

The authors express their thanks to Merck (Darmstadt) for supplying the crystals.

#### References

- ERIKSSON, B. (1982). *Acta Chem. Scand. Ser. A*, **36**, 186–188.  
ERIKSSON, B., LARSSON, L. O., NIINISTÖ, L. & VALKONEN, J. (1980). *Inorg. Chem.* **19**, 1207–1210.

- FULLER, C. C. & JACOBSON, R. A. (1976). *Cryst. Struct. Commun.* **5**, 349–352.  
*International Tables for X-ray Crystallography* (1962). Vol. III. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)  
MILINSKI, N., RIBÁR, B. & SATARIĆ, M. (1980). *Cryst. Struct. Commun.* **9**, 473–477.  
RIBÁR, B., MILINSKI, N., BUDOVALČEV, Ž. & KRSTANOVIĆ, I. (1980). *Cryst. Struct. Commun.* **9**, 203–206.  
ROGERS, D. J., TAYLOR, N. J. & TOOGOOD, G. E. (1983). *Acta Cryst.* **C39**, 939–941.

*Acta Cryst.* (1986). **C42**, 1452–1454

## Structures of (+)-8,10- and (+)-9,10-Dibromocamphor\*

BY STEVEN J. RETTIG AND JAMES TROTTER

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

(Received 6 December 1985; accepted 26 June 1986)

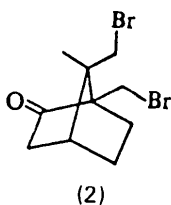
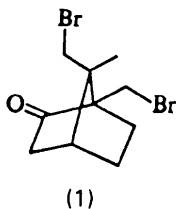
**Abstract.** C<sub>10</sub>H<sub>14</sub>Br<sub>2</sub>O, *M<sub>r</sub>* = 310.03, Mo Kα, λ<sub>α1</sub> = 0.70930 Å, *T* = 295 K; 8,10 isomer (1): monoclinic, *P*2<sub>1</sub>, *a* = 7.438 (1), *b* = 7.293 (1), *c* = 10.177 (1) Å, β = 95.689 (5)°, *V* = 549.3 (1) Å<sup>3</sup>, *Z* = 2, *D<sub>x</sub>* = 1.874 (1) g cm<sup>-3</sup>, μ = 73 cm<sup>-1</sup>, *F*(000) = 304, *R* = 0.051 for 891 reflections; 9,10 isomer (2): orthorhombic, *P*2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>, *a* = 10.607 (1), *b* = 10.610 (1), *c* = 9.8630 (3) Å, *V* = 1109.98 (12) Å<sup>3</sup>, *Z* = 4, *D<sub>x</sub>* = 1.855 (1) g cm<sup>-3</sup>, μ = 72 cm<sup>-1</sup>, *F*(000) = 608, *R* = 0.047 for 805 reflections. The geometries and dimensions of the camphor ring systems are similar to those in related molecules; the C(1)–C(7)–C(4) bridgehead angles are 94.0 (7) and 92.8 (9)°, and the mean C–Br bond distances are 1.968 (6) and 1.960 (9) Å.

**Experimental.** Colourless crystals, dimensions 0.25 × 0.30 × 0.21 mm for 8,10-dibromo isomer (1) [0.48 × 0.48 × 0.28 mm for 9,10-dibromo isomer (2)], crystal faces {001}, (1̄10), (100), (101) [{100}, {011}]; Enraf–Nonius CAD-4F diffractometer; lattice parameters from 25 reflections with θ = 20–26° [18–

24°]. Intensities for θ ≤ 30° [25°]; *hkl*: 0 to 10, 0 to 10, –14 to 14 [0 to 13, 0 to 13, 0 to 12]; ω–2θ scan, ω scan width (0.65 + 0.35 tan θ)° at 1.3–10° min<sup>-1</sup>,

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

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> <sub>eq</sub> *
8,10-Dibromocamphor				
Br(1)	9521 (16)	40000	103038 (9)	60
Br(2)	21598 (17)	36412 (29)	36724 (9)	72
O	2248 (11)	331 (11)	6044 (8)	56
C(1)	2994 (12)	3586 (14)	6515 (8)	33
C(2)	2906 (13)	1523 (16)	6761 (10)	36
C(3)	3833 (15)	1206 (16)	8144 (10)	38
C(4)	4418 (15)	3119 (15)	8571 (10)	85
C(5)	5970 (12)	3707 (19)	7761 (12)	54
C(6)	5018 (12)	3927 (19)	6365 (9)	45
C(7)	2822 (11)	4311 (14)	7928 (8)	29
C(8)	984 (12)	3861 (21)	8383 (7)	43
C(9)	3086 (17)	6387 (18)	8094 (11)	47
C(10)	1629 (14)	4292 (20)	5467 (8)	46
9,10-Dibromocamphor				
Br(1)	40245 (17)	33271 (17)	97060 (15)	68
Br(2)	25380 (20)	29256 (14)	30022 (13)	58
O	590 (9)	1241 (8)	4951 (12)	53
C(1)	2490 (14)	2231 (9)	5858 (10)	34
C(2)	1349 (13)	1337 (12)	5843 (17)	39
C(3)	1390 (13)	623 (14)	7163 (17)	46
C(4)	2564 (16)	1161 (11)	7853 (14)	46
C(5)	3710 (13)	667 (16)	7102 (20)	52
C(6)	3643 (12)	1320 (12)	5727 (16)	38
C(7)	2518 (16)	2574 (11)	7413 (12)	36
C(8)	1360 (13)	3289 (15)	7885 (17)	49
C(9)	3694 (13)	3301 (14)	7748 (13)	40
C(10)	2442 (14)	3362 (12)	4915 (11)	39



\* (1*R*,4*S*,7*R*)- and (1*R*,4*S*,7*S*)-1,7-bis(bromomethyl)-7-methylbicyclo[2.2.1]heptan-2-one.

\* *U*<sub>eq</sub> = 1/3 trace of diagonalized *U* tensor.

extended 25% on each side for background measurement; three standard reflections (average 3% decay); Lp and intensity absorption corrections, transmission factors 0.139–0.287 [0.055–0.170]; 1715 [1458] independent reflections measured, 891 [805] with  $I \geq 3\sigma(I)$ ,  $\sigma^2(I) = S + 4(B_1 + B_2) + (0.04S)^2$ ,  $S = \text{scan}$ ,  $B_1, B_2 = \text{background counts}$ . Structure by Patterson and Fourier methods; refined by full-matrix least squares on  $F$ ; H atoms from difference map, but fixed in calculated positions in final cycles;  $w = 1/\sigma^2(F)$ ; scattering factors, including anomalous dispersion for Br, from *International Tables for X-ray Crystallography* (1974); locally written or locally modified versions of standard computer programs; final  $R = 0.051$ ,  $wR = 0.058$  for 891 reflections,  $S = 2.49$ , 117 parameters [0.047, 0.055 for 805 reflections, 1.68, 119 parameters, including extinction correction,  $g = 2.54(4) \times 10^{-4}$ ];  $R = 0.109$  for all 1715 reflections [0.099 for all 1458 reflexions];  $\Delta/\sigma = 0.01$  (mean),

0.08 (max.) [0.13, 0.50]; max. final difference density 0.6 [0.9]  $e \text{ \AA}^{-3}$  close to Br positions. Thermal parameters interpretable in terms of rigid-body motion; bond lengths corrected for libration. Absolute configuration from Dadson, Lam, Money & Piper (1983). Final positional parameters are in Table 1, bond lengths and angles in Table 2, and other data have been deposited.\*

**Related literature.** The crystal structure analyses were undertaken to assist in the characterization of the products in regiospecific bromination and selective debromination synthetic routes to optically active camphor derivatives (Dadson, Lam, Money & Piper, 1983). The molecular structures (Fig. 1) are similar to those of other camphor derivatives (*e.g.* Phillips & Trotter, 1977).

\* Lists of anisotropic thermal parameters, H-atom coordinates, torsion angles and structure factors have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 43043 (23 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Bond lengths ( $\text{\AA}$ ) and angles ( $^\circ$ ) with *e.s.d.*'s in parentheses

	8,10-Dibromocamphor ( $n = 8$ )		9,10-Dibromocamphor ( $n = 9$ )	
	Uncorr.	Corr.	Uncorr.	Corr.
Br(1)–C( $n$ )	1.960 (8)	1.965	1.963 (13)	1.968
Br(2)–C(10)	1.965 (9)	1.970	1.945 (11)	1.951
O–C(2)	1.207 (13)	1.211	1.198 (18)	1.201
C(1)–C(2)	1.53 (2)	1.534	1.538 (18)	1.542
C(1)–C(6)	1.548 (13)	1.554	1.564 (18)	1.570
C(1)–C(7)	1.549 (11)	1.555	1.576 (16)	1.582
C(1)–C(10)	1.489 (13)	1.492	1.519 (15)	1.522
C(2)–C(3)	1.522 (14)	1.529	1.507 (22)	1.513
C(3)–C(4)	1.51 (2)	1.518	1.529 (20)	1.534
C(4)–C(5)	1.54 (2)	1.549	1.518 (21)	1.524
C(4)–C(7)	1.563 (14)	1.568	1.562 (15)	1.567
C(5)–C(6)	1.532 (14)	1.538	1.524 (23)	1.529
C(7)–C(8)	1.522 (13)	1.527	1.516 (18)	1.522
C(7)–C(9)	1.53 (2)	1.539	1.503 (19)	1.507
C(2)–C(1)–C(6)	103.4 (8)		103.5 (8)	
C(2)–C(1)–C(7)	100.1 (8)		99.6 (11)	
C(2)–C(1)–C(10)	114.9 (9)		117.0 (11)	
C(6)–C(1)–C(7)	102.0 (7)		102.0 (11)	
C(6)–C(1)–C(10)	118.3 (8)		117.7 (11)	
C(7)–C(1)–C(10)	115.7 (8)		114.5 (9)	
O–C(2)–C(1)	129.1 (10)		126.1 (13)	
O–C(2)–C(3)	124.7 (10)		127.7 (12)	
C(1)–C(2)–C(3)	106.2 (8)		106.2 (12)	
C(2)–C(3)–C(4)	102.5 (9)		102.7 (11)	
C(3)–C(4)–C(5)	108.2 (10)		107.8 (11)	
C(3)–C(4)–C(7)	102.0 (8)		102.1 (12)	
C(5)–C(4)–C(7)	101.5 (8)		102.7 (12)	
C(4)–C(5)–C(6)	102.9 (8)		103.9 (11)	
C(1)–C(6)–C(5)	104.8 (7)		104.1 (11)	
C(1)–C(7)–C(4)	94.0 (7)		92.8 (9)	
C(1)–C(7)–C(8)	111.6 (7)		113.5 (13)	
C(1)–C(7)–C(9)	114.7 (8)		110.4 (12)	
C(4)–C(7)–C(8)	114.9 (9)		114.8 (13)	
C(4)–C(7)–C(9)	114.8 (8)		113.9 (13)	
C(8)–C(7)–C(9)	106.7 (9)		110.4 (10)	
Br(1)–C( $n$ )–C(7)	113.1 (6)		111.8 (8)	
Br(2)–C(10)–C(1)	113.4 (7)		113.8 (8)	

Shortest intermolecular distance

Br...Br 3.467 (1)

3.638 (2)

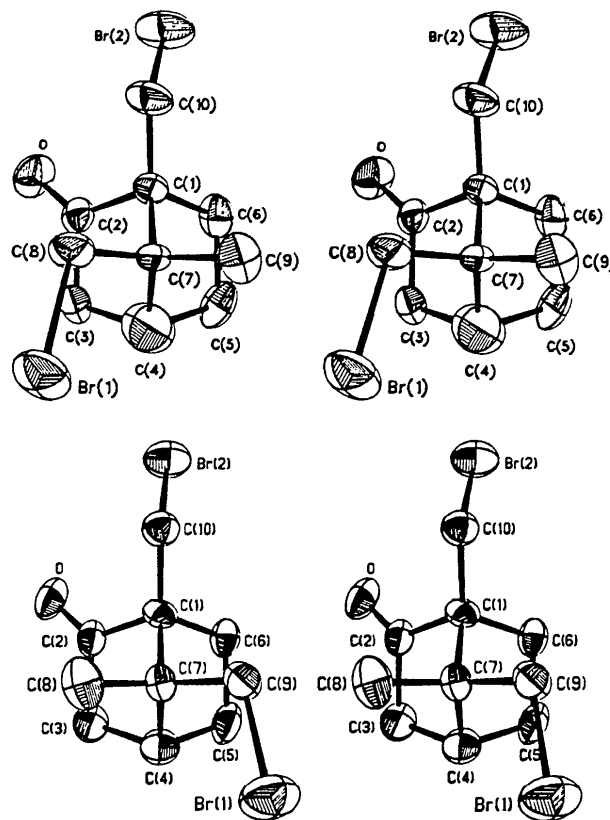


Fig. 1. Stereoviews of the dibromocamphor molecules, 8,10 isomer (top) and 9,10 isomer (bottom).

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

## References

- DADSON, W. M., LAM, M., MONEY, T. & PIPER, S. E. (1983). *Can. J. Chem.* **61**, 343–346.  
*International Tables for X-ray Crystallography* (1974). Vol. IV, pp. 99–102, 149. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)  
 PHILLIPS, S. E. V. & TROTTER, J. (1977). *Acta Cryst.* **B33**, 200–202.

*Acta Cryst.* (1986). **C42**, 1454–1455

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

BY STEVEN J. RETTIG AND JAMES TROTTER

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

(Received 6 December 1985; accepted 26 June 1986)

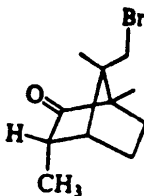
**Abstract.**  $C_{11}H_{17}BrO$ ,  $M_r = 245.16$ , orthorhombic,  $P2_12_12$ ,  $a = 14.887$  (1),  $b = 21.590$  (2),  $c = 7.0528$  (3) Å,  $V = 2266.8$  (3) Å<sup>3</sup>,  $Z = 8$  (2 molecules per asymmetric unit),  $D_x = 1.436$  (1) g cm<sup>-3</sup>, Mo  $K\alpha$ ,  $\lambda\alpha_1 = 0.70930$  Å,  $\mu = 36$  cm<sup>-1</sup>,  $F(000) = 1008$ ,  $T = 295$  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  $0.15 \times 0.12 \times 0.60$  mm, faces {010}, {001}, {120}; Enraf–Nonius CAD-4F diffractometer; lattice parameters from 25 reflections with  $\theta = 15$ – $21^\circ$ . Intensities for  $\theta \leq 25^\circ$ ,  $hkl$ : 0 to 17, 0 to 25, 0 to 8;  $\omega$ – $2\theta$  scan,  $\omega$  scan width  $(0.65 + 0.35 \tan \theta)^\circ$  at  $1.3$ – $10^\circ$  min<sup>-1</sup>, 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  $I \geq 3\sigma(I)$ ,  $\sigma^2(I) = S + 4(B_1 + B_2) + (0.04S)^2$ ,  $S = \text{scan}$ ,  $B_1$ ,  $B_2 = \text{background counts}$ . 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<sup>-3</sup>. Final positional parameters are in Table 1, bond distances

Table 1. Final positional (fractional,  $\times 10^4$ , Br  $\times 10^5$ ) and equivalent isotropic thermal parameters ( $U \times 10^3$  Å<sup>2</sup>) 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



\* (1R,3S,4R,7R)-7-Bromomethyl-1,3,7-trimethylbicyclo[2.2.1]heptan-2-one.

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