Ideally, the symmetry of the oxonium ion is C_{3v}. In some cases the site symmetry of H_3O^+ in a crystal is C_{3v}, *e.g.* $H_3O^+Br^-$ (Lundgren, 1970). In other cases, the environment of H_3O^+ has a virtual threefold symmetry where the hydrogen-bond acceptors are situated at the corners of a slightly distorted equilateral triangle, *e.g.* oxonium *p*-toluenesulphonate (Lundgren & Williams, 1973). No noticeable deviation from C_{3v} symmetry for H_3O^+ could be found in this latter case. The O—H distances were 1·008–1·013 (8) Å and the H—O—H angles 109·2–111·2 (5)°.

In the present structure the hydrogen-bond acceptors are arranged asymmetrically around H_3O^+. The O(w)···O bonds are not equal (2·522, 2·579 and 2·673 Å) and the O···O(w)···O angles are also very different (101·8, 108·3 and 133·0°). Nevertheless, the geometry of the oxonium ion is reasonably regular. The incorporation of the oxonium ion in the structure results in a moderate distortion of the ion from its ideal C_{3v} symmetry and in the formation of bent O—H···O hydrogen bonds. The O—H bonds are very similar: 0·986, 0·997 and 0·998 (5) Å, and the H—O—H angles are 110·9, 111·7 and 116·0 (4)°. The O—H···O angles subtended at H(1), H(2) and H(3) assume the values 162·0, 170·7 and 169·2 (4)°, respectively. It should be noted that the longest (and weakest) hydrogen bond, 2·673 Å, is also the most bent. Although the O—H bond distances are not significantly different, it is seen that the shortest O—H bond, 0·986 Å, is associated with the longest O(w)···O bond, in agreement with the well known relation between O—H and O···O distances (*e.g.* Olovsson & Jönsson, 1976).

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2*a*-Phenoxy-*trans*-1-oxadecalin

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Abstract. $C_{15}H_{20}O_2$, $M_r = 232·23$, triclinic, $P\bar{1}$, $a = 11·358$ (4), $b = 10·429$ (4), $c = 5·455$ (2) Å, $\alpha = 102·26$ (2), $\beta = 92·16$ (3), $\gamma = 92·85$ (2)°, $U = 629·9$ Å^3, $Z = 2$, $D_x = 1·225$ g cm^{-3}, μ(Cu $K\alpha$) = 5·5 cm^{-1}. The structure was refined to $R = 0·047$ for 1719 unique reflexions. The intra-annular C—O bond lengths are significantly different (1·405 and 1·448 Å).

Introduction. As part of a systematic examination of the effects of ground-state structure on the reactivity of acetals (Jones, Kennard, Kirby & Osborne, 1978; Jones, Kennard, Kirby, Osborne & Malik, 1978) we have prepared axial and equatorial anomers of several 2-aryloxy-*trans*-1-oxadecalins (Chandrasekhar & Kirby, 1978). The factors responsible for the anomeric effect may also control reactivity in such systems; thus the detailed structure of the acetal group is relevant to the question of stereo-electronic control. This paper

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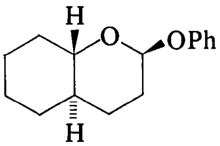
reports the crystal and molecular structure of the acetal (I).



(I)

Colourless plate-like crystals were obtained from methylene dichloride/petroleum spirit; because the compound is slowly attacked by atmospheric moisture, crystals were sealed in Lindemann tubes. Intensities were measured on a Philips PW 1100 diffractometer with graphite-monochromated Cu $K\alpha$ radiation, and a crystal 0·8 × 0·7 × 0·2 mm. Cell dimensions were obtained by least squares from 25 strong reflexions. 1860 reflexions were collected in the range $0 < 2\theta < 120°$; after application of Lp corrections, averaging equivalent reflexions gave 1721 unique reflexions with $F > 4\sigma(F)$.

Table 1. *Fractional atomic coordinates* (×10^4)

The overall isotropic temperature factor for the H atoms is 0·062 (1) Å^2.

	x	*y*	*z*
O(1)	1903 (1)	8893 (1)	9065 (2)
C(2)	3138 (1)	8854 (1)	9076 (3)
C(3)	3545 (1)	7500 (2)	8071 (3)
C(4)	2949 (1)	6887 (2)	5495 (3)
C(10)	1619 (1)	6965 (1)	5612 (3)
C(5)	959 (2)	6450 (2)	3062 (3)
C(6)	−362 (2)	6550 (2)	3259 (3)
C(7)	−669 (2)	7949 (2)	4399 (3)
C(8)	4 (1)	8473 (2)	6937 (3)
C(9)	1313 (1)	8375 (1)	6630 (3)
O(10)	3643 (1)	9687 (1)	7564 (2)
C(11)	3578 (1)	11028 (1)	8319 (3)
C(12)	2998 (1)	11643 (2)	10412 (3)
C(13)	3007 (1)	13006 (2)	10998 (3)
C(14)	3574 (2)	13746 (2)	9543 (3)
C(15)	4141 (2)	13120 (2)	7446 (3)
C(16)	4149 (1)	11760 (2)	6842 (3)
H(21)	3426	9182	11028
H(31)	4489	7566	7895
H(32)	3332	6881	9367
H(41)	3159	5870	4957
H(42)	3265	7413	4123
H(101)	1337	6339	6849
H(51)	1142	5433	2384
H(52)	1264	7023	1752
H(61)	−785	6251	1403
H(62)	−681	5906	4429
H(71)	−439	8576	3127
H(72)	−1606	7960	4673
H(81)	−277	7898	8263
H(82)	−182	9488	7628
H(91)	1601	8935	5285
H(121)	2548	11069	11561
H(131)	2561	13492	12622
H(141)	3580	14806	10026
H(151)	4577	13695	6280
H(161)	4600	11276	5224

The structure was solved by multisolution $\sum_2$ sign expansion using the program *SHELX* (G. M. Sheldrick); the best E map gave positions for all non-H atoms. Least-squares refinement proceeded to $R = 0{\cdot}13$ (all heavy atoms anisotropic), when a difference synthesis showed all H atoms. In the final stages of refinement all C—H bond lengths and H—C—H angles were constrained to be 1·08 Å and 109·5° respectively; an overall isotropic temperature factor for H atoms was refined. Two reflexions clearly in error were omitted. A parameter x was included in the refinement to allow for extinction; this refined to $1{\cdot}9\,(1) \times 10^{-5}$, where F_c was multiplied by $(1 - xF_c^2/\sin\theta)$. The final $R' = \sum w^{1/2}\Delta/\sum w^{1/2}|F_o|$ was 0·064, with a corresponding R of 0·047; the weighting scheme was $w = 1/[\sigma^2(F) + 0{\cdot}0015F^2]$. A final difference map showed no peaks >0·3 e Å^{-3}.

Final atomic coordinates are given in Table 1,* with derived bond lengths, bond angles and torsion angles in Tables 2–4. Diagrams of the structure are given in Figs. 1 and 2.

Discussion. The most striking feature of the structure is the difference in lengths of the ring C—O bonds [C(2)—O(1) 1·405, C(9)—O(1) 1·448 Å]. A similar alternation has been observed in halogeno derivatives

Table 2. *Bond lengths* (Å)

C(2)—O(1)	1·405 (3)	C(2)—C(3)	1·505 (4)
C(2)—O(10)	1·433 (3)	C(3)—C(4)	1·532 (4)
C(4)—C(10)	1·520 (4)	C(5)—C(10)	1·533 (4)
C(5)—C(6)	1·516 (4)	C(6)—C(7)	1·523 (4)
C(7)—C(8)	1·535 (4)	C(8)—C(9)	1·509 (4)
C(9)—O(1)	1·448 (3)	C(9)—C(10)	1·520 (4)
C(11)—O(10)	1·378 (3)	C(11)—C(12)	1·391 (3)
C(11)—C(16)	1·379 (3)	C(12)—C(13)	1·388 (3)
C(13)—C(14)	1·375 (4)	C(14)—C(15)	1·389 (3)
C(15)—C(16)	1·387 (3)		

Table 3. *Bond angles* (°)

C(2)—O(1)—C(9)	114·0 (2)	O(1)—C(2)—C(3)	113·1 (2)
O(1)—C(2)—O(10)	111·6 (2)	C(3)—C(2)—O(10)	106·4 (2)
C(2)—C(3)—C(4)	111·4 (2)	C(3)—C(4)—C(10)	109·9 (2)
C(4)—C(10)—C(5)	113·2 (2)	C(4)—C(10)—C(9)	110·1 (2)
C(5)—C(10)—C(9)	109·1 (2)	C(10)—C(5)—C(6)	111·5 (2)
C(5)—C(6)—C(7)	111·4 (2)	C(6)—C(7)—C(8)	111·2 (2)
C(7)—C(8)—C(9)	109·9 (2)	O(1)—C(9)—C(10)	110·9 (2)
O(1)—C(9)—C(8)	107·6 (2)	C(10)—C(9)—C(8)	111·4 (2)
C(2)—O(10)—C(11)	118·9 (2)	O(10)—C(11)—C(12)	124·4 (2)
O(10)—C(11)—C(16)	115·1 (2)	C(12)—C(11)—C(16)	120·6 (2)
C(11)—C(12)—C(13)	119·0 (3)	C(12)—C(13)—C(14)	121·0 (2)
C(13)—C(14)—C(15)	119·5 (3)	C(14)—C(15)—C(16)	120·3 (3)
C(11)—C(16)—C(15)	119·7 (2)		

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33648 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 4. *Selected torsion angles (°) involving non-hydrogen atoms*

The sign is as defined by Klyne & Prelog (1960).

C(9)–O(1)–C(2)–C(3)	−55·3 (3)	C(2)–C(3)–C(4)–C(10)	−51·6 (3)	C(5)–C(10)–C(9)–C(8)	59·1 (3)
C(9)–O(1)–C(2)–O(10)	64·6 (2)	C(3)–C(4)–C(10)–C(5)	176·3 (2)	C(10)–C(5)–C(6)–C(7)	55·2 (3)
C(2)–O(1)–C(9)–C(10)	57·3 (3)	C(3)–C(4)–C(10)–C(9)	53·9 (3)	C(5)–C(6)–C(7)–C(8)	−54·3 (3)
C(2)–O(1)–C(9)–C(8)	179·4 (2)	C(4)–C(10)–C(5)–C(6)	−179·8 (2)	C(6)–C(7)–C(8)–C(9)	55·7 (3)
O(1)–C(2)–C(3)–C(4)	52·0 (3)	C(9)–C(10)–C(5)–C(6)	−56·8 (3)	C(7)–C(8)–C(9)–O(1)	179·4 (2)
O(10)–C(2)–C(3)–C(4)	−70·8 (3)	C(4)–C(10)–C(9)–O(1)	−56·3 (3)	C(7)–C(8)–C(9)–C(10)	−58·8 (2)
O(1)–C(2)–O(10)–C(11)	66·9 (2)	C(4)–C(10)–C(9)–C(8)	−176·1 (2)	C(2)–O(10)–C(11)–C(12)	−3·4 (3)
C(3)–C(2)–O(10)–C(11)	−169·4 (2)	C(5)–C(10)–C(9)–O(1)	178·9 (2)	C(2)–O(10)–C(11)–C(16)	176·2 (2)

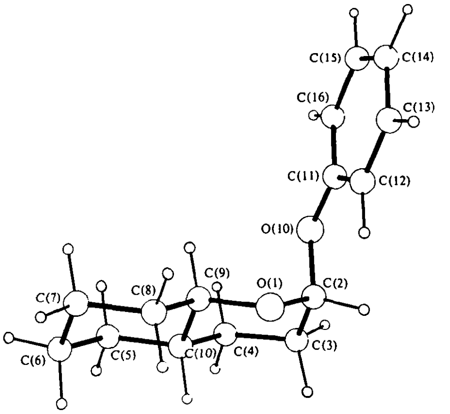

Fig. 1. The title compound showing the labelling scheme for the non-H atoms. H atoms are numbered such that H(*mn*) is the *n*th H atom on C(*m*).

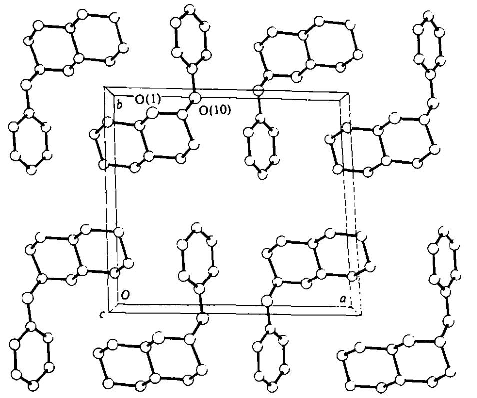

Fig. 2. Packing diagram viewed down **c**; H atoms have been omitted.

of 1,4-dioxane, where the bonds to axial halogens are lengthened (Romers, Altona, Buys & Havinga, 1969); and in two methoxymethoxy enol ethers CH_3OCH_2OR, where R is significantly more electronegative than Me and comparable with the phenyl group of (I) (Jones, Kennard, Kirby & Osborne, 1978; Jones, Kennard, Kirby, Osborne & Malik, 1978). It is not clear whether the exocyclic bond C(2)–O(10) is lengthened in (I); a comparison with the equatorial anomer should prove instructive, and its structure determination is being attempted.

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