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Structure of 9-Bromo-3-*endo*-methylcamphor*

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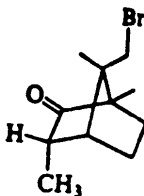*Department of Chemistry, University of British Columbia, Vancouver, BC, Canada V6T 1Y6*

Abstract. $C_{11}H_{17}BrO$, $M_r = 245.16$, orthorhombic, $P2_12_12$, $a = 14.887$ (1), $b = 21.590$ (2), $c = 7.0528$ (3) Å, $V = 2266.8$ (3) Å^3, $Z = 8$ (2 molecules per asymmetric unit), $D_x = 1.436$ (1) g cm^{-3}, Mo $K\alpha$, $\lambda\alpha_1 = 0.70930$ Å, $\mu = 36$ cm^{-1}, $F(000) = 1008$, $T = 295$ K, $R = 0.058$ for 1178 reflections. The geometry and dimensions of the molecule are similar to those in related materials; the C(1)—C(7)—C(4) bridgehead angle is 92.7 (7)°, and the mean C—Br bond distance is 1.98 (1) Å.

Experimental. Colourless crystals, dimensions 0.15 × 0.12 × 0.60 mm, faces {010}, {001}, {120}; Enraf–Nonius CAD-4F diffractometer; lattice parameters from 25 reflections with $\theta = 15$–21°. Intensities for $\theta \leq 25°$, *hkl*: 0 to 17, 0 to 25, 0 to 8; ω–2θ scan, ω scan width $(0.65 + 0.35\tan\theta)°$ at 1.3–10° min^{-1}, extended 25% on each side for background measurement; three standard reflections (no decay); Lp and intensity absorption corrections, transmission factors 0.38–0.57; 2304 independent reflections measured, 1178 with $I \geq 3\sigma(I)$, $\sigma^2(I) = S + 4(B_1 + B_2) + (0.04S)^2$, $S =$ scan, B_1, $B_2 =$ background counts. Structure by Patterson and Fourier methods; refined by full-matrix least squares on F; H atoms in calculated positions;

enantiomorph gave significantly poorer agreement ($R = 0.079$ *vs* 0.071 before final refinement); $w = 1/\sigma^2(F)$; scattering factors from *International Tables for X-ray Crystallography* (1974); locally written, or locally modified versions of standard computer programs; final $R = 0.058$, $wR = 0.076$ for 1178 reflections, $S = 3.12$, 235 parameters; $R = 0.121$ for all 2304 reflections; $\Delta/\sigma = 0.06$ (mean), 0.29 (max.); max. final difference density -0.8 to $+0.6$ e Å^{-3}. Final positional parameters are in Table 1, bond distances

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

	x	*y*	*z*	U_{eq}*
Br	36185 (15)	25692 (9)	42254 (24)	89
O	5701 (8)	1496 (5)	11381 (19)	157
C(1)	5286 (11)	1524 (6)	8040 (19)	45
C(2)	5460 (10)	1801 (8)	9919 (22)	53
C(3)	5399 (9)	2478 (7)	9888 (23)	48
C(4)	5155 (9)	2564 (6)	7773 (17)	40
C(5)	5964 (10)	2443 (9)	6583 (22)	66
C(6)	6136 (10)	1754 (8)	6840 (24)	64
C(7)	4536 (9)	1993 (7)	7392 (20)	46
C(8)	3665 (11)	1942 (7)	8540 (21)	62
C(9)	4305 (11)	1876 (7)	5278 (21)	60
C(10)	5091 (14)	826 (8)	7872 (29)	93
C(11)	6199 (13)	2819 (9)	10703 (24)	93
Br'	36392 (16)	39594 (9)	106580 (24)	89
O'	2172 (8)	5521 (6)	3693 (17)	86
C(1')	2222 (9)	5189 (7)	6994 (21)	46
C(2')	2605 (11)	5331 (7)	5030 (24)	54
C(3')	3586 (11)	5210 (6)	5112 (17)	46
C(4')	3716 (9)	5033 (6)	7193 (19)	42
C(5')	3594 (14)	5597 (7)	8407 (22)	64
C(6')	2618 (13)	5749 (8)	8145 (24)	65
C(7')	2846 (9)	4650 (6)	7569 (17)	40
C(8')	2745 (12)	4085 (7)	6350 (23)	64
C(9')	2683 (10)	4507 (7)	9604 (20)	59
C(10')	1258 (14)	5126 (9)	7013 (28)	89
C(11')	4165 (13)	5760 (10)	4361 (29)	95

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

and angles in Table 2, and other data have been deposited.* The two independent molecules (Fig. 1) have similar structure and conformation, the molecular geometry being generally close to that observed for other camphor derivatives (Rettig & Trotter, 1986). Absolute configuration given by Hutchinson & Money (1984).

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* Lists of anisotropic thermal parameters, H-atom positions, torsion angles and structure factors have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 43044 (16 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* (°), *with e.s.d.'s in parentheses*

Br—C(9)	1·957 (14)	Br'—C(9')	1·994 (15)
O—C(2)	1·27 (2)	O'—C(2')	1·21 (2)
C(1)—C(2)	1·48 (2)	C(1')—C(2')	1·53 (2)
C(1)—C(6)	1·60 (2)	C(1')—C(6')	1·57 (2)
C(1)—C(7)	1·58 (2)	C(1')—C(7')	1·54 (2)
C(1)—C(10)	1·54 (2)	C(1')—C(10')	1·44 (2)
C(2)—C(3)	1·46 (2)	C(2')—C(3')	1·48 (2)
C(3)—C(4)	1·55 (2)	C(3')—C(4')	1·53 (2)
C(3)—C(11)	1·51 (2)	C(3')—C(11')	1·56 (2)
C(4)—C(5)	1·49 (2)	C(4')—C(5')	1·50 (2)
C(4)—C(7)	1·56 (2)	C(4')—C(7')	1·56 (2)
C(5)—C(6)	1·52 (2)	C(5')—C(6')	1·50 (3)
C(7)—C(8)	1·53 (2)	C(7')—C(8')	1·50 (2)
C(7)—C(9)	1·55 (2)	C(7')—C(9')	1·49 (2)

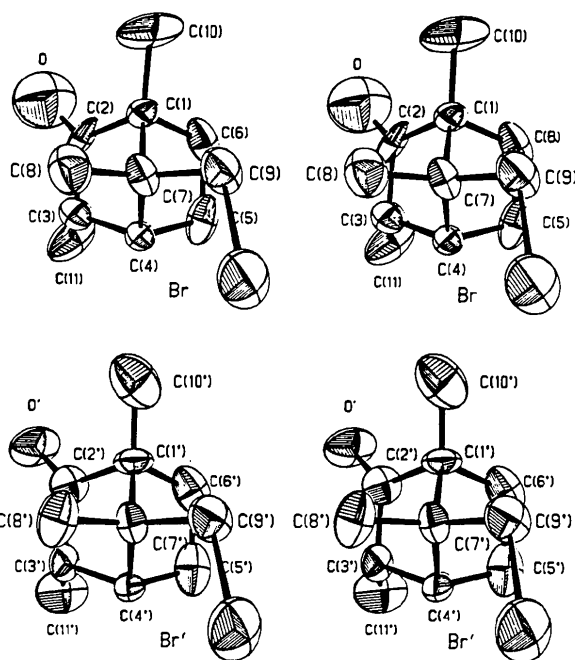|			
C(2)—C(1)—C(6)	102·1 (12)	C(2')—C(1')—C(6')	100·0 (12)
C(2)—C(1)—C(7)	97·1 (12)	C(2')—C(1')—C(7')	99·5 (11)
C(2)—C(1)—C(10)	120·0 (13)	C(2')—C(1')—C(10')	113·5 (15)
C(6)—C(1)—C(7)	101·9 (11)	C(6')—C(1')—C(7')	102·7 (11)
C(6)—C(1)—C(10)	114·4 (14)	C(6')—C(1')—C(10')	116·2 (14)
C(7)—C(1)—C(10)	118·3 (13)	C(7')—C(1')—C(10')	121·6 (14)
O—C(2)—C(1)	124·4 (15)	O'—C(2')—C(1')	125·0 (14)
O—C(2)—C(3)	123 (2)	O'—C(2')—C(3')	127·8 (14)
C(1)—C(2)—C(3)	112·4 (12)	C(1')—C(2')—C(3')	107·2 (13)
C(2)—C(3)—C(4)	98·6 (12)	C(2')—C(3')—C(4')	101·9 (12)
C(2)—C(3)—C(11)	115·5 (13)	C(2')—C(3')—C(11')	113·4 (13)
C(4)—C(3)—C(11)	119·5 (13)	C(4')—C(3')—C(11')	116·5 (14)
C(3)—C(4)—C(5)	109·4 (12)	C(3')—C(4')—C(5')	109·3 (11)
C(3)—C(4)—C(7)	102·1 (11)	C(3')—C(4')—C(7')	101·0 (11)
C(5)—C(4)—C(7)	103·9 (11)	C(5')—C(4')—C(7')	103·5 (11)
C(4)—C(5)—C(6)	103·9 (13)	C(4')—C(5')—C(6')	103·0 (13)
C(1)—C(6)—C(5)	103·5 (12)	C(1')—C(6')—C(5')	104·9 (13)
C(1)—C(7)—C(4)	92·3 (10)	C(1')—C(7')—C(4')	93·1 (10)
C(1)—C(7)—C(8)	113·5 (12)	C(1')—C(7')—C(8')	113·8 (12)
C(1)—C(7)—C(9)	109·4 (11)	C(1')—C(7')—C(9')	108·2 (12)
C(4)—C(7)—C(8)	117·7 (11)	C(4')—C(7')—C(8')	114·6 (11)
C(4)—C(7)—C(9)	115·1 (13)	C(4')—C(7')—C(9')	114·1 (12)
C(8)—C(7)—C(9)	108·0 (12)	C(8')—C(7')—C(9')	111·6 (13)
Br—C(9)—C(7)	110·9 (10)	Br'—C(9')—C(7')	111·5 (10)

Fig. 1. Stereoviews of the two independent 9-bromo-3-*endo*-methylcamphor molecules.

Related literature.

The crystal structure analysis was undertaken to assist in the characterization of the products of bromination of camphor derivatives (Hutchinson & Money, 1984).

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