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Structure of Benzo[*b*]biphenylene

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Abstract. $C_{16}H_{10}$, $M_r = 202{\cdot}25$, triclinic, $P\bar{1}$, $a = 6{\cdot}000\,(1)$, $b = 7{\cdot}841\,(1)$, $c = 11{\cdot}933\,(3)$ Å, $\alpha = 106{\cdot}16\,(2)$, $\beta = 99{\cdot}08\,(2)$, $\gamma = 93{\cdot}38\,(1)^\circ$, $V = 529{\cdot}3\,(2)$ Å^3, $Z = 2$, $D_m = 1{\cdot}24\,(1)$, $D_x = 1{\cdot}269\,(1)$ g cm^{-3}, $\lambda(\text{Mo}\ K\alpha) = 0{\cdot}71073$ Å, $\mu = 0{\cdot}668$ cm^{-1}, $F(000) = 212$, $T = 295$ K, $R(F) = 0{\cdot}125$ and $wR(F) = 0{\cdot}077$ for 2751 unique reflections $I \geq 0$ and $R(F) = 0{\cdot}051$, $wR(F) = 0{\cdot}059$ for 1227 reflections with $I \geq 3\sigma(I)$. The asymmetric unit consists of two independent half molecules of benzo[*b*]biphenylene, with each molecule disordered about a crystallographically imposed center of inversion at the center of the molecule.

Introduction. Diarylalkynes can be synthesized by coupling and dehydrohalogenating (dibromomethyl)arenes under basic conditions (Vernigor, Shalaev & Luk'yanets, 1981). In this regard the reaction of 1,2-bis(dibromomethyl)benzene with potassium *tert*-butoxide in Me_2SO was examined and resulted in the synthesis of benzo[*b*]biphenylene. The previously reported reaction of 1,2-bis(dibromomethyl)benzene with potassium *tert*-butoxide in tetrahydrofuran gave 1,4-dibromobenzo[*b*]biphenylene (Jensen & Coleman, 1959). There are other syntheses available for the preparation of benzo[*b*]biphenylene (Baker, Barton, McOmie & Searle, 1962; Barton, Shepherd & Willis, 1986) but this synthesis represents the only method using one step, although in extremely low yield. Crystals of benzo[*b*]biphenylene were obtained and an X-ray structural study was undertaken.

Experimental. Potassium *tert*-butoxide (27·00 g, 0·2406 mol, Aldrich) was added to a solution of 1,2-bis(dibromomethyl)benzene (12·65 g, 0·0300 mol, Fluka) in 90 ml of Me_2SO. The mixture was stirred for 1 h and acidified with dilute HCl. The mixture was filtered and the resulting dark brown solid was sublimed at 393 K and 0·05 Pa. Recrystallization of the sublimed product from toluene afforded 0·031 g (1% yield) of benzo[*b*]biphenylene.

Slow evaporation of a tetrahydrofuran solution of the title compound gave colorless triclinic crystals suitable for X-ray diffraction work. One of the crystals (0·17 × 0·46 × 0·36 mm) was mounted on a glass fiber with epoxy cement. Data were collected on a Syntex $P2_1$ diffractometer with graphite-monochromated Mo $K\alpha$ radiation. Unit-cell parameters from refinement of the indices and angles of 15 well centered reflections with 2θ in the range 20·0 to 30·0°. D_m determined by flotation in aqueous sodium chloride. Intensity data collected [2θ–θ scans; $(\sin\theta_{\max})/\lambda = 0{\cdot}729$ Å^{-1}; $0 \leq h \leq 8$, $-11 \leq k \leq 10$, $-17 \leq l \leq 15$; 3902 reflections measured; standard reflections: $0\bar{4}7$, $2\bar{3}\bar{3}$, 401; no indication of standard reflection decay]. Unique reflections $I \geq 0$: 2751; unique reflections $I \geq 3\sigma(I)$: 1227; unobserved reflections: 789. $|E|$ statistics consistent with centrosymmetric space group. Data corrected for Lorentz and polarization effects. Structure solved by direct methods (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980), and refined by Fourier and difference Fourier methods, and least-squares refinement (Strouse, 1985) in $P\bar{1}$. Solution of structure in $P1$ also gave disordered structure. H atoms found in difference Fourier synthesis and refined isotropically. Disordered atoms were set to an occupancy of 0·500. Parameters refined: 230. $R(F) = 0{\cdot}125$, $wR(F) = 0{\cdot}077$, $S = 1{\cdot}45$, $w = 1/(\sigma_{F(\text{obs})})^2$, $(\Delta/\sigma)_{\max} \leq 0{\cdot}01$, max. residual electron density: 0·71 e Å^{-3}, scattering factors from *International Tables for X-ray Crystallograph* (1974) [for the 1227 data $I \geq 3\sigma(I)$ $R = 0{\cdot}051$ and $wR(F) = 0{\cdot}059$; refined and derived quantities from refinement of the 2751 data $I \geq 0$]. All programs used are available in *The UCLA Crystallographic Computing Package* (Strouse, 1985). Final atomic coordinates, equivalent isotropic thermal parameters for carbon (Hamilton, 1959) and isotropic thermal parameters for hydrogen are listed in Table 1.†

Discussion. Bond lengths and bond angles are listed in Table 2. The structure has several interesting features (Figs. 1 and 2). The central cyclobutadiene and benzene rings are disordered about a crystallographically imposed center of inversion. This results in relatively

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† Lists of structure factors, anisotropic thermal parameters and least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51230 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. *Final atomic coordinates for all atoms, equivalent isotropic thermal parameters for carbon atoms and isotropic thermal parameters for hydrogen atoms*

$$U_{eq} = \tfrac{1}{3}\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq} or U(Å^2 × 10^2)
C(01)	0·5941 (3)	0·1715 (3)	0·1696 (2)	4·7 (2)
C(02)	0·3756 (3)	0·0799 (3)	0·1556 (2)	4·8 (2)
C(03)	0·2594 (4)	0·1094 (3)	0·2499 (2)	5·8 (2)
C(04)	0·3622 (5)	0·2307 (4)	0·3569 (2)	6·8 (2)
C(05)	0·5746 (5)	0·3211 (3)	0·3705 (2)	6·6 (2)
C(06)	0·6928 (4)	0·2928 (3)	0·2770 (2)	5·5 (2)
C(07*A*)	0·7175 (9)	0·1352 (7)	0·0683 (5)	5·0 (4)
C(07*B*)	0·6077 (8)	0·0804 (7)	0·0479 (5)	4·4 (4)
C(08*A*)	0·2718 (9)	−0·0512 (7)	0·0404 (5)	4·8 (4)
C(08*B*)	0·3846 (8)	−0·0148 (7)	0·0331 (5)	4·4 (4)
C(11)	0·8564 (3)	0·6509 (3)	0·1363 (2)	4·6 (2)
C(12)	1·0729 (3)	0·6072 (3)	0·1821 (2)	4·7 (2)
C(13)	1·1574 (4)	0·6660 (3)	0·3037 (2)	5·4 (2)
C(14)	1·0259 (4)	0·7669 (3)	0·3772 (2)	6·4 (2)
C(15)	0·8144 (4)	0·8085 (3)	0·3323 (2)	6·3 (2)
C(16)	0·7283 (4)	0·7508 (3)	0·2118 (2)	5·4 (2)
C(17*A*)	0·7679 (9)	0·5867 (6)	0·0075 (5)	4·7 (4)
C(17*B*)	0·8807 (9)	0·5559 (7)	0·0151 (5)	4·5 (4)
C(18*A*)	1·2092 (9)	0·4957 (6)	0·0993 (5)	5·1 (4)
C(18*B*)	1·1023 (8)	0·5123 (6)	0·0638 (6)	4·4 (4)
H(03)	0·118 (4)	0·042 (3)	0·244 (2)	4·0 (7)
H(04)	0·292 (4)	0·249 (3)	0·427 (2)	4·6 (7)
H(05)	0·635 (3)	0·401 (3)	0·444 (2)	2·6 (6)
H(06)	0·832 (4)	0·356 (3)	0·285 (2)	3·6 (7)
H(07)	0·854 (6)	0·204 (4)	0·078 (3)	0·8 (9)
H(08)	0·136 (6)	−0·122 (5)	0·034 (3)	1·2 (10)
H(13)	1·312 (4)	0·647 (3)	0·334 (2)	3·4 (6)
H(14)	1·085 (4)	0·812 (3)	0·458 (2)	3·1 (6)
H(15)	0·716 (4)	0·881 (3)	0·386 (2)	3·9 (7)
H(16)	0·578 (3)	0·774 (2)	0·180 (2)	1·9 (5)
H(17)	0·616 (7)	0·612 (5)	−0·020 (3)	2·2 (10)
H(18)	1·358 (6)	0·470 (4)	0·128 (3)	0·9 (9)

Table 2. *Unique bond lengths* (Å) *and angles* (°) *for all atoms in the asymmetric unit*

C(01)–C(02)	1·420 (3)	C(11)–C(12)	1·427 (3)
C(01)–C(06)	1·383 (3)	C(11)–C(16)	1·383 (3)
C(01)–C(07*A*)	1·485 (6)	C(11)–C(17*A*)	1·476 (6)
C(01)–C(07*B*)	1·447 (6)	C(11)–C(17*B*)	1·465 (7)
C(02)–C(03)	1·388 (3)	C(12)–C(13)	1·394 (3)
C(02)–C(08*A*)	1·484 (6)	C(12)–C(18*A*)	1·506 (6)
C(02)–C(08*B*)	1·455 (6)	C(12)–C(18*B*)	1·445 (6)
C(03)–C(04)	1·386 (3)	C(13)–C(14)	1·387 (3)
C(03)–H(03)	0·950 (22)	C(13)–H(13)	0·973 (20)
C(04)–C(05)	1·385 (3)	C(14)–C(15)	1·391 (3)
C(04)–H(04)	0·979 (23)	C(14)–H(14)	0·936 (20)
C(05)–C(06)	1·388 (3)	C(15)–C(16)	1·384 (3)
C(05)–H(05)	0·926 (20)	C(15)–H(15)	1·015 (21)
C(06)–H(06)	0·923 (21)	C(16)–H(16)	0·969 (18)
C(07*A*)–C(08*B*′)	1·339 (8)	C(17*A*)–C(18*B*′)	1·359 (7)
C(07*A*)–H(07)	0·928 (34)	C(17*A*)–H(17)	0·975 (39)
C(07*B*)–C(08*A*′)	1·346 (7)	C(17*B*)–C(18*A*′)	1·324 (8)
C(07*B*)–C(08*B*)	1·454 (6)	C(17*B*)–C(18*B*)	1·464 (5)
C(08*A*)–H(08)	0·938 (37)	C(18*A*)–H(18)	0·957 (33)

C(02)–C(01)–C(06)	120·4 (2)	C(12)–C(11)–C(16)	120·5 (2)
C(02)–C(01)–C(07*A*)	119·6 (3)	C(12)–C(11)–C(17*A*)	119·6 (3)
C(02)–C(01)–C(07*B*)	90·7 (2)	C(12)–C(11)–C(17*B*)	90·7 (2)
C(06)–C(01)–C(07*A*)	120·0 (3)	C(16)–C(11)–C(17*A*)	119·9 (3)
C(06)–C(01)–C(07*B*)	148·9 (3)	C(16)–C(11)–C(17*B*)	148·8 (3)
C(01)–C(02)–C(03)	120·4 (2)	C(11)–C(12)–C(13)	120·0 (2)
C(01)–C(02)–C(08*A*)	120·1 (3)	C(11)–C(12)–C(18*A*)	120·1 (3)
C(01)–C(02)–C(08*B*)	90·7 (2)	C(11)–C(12)–C(18*B*)	90·7 (2)
C(03)–C(02)–C(08*A*)	119·5 (3)	C(13)–C(12)–C(18*A*)	119·9 (3)
C(03)–C(02)–C(08*B*)	148·9 (3)	C(13)–C(12)–C(18*B*)	149·2 (3)
C(02)–C(03)–C(04)	118·2 (2)	C(12)–C(13)–C(14)	118·2 (2)
C(02)–C(03)–H(03)	121·2 (13)	C(12)–C(13)–H(13)	119·9 (12)
C(04)–C(03)–H(03)	120·5 (13)	C(14)–C(13)–H(13)	121·6 (12)
C(03)–C(04)–C(05)	121·5 (2)	C(13)–C(14)–C(15)	121·6 (2)
C(03)–C(04)–H(04)	121·0 (13)	C(13)–C(14)–H(14)	118·6 (13)
C(05)–C(04)–H(04)	117·3 (13)	C(15)–C(14)–H(14)	119·7 (13)
C(04)–C(05)–C(06)	121·0 (3)	C(14)–C(15)–C(16)	120·8 (2)
C(04)–C(05)–H(05)	118·1 (13)	C(14)–C(15)–H(15)	121·9 (12)
C(06)–C(05)–H(05)	120·9 (13)	C(16)–C(15)–H(15)	117·2 (12)
C(01)–C(06)–C(05)	118·5 (2)	C(11)–C(16)–C(15)	118·8 (2)
C(01)–C(06)–H(06)	120·2 (13)	C(11)–C(16)–H(16)	119·9 (11)
C(05)–C(06)–H(06)	121·2 (13)	C(15)–C(16)–H(16)	121·2 (11)
C(01)–C(07*A*)–C(08*B*′)	117·5 (5)	C(11)–C(17*A*)–C(18*B*′)	118·3 (5)
C(01)–C(07*A*)–H(07)	118·0 (23)	C(11)–C(17*A*)–H(17)	117·6 (23)
C(08*B*′)–C(07*A*)–H(07)	124·3 (23)	C(18*B*′)–C(17*A*)–H(17)	124·0 (24)
C(01)–C(07*B*)–C(08*A*′)	148·6 (5)	C(11)–C(17*B*)–C(18*A*′)	147·7 (6)
C(01)–C(07*B*)–C(08*B*)	89·6 (4)	C(11)–C(17*B*)–C(18*B*)	88·5 (4)
C(08*A*′)–C(07*B*)–C(08*B*)	121·7 (7)	C(18*A*′)–C(17*B*)–C(18*B*)	123·9 (8)
C(02)–C(08*A*)–C(07*B*′)	117·9 (5)	C(12)–C(18*A*)–C(17*B*′)	116·9 (5)
C(02)–C(08*A*)–H(08)	120·1 (23)	C(12)–C(18*A*)–H(18)	121·5 (21)
C(07*B*′)–C(08*A*)–H(08)	121·5 (23)	C(17*B*′)–C(18*A*)–H(18)	121·5 (22)
C(02)–C(08*B*)–C(07*A*′)	147·8 (5)	C(12)–C(18*B*)–C(17*A*′)	148·5 (6)
C(02)–C(08*B*)–C(07*B*)	89·0 (4)	C(12)–C(18*B*)–C(17*B*)	90·1 (4)
C(07*A*′)–C(08*B*)–C(07*B*)	123·2 (7)	C(17*A*′)–C(18*B*)–C(17*B*)	121·4 (8)

large standard deviations for the positional and thermal parameters of the disordered atoms. There is also anomalous shortening and lengthening of some bonds as a result of the constraint that the two terminal benzo groups be identical. The asymmetric unit consists of two independent half molecules of benzo[*b*]biphenylene with the unit cell containing two molecules (Fig. 3). Fig. 1 shows the first molecule of benzo[*b*]biphenylene labeled (Tables 1 and 2) with only the atoms corresponding to one of the two molecules of the disorder model about the ($\frac{1}{2}$, 0, 0) center of inversion. Fig. 2 shows both molecules of the disordered model about the ($\frac{1}{2}$, 0, 0) center of inversion. The second disordered set of atoms is shown with open bonds and no shading. The second half-molecule and its symmetry-related atoms generated by the center of inversion has the same numbering scheme except that the first digit in the atom name is 1 instead of 0.

The structures of the half-molecules of the asymmetric unit are identical within experimental error (Stout & Jensen, 1968). Equivalent bond lengths and angles (assuming *mm* symmetry for an individual molecule) for the two half molecules have been averaged together for the following discussion using the formula

$$\sigma = [\sum_m (l_m - \bar{l})^2/(m-1)]^{1/2}$$

to determine the standard deviation of the mean value (Stout & Jensen, 1968). Corresponding mean bond lengths observed in biphenylene (1) (Fawcett & Trotter, 1959) and the series biphenyleno[2,3-*c*]phenanthrene (2), benzo[*g*]biphenyleno[2,3-*c*]phenanthrene (3) and biphenyleno[2,3-*a*]pentahelicene (4) (Van Meerssche, Germain, Declercq, Soubrier-Payen, Figeys & Vanommeslaeghe, 1981) are discussed below. Compounds (2), (3) and (4) contain both a benzo[*b*]biphenylene fragment and a helicene fragment. The mean values for the benzo[*b*]biphenylene fragments in (2), (3) and (4) have been averaged for the following discussion.

The bond length between C(01) and C(02) [or the equivalent C(01′) and C(02′)] is the same for benzo[*b*]biphenylene, (1) and (2)–(4) at 1·424 (5), 1·426 (3) and 1·423 (3) Å, respectively. The bond lengths between C(01) and C(06) [or equivalently C(02) and C(03), C(02′) and C(03′) or C(01′) and C(06′)] in benzo[*b*]biphenylene, (1) and (2)–(4) [1·387 (5), 1·372 (2) and 1·371 (7) Å, respectively] are the same at the 75% confidence level (Stout & Jensen, 1968). The remaining bond lengths in the terminal benzo group [mean of C(03)—C(04) and C(05)—C(06) = 1·386 (2), C(04)—C(05) = 1·388 (4) Å] in benzo[*b*]biphenylene are equal within experimental error to the bond between C(01) and C(06). In structures (1) and (2)–(4) these bonds show slight alternation. The mean bond length between atoms C(01′) and C(07*B*′) [1·462 (17) Å], which corresponds to the single bond of the cyclobutadiene ring, is significantly shorter than the single bond in (1) [1·514 (3) Å] and (2)–(4) [1·511 (3) Å]. The bond length between C(01) and C(07*A*) [1·488 (13) Å] is considerably longer than the corresponding bond lengths in (1) and (2)–(4) [1·372 (2) and 1·440 (4) Å, respectively]. The bond lengths between the disordered atoms [C(07*B*′)—C(08*B*′) = 1·459 (17) and C(07*B*′)—C(08*A*) = 1·342 (15) Å] are similar in length to the values in (1) [1·426 (3) and 1·372 (2) Å, respectively] and (2)–(4) [1·426 (2) and 1·346 (6) Å, respectively]. Statistically significant alternation of double and single bonds is observed in the cyclohexatriene ring in the hexa(trimethylsilyl) derivative of tris(benzocyclobutadieno)benzene (Diercks & Vollhardt, 1986) with single and double bond lengths in the six-membered ring being 1·494 (8) and 1·335 (2) Å, respectively.

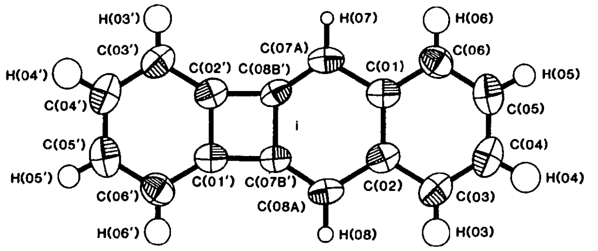

Fig. 1. *ORTEP* (Johnson, 1971) labeling diagram for one molecule of benzo[*b*]biphenylene contained in the unit cell. The thermal ellipsoids are drawn at the 50% probability level. The second molecule is numbered C(11)–C(18*B*). i denotes the center of inversion.

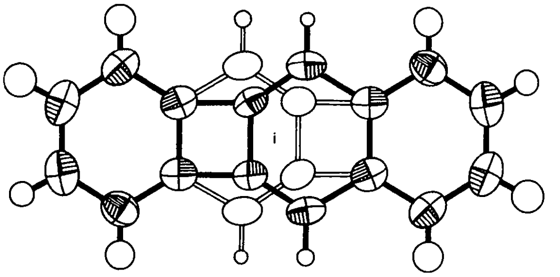

Fig. 2. *ORTEP* (Johnson, 1971) diagram of one molecule of benzo[*b*]biphenylene showing the disordered atoms about the center of inversion (i). The thermal ellipsoids are drawn at the 50% probability level.

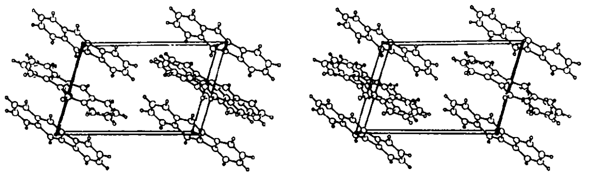

Fig. 3. *ORTEP* (Johnson, 1971) stereoview packing diagram of benzo[*b*]biphenylene. The *b* axis is vertical and the *c* axis is horizontal. The thermal ellipsoids are drawn at the 20% probability level.

The mean angle defined by C(02′)—C(01′)—C(07*B*′), for example, is 90·0 (9)° for benzo[*b*]biphenylene, 90·0 (2)° for (1), 90·0 (6)° for (2)–(4). The angle defined by C(01′)—C(02′)—C(03′) [or equivalently C(01)—C(02)—C(03)] is 120·3 (2)° in benzo[*b*]biphenylene, 122·6 (2)° in (1) and 122·5 (8)° in (2)–(4). The angle defined by C(02′)—C(03′)—C(04′) [or equivalently C(02)—C(03)—C(04)] is 118·4 (3) and 115·2 (2)° for benzo[*b*]biphenylene and (1). In the benzo groups fused to the cyclobutadiene ring in (2)–(4) [*i.e.* C(02′)—C(03′)—C(04′)] the angle is 116·0 (11)° and in the benzo group fused to biphenylene [*i.e.* C(02)—C(03)—C(04)] the angle ranges from 117·8 (3) to 120·5 (8)° for carbons which are fused and not fused to a helicene fragment, respectively. The angle defined by C(03′)—C(04′)—C(05′) is 121·2 (2), 122·2 (2) and 122·5 (9)° in benzo[*b*]biphenylene, (1) and (2)–(4), respectively. The angle defined by C(03)—C(04)—C(05) is 121·2 (2)° and the corresponding values in (1) and (2)–(4) are 121·9 (5) and 118·9 (8)°. The mean C—H bond length [0·96 (3) Å] and C—C—H angle [120 (2)°] are close to their expected values (Hamilton & Ibers, 1968; Churchill, 1973).

The molecules of benzo[*b*]biphenylene are planar within experimental error (r.m.s.d. = 0·011 and 0·007 Å for molecules 1 and 2, respectively). The molecules of benzo[*b*]biphenylene are slip stacked (Fig. 3) with interplanar spacings of 2·560 (2) and 2·455 (1) Å for molecules 1 and 2, respectively. The interplanar spacing is the perpendicular distance between the least-squares planes defined by two neighboring molecules in a stack. The two separate stacks of benzo[*b*]biphenylene molecules are slipped from molecule over molecule stacking by the slip angles 64·7 and 65·3° for molecules 1 and 2, respectively, with no significant overlap between π orbitals on the molecules along the stacking axis (*a* axis). The slip angle is the angle between the stacking axis (*a* axis) and the least-squares planes of the molecule. Many planar systems stack in a slip-stacked manner (Ferrara, Tessier-Youngs & Youngs, 1985).

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Structure of a 2,3,4-Tri-*O*-acetyl-*β*-D-glucopyranosuronic Acid Derivative

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Abstract. Methyl {1-[(1′*R*)-1′-methoxybutyl]-2,3,4-tri-*O*-acetyl-*β*-D-glucopyranosid}uronate, $C_{18}H_{28}O_{11}$, $M_r = 420{\cdot}41$, orthorhombic, $P2_12_12_1$, $a = 8{\cdot}604$ (3), $b = 13{\cdot}500$ (2), $c = 18{\cdot}664$ (4) Å, $V = 2167{\cdot}9$ Å^3, $Z = 4$, $D_x = 1{\cdot}288$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å, $\mu = 0{\cdot}10$ mm^{-1}, $F(000) = 896$, $T = 298$ K, $R = 0{\cdot}055$ for 1592 observed reflections. The structure was investigated to determine the relative configuration, which could not be established unambiguously by NMR. The endocyclic acetal bond is longer than the exocyclic by 0·045 (7) Å.

Introduction. In a series of papers (Jones, Sheldrick, Kirby & Glenn, 1982*a*,*b*; Edwards, Jones & Kirby, 1986; Allen & Kirby, 1984; Briggs, Glenn, Jones, Kirby & Ramaswamy, 1984; Jones & Kirby, 1984) a large number of organic compounds containing C—O single bonds were investigated to derive crystal structure–reactivity correlations in acetals and glucosides. In general the length of the bond R_1—OR_2 increases with increasing $+I$ and $+M$ effects of the group R_1, and with increasing electron withdrawal by the group OR_2. The lengths of the C—O bonds vary over a range of more than 0·1 Å (Allen & Kirby, 1984). We report here the crystal structure determination of the title compound (see Fig. 1).

Experimental. Crystal size 0·6 × 0·5 × 0·4 mm. Intensity data were collected on a Stoe–Siemens four-circle diffractometer with monochromated Mo $K\alpha$ radiation using the profile-fitting mode involving variable scan width and speed (Clegg, 1981). 2173 reflections were measured with $2\theta_{\max}$ 50°, $+h+k+l$ and three check reflections with no significant intensity change. 2173 unique reflections were recorded, of which 1592 with $F > 4\sigma(F)$ were used for all calculations (*SHELXS*86, Sheldrick, 1985; *SHELX*76, Sheldrick, 1976). Cell constants were refined from $\pm 2\theta$ values of 44 reflec-

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