Acta Cryst. (1975). B31, 903

(+)-8-Bromocamphor

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(*Received* 20 *September* 1974; *accepted* 18 *November* 1974)

Abstract. $C_{10}H_{15}BrO$, monoclinic, $P2_1$, $a=9{\cdot}411$ (1), $b=7{\cdot}928$ (2), $c=6{\cdot}873$ (2) Å, $\beta=92{\cdot}71$ (2)°, $Z=2$, $D_x=1{\cdot}50$ g cm^{-3}, μ(Cu $K\alpha$) = 56 cm^{-1}. The norbornane skeleton is regular with normal bond lengths and angles, the C–C–C bridge angle being 94°.

Introduction. Crystals are white and poorly formed, with no definite faces. Unit-cell and intensity data were measured on a Datex-automated G.E. XRD 6 diffractometer with Cu $K\alpha$ radiation and the usual θ–2θ scan technique. During the data collection the intensity of a check reflexion decreased by 25% as a result of crystal decomposition, and the data were corrected appropriately. Of 1102 reflexions with $2\theta \leq 145°$, 794 (72%) had intensity greater than 3σ above background [$\sigma^2(I)=S+B+(0{\cdot}05S)^2$, where S = scan and B = background count]. Absorption corrections were not made because of the difficulty of defining the crystal shape accurately. The structure was determined by Patterson and electron-density methods, and refined by full-matrix least-squares techniques with minimization of $\sum w(F_o-F_c)^2$; best constancy of average values of $w(F_o-F_c)^2$, taken as a function of F_o, was given by the weighting scheme: $\sqrt{w}=1$ when $|F_o| \leq 10$, $\sqrt{w}=10/|F_o|$ when $|F_o|>10$. Anisotropic thermal parameters and bromine anomalous dispersion corrections were introduced, but the hydrogen atoms could not be located, possibly because of the rather poor quality of the data resulting from the poor crystal specimen, crystal decomposition, and lack of absorption corrections. The

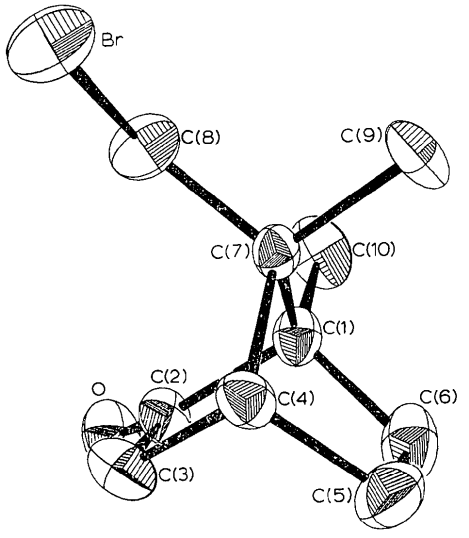

Fig. 1. View of the structure and absolute configuration of (+)-8-bromocamphor.

Table 1. *Atomic positional and thermal parameters* ($\times 10^4$)

The thermal factor expression is $f=f^0 \exp[-(b_{11}h^2+\ldots+2b_{12}hk+\ldots)]$.

	x/a	*y/b*	*z/c*
Br	11274 (1)	5000	1821 (3)
O	6106 (13)	3411 (22)	−782 (15)
C(1)	6885 (16)	3705 (20)	2586 (21)
C(2)	6668 (15)	4216 (25)	459 (21)
C(3)	7318 (19)	6004 (24)	333 (25)
C(4)	7894 (16)	6296 (20)	2390 (18)
C(5)	6620 (19)	6666 (29)	3640 (30)
C(6)	5931 (17)	4903 (50)	3722 (23)
C(7)	8371 (14)	4528 (15)	3070 (16)
C(8)	9487 (15)	3692 (22)	1834 (24)
C(9)	8880 (19)	4418 (23)	5250 (20)
C(10)	6746 (22)	1789 (24)	2918 (27)

Table 1 (*cont.*)

	b_{11}	b_{22}	b_{33}	b_{12}	b_{13}	b_{23}
Br	127 (2)	232 (3)	489 (6)	−11 (3)	26 (2)	−50 (6)
O	194 (16)	407 (36)	244 (25)	−69 (22)	−46 (16)	−44 (27)
C(1)	117 (17)	203 (26)	207 (32)	−27 (17)	19 (19)	−15 (25)
C(2)	108 (16)	303 (35)	158 (30)	−12 (21)	−48 (19)	−36 (28)
C(3)	165 (23)	233 (35)	286 (39)	−9 (25)	−43 (25)	65 (31)
C(4)	152 (20)	157 (23)	194 (32)	12 (17)	−20 (19)	−9 (22)
C(5)	140 (21)	329 (46)	355 (50)	46 (26)	16 (26)	−50 (41)
C(6)	143 (18)	463 (62)	279 (34)	−57 (48)	41 (20)	−25 (58)
C(7)	123 (14)	119 (23)	157 (26)	3 (11)	14 (16)	−16 (16)
C(8)	114 (15)	217 (29)	384 (44)	−28 (18)	20 (20)	−27 (3)
C(9)	216 (23)	251 (33)	179 (28)	8 (22)	−58 (21)	53 (25)
C(10)	235 (27)	204 (32)	364 (46)	−89 (25)	7 (29)	49 (32)

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.

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

* 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.

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)

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Acta Cryst. (1975). B**31**, 904

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

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Abstract. $C_{10}H_{14}Br_2O$, orthorhombic, $P2_12_12_1$, $a=15{\cdot}919$ (4), $b=6{\cdot}642$ (1), $c=10{\cdot}965$ (1) Å, $Z=4$, $D_x=1{\cdot}78$ g cm^{-3}, μ(Cu $K\alpha$)$=95$ cm^{-1}. 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 $2\theta \leq 125°$, 885 (80%) had intensity greater than 3σ. The structure was determined and re-

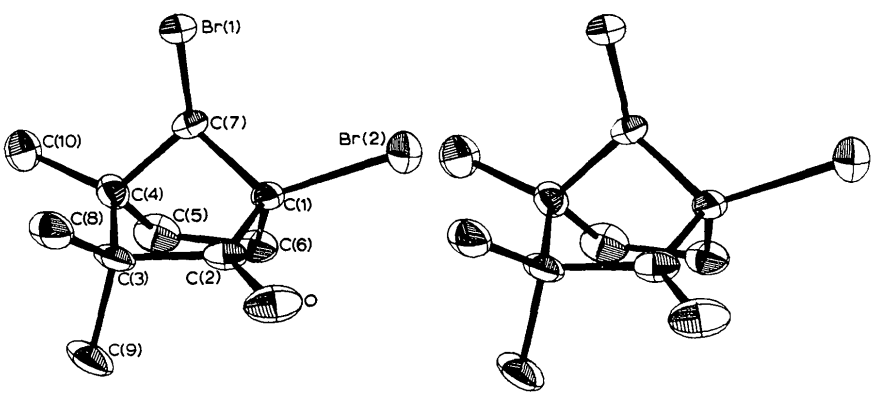

Fig. 1. (–)-3,3,4-Trimethyl-1,7-dibromonorbornan-2-one.