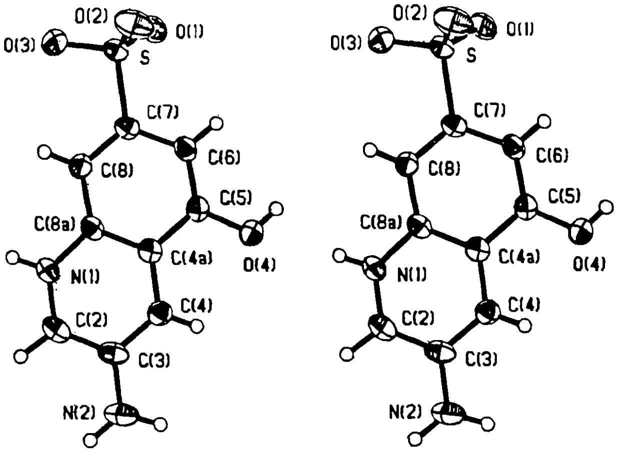

Fig. 1. Stereoview of the 3-amino-5-hydroxyquinoline-7-sulfonic acid molecule.

atom. The quinoline ring system is slightly folded, with an angle of 1·5° between the almost planar six-membered rings; the NH_2 group is slightly non-planar [N(2) is displaced by –0·083 (3) Å from the plane of the three bonded atoms, with this plane rotated 8·8° out of the attached ring plane], and S is 0·118 (1) Å from the plane of the six-membered ring to which it is bonded. Bond lengths and angles (Table 2) are generally close to those in related molecules, *e.g.* 8-hydroxy-2-methylquinoline-5-sulfonic acid monohydrate (Merritt & Duffin, 1970). The molecules are linked by a system of hydrogen bonds (Table 2) involving all the active H atoms.

We thank Drs R. W. Kennedy and J. P. Kutney for suggesting the problem, the Natural Sciences and Engineering Research Council of Canada for financial support, and the University of British Columbia Computing Centre for assistance.

Table 2. *Bond lengths* (Å) *and angles* (°), *with e.s.d.'s in parentheses*

	Uncorr.	Corr.		Uncorr.	Corr.
S–O(1)	1·450 (2)	1·461	C(3)–C(4)	1·384 (4)	1·387
S–O(2)	1·449 (2)	1·460	C(4)–C(4a)	1·402 (4)	1·405
S–O(3)	1·447 (2)	1·459	C(4a)–C(5)	1·428 (4)	1·431
S–C(7)	1·779 (3)	1·787	C(4a)–C(8a)	1·407 (4)	1·412
O(4)–C(5)	1·346 (4)	1·349	C(5)–C(6)	1·368 (4)	1·370
N(1)–C(2)	1·324 (4)	1·326	C(6)–C(7)	1·408 (4)	1·413
N(1)–C(8a)	1·372 (4)	1·375	C(7)–C(8)	1·364 (4)	1·367
N(2)–C(3)	1·375 (4)	1·377	C(8)–C(8a)	1·406 (4)	1·408
C(2)–C(3)	1·390 (5)	1·395			

O(1)–S–O(2)	111·84 (14)	C(4)–C(4a)–C(8a)	119·7 (3)
O(1)–S–O(3)	112·86 (14)	C(5)–C(4a)–C(8a)	118·0 (3)
O(1)–S–C(7)	106·24 (14)	O(4)–C(5)–C(4a)	115·3 (3)
O(2)–S–O(3)	113·49 (15)	O(4)–C(5)–C(6)	125·2 (3)
O(2)–S–C(7)	105·45 (14)	C(4a)–C(5)–C(6)	119·5 (3)
O(3)–S–C(7)	106·23 (13)	C(5)–C(6)–C(7)	120·6 (3)
C(2)–N(1)–C(8a)	124·4 (3)	S–C(7)–C(6)	117·9 (2)
N(1)–C(2)–C(3)	120·7 (3)	S–C(7)–C(8)	119·9 (2)
N(2)–C(3)–C(2)	120·1 (3)	C(6)–C(7)–C(8)	122·1 (3)
N(2)–C(3)–C(4)	122·1 (3)	C(7)–C(8)–C(8a)	117·6 (3)
C(2)–C(3)–C(4)	117·8 (3)	N(1)–C(8a)–C(4a)	116·5 (3)
C(3)–C(4)–C(4a)	120·9 (3)	N(1)–C(8a)–C(8)	121·2 (3)
C(4)–C(4a)–C(5)	122·3 (3)	C(4a)–C(8a)–C(8)	122·2 (3)

Hydrogen bonds

	$X\cdots O$	X–H		
O(5)–$H_a\cdots$O(2)	2·794 (3)	1·00 (5)	1·80 (5)	172 (5)
O(5)–$H_b\cdots$O(1)	2·807 (4)	0·85 (4)	2·00 (4)	158 (4)
O(4)–H···O(5)	2·662 (3)	0·78 (4)	1·89 (4)	174 (4)
N(1)–H···O(5)	2·901 (4)	0·84 (4)	2·24 (4)	136 (3)
N(1)–H···O(3)	2·925 (4)	0·84 (4)	2·37 (4)	124 (3)
N(2)–$H_a\cdots$O(1)	2·827 (4)	0·95 (4)	2·20 (4)	123 (3)
N(2)–$H_b\cdots$O(3)	3·135 (5)	0·91 (5)	2·40 (5)	138 (4)

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Structure of 8-Bromo-6-*endo*-methylisofenchone*

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(*Received* 19 *March* 1986; *accepted* 25 *June* 1986)

Abstract. $C_{11}H_{17}BrO$, $M_r = 245{\cdot}16$, orthorhombic, $P2_12_12$, $a = 10{\cdot}957$ (1), $b = 22{\cdot}409$ (2), $c = 9{\cdot}2455$ (6) Å, $V = 2270{\cdot}1$ (3) Å^3, $Z = 8$ (two molecules per asymmetric unit), $D_x = 1{\cdot}434$ (1) g cm^{-3}, Mo $K\alpha$, $\lambda\alpha_1 = 0{\cdot}70930$ Å, $\mu = 35$ cm^{-1}, $F(000) = 1008$, $T = 295$ K, $R = 0{\cdot}054$ for 988 reflections. The geometry and dimensions of the molecule are similar to

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* (1*S*,4*S*,5*S*,6*S*)-5-Bromomethyl-1,5,6-trimethylbicyclo[2.2.1]-heptan-2-one.

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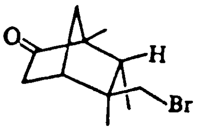
those in related materials; the C(1)—C(7)—C(4) bridgehead angle is 96·5 (9)°, and the mean C—Br bond distance is 1·99 (2) Å.

Introduction. Attempted bromination of 9-bromo-3-*endo*-methylcamphor (Hutchinson & Money, 1984; Rettig & Trotter, 1986) resulted in isomerization to 8-bromo-6-*endo*-methylisofenchone (Money, 1985), whose structure has been confirmed by the present analysis.



Experimental. Colourless crystals, dimensions 0·48 × 0·18 × 0·45 mm, faces {100}, {010}, {001}, ±(1$\bar{1}$0), ±(2$\bar{1}\bar{1}$), Enraf–Nonius CAD-4F diffractometer, lattice parameters from 25 reflections with $\theta = 15$–18°. Intensities for $\theta \leq 25°$, *hkl*: 0 to 13, 0 to 26, 0 to 10, ω–2θ scan, ω-scan width $(0{\cdot}50 + 0{\cdot}35\tan\theta)°$ at 1·1–10° min^{-1}, extended 25% on each side for background measurement, three standard reflections (no decay), Lp and intensity absorption corrections, transmission factors 0·35–0·55, 2292 independent reflections measured, 988 with $I \geq 3\sigma(I)$, where $\sigma^2(I) = S + 4(B_1 + B_2) + (0{\cdot}04S)^2$, S = scan, B_1 and B_2 = background counts. Structure by Patterson and Fourier methods, refined by full-matrix least squares on F, H atoms in calculated positions, $w = 1/\sigma^2(F)$, scattering factors from *International Tables for X-ray Crystallography* (1974), locally written, or locally modified versions of standard programs,* final $R = 0{\cdot}054$, $wR = 0{\cdot}071$ (0·077 for enantiomorph) for 988 reflections, $S = 3{\cdot}02$, 235 parameters, $R = 0{\cdot}146$ for all 2292 reflections, $\Delta/\sigma = 0{\cdot}04$ (mean), 0·23 (maximum), maximum final difference density one peak of +0·6, otherwise <0·34 e Å^{-3}.

Discussion. Final positional parameters are in Table 1, and other data have been deposited.† The two independent molecules (Fig. 1) have similar structure and conformation, the molecular geometry (Table 2) being generally close to that observed for other camphor-like

Table 1. *Final positional (fractional* ×10^4, Br ×10^5) *and equivalent isotropic thermal parameters* (U ×10^3 Å^2) *with e.s.d.'s in parentheses*

	x	y	z	U_{eq}*
Br	27454 (19)	9103 (10)	5345 (21)	80
O	2201 (14)	1627 (6)	7269 (14)	170
C(1)	3653 (16)	1518 (8)	5272 (20)	51
C(2)	2810 (21)	1252 (13)	6420 (18)	79
C(3)	2633 (24)	639 (9)	6347 (20)	77
C(4)	3389 (20)	519 (8)	4974 (20)	58
C(5)	2705 (16)	760 (7)	3677 (19)	41
C(6)	2908 (17)	1457 (7)	3853 (16)	35
C(7)	4424 (16)	967 (9)	5148 (18)	61
C(8)	4267 (20)	2118 (9)	5560 (23)	98
C(9)	3370 (23)	530 (10)	2331 (19)	82
C(10)	1346 (17)	549 (9)	3506 (25)	75
C(11)	1767 (22)	1856 (9)	3732 (22)	81
Br′	32428 (23)	37466 (13)	42683 (22)	97
O′	1638 (14)	3966 (6)	−2447 (15)	83
C(1′)	1917 (13)	3290 (7)	−427 (19)	34
C(2′)	2309 (21)	3703 (10)	−1600 (18)	57
C(3′)	3648 (18)	3762 (11)	−1584 (21)	66
C(4′)	3938 (15)	3409 (9)	−216 (22)	55
C(5′)	3477 (16)	3752 (8)	1083 (17)	41
C(6′)	2071 (14)	3657 (7)	947 (16)	30
C(7′)	3001 (18)	2895 (8)	−307 (20)	65
C(8′)	721 (18)	3004 (8)	−549 (25)	76
C(9′)	3960 (17)	3457 (9)	2419 (24)	68
C(10′)	3885 (16)	4391 (8)	1109 (20)	59
C(11′)	1290 (16)	4213 (9)	1013 (22)	67

* $U_{eq} = \frac{1}{3}$ trace of diagonalized **U** tensor.

Table 2. *Bond lengths* (Å) *and angles* (°), *with e.s.d.'s in parentheses*

Br—C(9)	1·99 (2)	Br′—C(9′)	1·99 (2)
O—C(2)	1·33 (2)	O′—C(2′)	1·23 (2)
C(1)—C(2)	1·53 (3)	C(1′)—C(2′)	1·49 (2)
C(1)—C(6)	1·55 (2)	C(1′)—C(6′)	1·52 (2)
C(1)—C(7)	1·50 (2)	C(1′)—C(7′)	1·49 (2)
C(1)—C(8)	1·53 (2)	C(1′)—C(8′)	1·46 (2)
C(2)—C(3)	1·39 (3)	C(2′)—C(3′)	1·47 (3)
C(3)—C(4)	1·54 (3)	C(3′)—C(4′)	1·53 (3)
C(4)—C(5)	1·51 (2)	C(4′)—C(5′)	1·51 (2)
C(4)—C(7)	1·52 (3)	C(4′)—C(7′)	1·54 (2)
C(5)—C(6)	1·59 (2)	C(5′)—C(6′)	1·56 (2)
C(5)—C(9)	1·53 (2)	C(5′)—C(9′)	1·50 (3)
C(5)—C(10)	1·57 (3)	C(5′)—C(10′)	1·50 (2)
C(6)—C(11)	1·54 (3)	C(6′)—C(11′)	1·51 (2)
C(2)—C(1)—C(6)	103·6 (12)	C(2′)—C(1′)—C(6′)	103·9 (12)
C(2)—C(1)—C(7)	94 (2)	C(2′)—C(1′)—C(7′)	101·2 (15)
C(2)—C(1)—C(8)	119 (2)	C(2′)—C(1′)—C(8′)	118 (2)
C(6)—C(1)—C(7)	99·2 (14)	C(6′)—C(1′)—C(7′)	99·8 (13)
C(6)—C(1)—C(8)	117 (2)	C(6′)—C(1′)—C(8′)	113·6 (15)
C(7)—C(1)—C(8)	119 (2)	C(7′)—C(1′)—C(8′)	117·4 (15)
O—C(2)—C(1)	118 (2)	O′—C(2′)—C(1′)	126 (2)
O—C(2)—C(3)	126 (3)	O′—C(2′)—C(3′)	124 (2)
C(1)—C(2)—C(3)	116 (2)	C(1′)—C(2′)—C(3′)	110 (2)
C(2)—C(3)—C(4)	98 (2)	C(2′)—C(3′)—C(4′)	100 (2)
C(3)—C(4)—C(5)	108·9 (14)	C(3′)—C(4′)—C(5′)	108·9 (15)
C(3)—C(4)—C(7)	101·5 (15)	C(3′)—C(4′)—C(7′)	101·7 (15)
C(5)—C(4)—C(7)	102·5 (15)	C(5′)—C(4′)—C(7′)	101·6 (14)
C(4)—C(5)—C(6)	101·6 (13)	C(4′)—C(5′)—C(6′)	101·3 (14)
C(4)—C(5)—C(9)	106·7 (14)	C(4′)—C(5′)—C(9′)	108·2 (15)
C(4)—C(5)—C(10)	116 (2)	C(4′)—C(5′)—C(10′)	113·5 (15)
C(6)—C(5)—C(9)	110·4 (15)	C(6′)—C(5′)—C(9′)	110·8 (15)
C(6)—C(5)—C(10)	116·1 (15)	C(6′)—C(5′)—C(10′)	115·2 (14)
C(9)—C(5)—C(10)	106 (2)	C(9′)—C(5′)—C(10′)	107·7 (15)
C(1)—C(6)—C(5)	104·3 (13)	C(1′)—C(6′)—C(5′)	104·5 (13)
C(1)—C(6)—C(11)	116·0 (14)	C(1′)—C(6′)—C(11′)	114·5 (13)
C(5)—C(6)—C(11)	117 (2)	C(5′)—C(6′)—C(11′)	116·2 (14)
C(1)—C(7)—C(4)	97·6 (13)	C(1′)—C(7′)—C(4′)	95·3 (13)
Br—C(9)—C(5)	111·7 (15)	Br′—C(9′)—C(5′)	115·1 (13)

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* *ORFLS* and *ORFFE*, Busing, Martin & Levy (1962, 1964); *FORDAP*, Zalkin (unpublished); *ORTEP*II, Johnson (1976).

† Lists of anisotropic thermal parameters (some anomalies suggest limitations in the accuracy of the data), hydrogen positions, torsion angles, and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43190 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

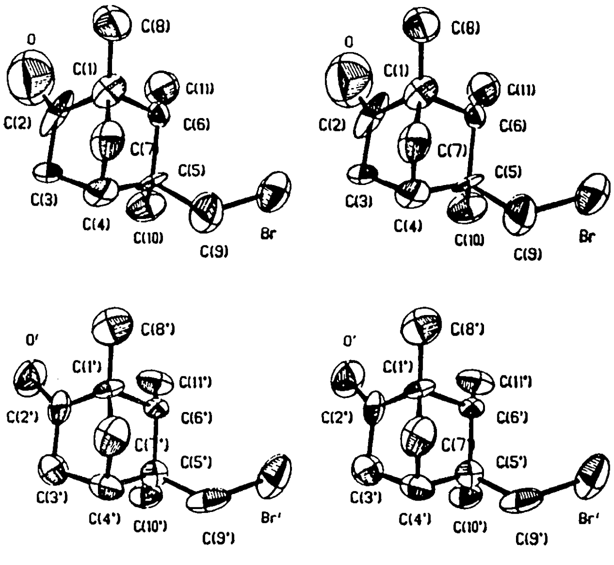

Fig. 1. Stereoviews of the two independent 8-bromo-6-*endo*-methylisofenchone molecules. [Crystallographic numbering for the peripheral methyl groups is in the order of the numbers of the skeletal carbon atoms to which they are bonded; in the usual chemical numbering, C(8), C(9), C(10) are numbered 10, 8, 9, respectively. With respect to the camphor precursor, the methyl groups are scrambled as a result of methyl shifts (Money, 1985).]

derivatives (Rettig & Trotter, 1986). The C(1)—C(7)—C(4) angle is 96·5 (10)°, and C—Br is 1·99 (2) Å. Intermolecular distances correspond to van der Waals interactions.

We thank Dr T. Money for the crystals, the Natural Sciences and Engineering Research Council of Canada for financial support, and the University of British Columbia Computing Centre for assistance.

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Structure of (1*R*,5*R*,6*R*)-1,5-Dimethyl-9-methylene-3-oxabicyclo[4.3.0]nonan-4-one

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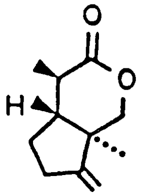
(*Received* 19 *March* 1986; *accepted* 25 *June* 1986)

Abstract. $C_{11}H_{16}O_2$, $M_r = 180·248$, monoclinic, $P2_1$, $a = 10·0945$ (3), $b = 6·5393$ (2), $c = 7·7191$ (2) Å, $\beta = 90·528$ (2)°, $V = 509·52$ (3) Å^3, $Z = 2$, $D_x = 1·174$ (1) g cm^{-3}, Cu $K\alpha$, $\lambda\alpha_1 = 1·54056$ Å, $\mu = 6·0$ cm^{-1}, $F(000) = 196$, $T = 295$ K, $R = 0·046$ for 1020 reflections. The molecule contains *trans*-fused five- and six-membered rings, with envelope and distorted half-chair conformations, respectively; bond lengths and angles are normal.

Introduction. In the development of a synthetic route from camphor to optically active terpenoids and steroids (Hutchinson, Money & Piper, 1984, 1986; Hutchinson & Money, 1985), a lactone intermediate was isolated; the present analysis was designed to establish the relative (and, tentatively, the absolute) configurations of the chiral centres of this lactone.



Experimental. Colourless crystals, dimensions 0·35 × 0·35 × 0·35 mm, {100}, {001}, {10$\bar{1}$} developed with additional small irregular faces, Enraf–Nonius CAD-4*F* diffractometer, lattice parameters from 25 reflections with $\theta = 40$–47°. Intensities for $\theta \leq 75°$, *hkl*: −12 to 12, 0 to 8, 0 to 9, ω–2θ scan, ω-scan width $(0·85 + 0·14\tan\theta)°$ at 1·3–10° min^{-1}, extended 25% on each side for background measurement, three standard reflections (no decay), Lp corrections, no

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