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The Crystal Conformation of 1,1,9,9-Tetramethylcyclohexadecane

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By semiempirical calculations of cyclohexadecane Dale¹ has found the square, diamond

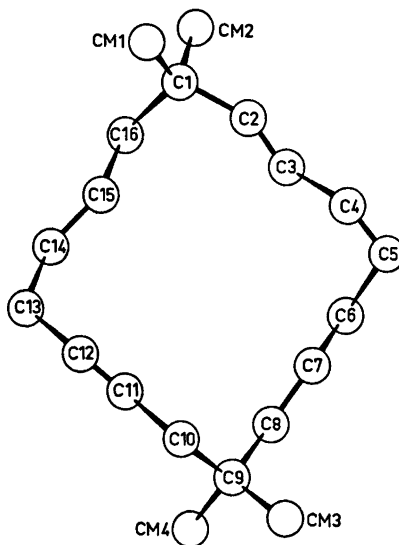


Fig. 1. Schematic drawing showing the molecular conformation.

Table 1. Final fractional coordinates and anisotropic thermal vibration parameters with estimated standard deviations (multiplied by 10^4) for carbon atoms. The symbol CM is used for the methyl carbons.

ATOM	X	Y	Z	B11	B22	B33	B12	B13	B23
C1A	506(11)	3460(4)	2755(4)	263(23)	26(3)	27(3)	41(15)	34(14)	-1(5)
C2A	1383(11)	3320(4)	3366(4)	321(24)	20(3)	23(3)	10(14)	15(14)	-9(4)
C3A	3197(11)	3114(4)	3374(4)	238(23)	20(3)	23(3)	-16(14)	-3(13)	-4(5)
C4A	3957(12)	2958(4)	3983(4)	334(27)	27(3)	26(3)	17(16)	-36(15)	-7(5)
C5A	5730(13)	2741(5)	4029(4)	368(28)	46(5)	29(3)	-24(19)	-75(16)	-0(6)
C6A	6083(10)	2154(5)	3687(4)	181(21)	38(4)	31(3)	-23(15)	-46(13)	-4(6)
C7A	5139(10)	1559(4)	3835(4)	237(23)	36(4)	24(3)	-1(15)	2(13)	-2(5)
C8A	5561(9)	976(4)	3511(3)	167(19)	29(3)	22(2)	21(13)	-9(11)	4(5)
C9A	4622(11)	348(4)	3616(4)	243(23)	27(4)	21(3)	58(15)	-4(14)	9(5)
C10A	2739(11)	433(4)	3525(4)	282(25)	22(3)	24(3)	4(14)	7(14)	3(5)
C11A	2023(10)	679(4)	2948(4)	208(21)	34(3)	24(3)	23(14)	5(13)	-5(5)
C12A	145(10)	816(4)	2912(4)	147(20)	28(3)	42(3)	-0(13)	12(13)	-0(5)
C13A	-640(10)	1080(4)	2347(4)	195(21)	31(4)	41(3)	5(15)	-42(14)	-22(6)
C14A	58(10)	1719(4)	2166(4)	230(22)	21(3)	35(3)	-12(14)	-72(13)	-12(5)
C15A	-129(10)	2254(4)	2575(3)	194(20)	28(3)	23(3)	-13(14)	-2(12)	-9(5)
C16A	548(9)	2472(4)	2374(3)	199(19)	24(3)	23(3)	23(13)	5(12)	6(5)
CM1A	1329(13)	4028(4)	2489(4)	508(33)	29(4)	29(3)	2(18)	6(17)	22(5)
CM2A	-1288(12)	3628(5)	2446(4)	339(27)	47(4)	39(3)	79(19)	-10(17)	-21(6)
CM3A	5069(12)	131(4)	4238(4)	395(29)	39(4)	31(3)	63(18)	-3(15)	24(5)
CM4A	5243(11)	-164(5)	3220(4)	332(27)	53(5)	34(3)	167(18)	39(15)	-18(6)
C1B	-461(11)	6475(4)	9728(3)	289(21)	24(4)	21(3)	4(14)	8(13)	7(5)
C2B	-2273(10)	6589(4)	9652(4)	156(18)	27(3)	25(3)	-31(13)	21(12)	-1(5)
C3B	-3029(10)	6440(4)	9989(4)	169(19)	31(3)	24(3)	-2(13)	14(12)	-3(5)
C4B	-4045(10)	6954(4)	9375(4)	177(22)	24(3)	37(3)	-37(14)	3(14)	2(5)
C5B	-5696(11)	7188(4)	8504(5)	195(22)	34(4)	49(4)	-15(16)	-7(15)	-14(6)
C6B	-5033(10)	7434(4)	8286(4)	183(21)	30(4)	31(3)	37(14)	-69(13)	-12(5)
C7B	-5187(10)	8395(4)	8699(4)	175(20)	29(4)	23(2)	-5(14)	13(12)	-8(5)
C8B	-4531(9)	9317(4)	8451(3)	178(19)	29(3)	17(2)	-14(13)	-3(11)	-3(4)
C9B	-4526(10)	9501(4)	8863(3)	208(21)	28(3)	13(2)	17(14)	11(12)	4(4)
C10B	-3601(10)	9178(4)	9439(3)	234(21)	24(3)	15(2)	8(13)	30(12)	-4(4)
C11B	-1811(10)	9253(4)	9440(3)	210(21)	31(3)	21(2)	-11(14)	-2(12)	3(4)
C12B	-1044(10)	9110(4)	10039(4)	186(21)	27(3)	23(3)	-28(14)	1(12)	-1(4)
C13B	798(11)	8599(4)	10007(4)	262(23)	30(4)	29(3)	-37(15)	-11(14)	4(5)
C14B	1056(9)	8284(4)	9705(3)	153(18)	35(3)	19(2)	-5(13)	-5(11)	1(5)
C15B	142(10)	7693(4)	9932(4)	193(20)	23(3)	27(3)	-31(13)	49(12)	-0(5)
C16B	553(9)	7983(4)	9609(3)	201(19)	32(3)	22(2)	-2(14)	32(11)	-7(5)
CM1B	114(11)	6264(4)	10343(4)	300(25)	31(4)	29(3)	-17(16)	-4(15)	14(5)
CM2B	96(11)	5953(4)	9336(4)	254(22)	35(4)	49(3)	50(15)	1(14)	-9(6)
CM3B	-6333(11)	9791(4)	8929(4)	253(24)	39(4)	26(3)	76(15)	-11(14)	-2(5)
CM4B	-3686(11)	10160(4)	8557(3)	346(27)	26(3)	24(3)	-19(15)	5(14)	-1(5)

Table 2. Interatomic distances, bond angles and dihedral angles with estimated standard deviations.

DISTANCE	(Å)	DISTANCE	(Å)	DISTANCE	(Å)
C1A = C2A	1,85(1)	C2A = C3A	1,91(1)	C3A = C4A	1,53(1)
C4A = C5A	1,48(1)	C5A = C6A	1,58(1)	C6A = C7A	1,58(1)
C7A = C8A	1,48(1)	C8A = C9A	1,54(1)	C9A = C10A	1,51(1)
C10A = C11A	1,52(1)	C11A = C12A	1,52(1)	C12A = C13A	1,51(1)
C13A = C14A	1,51(1)	C14A = C15A	1,48(1)	C15A = C16A	1,49(1)
C16A = C1A	1,51(1)	C1A = CM1A	1,58(1)	C1A = CM2A	1,51(1)
C9A = CM3A	1,53(1)	C9A = CM4A	1,51(1)	C1B = C2B	1,51(1)
C2B = C3B	1,51(1)	C3B = C4B	1,50(1)	C4B = C5B	1,51(1)
C6B = C6B	1,55(1)	C6B = C7B	1,51(1)	C7B = C8B	1,55(1)
C8B = C9B	1,55(1)	C9B = C10B	1,49(1)	C10B = C11B	1,51(1)
C11B = C12B	1,50(1)	C12B = C13B	1,53(1)	C13B = C14B	1,52(1)
C14B = C15B	1,51(1)	C15B = C16B	1,53(1)	C16B = C1B	1,51(1)
C1B = CM1B	1,52(1)	C1B = CM2B	1,50(1)	C9B = CM3B	1,52(1)
C9B = CM4B	1,55(1)				

ANGLE	(°)	ANGLE	(°)
C1A = C2A = C3A	114,2(7)	C2A = C3A = C4A	111,4(8)
C3A = C4A = C5A	114,9(8)	C4A = C5A = C6A	116,8(8)
C5A = C6A = C7A	114,7(8)	C6A = C7A = C8A	114,8(8)
C7A = C8A = C9A	118,8(7)	C8A = C9A = C10A	112,1(6)
C9A = C10A = C11A	116,8(7)	C10A = C11A = C12A	113,1(8)
C11A = C12A = C13A	115,6(8)	C12A = C13A = C14A	115,4(7)
C13A = C14A = C15A	114,2(8)	C14A = C15A = C16A	112,2(7)
C15A = C16A = C1A	118,5(7)	C16A = C1A = C2A	111,8(7)
C16A = C1A = CM1A	118,1(8)	C16A = C1A = CM2A	118,2(7)
C2A = C1A = CM1A	118,4(7)	C2A = C1A = CM2A	105,1(8)
CM1A = C1A = CM2A	118,8(8)	C1B = C2B = C3B	117,7(7)
C2B = C3B = C4B	111,7(7)	C3B = C4B = C5B	113,3(8)
C4B = C5B = C6B	116,8(7)	C6B = C6B = C7B	113,1(8)
C6B = C7B = C8B	118,4(7)	C7B = C8B = C9B	114,2(6)
C8B = C9B = C10B	113,4(6)	C9B = C10B = C11B	116,1(7)
C10B = C11B = C12B	111,2(7)	C11B = C12B = C13B	115,2(7)
C12B = C13B = C14B	112,4(7)	C13B = C14B = C15B	116,7(7)
C14B = C15B = C16B	113,8(7)	C15B = C16B = C1B	117,5(7)
C16B = C1B = C2B	111,2(6)	C16B = C1B = CM1B	108,6(7)
C16B = C1B = CM2B	108,6(7)	C2B = C1B = CM1B	109,2(7)
C2B = C1B = CM2B	111,2(7)	CM1B = C1B = CM2B	107,9(7)
C6B = C9B = CM3B	108,8(6)	C6B = C9B = CM4B	105,8(6)
C10B = C9B = CM3B	110,1(7)	C10B = C9B = CM4B	110,2(7)
CM3B = C9B = CM4B	108,3(7)		

DIMEDRAL ANGLE	(°)	DIMEDRAL ANGLE	(°)
C1A = C2A = C3A = C4A	-177,6(7)	C2A = C3A = C4A = C5A	179,8(7)
C3A = C4A = C5A = C6A	-58,1(11)	C4A = C5A = C6A = C7A	-89,3(11)
C5A = C6A = C7A = C8A	-177,4(7)	C6A = C7A = C8A = C9A	-178,1(7)
C7A = C8A = C9A = C10A	54,6(10)	C8A = C9A = C10A = C11A	55,1(10)
C9A = C10A = C11A = C12A	-175,6(7)	C10A = C11A = C12A = C13A	-179,9(7)
C11A = C12A = C13A = C14A	-58,8(10)	C12A = C13A = C14A = C15A	-68,3(10)
C13A = C14A = C15A = C16A	179,0(6)	C14A = C15A = C16A = C1A	-177,8(7)
C15A = C16A = C1A = C2A	54,5(10)	C16A = C1A = C2A = C3A	58,6(10)
C1B = C2B = C3B = C4B	-177,6(7)	C2B = C3B = C4B = C5B	-178,3(7)
C3B = C4B = C5B = C6B	-68,5(10)	C4B = C5B = C6B = C7B	-67,8(10)
C5B = C6B = C7B = C8B	-179,8(6)	C6B = C7B = C8B = C9B	-176,8(6)
C7B = C8B = C9B = C10B	54,1(9)	C8B = C9B = C10B = C11B	55,8(10)
C9B = C10B = C11B = C12B	-176,9(7)	C10B = C11B = C12B = C13B	-178,8(6)
C11B = C12B = C13B = C14B	-68,7(9)	C12B = C13B = C14B = C15B	-68,4(9)
C13B = C14B = C15B = C16B	-176,8(7)	C14B = C15B = C16B = C1B	-175,7(6)
C15B = C16B = C1B = C2B	53,1(10)	C16B = C1B = C2B = C3B	56,5(10)

lattice conformation to be of lowest enthalpy. Infrared spectroscopy of the low temperature crystal indicated the same conformation.³ As part of a study of conformational problems in *gem*-dimethylsubstituted cyclohexadecanes,³ the 1,1,9,9-tetramethylcyclohexadecane has been synthesized. Its crystal structure is now reported.

The crystals of C₃₀H₄₀ belong to the monoclinic system with space group *P*₂₁/*c* and cell dimensions *a* = 8.001(3) Å, *b* = 20.741(7) Å, *c* = 23.388(9) Å, β = 95.68(3)°. There are two independent molecules in the asymmetric unit. 1873

observed reflections were measured on an automatic four circle diffractometer at room temperature (MoK α -radiation).

The structure was solved by direct methods⁴ and refined by full-matrix least squares technique.⁵ * Anisotropic temperature factors were introduced for carbon atoms. Hydrogen positions were calculated, but not refined. Weights in least squares were obtained from the standard deviations in intensities, $\sigma(I)$, taken as

* All programs used (except those for phase determination) are included in this reference.

$$\sigma(I) = [C_T + (0.02C_N)^2]^{\frac{1}{2}}$$

where C_T is the total number of counts, and C_N the net count. The R -value arrived at was 9.5 % (weighted value $R_w = 6.8$ %) for 1873 observed reflections.

Final fractional coordinates and thermal parameters with estimated standard deviations for carbon atoms are given in Table 1. The expression for anisotropic vibration is:

$$\exp[-(B11h^2 + B22k^2 + B33l^2 + B12hk + B13hl + B23kl)].$$

The principal axes of the thermal vibration ellipsoids were calculated from the temperature parameters of Table 1. Maximum r.m.s. amplitudes range from 0.26 to 0.43 Å. Due to the size of the molecule, no rigid-body analysis of translational, librational and screw motion has been carried out.

Interatomic distances, bond angles and dihedral angles are listed in Table 2. The standard deviations, in parentheses, are estimated from the correlation matrix of the last least squares refinement cycle. A list of observed structure factors is available by request to the author.

The two independent molecules, A and B, have the same square, diamond lattice conformation shown in Fig. 1. Bond distances and angles are normal.

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