

The Crystal and Molecular Structure of Cannabinol

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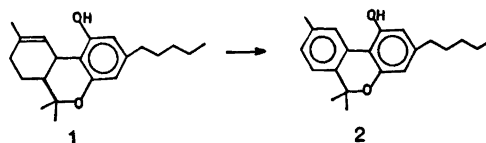
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The structure of cannabinol, C₂₁H₃₆O₂, has been determined by X-ray methods using 1401 reflections collected by counter methods. The compound crystallizes in the space group *P*2₁/*c* with two molecules in the asymmetric unit. Cell dimensions are: *a* = 20.658(8) Å; *b* = 18.166(4) Å; *c* = 13.517(5) Å; β = 133.56° (2). The structure model was refined to a conventional *R* of 0.135 and a weighted *R* of 0.091. The main difference between the two conformers in the asymmetric unit is found in the pentyl side chain. Both phenyl rings in each molecule are planar and the mean angle between the two planes is 20.2°. The mean bond distance between the phenyl rings is 1.46(2) Å. The phenol group is bent out of the benzene plane in both molecules. There is one *inter*-molecular hydrogen bond [2.73(2) Å] from a phenol group to a pyrane oxygen, and one between the phenol groups in the two molecules [2.92(2) Å]. The cannabinol molecule is considerably more planar than the Δ^9 -tetrahydrocannabinol molecule. While the methyl substituent on the cyclohexene ring in Δ^9 -tetrahydrocannabinol is 2.6 Å above a plane through the phenol ring, the corresponding distance in cannabinol is only 1.2 Å.

Cannabinol (CBN, 2) was the first cannabinoid to be structurally defined and synthesized.¹ CBN is rarely found in freshly prepared marijuana,² since Δ^9 -tetrahydrocannabinol (Δ^9 -THC, 1) is the precursor of CBN. In the presence of oxygen Δ^9 -THC is slowly converted into CBN. The aromatisation of the cyclohexene ring leads to a loss of the psychotomimetic activity,³ however, in experiments with rabbits a synergistic effect occurs⁴ when CBN is given together with Δ^9 -THC.

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Current interest in structural activity relationship of cannabinoids and how interaction between them alters biological activity, has created a need for detailed structural information on the conformation and hydrogen-bonding pattern. We found that a comparison of the three-dimensional structure of the "active" Δ^9 -THC molecule and the "inactive" CBN would be of interest.



EXPERIMENTAL AND STRUCTURE DETERMINATION

A square plate-formed crystal of dimensions 0.05 × 0.16 × 0.16 mm was used for the crystallographic work. A computer-controlled Syntex PI four-circle diffractometer with graphite-monochromatized MoK α radiation was utilized in the preliminary experiments and the collection of intensity data. The axial solutions (computer program written by R. Sparks which is part of the diffractometer program library) from the angular coordinates of eleven reflections indicated a monoclinic system. The systematic absences found in a fast low-angle data collection implied the space group *P*2₁/*c* with eight molecules in the unit cell, *i.e.* two molecules per asymmetric unit. Cell dimensions and their standard deviations were determined by a least-squares treatment of the angular coordinates of eleven reflections with 2θ -values between 4.5 and 11°.

Three-dimensional intensity data were recorded using the ω - 2θ scanning mode with scan speed variable from 3° to 12° min⁻¹ de-

Table 1. Fractional atomic coordinates and thermal parameters for nonhydrogen atoms. The anisotropic temperature factor is given by $\exp(-2\pi^2 [U_{11}(a^*h)^2 + U_{22}(b^*k)^2 + U_{33}(c^*l)^2 + 2U_{12}(a^*b^*hk) + 2U_{13}(a^*c^*hl) + 2U_{23}(b^*c^*kl)])$. Estimated standard deviations in parentheses.

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> ₂₃	<i>B</i>
C16A	.2838(14)	.0863(12)	-.0584(22)	.066(17)	.079(19)	.115(20)	.022(15)	.062(17)	.007(16)	4.7(3)
C17A	.6032(14)	.0856(11)	-.0522(21)	.101(19)	.067(17)	.098(18)	.009(15)	.086(17)	.009(16)	3.7(5)
C18A	.4836(15)	.1459(11)	-.2804(17)	.125(21)	.082(18)	.024(13)	-.006(16)	.051(15)	.001(12)	4.3(6)
C19A	.8016(14)	.3449(12)	.3463(18)	.078(19)	.085(21)	.036(14)	-.049(16)	.034(15)	-.043(14)	4.7(5)
C20A	.7916(14)	.4150(12)	.3879(20)	.089(20)	.070(19)	.061(16)	-.016(16)	.057(16)	-.030(14)	4.7(6)
C21A	.8681(14)	.4693(13)	.4555(21)	.051(17)	.050(17)	.063(17)	-.026(15)	.017(14)	.002(14)	2.7(4)
C22A	.8636(16)	.5405(14)	.5029(23)	.068(20)	.068(20)	.095(22)	-.007(17)	.040(19)	.031(18)	2.9(4)
C23A	.9342(16)	.5936(13)	.5523(23)	.091(20)	.077(20)	.109(22)	-.039(17)	.061(19)	.046(15)	3.1(4)
C16B	.4565(14)	.0847(12)	.3702(21)	.081(18)	.103(20)	.103(19)	-.012(15)	.073(17)	.003(14)	4.7(3)
C17B	.2041(14)	.2120(11)	.4583(21)	.115(20)	.066(19)	.083(17)	.031(16)	.069(17)	.046(15)	3.7(5)
C18B	.1065(15)	.2431(12)	.2105(22)	.077(20)	.099(21)	.081(19)	.019(16)	.048(17)	.016(16)	4.3(6)
C19B	.0880(18)	.0919(15)	.4655(29)	.167(29)	.109(25)	.174(31)	-.012(21)	.155(29)	-.010(22)	4.7(5)
C20B	.1148(21)	-.0870(17)	.5988(37)	.169(32)	.167(34)	.225(39)	-.043(25)	.171(33)	.006(30)	4.7(6)
C21B	.0744(31)	-.1423(30)	.6229(40)	.233(63)	.422(74)	.155(41)	-.036(47)	.168(44)	.000(44)	2.7(4)
C22B	.1177(25)	-.1653(23)	.7444(39)	.147(37)	.219(46)	.167(37)	-.065(31)	.107(36)	-.022(37)	2.9(4)
C23B	.0783(23)	-.2244(19)	.7681(32)	.200(39)	.190(38)	.144(31)	-.019(29)	.131(32)	-.005(27)	3.1(4)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i>	Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i>
O1A	.5649(7)	.2163(6)	-.0789(12)	3.5(3)	O1B	.1090(9)	.1233(7)	.2790(12)	4.7(3)
C2A	.6052(12)	.2303(10)	.0550(18)	3.6(5)	C2B	.1508(13)	.0595(10)	.3564(17)	3.7(5)
C3A	.6773(13)	.2770(11)	.1312(19)	3.9(5)	C3B	.1022(12)	.0171(11)	.3716(19)	4.3(6)
C4A	.7211(13)	.2951(10)	.2667(20)	4.2(6)	C4B	.1366(15)	-.0475(12)	.4404(21)	4.7(5)
C5A	.6878(12)	.2614(10)	.3170(18)	4.1(5)	C5B	.2226(14)	-.0679(10)	.5049(19)	4.7(6)
C6A	.6169(12)	.2156(10)	.2406(18)	3.3(4)	C6B	.2700(12)	-.0249(10)	.4888(16)	2.7(4)
C7A	.5681(10)	.1972(8)	.1020(16)	1.7(4)	C7B	.2346(12)	.0420(10)	.4092(17)	2.9(4)
C8A	.4858(12)	.1542(10)	.0087(18)	3.0(4)	C8B	.2759(11)	.0884(10)	.3775(17)	2.9(4)
C9A	.4251(13)	.1395(10)	.0214(18)	3.6(6)	C9B	.3450(12)	.0645(9)	.3896(16)	3.1(4)
C10A	.3461(13)	.1021(11)	-.0803(20)	4.0(5)	C10B	.3811(13)	.1121(11)	.3551(19)	4.4(5)
C11A	.3273(14)	.0748(11)	-.1929(21)	5.8(6)	C11B	.3464(12)	.1810(12)	.3130(21)	6.1(6)
C12A	.3885(14)	.0912(11)	-.2034(20)	4.9(5)	C12B	.2780(14)	.2055(11)	.2979(20)	5.2(5)
C13A	.4646(13)	.1293(10)	-.1110(19)	3.9(5)	C13B	.2416(12)	.1605(10)	.3312(18)	3.2(4)
C14A	.5260(13)	.1404(11)	-.1321(18)	3.6(5)	C14B	.1673(15)	.1835(12)	.3189(23)	5.4(5)
O15A	.5859(8)	.1819(6)	.2961(11)	4.0(3)	O15B	.3551(8)	-.0436(7)	.5530(12)	4.7(3)

pending on the intensity of the reflection, and a scan range from $2\theta(\alpha_1) - 0.8^\circ$ to $2\theta(\alpha_2) + 0.8^\circ$. Background counting time was equal to $0.35 \times$ (scan time) on each side of the scan range. The temperature was kept constant within 2° at 24°C . The variations in the intensities of three standard reflections which were remeasured after every hundred reflections, were random. Accordingly no corrections were applied to the intensities for these variations.

The estimated standard deviations were taken as the square root of the total counts with a 2% addition of the net intensity for experimental uncertainties. Of the 4835 symmetry-independent reflections measured ($2\theta_{\text{max}} = 45^\circ$), 1401 had intensities larger than twice their standard deviations. These were regarded as observed reflections, and the remaining were excluded from further calculations. The intensities were corrected for Lorentz and polarization effects.⁵

The phase problem was solved by the MULTAN⁶ program package. The structure model was refined to a conventional R of 0.17. The computer programs used as well as programs subsequently employed, are part of a local (Oslo) assembly of computer programs which is described in Ref. 7. Atomic scattering factors used were those of Doyle and Turner⁸ for carbon and oxygen, and of Stewart *et al.*⁹ for

hydrogen. Because of the relatively small number of observed reflections anisotropic thermal parameters were only introduced for the atoms in the sidechains (eight atoms in each molecule, see Table 1) where the thermal motion is largest. This gives an overdetermination ratio of 5.3. Least-squares refinement lowered R to 0.16.

At this point the hydrogen atoms were placed in calculated positions and included in the structure factor calculations with estimated isotropic thermal parameters. The distances indicated two *inter*-molecular hydrogen bonds and the two hydrogens bonded to oxygens (A15 and B15) were placed accordingly. Full-matrix least-squares refinement of all non-hydrogen atomic parameters converged to a weighted R_w of 0.091 and an R of 0.135. The weights used in the refinement were $1/\sigma^2(F_{\text{obs}})$.

Atomic parameters for nonhydrogen atoms are given in Table 1, and those used for hydrogen atoms are listed in Table 2. A list of observed and calculated structure factors is available upon request from: Department of Chemistry, University of Oslo, Oslo 3, Norway.

CRYSTAL DATA

Cannabinol, $\text{C}_{21}\text{H}_{28}\text{O}_2$, $M = 310.43$ amu, space group $P2_1/c$, $a = 20.658(8)$ Å, $b = 18.166(4)$ Å,

Table 2. Calculated fractional coordinates and estimated thermal parameters for hydrogen atoms.

Atom	x	y	z	B	Atom	x	y	z	B
A3	.699	.299	.090	4.5	B3	.042	.034	.332	5.0
A5	.718	.272	.414	4.5	B5	.250	-.114	.563	5.0
A9	.440	.156	.106	4.5	B9	.370	.014	.424	5.0
A11	.273	.044	-.262	5.5	B11	.371	.215	.288	5.5
A12	.374	.073	-.287	5.0	B12	.256	.257	.267	5.5
A15	.579	.217	.340	4.5	B15	.374	-.089	.602	5.2
A16,1	.230	.060	-.141	6.0	B16,1	.474	.125	.341	6.0
A16,2	.265	.134	-.048	6.0	B16,2	.436	.041	.311	6.0
A16,3	.316	.056	.024	6.0	B16,3	.508	.072	.468	6.0
A17,1	.579	.035	-.086	6.0	B17,1	.245	.254	.489	6.0
A17,2	.637	.089	.047	6.0	B17,2	.237	.171	.526	6.0
A17,3	.644	.698	-.066	6.0	B17,3	.153	.228	.446	6.0
A18,1	.532	.153	-.280	6.0	B18,1	.059	.256	.210	6.0
A18,2	.442	.189	-.326	6.0	B18,2	.079	.224	.120	6.0
A18,3	.450	.100	-.330	6.0	B18,3	.143	.238	.235	6.0
A19,1	.853	.318	.430	6.0	B19,1	.024	-.076	.396	6.5
A19,2	.813	.356	.287	6.0	B19,2	.094	-.144	.452	6.5
A20,1	.786	.404	.454	6.5	B20,1	.101	-.036	.605	6.5
A20,2	.736	.439	.305	6.5	B20,2	.181	-.096	.667	6.5
A21,1	.872	.481	.388	6.5	B21,1	.052	-.182	.560	7.0
A21,2	.924	.444	.537	6.5	B21,2	.021	-.113	.597	7.0
A22,1	.867	.531	.579	7.0	B22,1	.136	-.124	.806	7.5
A22,2	.805	.564	.425	7.0	B22,2	.174	-.190	.774	7.5
A23,1	.925	.640	.583	7.5	B23,1	.122	-.238	.866	7.5
A23,2	.933	.605	.480	7.5	B23,2	.061	-.267	.710	7.5
A23,3	.994	.572	.633	7.5	B23,3	.024	-.202	.743	7.5

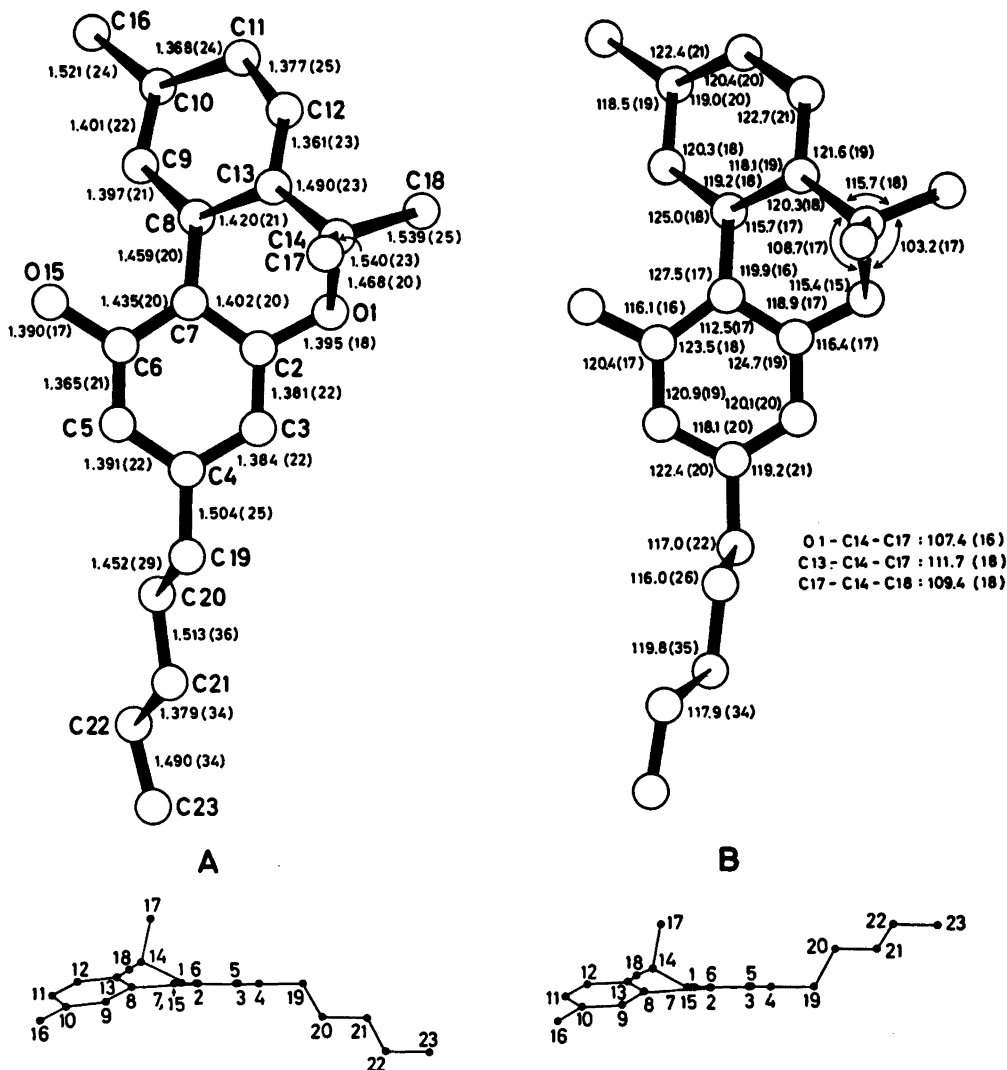


Fig. 1. Mean bond lengths (Å) and bond angles (°) and a view along the plane of the benzene ring, showing the conformation of the two molecules A and B, in the asymmetric unit.

$c = 13.517(5)$ Å, $\beta = 135.56^\circ (2)$, $V = 3675.8(18)$ Å³, $Z = 8$, $D_{\text{calc}} = 1.222$ g cm⁻³, $F(000) = 1344$.

DISCUSSION

The molecular structure and atomic numbering is shown in Fig. 1, together with a view along the plane of the phenol ring, showing the conformation. The asymmetric unit in the crystal consists of two CBN molecules, denoted A and B, respectively. Bond lengths and

angles are given in Table 3 and mean molecular parameters are shown in Fig. 1. The standard deviations are relatively high, so a detailed discussion of bond distances and angles is not meaningful. The mean values of the molecular parameters are all normal within their estimated standard deviations, except for the shortening of the C19–C20 and C21–C22 bonds, probably caused by the large thermal vibrations in the side chain.

Table 3. Molecular parameters (Å and °) with estimated standard deviations. See Fig. 1 for mean bond lengths and bond angles.

Bond length	A	B	Angle	A	B
O1-C2	1.40(2)	1.39(2)	C2-O1-C14	115(1)	115(2)
O1-C14	1.51(2)	1.43(2)	O1-C2-C3	117(2)	116(2)
C2-C3	1.37(2)	1.39(2)	O1-C2-C7	119(2)	119(2)
C2-C7	1.42(2)	1.38(2)	C3-C2-C7	124(2)	125(2)
C3-C4	1.41(2)	1.36(2)	C2-C3-C4	121(2)	119(2)
C4-C5	1.40(2)	1.38(2)	C3-C4-C5	116(2)	120(2)
C5-C6	1.35(2)	1.38(2)	C3-C4-C19	118(2)	120(2)
C6-C7	1.43(2)	1.44(2)	C5-C4-C19	126(2)	119(2)
C7-C8	1.46(2)	1.46(2)	C4-C5-C6	122(2)	119(2)
C8-C9	1.40(2)	1.39(2)	C5-C6-C7	124(2)	123(2)
C8-C13	1.43(2)	1.41(2)	C5-C6-O15	121(2)	120(2)
C9-C10	1.39(2)	1.42(2)	C7-C6-O15	115(2)	117(2)
C10-C11	1.38(2)	1.36(2)	C2-C7-C6	112(2)	113(2)
C11-C12	1.40(2)	1.35(3)	C2-C7-C8	120(2)	120(2)
C12-C13	1.34(2)	1.38(2)	C6-C7-C8	128(2)	127(2)
C13-C14	1.49(2)	1.49(2)	C7-C8-C9	127(2)	123(2)
C6-O15	1.42(2)	1.36(2)	C7-C8-C13	115(2)	117(2)
C10-C16	1.53(2)	1.51(2)	C9-C8-C13	118(2)	120(2)
C14-C17	1.52(2)	1.56(2)	C8-C9-C10	121(2)	120(2)
C14-C18	1.54(2)	1.54(2)	C9-C10-C11	121(2)	117(2)
C4-C19	1.51(2)	1.50(3)	C9-C10-C16	118(2)	119(2)
C19-C20	1.46(2)	1.44(3)	C11-C10-C16	121(2)	124(2)
C20-C21	1.52(3)	1.50(5)	C10-C11-C12	117(2)	124(2)
C21-C22	1.47(3)	1.28(4)	C11-C12-C13	125(2)	120(2)
C22-C23	1.47(3)	1.51(4)	C8-C13-C12	118(2)	118(2)
			C8-C13-C14	122(2)	118(2)
			C12-C13-C14	120(2)	123(2)
			O1-C14-C13	106(2)	111(2)
			O1-C14-C17	108(1)	107(2)
			O1-C14-C18	102(1)	105(2)
			C13-C14-C17	113(2)	111(2)
			C13-C14-C18	117(2)	114(2)
			C17-C14-C18	110(2)	109(2)
			C4-C19-C20	115(2)	119(3)
			C19-C20-C21	116(2)	117(3)
			C20-C21-C22	119(2)	121(5)
			C21-C22-C23	116(2)	120(4)

Dihedral angles (°) The angles are positive in a right-hand screw.

	A	B		A	B
C14-O1-C2-C7	-34.5(22)	-33.5(25)	O1-C2-C7-C8	-4.5(25)	-3.0(26)
C2-C7-C8-C13	19.3(24)	19.5(25)	C7-C8-C13-C14	5.4(26)	0.2(25)
C8-C13-C14-O1	-39.5(23)	-34.0(26)	C8-C13-C14-C17	78.3(24)	84.9(23)
C8-C13-C14-C18	-152.0(18)	-152.4(18)	C13-C14-O1-C2	53.6(19)	50.8(23)
C17-C14-O1-C2	-67.5(18)	-70.2(21)	C18-C14-O1-C2	176.7(15)	174.6(15)
C3-C4-C19-C20	117.5(20)	-98.9(32)	C4-C19-C20-C21	-174.0(18)	-169.6(32)
C19-C20-C21-C22	-178.7(19)	149.0(47)	C20-C21-C22-C23	-173.1(19)	-176.1(35)

Compared to the structure of the Δ^9 -THC skeleton, studied in Δ^9 -THC Acid B¹⁰ and 8β -hydroxy- Δ^9 -THC,¹¹ the aromatisation of the cyclohexane ring has led to considerable changes in the over-all geometry, as well as

a loss of biological activity. CBN is a much more planar molecule than Δ^9 -THC. The methyl group C16 is approximately 1.2 Å above the plane through the phenol ring in CBN, while the corresponding distance in Δ^9 -

Table 4. Deviations from least-squares planes ($\text{\AA} \times 10^3$). The deviations for those atoms used to define the plane are given in italicized figures.^a

Atom	Plane A1	Plane B1	Atom	Plane A2	Plane B2
O1	7	2	O1	846	757
C2	10	9	C2	531	423
C3	5	19	C7	52	12
C4	-12	-34	C8	-9	-2
C5	5	18	C9	-10	0
C6	10	9	C10	24	7
C7	-17	-23	C11	-19	-12
C8	-147	-130	C12	-1	10
C13	190	225	C13	14	-3
C14	750	680	C14	-24	5
O15	44	80	C16	-10	0
C19	26	60			

^a Angle between: A1 and A2, 21.0°; B1 and B2, 19.4°.

THC is about 2.6 \AA . It is obvious that this difference will be of great importance for the matching of the receptor and the drug molecule.

The two crystallographic independent molecules differ mainly in the conformation of the side chain. The side chain in molecule A is rotated 180° around the C4–C19 bond compared to molecule B. Except for the C20–C21 bond which is more eclipsed in B than in A [dihedral angle C19–C20–C21–C22 is 149.0° (4.7) in B and -178.7° (1.9) in A], the chain in both molecules is fully extended and all-*anti*.

Both aromatic rings are nearly planar, but while all atoms directly attached to ring 2 (Plane A2 and B2, Table 4) are less than 0.05 \AA out of the plane, both the phenol oxygen O15 and C8 are bent significantly out of the plane through ring 1 (Plane A1 and B1). The mean deviation from the plane is +0.062 \AA for O15 and -0.138 \AA for C8. The short distance between the hydrogen atom on C9 and the phenol group (2.25 \AA) indicates considerable steric interaction which probably is the cause of this effect. The same result is also observed in the Δ^9 -THC structures studied.^{10,11} In all the four independent determinations of this parameter we observe a C7–C6–O15 bond angle less than 120° (mean value 117.1°), while Archer *et al.*¹² have calculated by Westheimer calculations the bond angle to open slightly (122–124°).

The bond angles in the phenol ring vary considerably in both molecules (112–125°) and in the same way as in the other cannabinoids studied.^{10,11,13} This has been interpreted by strains in the aromatic system.¹⁰ In the other aromatic ring in CBN the angles are not significantly different from benzene values.

The mean bond distance between the two phenyl rings is 1.46(2) \AA , which is not significantly different from the corresponding bond in biphenyl in the crystalline phase¹⁴ (1.494(3) \AA).

The planes through the phenyl rings make an angle of 19.4° in molecule B and 21.1° in molecule A. The dihedral angle C2–C7–C8–C13 is 19.3° in A and 19.5° in B. In the two Δ^9 -THC structures,^{10,11} this angle was observed to 15.1° and 16.6°, while Westheimer calculations¹² gave 13° for CBN.

The pyrane oxygen in molecule A (O1A) acts as acceptor for a hydrogen bond [2.73(2) \AA] from the phenol group (O15A) in another A molecule in position: $x, \frac{1}{2} - y, \frac{1}{2} + z$. There are no short contacts from the pyrane oxygen in molecule B to a hydrogen donor. The C14–O1 bond distance is probably significantly longer in molecule A, [1.51(2) \AA] where O1 is hydrogen-bond acceptor, than in B [1.43(2) \AA]. There is another hydrogen bond [2.92(2) \AA] from the phenol group in molecule B to the phenol group in molecule A in position: $1 - x, -y, 1 - z$. There are no other contacts shorter than 3.5 \AA between the heavy atoms in A and B.

REFERENCES

1. Adams, R., Baker, B. R. and Wearn, R. B. *J. Am. Chem. Soc.* 62 (1940) 2204.
2. Turner, C. E., Hadley, K. W. and Davis, K. H. *Acta Pharm. Jugosl.* 23 (1973) 89.
3. Mechoulam, R., Shani, A., Edery, H. and Grunfeld, Y. *Science* 169 (1970) 611.
4. Takahashi, R. N. and Karniol, I. G. *Psychopharmacologia* 41 (1975) 277.
5. Ottersen, T. *LP-76 computer program, cryst. prog. lib.*, Department of Chemistry, University of Hawaii, Honolulu 1976.
6. Germain, G., Main, P. and Woolfson, M. M. *Acta Crystallogr. A* 27 (1971) 368.
7. Groth, P. *Acta Chem. Scand.* 27 (1973) 1837.
8. Doyle, P. A. and Turner, P. S. *Acta Crystallogr. A* 24 (1968) 390.
9. Stewart, R. F., Davidson, E. R. and Simpson, W. T. *J. Chem. Phys.* 42 (1965) 3175.
10. Rosenqvist, E. and Ottersen, T. *Acta Chem. Scand. B* 29 (1975) 379.
11. Ottersen, T. and Rosenqvist, E. *Acta Chem. Scand. B* 31 (1977) 749.
12. Archer, R. H., Boyd, D. B., Demarco, P. V., Tyminski, I. J. and Allinger, N. L. *J. Am. Chem. Soc.* 92 (1970) 5200.
13. Bandaranayake, W. M., Begley, M. J., Brown, B. O., Clarke, D. G., Crombie, L. and Whitig, D. A. *J. Chem. Soc. Perkin Trans. 1* (1974) 998.
14. Robertson, G. B. *Nature* 191 (1961) 593.

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