

Table 2. Fractional atomic coordinates and equivalent isotropic displacement parameters ( $\text{\AA}^2$ ) for (II)
$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j.$$

	x	y	z	$U_{\text{eq}}$
F	-0.1442 (9)	0.4238 (6)	0.0957 (1)	0.119 (4)
S	0.1995 (3)	0.0120 (3)	0.1285 (1)	0.065 (1)
O1	0.1352 (8)	0.2062 (6)	0.1442 (1)	0.076 (3)
O2	0.0905 (9)	-0.1231 (7)	0.1530 (2)	0.086 (4)
O3	0.4350 (7)	0.0138 (8)	0.1269 (2)	0.093 (4)
C0	-0.1610 (17)	-0.0423 (11)	-0.0457 (3)	0.093 (6)
C1	-0.0989 (13)	0.2475 (10)	0.1544 (2)	0.077 (5)
C2	-0.1385 (15)	0.4388 (10)	0.1389 (2)	0.076 (6)
C3	-0.3578 (13)	0.5215 (12)	0.1529 (2)	0.085 (6)
C4	-0.3555 (20)	0.6021 (13)	0.1956 (3)	0.103 (7)
C5	-0.3466 (18)	0.4707 (16)	0.2309 (3)	0.109 (7)
C6	-0.1713 (24)	0.3278 (16)	0.2297 (3)	0.142 (8)
C7	-0.1403 (26)	0.2141 (16)	0.1994 (3)	0.163 (9)
C61	0.0951 (10)	0.0002 (8)	0.0777 (2)	0.054 (3)
C62	-0.1099 (12)	-0.0784 (9)	0.0706 (2)	0.066 (5)
C63	-0.1889 (14)	-0.0883 (10)	0.0309 (3)	0.073 (5)
C64	-0.0690 (12)	-0.0231 (9)	-0.0025 (2)	0.068 (5)
C65	0.1354 (13)	0.0556 (9)	0.0057 (2)	0.055 (6)
C66	0.2189 (12)	0.0684 (8)	0.0451 (2)	0.059 (4)

Table 3. Selected bond distances ( $\text{\AA}$ ) and angles ( $^\circ$ ) in compounds (I) and (II)

	(I)	(II)
S—O1	1.570 (2)	1.558 (5)
S—O2	1.424 (2)	1.428 (6)
S—O3	1.426 (3)	1.419 (4)
S—C61	1.749 (3)	1.758 (7)
F—C2	1.405 (3)	1.399 (7)
C61—C62	1.380 (4)	1.381 (9)
C62—C63	1.374 (4)	1.369 (12)
C63—C64	1.381 (4)	1.383 (13)
C64—C65	1.378 (5)	1.385 (11)
C65—C66	1.371 (5)	1.370 (12)
C66—C61	1.387 (4)	1.382 (9)
CO—C64	1.496 (5)	1.507 (14)
C1—O1	1.468 (3)	1.479 (9)
C1—C2	1.495 (4)	1.506 (10)
C2—C3	1.516 (4)	1.521 (12)
C3—C4	1.515 (4)	1.499 (14)
C4—C5	1.501 (6)	1.492 (16)
C5—C6	1.523 (6)	1.487 (17)
C6—C1	1.516 (4)	—
C6—C7	—	1.298 (15)
C7—C1	—	1.494 (12)
O1—S—O2	109.6 (1)	109.7 (3)
O2—S—O3	119.7 (2)	119.0 (4)
O3—S—O1	103.8 (1)	104.6 (4)
C61—S—O1	104.6 (1)	105.0 (3)
C61—S—O2	109.0 (1)	108.6 (3)
C61—S—O3	109.1 (2)	109.0 (3)
S—O1—C1	119.4 (2)	119.7 (5)
S—C61—C62	120.2 (2)	119.6 (6)
S—C61—C66	120.0 (2)	119.9 (5)
C62—C61—C66	119.9 (3)	120.5 (7)
C61—C62—C63	119.4 (3)	119.1 (7)
C62—C63—C64	121.9 (3)	122.0 (7)
C63—C64—C65	117.5 (3)	117.3 (7)
C64—C65—C66	122.0 (3)	122.1 (7)
C65—C66—C61	119.3 (3)	118.9 (7)
CO—C64—C63	120.1 (3)	119.8 (7)
CO—C64—C65	122.3 (3)	122.8 (8)
O1—C1—C2	106.9 (2)	105.5 (6)
F—C2—C1	108.3 (2)	105.2 (5)
F—C2—C3	108.2 (3)	107.8 (5)
C1—C2—C3	111.3 (2)	114.1 (7)
C2—C3—C4	110.3 (3)	114.9 (7)
C3—C4—C5	110.4 (3)	116.7 (9)
C4—C5—C6	111.0 (3)	117.3 (9)
C5—C6—C1	110.5 (3)	—

O1—C1—C6	109.0 (2)	—
C2—C1—C6	111.1 (2)	—
O1—C1—C7	—	110.1 (7)
C5—C6—C7	—	125.0 (11)
C6—C7—C1	—	130.7 (9)
C7—C1—C2	—	116.6 (7)

The authors thank the Deutsche Akademische Austauschdienst for financial support, J. W. Bats for help during data collection, and H. Fueb for helpful discussions.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry, along with unit-cell diagrams have been deposited with the IUCr (Reference: AB1200). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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*Acta Cryst.* (1995). **C51**, 2707–2710

## *N*-Acetyl-3,6-di-*tert*-butyl-2-(3,6-di-*tert*-butyl-3*H*-azepin-2-yl)-2,3-dihydro-1*H*-azepine

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(Received 16 May 1995; accepted 28 June 1995)

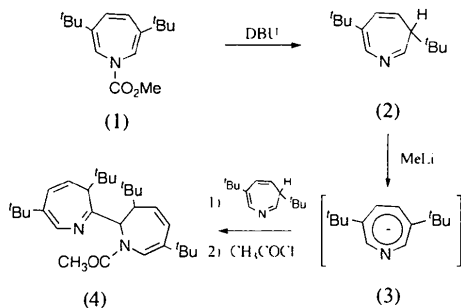
## Abstract

The deprotonation reaction of 3,6-di-*tert*-butyl-3*H*-azepine, (2), using methyllithium, and subsequent quenching of the resulting anionic species with acetyl

chloride gave the title compound, C<sub>30</sub>H<sub>48</sub>N<sub>2</sub>O, (4). The structure of (4) consists of a boat-shaped 3*H*-azepine ring and an envelope-shaped 2,3-dihydro-1*H*-azepine ring which are linked by a C—C single bond between the 2 position of the 3*H*-azepine moiety and the 2 position of the 2,3-dihydro-1*H*-azepine moiety.

### Comment

The elegant conversion of methyl 1*H*-azepine-1-carboxylate into a labile 3*H*-azepine, which was characterized by low-temperature <sup>1</sup>H NMR spectroscopy, has been accomplished using iodotrimethylsilane as a demethoxycarbonylating agent (Vogel, Altenbach, Drossard, Schmickler & Stegelmeier, 1980). Recently, we established an effective demethoxycarbonylation reaction of methyl 3,6-di-*tert*-butyl-1*H*-azepine-1-carboxylate, (1), using 1,8-diazabicyclo[5.4.0]undec-7-ene (DBU) for the synthesis of 3*H*-azepine derivatives that are stabilized by bulky *tert*-butyl substituents on the seven-membered ring (Satake *et al.*, 1991, 1994). The chemistry of the 3*H*-azepine is of interest in connection with the tautomeric 1*H*-azepine/3*H*-azepine anion electronic systems that can be regarded as anti-Hückel eight- $\pi$ -electron systems (Hafner & König, 1963; Kao, 1988). Considering the synthesis and observation of the chemical properties of anti-Hückel eight- $\pi$ -electron species, a proton abstraction reaction of 3*H*-azepine was attempted using methyllithium. To a dry THF solution of the 3*H*-azepine (2), an equimolar THF solution of methyllithium was added slowly at 293 K within 2 h. Equimolar acetyl chloride was added to the resulting deep-red solution in order to quench the anionic species formed. The solvent was removed and the residue was chromatographed on silica gel, giving the dimer (4) in 22% yield as colourless prisms.



The boat-shaped 3*H*-azepine ring of the dimer (4) (Fig. 1) consists of a bow plane [C(7), C(8), C(9)], a stern plane [N(2), C(12), C(11), C(10)] and a bottom plane [N(2), C(7), C(9), C(10)]. The bow plane ( $\delta_1$ ) and the stern plane ( $\delta_2$ ) form dihedral angles of 41.4 (4) 23.3 (3)°, respectively, with the bottom plane. These angles are in good agreement with those predicted (41.0 for  $\delta_1$  and 22.3° for  $\delta_2$ ) by MNDO calculations (Dewar

& Thiel, 1977) for 3*H*-azepine without any substituents. A bond alternation between single and double bonds in the 2-azatriene system of the ring is observed. A similar boat-shaped seven-membered ring has been reported for other 3*H*-azepine derivatives (Carstensen-Oeser, 1972; Lindner & von Gross, 1973; Streef, van der Plas & van Veldhuizen, 1984).

On the other hand, six atoms [N(1), C(1), C(2), C(3), C(4) and C(5)] in the 2,3-dihydro-1*H*-azepine ring, which is linked by a C(6)—C(7) single bond to the 3*H*-azepine ring, are substantially coplanar. It is thought that this planarity is a reflection of extended conjugation from the C(4)=C(3)—C(2)=C(1) diene system to the N(1)—C(13)=O(1) carbamoyl moiety.

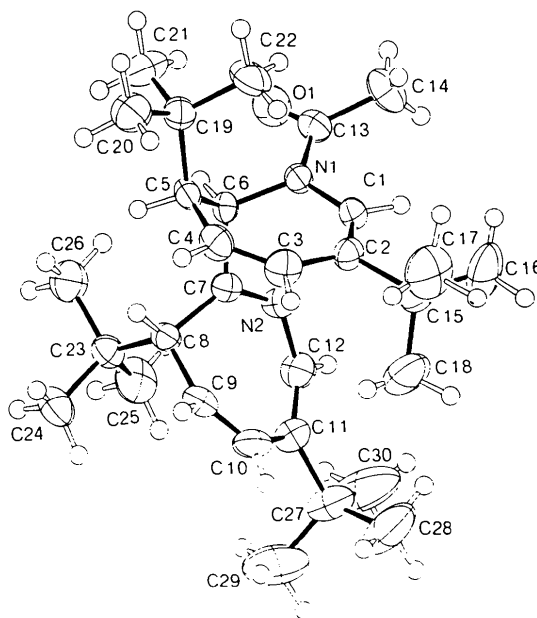


Fig. 1. Displacement ellipsoids at 50% probability and the atomic numbering scheme of the title molecule. H atoms are represented by spheres corresponding to  $B = 1.0 \text{ \AA}^2$ .

### Experimental

Compound (4) was newly synthesized (see *Comment*) and crystals were grown by slow evaporation of a methanol solution.

#### Crystal data

C<sub>30</sub>H<sub>48</sub>N<sub>2</sub>O  
 $M_r = 452.7$   
 Monoclinic  
 $P2_1/a$   
 $a = 12.421 (8) \text{ \AA}$   
 $b = 12.670 (7) \text{ \AA}$   
 $c = 18.450 (9) \text{ \AA}$   
 $\beta = 95.48 (5)^\circ$   
 $V = 2890 (3) \text{ \AA}^3$   
 $Z = 4$   
 $D_c = 1.040 \text{ Mg m}^{-3}$

Mo  $K\alpha$  radiation  
 $\lambda = 0.71073 \text{ \AA}$   
 Cell parameters from 25 reflections  
 $\theta = 10\text{--}11^\circ$   
 $\mu = 0.058 \text{ mm}^{-1}$   
 $T = 298 \text{ K}$   
 Prismatic  
 $0.50 \times 0.25 \times 0.13 \text{ mm}$   
 Colourless

## Data collection

Rigaku AFC-5R diffractometer	$R_{\text{int}} = 0.024$
$\omega/2\theta$ scans	$\theta_{\text{max}} = 26.0^\circ$
Absorption correction: none	$h = 0 \rightarrow 14$
5352 measured reflections	$k = 0 \rightarrow 15$
5091 independent reflections	$l = -21 \rightarrow 20$
2046 observed reflections [ $I > 3\sigma(I)$ ]	3 standard reflections monitored every 97 reflections
	intensity decay: 1.0%

## Refinement

Refinement on $F^2$	$\Delta\rho_{\text{max}} = 0.15 \text{ e } \text{\AA}^{-3}$
$R = 0.047$	$\Delta\rho_{\text{min}} = -0.15 \text{ e } \text{\AA}^{-3}$
$wR = 0.030$	Extinction correction:
$S = 1.33$	$I_{\text{corr}} = I_o(1 + gI_c)$
2046 reflections	Extinction coefficient:
443 parameters	$g = 1.09 \times 10^{-6}$
Only coordinates of H atoms refined	Atomic scattering factors from <i>International Tables for X-ray Crystallography</i> (1974, Vol. IV)
$w = 1/\sigma^2(F)$	
$(\Delta/\sigma)_{\text{max}} = 0.37$	

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

	$x$	$y$	$z$	$B_{\text{eq}}$
O(1)	0.5346 (2)	0.1358 (2)	0.1001 (1)	4.5 (2)
N(1)	0.6921 (2)	0.0605 (2)	0.1475 (1)	2.5 (1)
N(2)	0.7027 (2)	0.1585 (2)	0.2736 (2)	3.1 (2)
C(1)	0.7404 (3)	-0.0265 (3)	0.1851 (2)	2.9 (2)
C(2)	0.8442 (3)	-0.0456 (3)	0.2039 (2)	2.9 (2)
C(3)	0.9352 (3)	0.0190 (3)	0.1844 (2)	3.6 (2)
C(4)	0.9374 (3)	0.1057 (3)	0.1438 (2)	3.4 (2)
C(5)	0.8449 (3)	0.1663 (3)	0.1069 (2)	2.7 (2)
C(6)	0.7466 (3)	0.1635 (3)	0.1511 (2)	2.4 (2)
C(7)	0.7662 (3)	0.1995 (3)	0.2317 (2)	2.5 (2)
C(8)	0.8553 (3)	0.2795 (3)	0.2530 (2)	2.7 (2)
C(9)	0.9282 (3)	0.2294 (3)	0.3130 (2)	3.2 (2)
C(10)	0.9016 (3)	0.1870 (3)	0.3741 (2)	3.6 (2)
C(11)	0.7948 (3)	0.1730 (3)	0.3977 (2)	3.4 (2)
C(12)	0.7079 (3)	0.1673 (3)	0.3493 (2)	3.6 (2)
C(13)	0.5854 (3)	0.0567 (3)	0.1188 (2)	3.2 (2)
C(14)	0.5348 (4)	-0.0505 (4)	0.1077 (3)	4.8 (3)
C(15)	0.8763 (3)	-0.1421 (3)	0.2518 (2)	4.0 (2)
C(16)	0.7802 (5)	-0.2117 (4)	0.2656 (3)	6.8 (4)
C(17)	0.9556 (4)	-0.2117 (4)	0.2148 (3)	6.7 (3)
C(18)	0.9285 (5)	-0.1033 (4)	0.3255 (3)	7.1 (4)
C(19)	0.8240 (3)	0.1417 (3)	0.0237 (2)	3.4 (2)
C(20)	0.9282 (4)	0.1658 (4)	-0.0108 (2)	4.8 (3)
C(21)	0.7351 (4)	0.2129 (4)	-0.0104 (3)	5.4 (3)
C(22)	0.7958 (4)	0.0254 (4)	0.0072 (3)	4.8 (3)
C(23)	0.8191 (3)	0.3959 (3)	0.2649 (2)	3.3 (2)
C(24)	0.9204 (4)	0.4594 (4)	0.2904 (3)	4.9 (3)
C(25)	0.7368 (4)	0.4099 (4)	0.3204 (3)	5.1 (3)
C(26)	0.7704 (4)	0.4382 (4)	0.1918 (3)	4.8 (3)
C(27)	0.7855 (4)	0.1588 (4)	0.4794 (2)	5.3 (3)
C(28)	0.8438 (5)	0.0559 (6)	0.5053 (3)	8.8 (4)
C(29)	0.8440 (5)	0.2514 (7)	0.5206 (3)	9.0 (5)
C(30)	0.6701 (6)	0.1574 (7)	0.4987 (3)	10.4 (5)

Table 2. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

N(1)—C(1)	1.406 (4)	C(7)—C(8)	1.525 (5)
N(1)—C(6)	1.469 (4)	C(8)—C(9)	1.502 (5)
C(1)—C(2)	1.326 (5)	C(9)—C(10)	1.320 (5)
C(2)—C(3)	1.468 (5)	C(10)—C(11)	1.446 (5)
C(3)—C(4)	1.331 (5)	C(11)—C(12)	1.336 (5)
C(4)—C(5)	1.490 (5)	C(6)—C(7)	1.552 (4)

C(5)—C(6)	1.532 (5)	N(1)—C(13)	1.380 (4)
N(2)—C(7)	1.267 (4)	O(1)—C(13)	1.217 (4)
N(2)—C(12)	1.396 (4)	C(13)—C(14)	1.502 (6)
N(1)—C(1)—C(2)	129.1 (4)	C(5)—C(6)—C(7)	116.3 (3)
C(1)—N(1)—C(6)	120.1 (3)	N(2)—C(7)—C(6)	114.5 (3)
C(1)—C(2)—C(3)	125.9 (4)	C(6)—C(7)—C(8)	119.1 (3)
C(2)—C(3)—C(4)	131.0 (4)	O(1)—C(13)—N(1)	122.2 (4)
C(3)—C(4)—C(5)	128.7 (4)	O(1)—C(13)—C(14)	120.5 (4)
C(4)—C(5)—C(6)	111.4 (3)	N(1)—C(13)—C(14)	117.2 (4)
N(1)—C(6)—C(5)	112.5 (3)	C(1)—N(1)—C(13)	120.7 (3)
N(2)—C(12)—C(11)	129.1 (4)	C(1)—C(2)—C(15)	119.2 (4)
C(7)—N(2)—C(12)	127.2 (3)	C(3)—C(2)—C(15)	114.9 (3)
N(2)—C(7)—C(8)	126.5 (3)	C(4)—C(5)—C(19)	113.2 (3)
C(7)—C(8)—C(9)	106.3 (3)	C(6)—N(1)—C(13)	118.0 (3)
C(8)—C(9)—C(10)	128.2 (4)	C(6)—C(5)—C(19)	116.8 (3)
C(9)—C(10)—C(11)	128.3 (4)	C(7)—C(8)—C(23)	116.8 (3)
C(10)—C(11)—C(12)	120.7 (4)	C(9)—C(8)—C(23)	117.2 (3)
C(12)—C(11)—C(27)	121.3 (4)	C(10)—C(11)—C(27)	117.9 (4)
N(1)—C(6)—C(7)	109.5 (3)	C(3)—C(4)—C(5)—C(6)	33.4 (6)
C(3)—C(4)—C(5)—C(6)	33.4 (6)	C(1)—C(2)—C(3)—C(4)	-2.2 (7)
N(1)—C(1)—C(2)—C(3)	-4.6 (6)	C(7)—N(2)—C(12)—C(11)	-35.0 (7)
N(1)—C(6)—C(5)—C(4)	-73.5 (4)	C(7)—C(8)—C(9)—C(10)	-52.5 (5)
N(2)—C(7)—C(6)—C(5)	-152.6 (3)	C(2)—C(1)—N(1)—C(6)	-27.0 (6)
C(5)—C(6)—C(7)—C(8)	28.4 (5)	C(2)—C(3)—C(4)—C(5)	2.2 (7)
N(2)—C(7)—C(8)—C(9)	56.8 (5)	C(8)—C(7)—N(2)—C(12)	-8.2 (6)
N(2)—C(12)—C(11)—C(10)	9.8 (7)	C(8)—C(9)—C(10)—C(11)	3.4 (7)
C(1)—N(1)—C(6)—C(5)	72.9 (4)	C(9)—C(10)—C(11)—C(12)	24.5 (7)
C(1)—N(1)—C(6)—C(7)	-58.1 (4)	C(1)—N(1)—C(13)—O(1)	163.8 (4)

H atoms were located from a difference Fourier map; their coordinates were refined but the displacement parameter of each was fixed at the value of the equivalent isotropic displacement parameter of the C atom to which it is attached. Calculations were performed on a VAX 3100 computer using *TEXSAN* (Molecular Structure Corporation, 1985) at the X-ray Laboratory of Okayama University.

Data collection: Rigaku AFC system (Rigaku Corporation, 1990). Cell refinement: Rigaku AFC system. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1985). Program(s) used to solve structure: *MITHRIL* (Gilmore, 1984). Program(s) used to refine structure: *TEXSAN*. Molecular graphics: *ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *TEXSAN*.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AS1196). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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*Acta Cryst.* (1995). **C51**, 2710–2712

## Anomalin. A Dihydropyranocoumarin Derivative from the Plant *Lingusticum elatum*

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(Received 27 January 1995; accepted 21 June 1995)

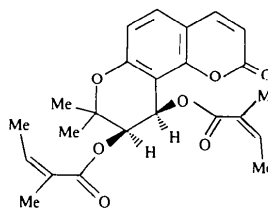
### Abstract

The title compound, 9,10-dihydro-8,8-dimethyl-2-oxo-2*H*,8*H*-benzo[1,2-*b*:3,4-*b'*]dipyran-9,10-diyl 2-methyl-2-butenolate, C<sub>24</sub>H<sub>26</sub>O<sub>7</sub>, contains a highly planar coumarin nucleus and a substituted dihydropyran ring (C), which has a distorted half-chair conformation, with an 8 $\alpha$ ,9 $\beta$  orientation. The conformation of ring C is further supported by the two angelyloxy (2-methyl-2-butenoyloxy) substituents at positions C9 and C10, which are *cis* oriented and thus cannot both occupy equatorial positions with respect to the plane of ring C. The conformations of the two angelyloxy substituents are different, as indicated by their endocyclic torsion angles. The most striking of these angles are O1'—C2'—C4'=C6' and O1'—C2'—C4'—C5' [−137.7 (5) and 43.7 (6)°, respectively, in the chain at C10 and 155.8 (5) and −24.7 (9)°, respectively, in the chain at C9]. These variations are due to two intramolecular hydrogen bonds, namely, C16—H161···O1' [C16···O1' 3.056 (7) Å] and C7''—H7Y···O3'' [C7''···O3'' 2.955 (12) Å]. The methyl substituents, C15 and C16, at position C8 are  $\alpha$  and  $\beta$  oriented, respectively. The

crystal structure is stabilized by a weak C4—H41···O3' hydrogen bond [C4···O3' 3.297 (6) Å] between the screw-related molecules.

### Comment

The present analysis of 9,10-dihydro-8,8-dimethyl-2-oxo-2*H*,8*H*-benzo[1,2-*b*:3,4-*b'*]dipyran-9,10-diyl 2-methyl-2-butenolate, (I), was aimed at exploring the shapes of the dihydropyran ring (C) and the two angelyloxy (2-methyl-2-butenoyloxy) side chains at positions C9 and C10 (Fig. 1). The absolute configuration of (I) was established as 9*R*,10*R* by comparison of its spectroscopic data with those of other dihydrocoumarins (Murray, Stewart & Brown, 1982) and confirmed by the present X-ray analysis.



(I)

The coumarin nucleus is highly planar, whereas the dihydropyran ring approaches a half-chair conformation, with an 8 $\alpha$ ,9 $\beta$  orientation and a ring displacement asymmetric parameter  $\Delta C_2^{2,9} = 3.7 (2)^\circ$  (Duax, Weeks & Rohrer, 1974).

The two angelyloxy substituents at positions C9 and C10 are *cis* oriented, with an O1'—C9—C10—O1'' torsion angle of  $-43.8 (4)^\circ$ , thus precluding them from both occupying equatorial positions with respect to the least-squares plane of ring C. The dihedral angles between the least-squares plane of the dihydropyran ring and those of the angelyloxy side chains at C9 and C10 are 104.6 (2) and 87.1 (1)°, respectively. The two angelyloxy side chains differ in their conformations and show variations in some of their corresponding geometric parameters. A comparison of the corresponding bond distances, bond angles and endocyclic torsion angles of the two side-chain substituents is presented in Table 2. These deviations seem to be due to the relative motion of neighbouring atoms attempting to attain minimum steric interference in the presence of two intramolecular hydrogen bonds [C16···O1' 3.056 (7), H161···O1' 2.60 (4) Å and C16—H161···O1' 112 (3)°; C7'···O3'' 2.955 (12), H7Y···O3'' 2.30 (9) Å and C7—H7Y···O3'' 117 (8)°]. The two methyl groups, C15 and C16, present at position C8 are  $\alpha$  and  $\beta$  oriented, respectively.

The crystal structure consists of molecules hydrogen bonded through donor C atoms and acceptor carbonyl O atoms of the symmetry-related molecule at