>0·18 e Å⁻³. Atom coordinates are given in Table 1, with derived parameters in Table 2 and Fig. 1.*

Discussion. The structure determination shows that the product adopts structure (II). The saturated ring thus possesses the chair conformation [maximum deviation from ideal torsion angles (Bucourt & Hainaut, 1965) is 7·5°]. Its substituents are equatorial. The junction between the saturated and unsaturated rings is diaxial;

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*Lists of structure factors, torsion angles and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35576 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
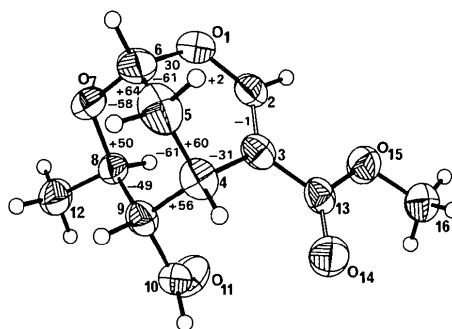

Fig. 1. The molecule of the title compound, showing the atom labelling and the ring torsion angles (to nearest degree). Torsion-angle e.s.d.'s were all < 0·5°. *N.B.* The labelling does not correspond to the IUPAC numbering of the title.

the latter ring adopts the sofa conformation (maximum torsion-angle deviation 5·3°).

The absolute configuration could not be deduced from the crystallographic data; the configuration given here is consistent with the known configuration of secologanin (Battersby, Burnett & Parsons, 1969) and the assumed reaction mechanism (Tietze & Uzar, to be published).

There are no unusually short non-bonded distances.

References

Battersby, A. R., Burnett, A. R. & Parsons, P. G. (1969). *J. Chem. Soc. C*, pp. 1187–1192.
Bucourt, R. & Hainaut, D. (1965). *Bull. Soc. Chim. Fr.* pp. 1366–1378.

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

C(2)–O(1)	1·353 (4)	C(2)–C(3)	1·341 (4)
C(3)–C(4)	1·497 (4)	C(4)–C(5)	1·523 (5)
C(4)–C(9)	1·556 (4)	C(5)–C(6)	1·490 (6)
C(6)–O(1)	1·452 (4)	C(6)–O(7)	1·398 (4)
C(8)–O(7)	1·449 (4)	C(8)–C(9)	1·524 (4)
C(10)–C(9)	1·499 (4)	C(10)–O(11)	1·188 (4)
C(12)–C(8)	1·507 (5)	C(13)–C(3)	1·464 (4)
C(13)–O(14)	1·204 (4)	C(13)–O(15)	1·338 (4)
C(16)–O(15)	1·438 (4)		
C(2)–O(1)–C(6)	117·1 (3)	O(1)–C(2)–C(3)	124·9 (3)
C(2)–C(3)–C(4)	119·3 (3)	C(2)–C(3)–C(13)	121·6 (3)
C(4)–C(3)–C(13)	119·0 (3)	C(3)–C(4)–C(5)	107·1 (3)
C(3)–C(4)–C(9)	112·1 (3)	C(5)–C(4)–C(9)	107·2 (3)
C(4)–C(5)–C(6)	107·0 (3)	O(1)–C(6)–C(5)	111·7 (3)
O(1)–C(6)–O(7)	110·1 (3)	C(5)–C(6)–O(7)	112·5 (4)
C(6)–O(7)–C(8)	114·9 (3)	O(7)–C(8)–C(9)	111·5 (3)
O(7)–C(8)–C(12)	105·5 (3)	C(9)–C(8)–C(12)	112·8 (3)
C(4)–C(9)–C(8)	111·4 (3)	C(4)–C(9)–C(10)	109·7 (3)
C(8)–C(9)–C(10)	111·2 (3)	C(9)–C(10)–O(11)	124·7 (3)
C(3)–C(13)–O(14)	123·3 (3)	C(3)–C(13)–O(15)	113·8 (3)
O(14)–C(13)–O(15)	122·9 (3)	C(13)–O(15)–C(16)	116·1 (3)

(6aRS,9RS,10aRS)-4-Bromo-1-methoxy-6,6,9-trimethyl-6a,7,8,9,10,10a-hexahydro-6H-dibenzo[b,d]pyran

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Abstract. $C_{17}H_{23}BrO_2$, $M_r = 339·28$, triclinic, $P\bar{1}$, $a = 7·378$ (4), $b = 9·740$ (5), $c = 11·406$ (6) Å, $\alpha = 80·83$ (4), $\beta = 81·26$ (4), $\gamma = 81·88$ (4)°, $U = 794·0$ Å³, $Z = 2$, $D_x = 1·419$ Mg m⁻³, $\mu = 2·6$ mm⁻¹. Final $R = 0·064$ for 1323 independent reflexions. The substitution pattern, which could not be unam-
biguously inferred from chemical or spectroscopic evidence, has been determined.

Introduction. The title compound (II) was synthesized from the cannabinoid analogue (I) (von Kiedrowski & Tietze, unpublished). A crystal structure determination

$C_{17}H_{23}BrO_2$

was undertaken to determine the position of the Br atom, which could not be inferred from chemical and spectroscopic evidence.

(I)   (II)
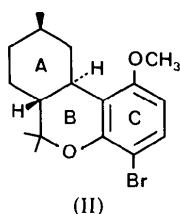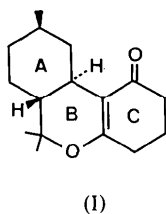
Large colourless crystals were grown from diisopropyl ether/petroleum spirit. An irregular fragment (0·3 × 0·3 × 0·15 mm) was used for data collection on a

Table 1. *Atom coordinates* (×10⁴) *and isotropic temperature factors* (Å² × 10³)

	x	y	z	U or U_{eq}
Br	6525 (1)	8005 (1)	6171 (1)	60 (1)*
C(1)	7707 (9)	4592 (9)	9399 (7)	39 (3)*
C(2)	7206 (10)	5962 (9)	9571 (8)	44 (4)*
H(2)	7041	6217	10362	56
C(3)	6940 (10)	6966 (9)	8601 (8)	46 (3)*
H(3)	6667	7931	8721	59
C(4)	7055 (9)	6630 (9)	7462 (7)	42 (3)*
C(41)	7540 (9)	5216 (8)	7297 (6)	33 (3)*
O(5)	7525 (7)	4927 (6)	6169 (5)	46 (2)*
C(6)	7546 (10)	3445 (8)	6013 (7)	39 (3)*
C(61)	9089 (10)	2628 (8)	6673 (6)	37 (3)*
H(61)	10141	3097	6315	50
C(7)	9611 (11)	1101 (8)	6461 (7)	47 (3)*
H(7a)	9843	1066	5614	61
H(7b)	8605	578	6810	61
C(8)	11307 (12)	458 (10)	7014 (7)	58 (4)*
H(8a)	11511	−521	6938	74
H(8b)	12342	907	6591	74
C(9)	11142 (11)	612 (8)	8344 (7)	47 (3)*
H(9)	10204	61	8768	62
C(10)	10547 (10)	2111 (8)	8540 (7)	38 (3)*
H(10a)	10343	2155	9386	52
H(10b)	11511	2666	8167	52
C(101)	8750 (10)	2717 (8)	8005 (6)	36 (3)*
H(101)	7804	2161	8409	47
C(102)	8005 (9)	4178 (8)	8232 (6)	37 (3)*
O(11)	7949 (7)	3515 (6)	10309 (4)	52 (2)*
C(12)	5680 (11)	2988 (10)	6478 (8)	55 (4)*
H(12a)	5677	2039	6346	71
H(12b)	4761	3587	6064	71
H(12c)	5408	3043	7321	71
C(13)	7954 (12)	3493 (10)	4652 (7)	56 (4)*
H(13a)	7951	2567	4464	76
H(13b)	9147	3798	4375	76
H(13c)	7032	4124	4266	76
C(14)	12941 (11)	45 (10)	8857 (7)	61 (4)*
H(14a)	13343	−906	8728	76
H(14b)	12766	100	9701	76
H(14c)	13859	630	8460	76
C(15)	7794 (12)	3835 (10)	11504 (7)	59 (4)*
H(15a)	8008	2964	12019	77
H(15b)	6585	4291	11746	77
H(15c)	8705	4427	11558	77

* U_{eq} is $\frac{1}{3}$ of the trace of the orthogonalized U matrix.

Stoe four-circle diffractometer with monochromated Mo $K\alpha$ radiation. 2953 reflexions were collected ($2\theta_{max}$ = 40°). After application of Lp corrections, averaging equivalents gave 1469 unique data, 1323 with F > $3\sigma(F)$. An attempted absorption correction (based on measurements of equivalent reflexions at different ψ values) proved unsatisfactory, probably because of irregular reflexion shape. Accurate cell constants were obtained from diffractometer-angle measurements of 20 strong reflexions (20 < 2θ < 25°); the cell quoted is the Niggli reduced cell.

The structure was solved by heavy-atom methods and refined to $R_w = \sum w^{1/2}\Delta/\sum w^{1/2}|F_o| = 0\cdot063$, $R = 0\cdot064$. The weighting scheme was $w^{-1} = \sigma^2(F) + 0\cdot001F^2$. All atoms except H were anisotropic; H atoms were included with a riding model [C–H 0·96 Å, H–C–H 109·5°, $U(H) = 1\cdot2U(C)$]. Final atomic parameters are given in Table 1, with derived bond lengths and angles in Table 2. Selected torsion angles are shown in Table 3.*

Discussion. A view of the molecule is shown in Fig. 1. The substitution pattern is unambiguously determined as (II). Ring A adopts the chair and ring B the half-chair conformation; the small deviations from ideal torsion angles (Bucourt & Hainaut, 1965) are comparable with those in (I) (Tietze, von Kiedrowski, Harms, Clegg & Sheldrick, 1980).

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35577 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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

Br–C(4)	1·875 (9)	C(1)–C(2)	1·370 (14)
C(1)–C(102)	1·428 (12)	C(1)–O(11)	1·366 (10)
C(2)–C(3)	1·373 (13)	C(3)–C(4)	1·377 (14)
C(4)–C(41)	1·409 (13)	C(41)–O(5)	1·363 (11)
C(6)–O(5)	1·479 (11)	C(6)–C(61)	1·520 (11)
C(61)–C(7)	1·531 (12)	C(7)–C(8)	1·499 (13)
C(8)–C(9)	1·533 (13)	C(10)–C(9)	1·506 (12)
C(10)–C(101)	1·546 (11)	C(101)–C(61)	1·516 (11)
C(101)–C(102)	1·499 (12)	C(102)–C(41)	1·395 (11)
C(12)–C(6)	1·501 (12)	C(13)–C(6)	1·530 (12)
C(14)–C(9)	1·528 (13)	C(15)–O(11)	1·430 (11)
C(2)–C(1)–C(102)	121·8 (8)	C(2)–C(1)–O(11)	123·7 (8)
C(102)–C(1)–O(11)	114·5 (8)	C(1)–C(2)–C(3)	119·4 (9)
C(2)–C(3)–C(4)	122·0 (9)	Br–C(4)–C(3)	121·1 (7)
Br–C(4)–C(41)	120·8 (7)	C(3)–C(4)–C(41)	118·1 (8)
C(4)–C(41)–O(5)	115·8 (7)	C(4)–C(41)–C(102)	122·0 (8)
O(5)–C(41)–C(102)	122·2 (8)	C(41)–O(5)–C(6)	117·7 (7)
O(5)–C(6)–C(61)	106·0 (7)	O(5)–C(6)–C(12)	109·4 (7)
C(61)–C(6)–C(12)	113·7 (7)	O(5)–C(6)–C(13)	102·6 (7)
C(61)–C(6)–C(13)	113·3 (7)	C(12)–C(6)–C(13)	111·0 (8)
C(6)–C(61)–C(7)	115·8 (8)	C(6)–C(61)–C(101)	112·5 (7)
C(7)–C(61)–C(101)	110·2 (7)	C(61)–C(7)–C(8)	111·2 (9)
C(7)–C(8)–C(9)	112·8 (8)	C(8)–C(9)–C(10)	111·1 (7)
C(8)–C(9)–C(14)	111·5 (8)	C(10)–C(9)–C(14)	111·1 (9)
C(9)–C(10)–C(101)	112·0 (8)	C(61)–C(101)–C(10)	108·1 (7)
C(61)–C(101)–C(102)	110·9 (7)	C(10)–C(101)–C(102)	115·1 (8)
C(1)–C(102)–C(41)	116·1 (8)	C(1)–C(102)–C(101)	122·9 (7)
C(41)–C(102)–C(101)	121·0 (8)	C(1)–O(11)–C(15)	118·6 (8)

Table 3. Torsion angles (°)

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

C(102)–C(1)–C(2)–C(3)	1·5 (13)	C(6)–C(61)–C(101)–C(10)	−168·9 (7)	C(4)–C(41)–C(102)–C(1)	8·7 (11)
C(2)–C(1)–C(102)–C(41)	−7·6 (12)	C(7)–C(61)–C(101)–C(10)	60·2 (9)	O(5)–C(41)–C(102)–C(1)	−171·3 (7)
O(11)–C(1)–C(102)–C(41)	172·0 (7)	C(61)–C(7)–C(8)–C(9)	53·4 (10)	C(41)–O(5)–C(6)–C(61)	−48·8 (8)
C(2)–C(1)–O(11)–C(15)	−4·3 (12)	C(7)–C(8)–C(9)–C(14)	−175·7 (8)	C(41)–O(5)–C(6)–C(13)	−167·9 (7)
C(1)–C(2)–C(3)–C(4)	3·9 (13)	C(14)–C(9)–C(10)–C(101)	178·8 (7)	O(5)–C(6)–C(61)–C(101)	62·3 (8)
C(2)–C(3)–C(4)–C(41)	−2·8 (12)	C(9)–C(10)–C(101)–C(102)	176·3 (7)	C(12)–C(6)–C(61)–C(101)	−57·9 (11)
Br–C(4)–C(41)–C(102)	177·7 (6)	C(61)–C(101)–C(102)–C(41)	6·3 (11)	C(13)–C(6)–C(61)–C(101)	174·1 (8)
C(3)–C(4)–C(41)–C(102)	−3·8 (12)	C(10)–C(101)–C(102)–C(41)	129·4 (8)	C(101)–C(61)–C(7)–C(8)	−58·7 (9)
C(102)–C(41)–O(5)–C(6)	15·4 (10)	O(11)–C(1)–C(2)–C(3)	−178·0 (8)	C(6)–C(61)–C(101)–C(102)	−41·9 (10)
C(4)–C(41)–C(102)–C(101)	−172·1 (8)	C(2)–C(1)–C(102)–C(101)	173·2 (8)	C(7)–C(61)–C(101)–C(102)	−172·8 (7)
O(5)–C(41)–C(102)–C(101)	8·0 (12)	O(11)–C(1)–C(102)–C(101)	−7·3 (11)	C(7)–C(8)–C(9)–C(10)	−51·3 (11)
C(41)–O(5)–C(6)–C(12)	74·1 (9)	C(102)–C(1)–O(11)–C(15)	176·2 (8)	C(8)–C(9)–C(10)–C(101)	54·1 (9)
O(5)–C(6)–C(61)–C(7)	−169·7 (7)	C(2)–C(3)–C(4)–Br	175·7 (7)	C(9)–C(10)–C(101)–C(61)	−59·1 (9)
C(12)–C(6)–C(61)–C(7)	70·1 (10)	Br–C(4)–C(41)–O(5)	−2·3 (10)	C(61)–C(101)–C(102)–C(1)	−174·5 (8)
C(13)–C(6)–C(61)–C(7)	−57·9 (10)	C(3)·C(4)–C(41) O(5)	176·1 (7)	C(10)–C(101)–C(102)–C(1)	−51·4 (10)
C(6)–C(61)–C(7)–C(8)	172·2 (7)	C(4)–C(41)–O(5)–C(6)	−164·6 (7)		

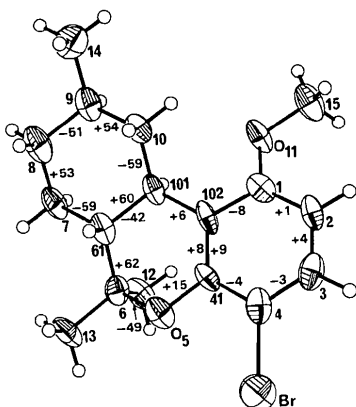Fig. 1. The molecule of (II), showing selected ring torsion angles (°) (e.s.d.'s 1°) and the atom labelling.

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TIETZE, L. F., VON KIEDROWSKI, G., HARMS, K., CLEGG, W. & SHELDRICK, G. M. (1980). Angew. Chem. 92, 130–131.

(2S,6aR,9R,10aR)-6,6,9-Trimethyl-1-oxo-2-phenylseleno-1,2,3,4,6a,7,8,9,10,10a-decahydro-6H-dibenzo[b,d]pyran

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Abstract. $C_{22}H_{28}O_2Se$, $M_r = 405·38$, monoclinic, $P2_1$, $a = 10·298$ (3), $b = 9·915$ (2), $c = 10·835$ (2) Å, $\beta = 114·62$ (4)°, $U = 1004·2$ Å³, $Z = 4$, $D_x = 1·341$ Mg m⁻³, μ(Mo $K\alpha$) = 1·86 mm⁻¹. The structure was solved with the aid of a novel Fourier-recycling procedure and refined to $R = 0·0496$ for 2388 independent data. The presence of the Se atom enabled the absolute configuration to be determined by refining both enantio-