THE CRYSTAL STRUCTURE OF *o*-(1,2-DICARBA-*closo*--DODECABORANE-1-YL)METHYL-CHOLESTEROL

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The structure of $C_2B_{10}H_{11}.CH_2C_{27}H_{45}O$ was determined by X-ray diffraction. This compound crystallizes in the orthorhombic systém with the $P2_12_12_1$ space group. The unit cell parameters are $a = 994{\cdot}0(4)$, $b = 3\,231{\cdot}0(7)$, $c = 1\,039{\cdot}6(2)$ pm, $V = 3\,338 \cdot 10^6$ pm^3, $Z = 4$, calculated density $D_c = 1{\cdot}080$ Mg m^{-3}. $M_r = 542{\cdot}9$, linear absorption coefficient μ(MoKα) = 0·54 cm^{-1} $F(000) = 1\,184$. Intensities were measured at room temperature, radiation used λ(MoKα) = = 71·073 pm. Final $R = 0{\cdot}063$ for 2 099 observed independent reflections. The studied molecule is built from *ortho*-carborane icosahedron connected with the cholesterol through the methylene CH_2 group bonded to the icosahedral carbon atom C(B1) (C—C= 151·0(7) pm) and to the oxygen atom of the cholesterol (C—O = 138·3(7) pm). Valence angle C(B1)—C—O = 108(2)°, torsion angle C(B1)—C—O—C(3) = 164(4)°.

In connection with the Czechoslovak project of Boron Neutron Capture therapy[1] a series of the steroid derivatives of *ortho*-carboranyl methyl ethers have been prepared in the Institute of Inorganic Chemistry[2] by insertion of the acetylene unit of 2-propynyl ethers of steroid alcohols into the decaborane skeleton[3]. In this paper the crystal structure of the *o*-(1,2-dicarba-*closo*-dodecaborane-1-yl) methyl cholesterol which served as a model compound of the above steroid derivatives of *ortho*-carborane is studied.

EXPERIMENTAL

Transparent colourless needle-like crystal measuring 0·2 × 0·2 × 0·3 mm was mounted on the Hilger and Watts four-circle diffractometer; MoKα radiation, Nb filter; cell parameters and standard deviations were refined by least squares from 60 reflections ($7° < \theta < 18°$) (ref.[4]); 3 344 unique independent reflections by learnt profile method[5] were measured by $\theta/2\theta$ scan to $\sin\theta/\lambda = 0{\cdot}594$ Å^{-1} for $h = 0 \to 11$, $k = 0 \to 38$, $l = 0 \to 12$. Reflections with $I > 3\sigma(I)$ were regarded as observed. Intensities of three standards (0 10 3, 2 4 4, 3 5 —1) were measured after every 30 reflections and showed no significant variation; data were corrected for Lorentz and polarization effects but not for absorption. Isotropic extinction correction of the type I was used[6] with Lorentz distribution and with the refined $g = 0{\cdot}329 \cdot 10^{-4}$.

The structure was solved by direct methods using MULTAN 80 (ref.[7]) and subsequent standard Fourier techniques. Full matrix weighted least squares refinement based on F of all

TABLE I

Fractional coordinates and temperature factors ($.10^{-4}$ pm^2) with the estimated standard deviations in the parentheses for the non-hydrogen atoms. $B_{eq} = (4/3) \sum_i \sum_j \beta_{ij} a_i a_j$

Atom	x	y	z	B_{eq}
C(B1)	1·0384(5)	0·0332(1)	−1·1740(4)	4·4(1)
C(B2)	0·8778(6)	0·0221(2)	−1·1713(5)	5·7(1)
B(3)	0·9850(8)	−0·0102(2)	−1·0953(6)	6·2(2)
B(4)	1·1306(7)	−0·0102(2)	−1·1928(6)	5·5(2)
B(5)	1·1013(8)	0·0235(2)	−1·3215(6)	5·8(2)
B(6)	0·9415(8)	0·0450(2)	−1·3044(6)	6·0(2)
B(7)	0·8535(8)	−0·0296(2)	−1·1834(8)	7·6(2)
B(8)	1·0112(9)	−0·0505(2)	−1·2019(7)	7·0(2)
B(9)	1·0813(7)	−0·0301(2)	−1·3424(6)	5·8(2)
B(10)	0·9673(9)	0·0044(2)	−1·4102(6)	7·3(2)
B(11)	0·8245(8)	0·0050(3)	−1·3150(8)	7·4(2)
B(12)	09·141(8)	−0·0420(2)	−1·3364(8)	7·3(2)
C	1·0879(6)	0·0684(2)	−1·0913(5)	5·8(1)
O	0·9782(4)	0·0872(1)	−1·0339(3)	6·2(1)
C(1)	0·9277(6)	0·1775(1)	−0·8185(4)	4·7(1)
C(2)	0·9024(6)	0·1471(1)	−0·9270(5)	5·3(1)
C(3)	1·0114(5)	0·1154(2)	−0·9301(4)	5·2(1)
C(4)	1·0164(6)	0·0923(1)	−0·8043(5)	5·3(1)
C(5)	1·0311(5)	0·1211(1)	−0·6900(4)	4·1(1)
C(6)	1·1202(5)	0·1140(1)	−0·6001(5)	4·8(1)
C(7)	1·1417(6)	0·1397(1)	−0·4828(5)	4·9(1)
C(8)	1·0282(5)	0·1698(1)	−0·4593(4)	3·9(1)
C(9)	0·9872(5)	0·1898(1)	−0·5869(4)	3·7(1)
C(10)	0·9356(5)	0·1575(1)	−0·6844(4)	3·8(1)
C(11)	0·8905(6)	0·2267(1)	−0·5683(5)	4·7(1)
C(12)	0·9339(6)	0·2569(1)	−0·4641(4)	4·7(1)
C(13)	0·9567(4)	0·2353(1)	−0·3363(4)	3·6(1)
C(14)	1·0646(5)	0·2021(1)	−0·3613(4)	3·9(1)
C(15)	1·1065(7)	0·1883(2)	−0·2284(5)	5·5(1)
C(16)	1·0981(7)	0·2271(2)	−0·1475(5)	5·5(1)
C(17)	1·0272(5)	0·2608(1)	−0·2295(4)	3·9(1)
C(18)	0·8259(5)	0·2165(1)	−0·2864(5)	5·1(1)
C(19)	0·7952(5)	0·1406(1)	−0·6471(5)	4·9(1)
C(20)	0·9447(5)	0·2913(1)	−0·1481(4)	4·5(1)
C(21)	0·8791(6)	0·3246(2)	−0·2295(5)	6·6(2)
C(22)	1·0330(6)	0·3116(1)	−0·0466(5)	5·7(1)
C(23)	0·9619(6)	0·3399(2)	0·0465(5)	5·9(7)
C(24)	1·0421(8)	0·3516(2)	0·1594(6)	8·3(2)
C(25)	0·9793(6)	0·3816(2)	0·2534(4)	6·2(1)
C(26)	0·978(1)	0·4236(2)	0·2079(8)	10·0(3)
C(27)	1·045(1)	0·3781(3)	0·3811(7)	11·6(3)

TABLE II

Fractional coordinates with their estimated standard deviations in parentheses and isotropic temperature factors of hydrogen atoms

Atoms	x	y	z	B_{iso}
H(B3)	0·991(5)	−0·008(1)	−0·985(5)	6·(1)
H(B4)	1·224(5)	−0·011(1)	−1·135(5)	6·(1)
H(B5)	1·198(7)	0·043(2)	−1·356(6)	11·(2)
H(B6)	0·913(5)	0·077(1)	−1·307(4)	5·(1)
H(B7)	0·747(7)	−0·035(2)	−1·120(6)	10·(2)
H(B8)	1·049(7)	−0·082(2)	−1·165(6)	11·(2)
H(B9)	1·146(7)	−0·048(2)	−1·394(6)	9·(2)
H(B10)	0·981(8)	0·010(2)	−1·517(8)	13·(2)
H(B11)	0·734(9)	0·014(2)	−1·334(8)	12 (2)
H(B12)	0·846(6)	−0·063(2)	−1·408(6)	8·(1)
H1(C)	1·163(4)	0·052(1)	−0·996(4)	4·8(9)
H2(C)	1·166(7)	0·090(2)	−1·130(6)	9·(2)
H1(C1)	1·012(5)	0·191(1)	−0·830(5)	6·(1)
H2(C1)	0·858(6)	0·200(2)	−0·811(5)	7·(1)
H1(C2)	0·910(4)	0·160(1)	−1·016(4)	3·3(8)
H2(C2)	0·806(8)	0·129(2)	−0·952(7)	12·(2)
H(C3)	1·131(6)	0·127(3)	−0·949(6)	8·(1)
H1(C4)	0·930(4)	0·076(1)	−0·789(4)	3·5(8)
H2(C4)	1·089(5)	0·071(1)	−0·795(5)	6·(1)
H(C6)	1·181(5)	0·091(1)	−0·608(5)	6·(1)
H1(C7)	1·148(5)	0·124(1)	−0·408(4)	5·(1)
H2(C7)	1·250(6)	0·158(2)	−0·498(5)	8·(1)
H(C8)	0·939(6)	0·155(1)	−0·428(4)	6·(1)
H(C9)	1·072(4)	0·201(1)	−0·620(3)	2·6(7)
H1(C11)	0·869(4)	0·239(1)	−0·639(4)	4·1(9)
H2(C11)	0·797(5)	0·213(1)	−0·548(4)	5·(1)
H1(C12)	0·857(4)	0·280(1)	−0·462(4)	4·6(9)
H2(C12)	1·012(4)	0·270(1)	−0·483(4)	3·8(9)
H(C14)	1·140(4)	0·214(1)	−0·392(3)	2·7(7)
H1(C15)	1·028(8)	0·162(2)	−0·221(7)	12·(2)
H2(C15)	1·184(5)	0·178(1)	−0·231(4)	5·(1)
H1(C16)	1·182(5)	0·237(1)	−0·096(5)	6·(1)
H2(C16)	1·056(6)	0·219(2)	−0·078(6)	8·(2)
H(C17)	1·100(3)	0·2791(9)	−0·281(3)	2·4(7)
H1(C18)	0·756(0)	0·239(0)	−0·279(0)	10·0
H2(C18)	0·794(0)	0·195(0)	−0·348(0)	10·0
H3(C18)	0·842(0)	0·204(0)	−0·200(0)	10·0
H1(C19)	0·798(0)	0·130(0)	−0·557(0)	10·0
H2(C19)	0·727(0)	0·163(0)	−0·654(0)	10·0
H3(C19)	0·770(0)	0·118(0)	−0·707(0)	10·0
H(C20)	0·869(0)	0·276(0)	−0·105(0)	10·0

TABLE II
(*Continued*)

Atoms	x	y	z	B_{iso}
H1(C21)	0·949(0)	0·338(0)	−0·285(0)	10·0
H2(C21)	0·808(0)	0·312(0)	−0·285(0)	10·0
H3(C21)	0·837(0)	0·346(0)	−0·172(0)	10·0
H1(C22)	1·077(0)	0·290(0)	0·007(0)	10·0
H2(C22)	1·103(0)	0·329(0)	−0·090(0)	10·0
H1(C23)	0·936(0)	0·366(0)	0·001(0)	10·0
H2(C23)	0·879(0)	0·326(0)	0·080(0)	10·0
H1(C24)	1·064(0)	0·326(0)	0·211(0)	10·0
H2(C24)	1·127(0)	0·365(0)	0·130(0)	10·0
H(C25)	0·882(0)	0·375(0)	0·264(0)	10·0
H1(C26)	0·915(0)	0·426(0)	0·134(0)	10·0
H2(C26)	0·948(0)	0·442(0)	0·279(0)	10·0
H3(C26)	1·071(0)	0·432(0)	0·180(0)	10·0
H1(C27)	1·031(0)	0·350(0)	0·416(0)	10·0
H2(C27)	1·144(0)	0·384(0)	0·372(0)	10·0
H3(C27)	1·005(0)	0·399(0)	0·442(0)	10·0

reflections was used with local programs system[8]. The 41 non-hydrogen atoms were refined anisotropically.

All 58 H atoms were found from the difference Fourier synthesis and refined with isotropic temperature factor. Some hydrogen atoms in the open part of the cholesterol molecule did not refine well. These hydrogen atoms were included in the calculated positions (C—H = 100 pm) with fixed isotropic thermal parameters ($5{\cdot}0 \cdot 10^{-6}$ pm^2) and refined as rigid methyl and methylene groups. Function minimized was $\sum w(|F_o| - |F_c|)^2$ with $w^{-1} = \sigma^2(|F_o|) + (0{\cdot}03|F_o|)^2$; in the final cycle $R = 0{\cdot}063$, $wR = 0{\cdot}083$, $S = 1{\cdot}87$ (for observed reflections), average $\Delta/\sigma = 0{\cdot}02$, $(\Delta/\sigma)_{max} = 0{\cdot}45$ for the H2(C2) atom. Max. and min. peak heights in final difference Fourier synthesis: $+0{\cdot}29 \cdot 10^{-6}$ and $-0{\cdot}24 \cdot 10^{-6}$ e pm^{-3}. Scattering factors from International Tables for X-ray Crystallography (1974) (ref.[9]). Calculation performed on a Siemens 7536 computer.

RESULTS AND DISCUSSION

Final fractional coordinates and the equivalent isotropic thermal parameters B_{eq} for non-hydrogen atoms[10] are given in Table I. Table II gives the fractional coordinates and isotropic thermal parameters for hydrogen atoms. Bond lengths for non-hydrogen atoms and for those hydrogen atoms which were individually refined are summarized in Tables III and IV. In Table III are also presented important valence angles. Torsion angles are given in Table V. Figure 1 shows the structure of the whole molecule with the numbering scheme. The structure consists of the

TABLE III

Interatomic distances (pm) with their estimated standard deviations in the parentheses, refering to the last decimal places for the non-hydrogen atoms and selected valence angles (°)

Atoms	Distance	Atoms	Distance
	The carborane polyhedron		
C(B1)—C(B2)	163·6(7)	B(5)—B(6)	174·(1)
C(B1)—B(3)	170·9(7)	B(5)—B(9)	175·8(8)
C(B1)—B(4)	168·7(7)	B(5)—B(10)	173·(1)
C(B1)—B(5)	168·6(8)	B(6)—B(10)	173·(1)
C(B1)—B(6)	170·6(8)	B(6)—B(11)	174·(1)
C(B1)—C	150·9(7)	B(7)—B(8)	172·(1)
C(B2)—B(3)	168·8(9)	B(7)—B(11)	179·(1)
C(B2)—B(6)	169·1(9)	B(7)—B(12)	175·(1)
C(B2)—B(7)	169·4(8)	B(8)—B(9)	175·(1)
C(B2)—B(11)	168·(1)	B(8)—B(12)	172·(1)
B(3)—B(4)	177·(1)	B(9)—B(10)	174·(1)
B(3)—B(7)	171·(1)	B(9)—B(12)	171·(1)
B(3)—B(8)	172·9(9)	B(10)—B(11)	173·(1)
B(4)—B(5)	175·0(9)	B(10)—B(12)	177·(1)
B(4)—B(8)	176·(1)	B(11)—B(12)	177·(1)
B(4)—B(9)	175·2(9)	C—O	138·3(7)
	The cholesterol part		
O—C(3)	145·1(6)	C(12)—C(13)	151·8(6)
C(1)—C(2)	151·5(7)	C(13)—C(14)	153·9(6)
C(1)—C(10)	153·8(6)	C(13)—C(17)	155·0(6)
C(2)—C(3)	149·2(7)	C(13)—C(18)	152·6(7)
C(3)—C(4)	150·6(7)	C(14)—C(15)	151·1(7)
C(4)—C(5)	151·5(7)	C(15)—C(16)	151·3(7)
C(5)—C(6)	130·7(7)	C(16)—C(17)	155·1(7)
C(5)—C(10)	151·4(6)	C(17)—C(20)	153·5(6)
C(6)—C(7)	149·1(7)	C(20)—C(21)	151·7(7)
C(7)—C(8)	150·8(7)	C(20)—C(22)	152·2(7)
C(8)—C(9)	153·2(6)	C(22)—C(23)	150·8(7)
C(8)—C(14)	150·4(6)	C(23)—C(24)	146·8(8)
C(9)—C(10)	154·2(5)	C(24)—C(25)	151·3(8)
C(9)—C(11)	154·2(6)	C(25)—C(26)	143·7(9)
C(10)—C(19)	154·7(7)	C(25)—C(27)	148·(1)
C(11)—C(12)	152·2(6)		

Atoms	Angle	Atoms	Angle
C(B1)—C—O	108·6(4)	C(23)—C(24)—C(25)	117·2(6)
C—O—C(3)	114·6(4)	C(24)—C(25)—C(26)	113·3(5)
C(22)—C(23)—C(24)	114·5(5)	C(24)—C(25)—C(27)	110·3(6)

ortho-carborane icosahedron connected with the cholesterol through the methylene group bonded to the icosahedral carbon atom C(B1). This methylene group links the carborane unit to cholesterol oxygen atom.

Ortho-carborane icosahedral cage is formed by ten B atoms and two C atoms with C(B1)—C(B2) = 163·6(7) pm, C—B distances range from 168(1) to 170·9(7) pm with the mean value of 168·(68) pm and B—B distances range from 171(1) to

TABLE IV

Interatomic distances (pm) including refined hydrogen atoms, estimated standard deviations in the parentheses refer to the last decimal places

Atoms	Distance	Atoms	Distance
C(B2)—H(CB2)	101·(6)	C(2)—H2(C2)	116·(7)
B(3)—H(B3)	115·(5)	C(3)—H(C3)	126·(6)
B(4)—H(B4)	111·(5)	C(4)—H1(C4)	102·(4)
B(5)—H(B5)	120·(7)	C(4)—H2(C4)	100·(5)
B(6)—H(B6)	109·(4)	C(6)—H(C6)	97·(5)
B(7)—H(B7)	126·(7)	C(7)—H1(C7)	94·(5)
B(8)—H(B8)	115·(7)	C(7)—H2(C7)	124·(6)
B(9)—H(B9)	102·(6)	C(8)—H(C8)	105·(5)
B(10)—H(B10)	114·(8)	C(9)—H(C9)	98·(4)
B(11)—H(B11)	96·(9)	C(11)—H1(C11)	87·(4)
B(12)—H(B12)	122·(6)	C(11)—H2(C11)	105·(4)
C—H1(C)	135·(4)	C(12)—H1(C12)	106·(4)
C—H2(C)	112·6()	C(12)—H2(C12)	91·(4)
		C(14)—H(C14)	90·(4)
		C(15)—H1(C15)	116·(8)
		C(15)—H2(C15)	84·(5)
C(1)—H1(C1)	95·(5)	C(16)—H1(C16)	104·(5)
C(1)—H2(C1)	101·(6)	C(16)—H2(C16)	87·(6)
C(2)—H1(C2)	102·(4)	C(17)—H(C17)	108·(3)

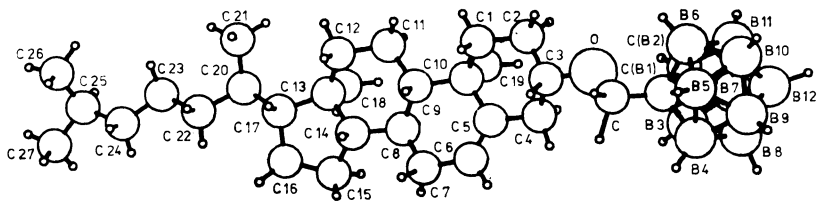

FIG. 1

View of the title compound with the numbering scheme

TABLE V

Torsion angles (°)

Sequence	Angle
Connection of the carbaborane and the cholesterol part of the studied molecule	
C(B1)—C—O—C(3)	164·4(4)
C—O—C(3)—C(1)	152·6(5)
C—O—C(3)—C(2)	151·4(4)

Sequence	Angle	
	A[a]	B[b]
Rings of the steroid skeleton		
C(1)—C(2)—C(3)—C(4)	59·5(5)	58(2)
C(1)—C(2)—C(3)—C(4)	59·5(5)	58(2)
C(1)—C(2)—C(3)—C(4)	59·5(5)	58(2)
C(2)—C(3)—C(4)—C(5)	−54·9(6)	−56(2)
C(3)—C(4)—C(5)—C(10)	49·1(6)	53(1)
C(4)—C(5)—C(10)—C(1)	−44·6(5)	−48(3)
C(5)—C(10)—C(1)—C(2)	49·4(6)	49(4)
C(10)—C(5)—C(6)—C(7)	−0·3(7)	1(2)
C(5)—C(6)—C(7)—C(8)	13·9(7)	13(1)
C(6)—C(7)—C(8)—C(9)	−42·9(5)	−43(2)
C(7)—C(8)—C(9)—C(10)	61·7(5)	62(1)
C(8)—C(9)—C(10)—C(5)	−47·4(5)	−47(2)
C(9)—C(10)—C(5)—C(6)	17·1(6)	16(2)
C(11)—C(9)—C(8)—C(14)	−45·1(5)	−49(1)
C(9)—C(8)—C(14)—C(13)	54·6(5)	57(1)
C(8)—C(14)—C(13)—C(12)	−60·4(5)	−61(1)
C(14)—C(13)—C(12)—C(11)	58·0(5)	56(1)
C(13)—C(12)—C(11)—C(9)	−54·8(5)	−55(1)
C(12)—C(11)—C(9)—C(8)	46·8(5)	50(2)
C(17)—C(13)—C(14)—C(15)	45·7(4)	47(1)
C(13)—C(14)—C(15)—C(16)	−34·9(5)	−34(1)
C(14)—C(15)—C(16)—C(17)	9·9(6)	8(2)
C(15)—C(16)—C(17)—C(13)	18·2(5)	21(2)
C(16)—C(17)—C(13)—C(14)	−38'3(4)	−41(2)

TABLE V
(*Continued*)

Sequence	Angle		
	A[a]	C[c]	D[d]
Side chain conformation of the cholesterol part of the studied molecule			
C(13)—C(17)—C(20)—C(22)	177·9(3)	178·4	−180·0
C(17)—C(20)—C(22)—C(23)	−175·6(4)	−120·2	168·6
C(20)—C(22)—C(23)—C(24)	167·1(5)	171·3	60·4
C(22)—C(23)—C(24)—C(25)	177·2(5)	−170·0	−171·4
C(23)—C(24)—C(25)—C(26)	−75·6(8)	65·9	61·4
C(23)—C(24)—C(25)—C(27)	158·4(6)	−174·8	−176·4

[a] Compound studied; [b] free cholesterol (average over 8 molecules); [c] methylcholesterol methylethers molecule 1; [d] methylcholesterol methylethers molecule 2.

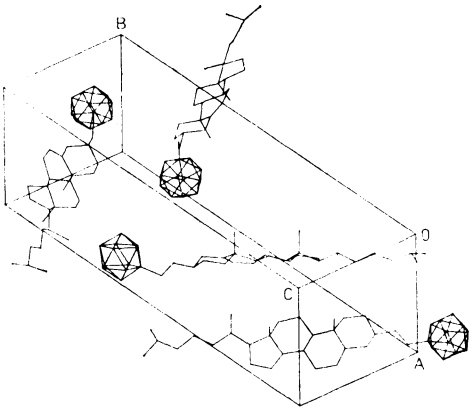

FIG. 2
Contents of the unit cell (H atoms are omitted)

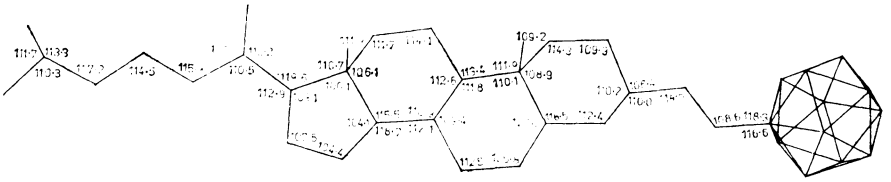

FIG. 3
Valence angles in the cholesterol part of the studied molecule

179(1) pm, mean B—B = 174·4(6) pm. These mean values are evidently shorter than usual values in the related carborane cages (171 and 178 pm respectively)[11-13] and also the e.s.d.'s of the bond lengths are higher.

Similar shortening of the bonds was found also in the 1,3,5-tris-[4-(C-*o*-carboranylmethyl)phenyl]benzene[14] and in 1,3,5-tris[4′-C-*o*-carboranyl)biphenyl-4-yl]benzene[15], where this fact was explained by the thermal motion of the carborane cages as terminal molecular fragments.

The eleven vertex hydrogen atoms bonded to the atoms of the icosahedron have C—H = 101(4) and average B—H = 113(6) pm. The methylene C atom is bonded to the C(B1) atom of the carborane cage with the bond length of 151·0(7) pm. This value agrees well with the distance C—C = 150·4(8) pm which was found in the structure of $(CCH_3)_2B_{10}H_8(CH_3)_2$ (ref.[16]). It is also comparable with some other bond distances of the same type between carbon atom in the carborane cage and C atom of the methylene, for instance 151·6 pm in $(MeC)_2B_8H_8$ (ref.[17]), 152·0 pm in $(MeC)_2B_9H_9$ (ref.[18]). The standard value for the C—C bond of the *closo*-carborane to an alkyl is 150·5 pm (ref.[19]).

The distances and angles in the cholesterol part in the studied compound are comparable with those known from the structures of the free cholesterol molecule[20] and its derivatives[21,22]. The C(5)—C(6) double bond distance, equals to 130·7(7) pm (compare 132·5 pm (ref.[20]), 132 pm (ref.[23])).

The conformation of the side chain of cholesterol can be defined by the torsion angles given in Table V in comparison with the same angles in methylcholesterol methylethers[24]. Cholesterol is connected with the methylene group through the C—O bond 138·3(7) pm long. This value does not differ much from the distance C—O = 136·0 pm found in the cholesterol *p*-octyloxybenzoate[25].

The packing of the molecules in the unit cell is illustrated in Fig. 2, valence angles are given in Fig. 3. Figures were drawn using the program PLUTO (ref.[26]).

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