWEENY, R., MASON, R. & TOWL, A. D. C. (1969). Discuss. Faraday Soc. 47, 20–26.
MATSUMOTO, M., YOSHIOKA, H., NAKATSU, K., YOSHIDA, T. & OTSUKA, S. (1974). J. Amer. Chem. Soc. 96, 3322–3324.
MESSMER, G. G. & AMMA, E. L. (1966). Inorg. Chem. 5, 1775–1781.
NYBURG, S. C., BROOK, A. G., PASCOE, J. D. & SZYMAŃSKI, J. T. (1972). Acta Cryst. B28, 1785–1791.
OKAYA, Y. & ASHIDA, T. (1966). Acta Cryst. 20, 461–471.
STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). J. Chem. Phys. 42, 3175–3187.

# Methyl Tri-O-acetyl-6-deoxy-6-methylsulphinyl(S)-α-D-glucopyranoside

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$C_{14}H_{22}O_9S$, orthorhombic, $P2_12_12_1$, $a = 14·559$ (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 monochromated 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 $\theta < 63°$ were collected with $\theta$–$2\theta$ scans of 2° scan width; stationary background measurements were