dimensional polymeric mode (Smith, O'Reilly & Kennard, 1980).

Packing of the dimer units in the cell is such that an unusually close intermolecular H···H contact exists between symmetry-related ring protons [H(3)···H(3)′] (2·55 Å). However, this does not appear to perturb the ring system in any way nor does it lead to any unusual effects in the temperature factors of the associated atoms.

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2,3:4,6-Di-*O*-isopropylidene-5-thio-α-D-glucopyranose

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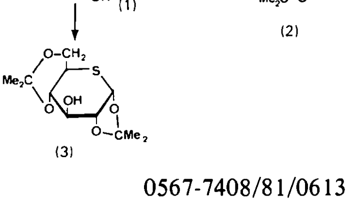
Abstract. $C_{12}H_{20}O_5S$, $M_r = 276{\cdot}4$, monoclinic, $P2_1$, $a = 5{\cdot}949\,(1)$, $b = 13{\cdot}082\,(4)$, $c = 9{\cdot}028\,(2)$ Å, $\beta = 94{\cdot}71\,(1)^\circ$, $U = 700{\cdot}2$ Å^3, $Z = 2$, $D_x = 1{\cdot}311$ Mg m^{-3}; final $R = 0{\cdot}068$ for 1326 unique diffractometer data. A S-containing pyranoid ring with a chair conformation is *trans*-fused to a 1,3-dioxolane ring system which has an envelope conformation.

Introduction. The compound (2) was obtained by reaction of 5-thio-D-glucose (1) with acetone and 2,2-dimethoxypropane (Clegg, Hughes & Al-Masoudi, 1979). The crystal structure was determined to verify that the product was the *trans*-fused system (2) rather than the *cis*-fused isomer (3).



Intensities were collected in the $2\theta/\omega$ scan mode on a Hilger & Watts Y290 diffractometer with Mo $K\alpha$ radiation ($\lambda = 0{\cdot}71069$ Å) and a Zr filter. Cell dimensions and the crystal-orientation matrix were obtained by least-squares refinement from 12 reflexions with $2\theta > 40^\circ$. 1326 unique data with $2\theta \leq 55^\circ$ and $I > 0$, corrected for crystal decomposition (assessed by periodic measurement of three standard reflexions, which showed a total decay of intensity of *ca* 35%) but not for absorption ($\mu = 0{\cdot}23$ mm^{-1}), were used for structure determination.

The structure was determined by a multi-solution tangent-refinement method and refined to a minimum value of $\sum w\Delta^2$ [$\Delta = |F_o| - |F_c|$; $w^{-1} = \sigma^2(F_o)$]. The H atom of the OH group was refined freely; all other H atoms were constrained [C—H = 0·96 Å, H—C—H = 109·5°; the H—C vector for X_3CH lies along the vector sum of the three C—X unit vectors; $U(\mathrm{H}) = 1{\cdot}1U_{\mathrm{eq}}(\mathrm{C})$]. Anisotropic thermal parameters were refined for C, O and S. The y coordinate of S(1) was fixed to define the origin. Final values of R and $R_g[=(\sum w\Delta^2/\sum wF_o^2)^{1/2}]$ are 0·068 and 0·062 respectively. Atom coordinates, and bond lengths and angles

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are given in Tables 1 and 2. Ring torsion angles are shown in Fig. 1.*

Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters* (Å$^2 \times 10^4$)

$U = \frac{1}{3}$(trace of the orthogonalized U_{ij} matrix).

	x	y	z	U
S(1)	4733 (4)	0	2190 (2)	717 (7)
C(2)	5949 (12)	−157 (5)	4165 (7)	620 (24)
O(3)	4383 (10)	−504 (4)	5092 (6)	735 (20)
H(3)	4431 (106)	−1359 (56)	4819 (70)	645 (191)
C(4)	6727 (11)	901 (5)	4611 (8)	564 (22)
O(5)	7826 (9)	940 (4)	6109 (6)	733 (19)
C(6)	7429 (11)	1929 (5)	6705 (7)	537 (22)
C(7)	9533 (12)	2563 (6)	6897 (8)	716 (29)
C(8)	6232 (13)	1801 (8)	8107 (7)	767 (31)
O(9)	5900 (7)	2447 (3)	5587 (4)	496 (14)
C(10)	4815 (10)	1640 (4)	4729 (6)	427 (18)
C(11)	3831 (10)	1949 (5)	3210 (6)	440 (18)
O(12)	2185 (7)	2726 (3)	3357 (4)	453 (13)
C(13)	1043 (11)	3030 (5)	1949 (7)	547 (22)
C(14)	−819 (11)	3727 (6)	2342 (9)	670 (27)
C(15)	2635 (13)	3585 (6)	1003 (8)	667 (27)
O(16)	7 (9)	2162 (4)	1246 (5)	689 (18)
C(17)	1473 (14)	1335 (6)	1014 (8)	717 (28)
C(18)	2730 (11)	1004 (5)	2486 (7)	544 (21)

Table 2. *Bond lengths* (Å) *and angles* (°)

S(1)–C(2)	1·880 (7)	S(1)–C(18)	1·808 (7)
C(2)–O(3)	1·379 (9)	C(2)–C(4)	1·504 (10)
O(3)–H(3)	1·147 (73)	C(4)–O(5)	1·454 (8)
C(4)–C(10)	1·503 (9)	O(5)–C(6)	1·429 (9)
C(6)–C(7)	1·499 (10)	C(6)–C(8)	1·511 (10)
C(6)–O(9)	1·467 (7)	O(9)–C(10)	1·432 (7)
C(10)–C(11)	1·502 (8)	C(11)–O(12)	1·424 (8)
C(11)–C(18)	1·522 (9)	O(12)–C(13)	1·447 (7)
C(13)–C(14)	1·499 (10)	C(13)–C(15)	1·512 (10)
C(13)–O(16)	1·417 (9)	O(16)–C(17)	1·416 (10)
C(17)–C(18)	1·533 (9)		

C(2)–S(1)–C(18)	98·4 (3)	S(1)–C(2)–O(3)	112·7 (5)
S(1)–C(2)–C(4)	103·9 (5)	O(3)–C(2)–C(4)	110·4 (6)
C(2)–O(3)–H(3)	99·2 (33)	C(2)–C(4)–O(5)	112·8 (6)
C(2)–C(4)–C(10)	113·1 (5)	O(5)–C(4)–C(10)	101·4 (5)
C(4)–O(5)–C(6)	107·8 (5)	O(5)–C(6)–C(7)	112·5 (6)
O(5)–C(6)–C(8)	108·6 (6)	C(7)–C(6)–C(8)	114·2 (6)
O(5)–C(6)–O(9)	105·7 (5)	C(7)–C(6)–O(9)	106·8 (5)
C(8)–C(6)–O(9)	108·6 (5)	C(6)–O(9)–C(10)	105·0 (4)
C(4)–C(10)–O(9)	101·7 (5)	C(4)–C(10)–C(11)	110·5 (5)
O(9)–C(10)–C(11)	114·8 (5)	C(10)–C(11)–O(12)	109·1 (5)
C(10)–C(11)–C(18)	107·1 (5)	O(12)–C(11)–C(18)	110·2 (5)
C(11)–O(12)–C(13)	113·1 (4)	O(12)–C(13)–C(14)	105·2 (5)
O(12)–C(13)–C(15)	110·9 (5)	C(14)–C(13)–C(15)	110·6 (6)
O(12)–C(13)–O(16)	109·1 (5)	C(14)–C(13)–O(16)	106·9 (5)
C(15)–C(13)–O(16)	113·7 (6)	C(13)–O(16)–C(17)	115·4 (5)
O(16)–C(17)–C(18)	110·5 (6)	S(1)–C(18)–C(11)	112·8 (5)
S(1)–C(18)–C(17)	110·9 (5)	C(11)–C(18)–C(17)	107·7 (5)

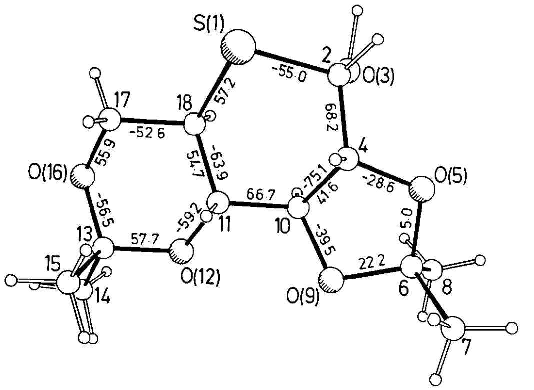

Fig. 1. Molecular structure of (2), with the atom numbering and ring torsion angles (e.s.d.'s 0·5–0·8°).

Discussion. Both six-membered rings have a chair conformation (Fig. 2). All substituents on the pyranoid ring, with the exception of the OH group, are equatorial. The five-membered 1,3-dioxolane ring has an envelope conformation: deviations of the atoms from the mean plane through C(4), O(5), C(6) and O(9) are: C(4) +0·018 (7), O(5) −0·028 (6), C(6) +0·027 (7), O(9) −0·017 (4), C(10) −0·613 (6) Å. The angle between this mean plane and the plane of C(4), O(9) and C(10) is 41 (1)°.

The main feature of interest is the *trans*-fusion of the five-membered ring to the pyranoid ring. Although acetal formation involving a *trans* fusion has been observed in seven-membered ring systems (Stevens, 1975; Kuszmann, Sohár & Horváth, 1976), this appears to be the only reported instance for a six-membered ring. As a general rule, *cis* fusion is considerably more favourable for five- and six-membered rings (Lemieux, 1964). The formation of (2) rather than (3) on isopropylidenation of (1) is, therefore, surprising, and is probably caused by the presence of a S atom in the pyranoid ring. Examination of models suggests that (3) has a greater distortion of the pyranoid chair conformation than (2), whereas the reverse is true for the analogous compounds with O replacing S (Hughes, 1979). Typical C—C single-bond lengths are intermediate between C—O and C—S, so that substitution of O and of S for CH_2 in a cyclohexane ring introduces opposite distortion effects.

I thank Dr N. A. Hughes for supplying crystals. Data were collected in Durham with the help of the late

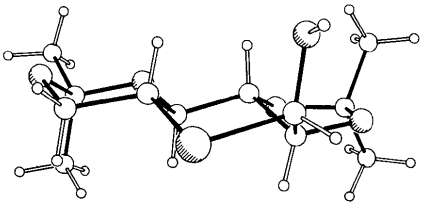

Fig. 2. Side view of the molecule, showing the ring conformations.

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* Lists of structure factors, anisotropic thermal parameters and H atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35920 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Dr H. M. M. Shearer. Programs, other than diffractometer-control software, were written by WC and Professor G. M. Sheldrick.

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SHORT COMMUNICATIONS

Contributions intended for publication under this heading should be expressly so marked; they should not exceed about 1000 *words; they should be forwarded in the usual way to the appropriate Co-editor; they will be published as speedily as possible.*

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Triclinic and orthorhombic barium dihydrogenphosphate: a statistical comparison.

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Abstract

Two independent X-ray structure analyses of triclinic $Ba(H_2PO_4)_2$ are compared by means of half-normal probability-plot analysis. Two independent determinations of orthorhombic $Ba(H_2PO_4)_2$ are likewise compared. The weighted mean coordinates for the triclinic and orthorhombic structures along with the resulting interatomic distances and angles have been deposited. These should be used in place of the results given in the earlier publications.

Two papers on the triclinic $Ba(H_2PO_4)_2$ structure have recently been published independently by Gilbert & Lenhert (1978) (GL) and Durif & Guitel (1978) (DG). Two papers on the orthorhombic form have also appeared [Gilbert, Lenhert & Wilson (1977) and Prelesnik, Herak, Čurić & Krstanović (1978) (GLW and PHCK)]. We have compared the two pairs of coordinate and thermal-parameter sets with the aid of half-normal probability plots (Abrahams & Keve, 1971) using the ranked moduli of Hamilton & Abrahams (1972). Important features of the four X-ray experiments are summarized in Table 1 with each experiment listed under the initials of the authors.

Table 1. *A summary of the four* $Ba(H_2PO_4)_2$ *analyses*

	Triclinic		Orthorhombic	
	GL	DG	GLW	PHCK
Number of reflections measured	13 381	1856	5792	1253
Maximum sin θ/λ (Å^{-1})	0·99	0·69	0·99	0·85
Radiation	Mo $K\alpha$, Nb filtered	Mo $K\beta$, monochromated	Mo K , Nb filtered	Mo $\kappa\alpha$, Zr filtered
Least-squares weights	Statistical	Statistical	Statistical	Unit weights
Absorption correction	Yes	No	Yes	None, sphere
Extinction correction	Yes	No	Yes	Yes
H atoms located	Refined	No	Yes	Refined?

In the course of our analysis we found a small discrepancy between the coordinates, cell parameters and bond distances and angles in the paper by DG. The half-normal probability-plot analysis, however, showed very little systematic difference between the coordinates of DG and GL for the triclinic structures.

The first plot for the orthorhombic coordinates showed clear evidence of systematic differences. The data set for PHCK was obtained and upon repeating the refinement, the R factor dropped from their value of 0·031 to 0·027 and the systematic differences were virtually eliminated. When modified Hughes weights were used (statistical weights were not available for PHCK) R dropped to 0·026 and the slope of the half-normal probability plot was very close to unity.

Comparison of the thermal parameters showed substantial systematic differences for both sets. Large differences are found for the heavy atoms, especially Ba. The systematic differences in the data sets, in particular, the treatment of absorption, weak reflections and the different sin θ/λ limits, may be responsible. For further details, the interested reader should consult the deposited material which includes the improved coordinate sets for both structures.*

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* All tables and plots, together with a more detailed discussion of the analysis have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36019 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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