

final  $R$  and  $R_w$  for 794 reflexions were 0.085 and 0.104\* [the opposite enantiomorph has the values 0.088 and 0.107 and hence may be rejected at the 0.5% significance level (Hamilton, 1965)]. Final positional and thermal parameters are in Table 1.

**Discussion.** The crystal structure analysis was undertaken to assist the study of a new stereospecific synthesis of 8-bromocamphor (Eck, Mills & Money, 1974). The norbornane skeleton (Fig. 1) has normal geometry; the angles between the three-atom bridge plane, C(1), C(7), C(4) and the four-atom planes of the boat-shaped six-membered ring are 124.5 and 124.2°. Bond lengths and angles (Table 2) are close to normal values, the bridgehead angle being 94°, and intermolecular distances correspond to van der Waals interactions.

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\* A table of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 30783 (11 pp., 1 microfiche). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1 NZ, England.

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### (-)-3,3,4-Trimethyl-1,7-dibromonorbornan-2-one

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**Abstract.**  $C_{10}H_{14}Br_2O$ , orthorhombic,  $P2_12_12_1$ ,  $a = 15.919$  (4),  $b = 6.642$  (1),  $c = 10.965$  (1) Å,  $Z = 4$ ,  $D_x = 1.78$  g cm<sup>-3</sup>,  $\mu(Cu K\alpha) = 95$  cm<sup>-1</sup>. The norbornane skeleton is slightly twisted and bond lengths and angles are normal, the C-C-C bridge angle being 95°.

**Introduction.** Crystals are white needles. Crystal data were measured as for 8-bromocamphor (Bear & Trotter, 1975) and were corrected for absorption. Of 1105 reflexions with  $2\theta \leq 125^\circ$ , 885 (80%) had intensity greater than  $3\sigma$ . The structure was determined and re-

Table 2. Bond lengths (Å) and angles (°), with standard deviations in parentheses

Br—C(8)	1.98 (1)	C(2)—C(3)	1.55 (2)
O—C(2)	1.17 (2)	C(3)—C(4)	1.51 (2)
C(1)—C(2)	1.52 (2)	C(4)—C(5)	1.54 (2)
C(1)—C(6)	1.54 (3)	C(4)—C(7)	1.54 (2)
C(1)—C(7)	1.57 (2)	C(5)—C(6)	1.54 (4)
C(1)—C(10)	1.54 (2)	C(4)—C(8)	1.53 (2)
		C(7)—C(9)	1.55 (2)
C(2)—C(1)—C(6)	106 (2)	C(3)—C(4)—C(7)	103 (1)
C(2)—C(1)—C(7)	100 (1)	C(5)—C(4)—C(7)	103 (1)
C(2)—C(1)—C(10)	113 (1)	C(4)—C(5)—C(6)	101 (2)
C(6)—C(1)—C(7)	100 (1)	C(1)—C(6)—C(5)	106 (1)
C(6)—C(1)—C(10)	118 (2)	C(1)—C(7)—C(4)	94 (1)
C(7)—C(1)—C(10)	117 (1)	C(1)—C(7)—C(8)	109 (1)
O—C(2)—C(1)	126 (2)	C(1)—C(7)—C(9)	114 (1)
O—C(2)—C(3)	129 (2)	C(4)—C(7)—C(8)	115 (1)
C(1)—C(2)—C(3)	105 (1)	C(4)—C(7)—C(9)	115 (1)
C(2)—C(3)—C(4)	102 (1)	C(8)—C(7)—C(9)	109 (1)
C(3)—C(4)—C(5)	107 (1)	Br—C(8)—C(7)	112 (1)

the University of British Columbia Computing Centre for assistance.

#### References

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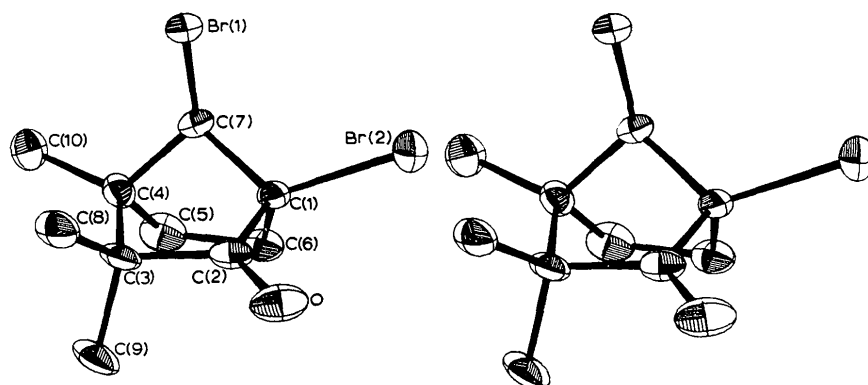


Fig. 1. (-)-3,3,4-Trimethyl-1,7-dibromonorbornan-2-one.

Table 1. Atomic positional and thermal parameters ( $\times 10^4$ ) in trimethyldibromonorbornanone
$$B(H) = 7 \text{ \AA}^2$$

$$f = f^0 \exp [-(b_{11}h^2 + \dots + 2b_{12}hk + \dots)]$$

	$x/a$	$y/b$	$z/c$		$x/a$	$y/b$	$z/c$
Br(1)	1086 (1)	1215 (2)	2015 (1)	H(51)	767	-4598	-853
Br(2)	-917 (1)	1133 (3)	556 (1)	H(52)	500	-4897	573
O	410 (7)	1767 (15)	-1544 (8)	H(61)	-372	-2693	-1201
C(1)	25 (6)	-533 (17)	109 (9)	H(62)	-696	-3237	172
C(2)	586 (8)	380 (18)	-818 (11)	H(71)	390	-1914	1777
C(3)	1420 (7)	-813 (19)	-809 (9)	H(81)	2094	1375	114
C(4)	1230 (7)	-2283 (18)	280 (11)	H(82)	2690	-265	-579
C(5)	582 (10)	-3893 (22)	-91 (13)	H(83)	2212	1481	-1364
C(6)	-215 (8)	-2675 (22)	-333 (11)	H(91)	1000	-2705	-2247
C(7)	645 (6)	-1082 (18)	1114 (8)	H(92)	1587	-852	-2732
C(8)	2165 (8)	603 (23)	650 (11)	H(93)	2018	-2790	-2062
C(9)	1518 (9)	-1922 (30)	-2071 (13)	H(101)	2355	-3944	329
C(10)	2020 (9)	-3117 (23)	919 (15)	H(102)	2373	-1979	1225
				H(103)	1851	-3990	1627

	$b_{11}$	$b_{22}$	$b_{33}$	$b_{12}$	$b_{13}$	$b_{23}$
Br(1)	40.9 (5)	316 (3)	70.3 (9)	4 (1)	-6.9 (5)	-38 (2)
Br(2)	35.7 (5)	506 (5)	129 (1)	41 (1)	-8.3 (7)	-53 (3)
O	86 (6)	319 (29)	75 (7)	-17 (10)	6 (6)	49 (13)
C(1)	30 (4)	257 (28)	59 (9)	-6 (8)	1 (5)	-10 (12)
C(2)	52 (5)	219 (29)	66 (9)	-21 (10)	5 (6)	-21 (15)
C(3)	44 (5)	360 (41)	59 (8)	-30 (11)	13 (5)	-62 (15)
C(4)	41 (5)	239 (29)	100 (11)	12 (10)	6 (7)	-21 (15)
C(5)	77 (8)	261 (34)	134 (15)	-30 (16)	5 (9)	-34 (24)
C(6)	46 (6)	320 (37)	88 (11)	-42 (12)	3 (6)	-28 (17)
C(7)	38 (4)	208 (25)	57 (7)	-14 (10)	-8 (4)	1 (14)
C(8)	44 (5)	509 (55)	94 (10)	-62 (14)	19 (6)	-40 (27)
C(9)	57 (7)	665 (75)	110 (12)	-45 (19)	25 (8)	-183 (28)
C(10)	67 (6)	324 (44)	150 (16)	63 (14)	8 (9)	-22 (22)

finned as for 8-bromocamphor, except that the weighting scheme change was at  $|F_o|=19$ , and hydrogen atoms were located on a difference map, and included but not refined.  $R$  and  $R_w$  were 0.063 and 0.073\* (0.065 and 0.076 for the opposite enantiomorph, which can therefore be rejected). Final positional and thermal parameters are in Table 1.

**Discussion.** The compound was an intermediate of unknown structure isolated during a new stereospecific synthesis of 8-bromocamphor, and the present X-ray study allowed formulation of the mechanisms of the reactions involved in the synthesis (Eck, Mills & Money, 1974). The norbornane skeleton (Fig. 1) is slightly twisted, probably as a result of steric interactions between neighbouring substituent groups; the angles between the C(1), C(7), C(4) plane and the four atom planes of the six-membered ring [C(1), C(2), C(3), C(4) and C(1), C(4), C(5), C(6)] are 127.1 and 119.1° (compare 124.5 and 124.2° in 8-bromocamphor). Bond lengths and angles (Table 2) are close to normal values, the bridgehead angle being 95°, and there are no unusual intermolecular contacts.

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Table 2. Bond lengths (Å) and angles (°) in trimethyldibromonorbornanone, with standard deviations in parentheses

Br(1)-C(7)	1.95 (1)	C(3)-C(4)	1.57 (2)
Br(2)-C(1)	1.93 (1)	C(3)-C(8)	1.52 (2)
O-C(2)	1.25 (1)	C(3)-C(9)	1.58 (2)
C(1)-C(2)	1.48 (2)	C(4)-C(5)	1.54 (2)
C(1)-C(6)	1.55 (2)	C(4)-C(7)	1.53 (1)
C(1)-C(7)	1.52 (1)	C(4)-C(10)	1.54 (2)
C(2)-C(3)	1.55 (2)	C(5)-C(6)	1.53 (2)
Br(2)-C(1)-C(2)	114.1 (8)	C(4)-C(3)-C(9)	113.4 (12)
Br(2)-C(1)-C(6)	114.4 (7)	C(8)-C(3)-C(9)	108.1 (10)
Br(2)-C(1)-C(7)	117.2 (7)	C(3)-C(4)-C(5)	111.0 (10)
C(2)-C(1)-C(6)	108.0 (9)	C(3)-C(4)-C(7)	104.3 (9)
C(2)-C(1)-C(7)	101.8 (8)	C(3)-C(4)-C(10)	114.3 (10)
C(6)-C(1)-C(7)	99.6 (9)	C(5)-C(4)-C(7)	96.5 (9)
O-C(2)-C(1)	127.2 (11)	C(5)-C(4)-C(10)	114.6 (12)
O-C(2)-C(3)	125.1 (11)	C(7)-C(4)-C(10)	114.3 (10)
C(1)-C(2)-C(3)	107.6 (9)	C(4)-C(5)-C(6)	103.6 (12)
C(2)-C(3)-C(4)	99.1 (8)	C(1)-C(6)-C(5)	103.0 (9)
C(2)-C(3)-C(8)	110.6 (11)	Br(1)-C(7)-C(1)	114.4 (8)
C(2)-C(3)-C(9)	108.6 (10)	Br(1)-C(7)-C(4)	119.5 (7)
C(4)-C(3)-C(8)	116.5 (10)	C(1)-C(7)-C(4)	95.0 (8)

## References

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