X-RAY CRYSTALLOGRAPHIC STUDIES OF RACEMIC AND OPTICALLY ACTIVE 4,4'-DIMETHYL-1,1'-BINAPHTHYL

by

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ABSTRACT

In contrast to 1,1'-binaphthyl, racemic 4,4'-dimethyl-1,1'-binaphthyl does not undergo spontaneous resolution upon heating from room temperature to just below the melting point. Optically active dimethyl binaphthyl may be obtained by seeding the racemic melt with optically active naphthidine. The crystal structures of both the racemic and optically active dimethyl binaphthyls were solved in the hope of understanding the above observations.

The racemate crystallizes in the monoclinic space group C2/c with cell parameters a=13.225, b=10.768, c=11.572 Å and β=114.04°. There are four molecules per unit-cell; two have the R and two have the S configuration. The structure was solved using direct methods and refined to R=0.074. There is a 3° bend in the plane of the naphthalene residues, which are cis-oriented with an angle of 68° between them.

The optically active form belongs to one of the tetragonal space groups $P4_12_12$ or $P4_32_12$ with cell parameters a = b = 8.3031 and c = 23.706 Å. Direct methods were used to solve the structure and the final R was 0.060. There are four molecules per unit-cell of identical configuration, but it could not be determined whether this was R or S. The naphthalene residues show a 2.7° bend and are also cis-oriented, but with an angle of 80° between them.

Bond lengths and angles are consistent with values

previously reported for 1,1'-binaphthyl and naphthalene. The racemate packs somewhat more efficiently and perhaps for this reason it is slightly more stable than the optically active form. It is difficult however to explain the difference in behaviour between the methylated and unmethylated binaphthyls on the basis of these results alone. Further studies would include the crystal structures of optically active 1,1'-binaphthyl and various naphthidines.

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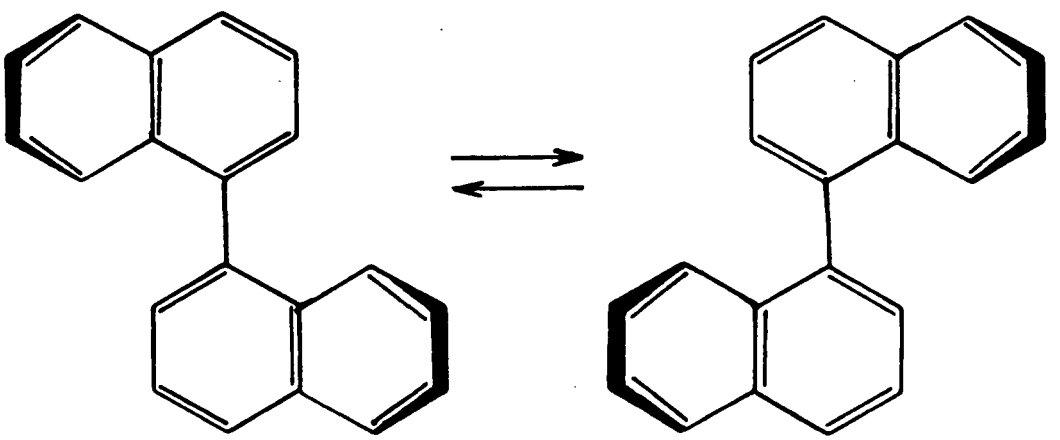
PART ONE

INTRODUCTION

This thesis describes the X-ray crystal structure analyses of racemic and optically active 4,4'-dimethyl-1,1'-binaphthyl. The background theory involved in x-ray techniques is covered in several standard texts,[1-5] and all nomenclature, conventions and crystallographic symbols used are consistent with those described in International Tables for X-ray Crystallography.[6]

There has been considerable interest in binaphthyls in relation to the spontaneous resolution of racemic mixtures into optically pure samples.[7-10] 1,1'-Binaphthyl is converted into its enantiomer by a simple rotation about the 1,1' bond (see Fig. 1). Two crystalline forms of 1,1'-binaphthyl are known;[11]



R-1,1'-Binaphthyl S-1,1'-Binaphthyl

Figure 1. Enantiomeric Conversion of 1,1'-Binaphthyl

the low melting form (m.p.=145°C) has been shown by X-ray structure analysis[12] to be the racemate with two R and two S molecules per unit-cell, and the high melting form (m.p. 158°C) to be the optically active form (i.e. only molecules of one enantiomer per unit-cell).

It has been found that in a certain temperature range, the optically active form is the more stable thermodynamically and so upon heating a racemic sample into this temperature range spontaneous resolution could occur.[9] The phase diagram corresponding to this is shown in Fig. 2 where the dotted line shows the behaviour of the racemate upon heating. It was found that one could control the choice of enantiomer to which the racemate would resolve by picking an original composition equivalent to a point (e.g. P) just to the right (or left) of the vertical dotted line and heating it. At all times the mixture would consist of the racemate plus S (or R) impurity, and crystals of the R (or S) form would not nucleate.

These observations of the behaviour of 1,1'-binaphthyl were not noticed in 4,4'-dimethyl-1,1'binaphthyl:[13] in the dimethyl compound the racemic form seems to be at all times the more stable, and no optical activity develops upon heating (experimentally the R-form of the dimethyl compound has been obtained by seeding the RS-melt with R-naphthidine.).

It is hoped that this might be better understood by comparing crystal structures of the racemic and optically active forms.

Preparation of Crystals (R.E. Pincock)

The preparation of the racemate[14] is illustrated in Fig. 3. 4-Bromo-1-methylnaphthalene (3) was prepared from 1-methylnaphthalene (1) by the sulfonation - bromodesulfonation procedure (developed by Fieser[15]) via (2) in 35% yield. Racemic 4,4'-dimethyl-1,1'-binaphthyl (4) was formed (26 - 41% yield)

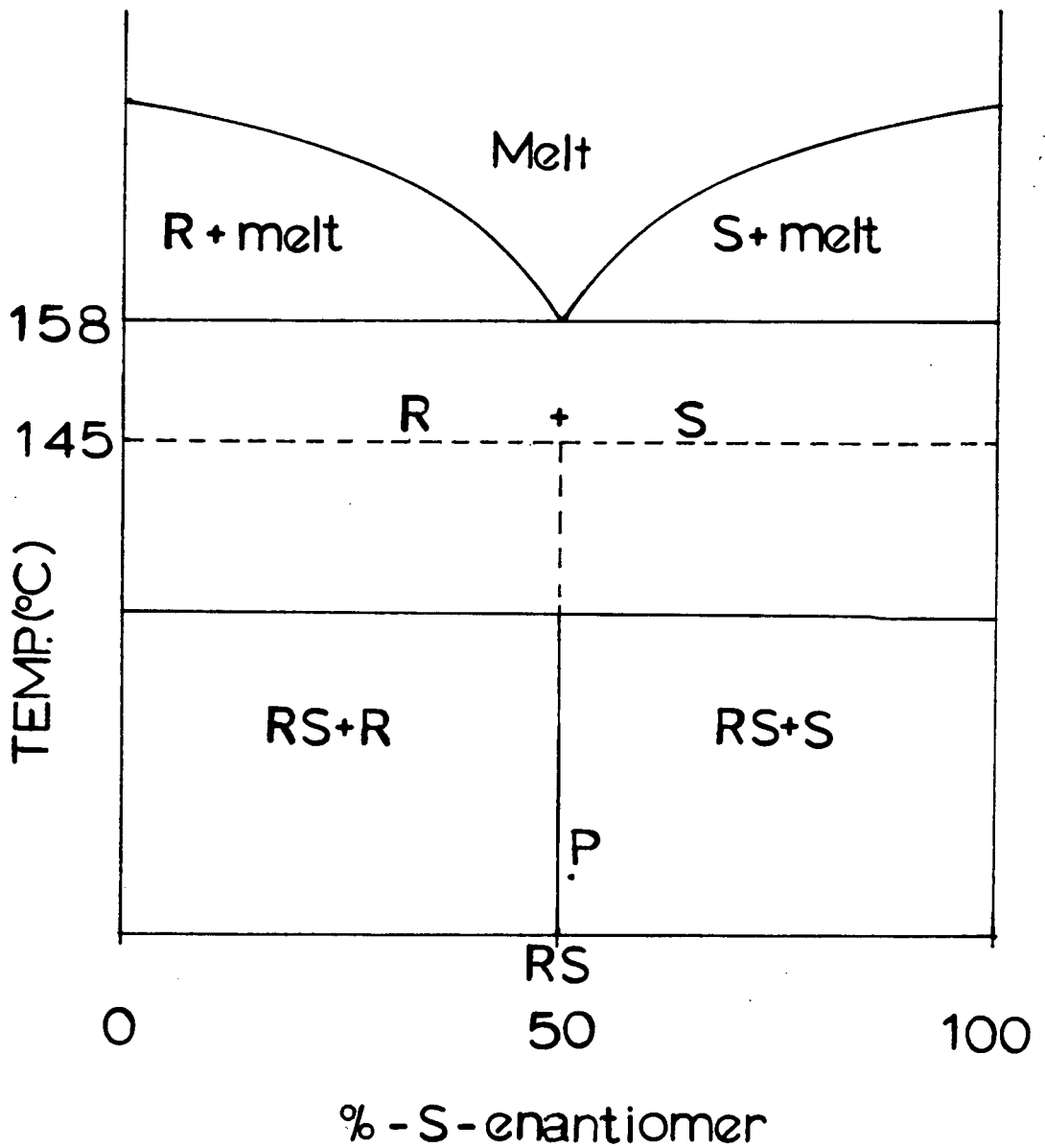


Figure 2. Phase Diagram for 1,1'-Binaphthyl

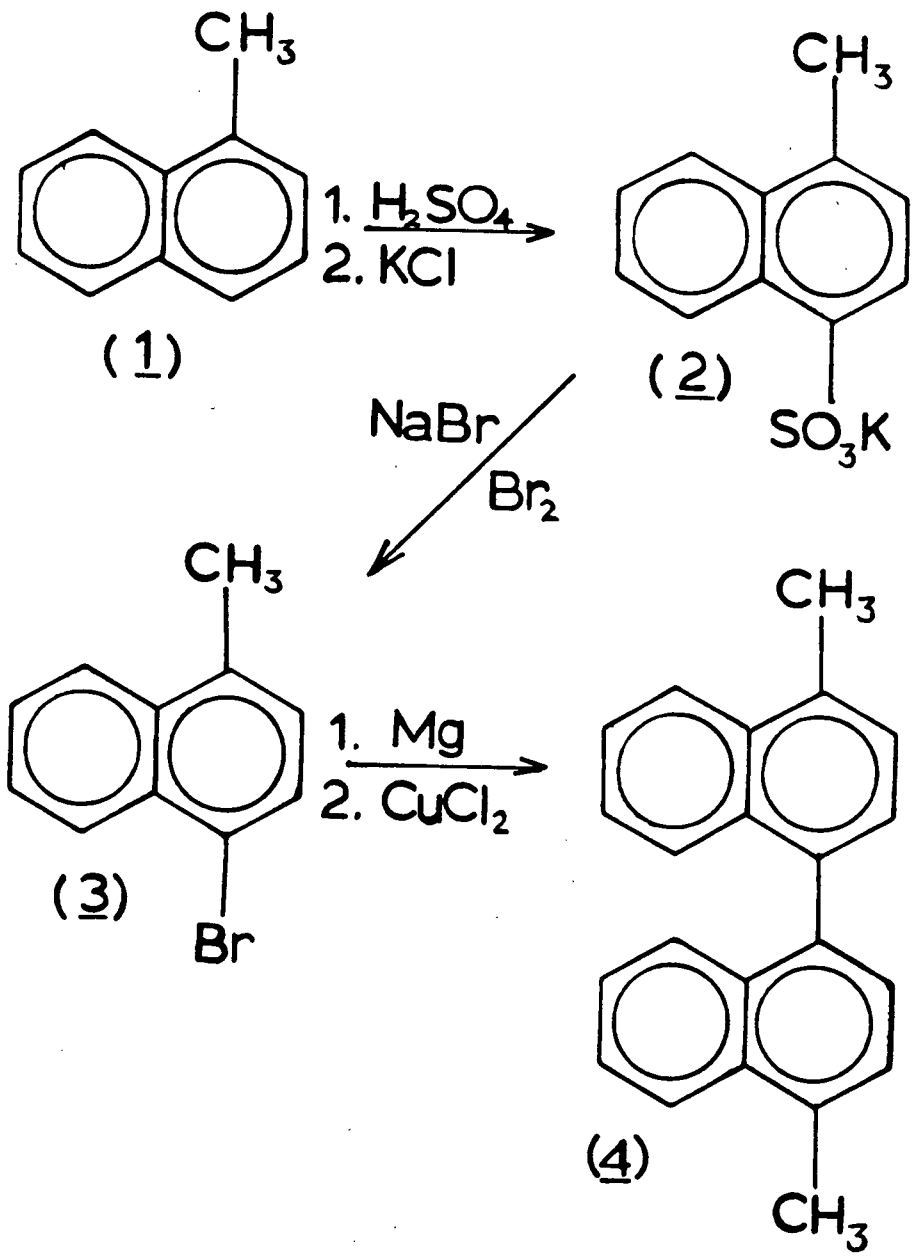


Figure 3. Preparation of Racemic 4,4'-Dimethyl-1,1'-binaphthyl

by the coupling of the Grignard reagent derived from (3), and was crystallized from acetone to give colorless rods.

Optically active 4,4'-dimethyl-1,1'-binaphthyl was prepared[14] by the induced resolution of the racemic compound using optically active naphthidine as a seed. Best results were obtained by weighing out 50mg samples of racemate containing 1% naphthidine into ampules which were sealed in air. These samples were held in a bath at 150°C for 4 minutes allowing the dimethyl binaphthyl to melt while the naphthidine (m.p. 206 - 207°C) remained solid and would seed the subsequent crystallization. Normally crystals formed overnight.

PART TWO:

CRYSTAL STRUCTURE OF

RACEMIC 4,4'-DIMETHYL-1,1'-BINAPHTHYL

Experimental.

Preliminary x-ray photography shows the well-formed colorless crystals to be monoclinic ; systematic absences (for general hkl, h+k=2n+1 ; for the h0l zone, l=2n+1) indicate that the space group is either C2/c or Cc. Unit-cell and intensity data were measured on a Datex-automated GE XRD 6 diffractometer with Cu K_α radiation utilizing the θ-2θ scan technique. Unit-cell parameters were refined by least-squares methods from manually obtained 2θ values of thirty reflections, and the final parameters are listed in Table I . The data were

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TABLE I

Crystal Data

$C_{22}H_{18}$	f.w. = 282.4 amu
Monoclinic	Z = 4
	Space Group = C2/c
a = 13.225(8) Å	
b = 10.768(4) Å	F(000) = 600
c = 11.572(7) Å	λ = 1.5418 Å
β = 114.04(2) ^{o}	Dc = 1.246(1) g/cc
V = 1505(1) Å^3	

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collected at a 2θ scan rate of 4^{o}min^{-1} over a range of (1.80+0.86tanθ)^o with 10 second background counts at each end of the scan. The 14 strongest reflections were remeasured with an attenuation of 28.5 . The 7 1 3 reflection was used as a standard and checked every 50 reflections. There were several large deviations in the scan counts of the standard reflection due to electronic difficulties with the diffractometer. The structure was solved with this set of data, but a new set was collected for refinement. In the second set the standard varied more smoothly and was used to scale the data. 611 of the 1497

unique reflections in the $0^0 \leq 2\theta \leq 120^0$ range had intensities less than $3.0\sigma(I)$, where $\sigma^2(I) = S + B + (0.06S)^2$, S is the scan count and B is the background count. Lorentz and polarization corrections were applied to give the structure amplitudes. No absorption correction was applied.

Structure Analysis.

A Wilson plot was used to determine overall temperature ($B = 3.59$ Å^2) and scale factors. E statistics (see Table II) indicate a centrosymmetric space group (i.e. C2/c) and 204 E's > 1.4 were obtained.

334 Σ_2-relationships were found from the largest 50 E's and used to select origin and symbol reflections. For the space group C2/c two origin determining phases are needed of suggested parities uug and ggu[6] (centered cell indices). The largest two E's (9 5 2 and 2 6 7) were chosen and three independent symbols selected providing 8 ($=2^3$) starting sets to be used in a symbolic addition and tangent refinement procedure.

An E-map was calculated from the best set of phased E's (that with the lowest R-Karle of 0.21) and all 11 carbons in the asymmetric unit could be located. 3 isotropic and 2 anisotropic least-squares refinement cycles were carried out before an electron-density difference map showed all but one (a methyl H) of the hydrogens. The methyl hydrogen positions were calculated geometrically, and three more least-squares refinement cycles (all reflections unit weight, 11 C's anisotropic, 9 H's isctropic) lowered the R value to 0.089 .

TABLE II

E Statistics

	OBSERVED	THEORETICAL	
		Centro.	Non-centro.
Mean $\|E\|$	0.6732	0.7980	0.8860
Mean $\|E\|^2$	1.0489	1.0000	1.0000
Mean $\|E^2-1\|$	1.2486	0.9680	0.7360
% Reflections with :			
E > 3.0	1.74	0.30	0.01
E > 2.0	5.81	5.00	1.80
E > 1.0	25.25	32.00	37.00

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Another four refinement cycles with polynomial weighting schemes[16] ($w=1/\{A+B|Fo|+C|Fo|^2+D|Fo|^3\}$, where A,B,C and D are recalculated after each cycle, final values being -0.01153, 0.08308, -0.01806 and 0.001396 respectively) reduced R to 0.078 and Rw to 0.074 . The anisotropic thermal parameters used in the refinement are the Bij in

$f=f_o \exp(-(B_{11}h^2+B_{22}k^2+B_{33}l^2+2B_{12}hk+2B_{13}hl+2B_{23}kl))$, where f_o is the tabulated structure factor and f is that corrected for thermal motion. The isotropic thermal parameters are the B's in $f=f_o\exp(-B\sin^2\theta/\lambda^2)$. Final parameters and temperature factors are shown in Tables III and IV , and a comparison of observed and calculated structure factors appears in Appendix I.

Results and Discussion.

The numbering scheme (see Fig. 4) is that generally used for naphthalene and hydrogens are given the same number as the carbon to which they are attached (H(111), H(112) and H(113) being the methyl hydrogens). The asymmetric unit is only one half of the dimethyl-binaphthyl molecule; the other half is generated by rotation about a two-fold axis.

Equations of and deviations from various planes through the asymmetric unit are shown in Table V . There is a slight bend in the unit, the rings lying at an angle of three degrees to each other. For a similar distortion in 1,1'-binaphthyl the close contacts between the two halves (e.g. C(8)...C(8')) have been suggested[12] as possible reasons. The dimethyl-binaphthyl shows close contacts at C(2)...C(2') (3.116(5)Å) and C(9)...C(9') (3.343(4)Å) but the C(8)...C(8') distance is

TABLE III

Fractional Atomic Parameters (Cx10⁴, Hx10³) With Their Standard Deviations

Atom	x/a	y/b	z/c
C(1)	4386(2)	2978(2)	2296(2)
C(2)	3898(2)	2039(2)	2686(2)
C(3)	2745(2)	1996(2)	2343(2)
C(4)	2056(2)	2886(2)	1586(2)
C(5)	1834(2)	4747(2)	229(2)
C(6)	2277(2)	5637(3)	-269(2)
C(7)	3428(2)	5685(3)	83(2)
C(8)	4112(2)	4838(2)	921(2)
C(9)	3681(2)	3892(2)	1492(2)
C(10)	2513(2)	3844(2)	1087(2)
C(11)	833(2)	2843(3)	1278(3)
H(2)	434(2)	136(2)	322(3)
H(3)	243(2)	133(3)	270(3)
H(5)	98(3)	470(3)	-43(3)
H(6)	176(2)	626(3)	-88(3)
H(7)	375(3)	632(3)	-26(3)
H(8)	494(3)	492(2)	114(3)
H(111)	37(3)	266(3)	34(3)
H(112)	52(2)	367(3)	144(3)
H(113)	68(3)	223(3)	178(4)

TABLE IV

Anisotropic Thermal Parameters Of The Carbon Atoms With Their Standard Deviations (x10⁴)

Atom	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
C(1)	43(2)	60(2)	57(2)	- 2(1)	11(1)	- 5(2)
C(2)	59(2)	64(2)	64(2)	- 5(2)	13(2)	8(2)
C(3)	63(2)	73(2)	69(2)	-23(2)	21(2)	6(2)
C(4)	44(2)	80(2)	59(2)	-16(1)	16(1)	- 9(2)
C(5)	42(2)	76(2)	65(2)	- 2(2)	9(1)	- 7(2)
C(6)	59(2)	70(2)	78(2)	5(2)	13(2)	13(2)
C(7)	63(2)	74(2)	83(2)	- 8(2)	25(2)	19(2)
C(8)	47(2)	73(2)	77(2)	- 7(2)	19(2)	6(2)
C(9)	42(2)	57(2)	55(2)	- 5(1)	13(1)	- 7(2)
C(10)	44(2)	60(2)	53(2)	- 9(1)	14(1)	-10(2)
C(11)	51(2)	118(3)	91(3)	-19(2)	24(2)	6(3)

Hydrogen Isotropic Thermal Parameters (A^2)

Atom	B
H(2)	3.1(5)
H(3)	4.4(6)
H(5)	5.0(7)
H(6)	3.9(6)
H(7)	5.5(8)
H(8)	3.8(6)
H(111)	6.1(8)
H(112)	5.1(7)
H(113)	7.0(9)

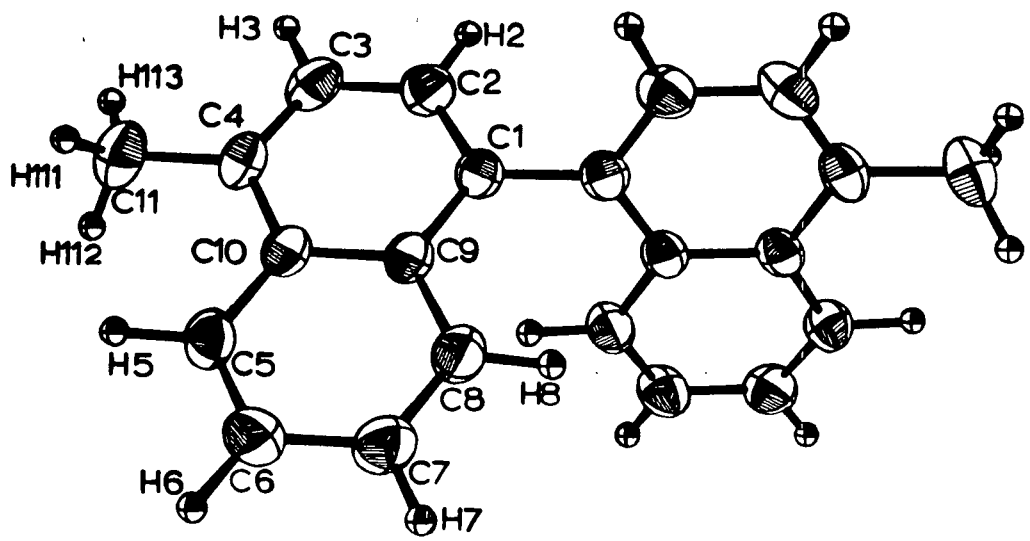

Figure 4. Molecular Drawing of Racemic 4,4'-Dimethyl-1,1'-binaphthyl.

TABLE V

Mean Planes

Equations of Planes: (lX+mY+nZ=p)

Plane	l	m	n	p
1	0.2380	-0.5615	-0.7925	-2.5779
2	0.2311	-0.6050	-0.7619	-2.7314
3	0.2250	-0.5866	-0.7780	-2.6741

Deviations from Planes:

Atom	Plane 1.	Plane 2.	Plane 3.
C(1)	-0.023(2) *	0.035(2)	-0.034(2) *
C(2)	0.021(2) *	0.141(2)	0.052(2) *
C(3)	0.010(3) *	0.131(3)	0.055(3) *
C(4)	-0.026(2) *	0.033(2)	-0.009(2) *
C(5)	0.068(3)	-0.007(3) *	0.009(3) *
C(6)	0.143(3)	0.004(3) *	0.041(3) *
C(7)	0.141(3)	0.002(3) *	0.026(3) *
C(8)	0.073(3)	-0.003(3) *	-0.013(3) *
C(9)	0.008(2) *	-0.007(2) *	-0.034(2) *
C(10)	0.013(2) *	0.004(2) *	-0.015(2) *
C(11)	-0.093(3)	-0.032(3)	-0.061(3) *
H(2)	0.06(3)	0.22(3)	0.11(3)
H(3)	-0.02(3)	0.14(3)	0.05(3)
H(5)	0.09(3)	0.01(3)	0.04(3)
H(6)	0.18(3)	0.00(3)	0.06(3)
H(7)	0.19(3)	0.00(3)	0.04(3)
H(8)	0.07(3)	-0.01(3)	-0.02(3)

*Atoms included in mean plane calculations.

Angles Between Normals to the Planes: (degrees)

Planes (1) and (2) -------- 3.1
Planes (1) and (3) -------- 1.8
Planes (2) and (3) -------- 1.4

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slightly too large (3.447(5)Å) to be used for such an argument. However the close approaches at C(2) and C(9) might still be the reason for distortion. There are no intermolecular steric repulsions of importance.

The two methyl-naphthalene residues are cis-oriented with an angle of 68.4(2)° between them. This is in agreement with the reported value[12] of 68° for 1,1'-binaphthyl (which also crystallizes in space group C2/c).

Bond lengths and bond angles are listed in Tables VI and VII, with standard deviations calculated from errors in positional parameters. They are not significantly different from those in 1,1'-binaphthyl or naphthalene. The C(1)-C(1') bond connecting the two asymmetric units has a length of 1.495(4)Å and is bisected by the two-fold axis.

TABLE VI

Bond Lengths (Å)

Carbon-Carbon Bonds

C(1)-C(2)	1.371(3)
C(1)-C(9)	1.433(3)
C(1)-C(1')	1.495(4)
C(2)-C(3)	1.411(3)
C(3)-C(4)	1.367(3)
C(4)-C(10)	1.432(3)
C(4)-C(11)	1.509(3)
C(5)-C(6)	1.367(4)
C(5)-C(10)	1.418(3)
C(6)-C(7)	1.408(4)
C(7)-C(8)	1.369(4)
C(8)-C(9)	1.422(3)
C(9)-C(10)	1.426(3)

Carbon-Hydrogen Bonds

C(2)-H(2)	0.98(3)
C(3)-H(3)	1.00(3)
C(5)-H(5)	1.05(3)
C(6)-H(6)	1.02(3)
C(7)-H(7)	0.97(3)
C(8)-H(8)	1.02(3)
C(11)-H(111)	1.02(3)
C(11)-H(112)	1.03(3)
C(11)-H(113)	0.95(4)

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TABLE VII

Bond Angles (°)

Involving Carbons Only		Involving Hydrogens	
C(9)-C(1)-C(2)	118.0(2)	C(1)-C(2)-H(2)	121(1)
C(1')-C(1)-C(2)	119.5(2)	C(3)-C(2)-H(2)	117(1)
C(1')-C(1)-C(9)	122.4(2)		
		C(2)-C(3)-H(3)	119(2)
C(1)-C(2)-C(3)	122.1(2)	C(4)-C(3)-H(3)	119(2)
C(2)-C(3)-C(4)	121.3(2)	C(10)-C(5)-H(5)	118(2)
		C(6)-C(5)-H(5)	120(2)
C(3)-C(4)-C(11)	120.0(2)		
C(3)-C(4)-C(10)	118.9(2)	C(5)-C(6)-H(6)	118(2)
C(10)-C(4)-C(11)	121.0(2)	C(7)-C(6)-H(6)	122(2)
C(10)-C(5)-C(6)	121.2(2)	C(6)-C(7)-H(7)	119(2)
		C(8)-C(7)-H(7)	121(2)
C(5)-C(6)-C(7)	120.1(2)		
		C(7)-C(8)-H(8)	117(2)
C(6)-C(7)-C(8)	120.4(2)	C(9)-C(8)-H(8)	122(2)
C(7)-C(8)-C(9)	121.1(2)	C(4)-C(11)-H(111)	112(2)
		C(4)-C(11)-H(112)	113(2)
C(8)-C(9)-C(10)	118.3(2)	C(4)-C(11)-H(113)	110(2)
C(8)-C(9)-C(1)	121.5(2)	H(111)-C(11)-H(112)	105(3)
C(1)-C(9)-C(10)	120.1(2)	H(111)-C(11)-H(113)	110(3)
		H(112-C(11)-H(113)	107(3)
C(9)-C(10)-C(4)	119.5(2)		
C(9)-C(10)-C(5)	118.9(2)		
C(4)-C(10)-C(5)	121.7(2)		

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PART THREE:

THE CRYSTAL STRUCTURE OF

OPTICALLY ACTIVE 4,4'-DIMETHYL-1,1'-BINAPHTHYL

Experimental.

From x-ray photography the crystal was found to be tetragonal and of one of the enantiomeric space groups $P4_12_12$ or $P4_32_12$ (systematic abscenses: 00l for $l \neq 4n$, h00 for $h \neq 2n$). Unit-cell parameters were refined by least-squares methods from the observed 2θ values of 28 planes (see Table VIII for final cell parameters).

TABLE VIII

Crystal Data

$C_{22}H_{18}$	f.w. = 282.4 amu
Tetragonal	Z = 4
	Space Group = $P4_12_12$ or $P4_32_12$
a = b = 8.3031(8) Å	
c = 23.706(7) Å	F(000) = 600
Dc = 1.1478(4) g/cc	λ = 1.5418 Å
V = 1634.3(5) Å^3	

Intensity data were collected as before for $0° \leq 2\theta \leq 160°$, the crystal mounted with (0,2,3) perpendicular to the goniostat axis and using 4 2 2 as the check reflection. The check reflection scan count showed only slight random variations and no data scaling was used. 395 out of 1117 unique reflections had intensities less than $3.0\sigma(I)$, and structure amplitudes were calculated as before.

Structure Analysis.

A Wilson plot yielded an overall temperature factor of 6.11 A^2 and an overall scale factor. E statistics (which are not conclusive for either a centrosymmetric or a non-centrosymmetric structure) are shown in Table IX and 172 E's are greater than 1.4. From the largest 50 E's 559 Σ_2-

TABLE IX

E Statistics

	OBSERVED	THEORETICAL	
		Centro.	Non-centro.
Mean $\|E\|$	0.8193	0.7980	0.8860
Mean $\|E\|^2$	0.9873	1.0000	1.0000
Mean $\|E^2-1\|$	0.9031	0.9680	0.7360
% Reflections with :			
E > 3.0	0.18	0.30	0.01
E > 2.0	3.38	5.00	1.80
E > 1.0	31.58	32.00	37.00

****

relations were obtained and used to aid in origin and symbol choice.

The spacegroups $P4_12_12$ and $P4_32_12$ belong to type 2P22 for which suggested origins[6] are $\phi(00u)=0$ or π and $\phi(hk0)=0$ or π (h+k=odd) or other sets of indices for which the structure factor amplitude expressions result in a choice of two phases differing by π. The (00u) reflections are systematically absent in these space groups and could not be used. The 6 7 0 reflection was assigned phase 0 (the amplitude expressions simplify to B=0, i.e. only phases 0 and π allowed), the 5 5 10 reflection (a structure invariant hence not origin determining, but must have phase 0 or π) was assigned phase 0, and the 3 3 17 reflection (for which amplitude expressions result in A=0 and hence only $\pm\pi/2$ are allowed) was given phase $+\pi/2$.

With two symbols, expressions may be derived for the phases of the 50 largest E's, so only two symbols, (0 7 7) and (2 4 15), with eight starting values each, were used to give 64 starting sets in a symbolic addition and tangent refinement procedure. The best starting set $(3\pi/2, 3\pi/2)$ refined to an R-Karle of 0.171 and determined 169 out of the 172 phases.

Using these 169 phased E's an E-map was calculated in which all 11 carbons in the asymmetric unit could be located. After 3 isotropic and 3 anisotropic least-squares refinement cycles, when the R value had dropped to 0.12, a difference map was calculated to locate one methyl hydrogen enabling all other hydrogen positions to be determined geometrically. 3 more least-squares cycles including the hydrogens reduced R to 0.063. A weighting scheme where $w=1/\{1+[(|Fo|-F^*)/G^*]^2\}$ was

introduced (first with F*=5 and G*=20 and then a further cycle with F*=6 and G*=15) and lowered R to its final value of 0.060. Structure factor tables appear in Appendix II, and final atomic parameters and temperature factors are listed in Tables X and XI .

Determination of the absolute configuration was attempted using anomalous dispersion techniques, but differences in R values between the two enantiomers were insignificantly small (perhaps understandably so as we are dealing with a hydrocarbon) yet all differences lean in the same direction; the slightly smaller residual factors suggest $P4_12_12$ (in which the structure was solved).

Results and Discussion.

Table XII shows the results of mean plane calculations. The molecule exhibits the same bending found in the racemic compound, the angle between the rings now being 2.7°. The two methyl-naphthalene residues are linked by bond C(1)-C(1') of length 1.510(8)Å and the angle between them is now 80.6(3)°. The slightly lesser bending might agree with the slightly greater dihedral angle than in the racemic dimethyl-binaphthyl, but it is hard to see why this bending should be three times that in binaphthyl.

Comparing close contacts for the two structures under study and binaphthyl[12] (see Table XIII), we see no obvious trend. It would seem that the C(8)...C(8') close approach should play a greater role in this bending than the other close approaches, but this is not evident from the results. More

TABLE X

Fractional Atomic Parameters ($Cx10^4$, $Hx10^3$) With Their Standard Deviations

Atom	x/a	y/b	z/c
C(1)	951(5)	271(5)	2401(2)
C(2)	2489(6)	276(7)	2618(2)
C(3)	3641(6)	1444(9)	2431(2)
C(4)	3269(6)	2563(7)	2035(2)
C(5)	1250(8)	3696(7)	1371(2)
C(6)	-243(8)	3636(9)	1130(3)
C(7)	-1377(7)	2577(8)	1325(2)
C(8)	-1014(6)	1493(6)	1742(2)
C(9)	525(5)	1450(6)	1988(2)
C(10)	1690(5)	2604(6)	1795(2)
C(11)	454(1)	374(1)	1836(4)
H(2)	271(5)	-57(5)	291(2)
H(3)	485(6)	118(5)	262(2)
H(5)	206(7)	434(7)	121(2)
H(6)	-42(7)	435(7)	81(2)
H(7)	-254(8)	259(7)	121(2)
H(8)	-177(7)	86(6)	185(2)
H(111)	482(8)	339(8)	139(3)
H(112)	562(8)	341(8)	201(3)
H(113)	408(9)	470(7)	192(3)

TABLE XI

Anisotropic Thermal Parameters Of The Carbon Atoms With Their Standard Deviations (x10⁴)

Atom	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
C(1)	190(8)	229(8)	21(1)	25(6)	3(2)	- 9(2)
C(2)	216(9)	333(12)	21(1)	49(9)	-10(2)	- 6(3)
C(3)	173(8)	425(15)	24(1)	-16(10)	2(3)	-22(3)
C(4)	213(9)	320(12)	25(1)	-36(8)	14(3)	-21(3)
C(5)	264(11)	273(11)	35(1)	- 8(10)	30(3)	24(3)
C(6)	260(12)	385(16)	43(2)	34(12)	15(4)	65(5)
C(7)	205(10)	372(14)	35(1)	67(10)	- 1(3)	35(4)
C(8)	172(8)	239(9)	28(1)	15(8)	- 2(2)	12(3)
C(9)	175(7)	230(8)	22(1)	15(6)	5(2)	- 4(2)
C(10)	172(8)	247(9)	26(1)	- 6(7)	22(2)	- 8(3)
C(11)	238(14)	513(25)	47(2)	-122(16)	31(5)	-16(6)

Hydrogen Isotropic Thermal Parameters (A^2)

Atom	B
H(2)	5(1)
H(3)	8(1)
H(5)	9(2)
H(6)	11(2)
H(7)	11(2)
H(8)	8(1)
H(111)	12(2)
H(112)	12(2)
H(113)	12(2)

TABLE XII

Mean Planes

Equations of Planes: (lX+mY+nZ=p)

Plane	l	m	n	p
1	0.2933	-0.6267	-0.7220	-4.0213
2	0.3105	-0.6556	-0.6883	-3.9098
3	0.3001	-0.6412	-0.7063	-3.9401

Deviations from Planes:

Atom	Plane 1.	Plane 2.	Plane 3.
C(1)	0.000(5) *	0.037(5)	0.013(4) *
C(2)	-0.005(6) *	0.096(6)	0.030(5) *
C(3)	0.003(5) *	0.130(5)	0.008(6) *
C(4)	0.003(4) *	0.090(4)	-0.018(6) *
C(5)	0.016(5)	-0.007(5) *	-0.012(6) *
C(6)	0.077(6)	-0.011(6) *	0.051(7) *
C(7)	0.135(7)	0.023(7) *	0.006(6) *
C(8)	0.056(6)	-0.017(6) *	-0.024(5) *
C(9)	0.006(4) *	-0.001(4) *	-0.029(4) *
C(10)	-0.007(4) *	0.013(4) *	-0.030(4) *
C(11)	0.04(1)	0.05(1)	-0.00(1) *
H(2)	0.11(4)	0.24(4)	0.04(4)
H(3)	-0.01(4)	0.17(4)	0.13(4)
H(5)	-0.03(5)	-0.04(5)	0.12(5)
H(6)	-0.01(6)	-0.13(6)	0.16(6)
H(7)	0.27(6)	0.11(6)	-0.09(6)
H(8)	0.20(5)	0.11(5)	-0.06(5)

---

*Atoms included in mean plane calculations.

Angles Between Normals to the Planes: (degrees)

Planes (1) and (2) -------- 2.7
Planes (1) and (3) -------- 1.3
Planes (2) and (3) -------- 1.5

****

TABLE XIII

Close Contacts

	Racemic dimethyl-binaphthyl	Optically active dimethyl-binaphthyl	1,1'-Binaphthyl
C(2)...C(2')	3.11 Å	3.31 Å	3.107 Å
C(9)...C(9')	3.34 Å	3.36 Å	3.277 Å
C(8)...C(8')	3.45 Å	3.63 Å	3.321 Å
Bending angle:	3 °	2.7 °	1 °
Angle between asymmetric units:	68.4 °	80 °	68 °

results from other structures would be necessary for a more conclusive argument.

Bond distances and angles are listed in Tables XIV and XV. The numbering scheme is the same as before (see molecular drawing Fig. 5), and standard deviations were obtained from errors in positional parameters only as unit-cell errors were negligible. The bond lengths and angles have normal values.

TABLE XIV

Bond Lengths (Å)

Carbon-Carbon Bonds

C(1)-C(2)	1.377(6)
C(1)-C(9)	1.429(5)
C(1)-C(1')	1.510(8)
C(2)-C(3)	1.432(8)
C(3)-C(4)	1.357(7)
C(4)-C(10)	1.429(6)
C(4)-C(11)	1.512(8)
C(5)-C(6)	1.366(8)
C(5)-C(10)	1.402(7)
C(6)-C(7)	1.369(8)
C(7)-C(8)	1.371(7)
C(8)-C(9)	1.405(6)
C(9)-C(10)	1.436(6)

Carbon-Hydrogen Bonds

C(2)-H(2)	1.01(4)
C(3)-H(3)	1.11(4)
C(5)-H(5)	0.94(6)
C(6)-H(6)	0.98(6)
C(7)-H(7)	1.02(6)
C(8)-H(8)	0.86(6)
C(11)-H(111)	1.12(7)
C(11)-H(112)	1.02(6)
C(11)-H(113)	0.91(6)

****

TABLE XV

Bond Angles (°)

Involving Carbons Only		Involving Hydrogens	
C(9)-C(1)-C(2)	118.9(4)	C(1)-C(2)-H(2)	115(2)
C(1')-C(1)-C(2)	120.6(4)	C(3)-C(2)-H(2)	125(2)
C(1')-C(1)-C(9)	120.5(3)		
		C(2)-C(3)-H(3)	110(2)
C(1)-C(2)-C(3)	120.4(5)	C(4)-C(3)-H(3)	128(2)
C(2)-C(3)-C(4)	121.8(5)	C(10)-C(5)-H(5)	119(3)
		C(6)-C(5)-H(5)	119(3)
C(3)-C(4)-C(11)	120.0(6)		
C(3)-C(4)-C(10)	120.0(5)	C(5)-C(6)-H(6)	116(4)
C(10)-C(4)-C(11)	120.0(6)	C(7)-C(6)-H(6)	123(4)
C(10)-C(5)-C(6)	120.8(5)	C(6)-C(7)-H(7)	124(3)
		C(8)-C(7)-H(7)	115(3)
C(5)-C(6)-C(7)	120.5(6)		
		C(7)-C(8)-H(8)	117(3)
C(6)-C(7)-C(8)	120.9(5)	C(9)-C(8)-H(8)	122(3)
C(7)-C(8)-C(9)	121.1(5)	C(4)-C(11)-H(111)	106(4)
		C(4)-C(11)-H(112)	109(4)
C(8)-C(9)-C(10)	117.7(4)	C(4)-C(11)-H(113)	102(5)
C(8)-C(9)-C(1)	121.6(3)	H(111)-C(11)-H(112)	97(5)
C(1)-C(9)-C(10)	120.6(4)	H(111)-C(11)-H(113)	120(6)
		H(112)-C(11)-H(113)	122(6)
C(9)-C(10)-C(4)	118.4(4)		
C(9)-C(10)-C(5)	119.0(4)		
C(4)-C(10)-C(5)	122.6(5)		

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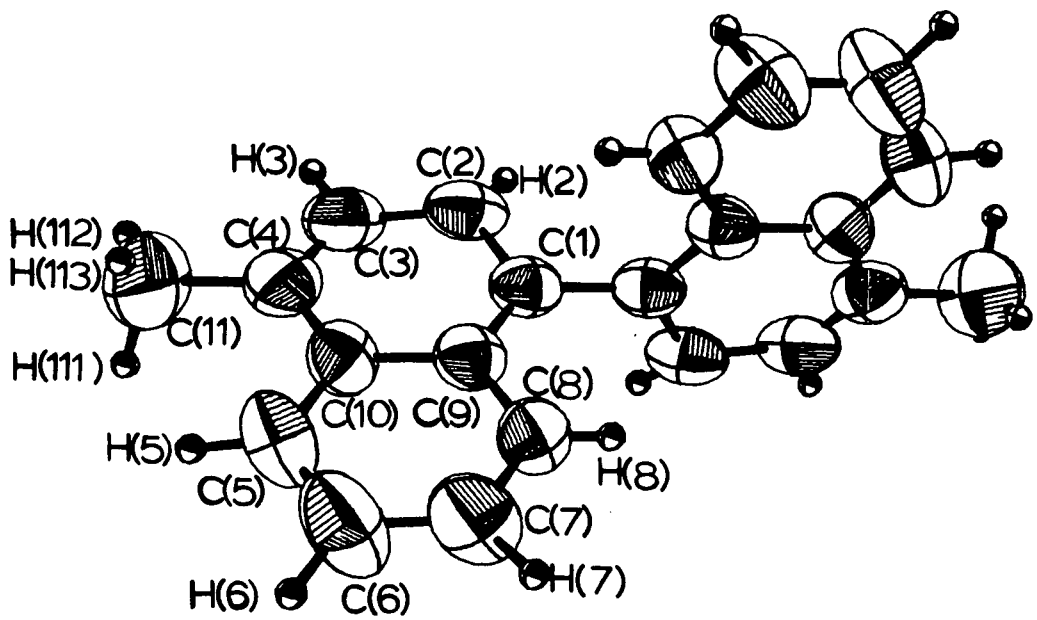

Figure 5. Molecular Drawing of Optically Active 4,4'-Dimethyl-1,1'-binaphthyl.

PART FOUR:

CONCLUSION

Bond Length and Angle Comparison

As shown in Fig. 6, the naphthalene residue consists of four types of bonds, here called types a, b, c and d. This figure also schematically shows the bond lengths and bond angles of both the racemic and optically active 4,4'-dimethyl-1,1'-binaphthyl to facilitate visual comparison. For each compound, the bond lengths have been averaged within each type, and the results compared to those of 1,1'-binaphthyl and naphthalene in Table XVI . These average bond lengths are very similar in all cases, and one may say that the structure of the naphthalene residue is reasonably invariant in these four compounds. Reasons for differences of stability will have to be sought in overall molecular geometry and intermolecular interactions, i.e. packing differences.

Intramolecular Differences.

These now reduce to differences in the angle between the residues and the differences in bending of the naphthalene unit. As mentioned before, it seems there is no correlation between the amount of bending and the dihedral angle (or the close contacts involved). The dihedral angle in each case is probably that with which the molecule packs most easily into its space group.

Cell Parameter Comparison.

The total mass content of the unit cells of the racemic and optically active forms is the same, but the cell volume of the racemate is smaller (1505 $Å^3$ as compared to 1634 $Å^3$) and

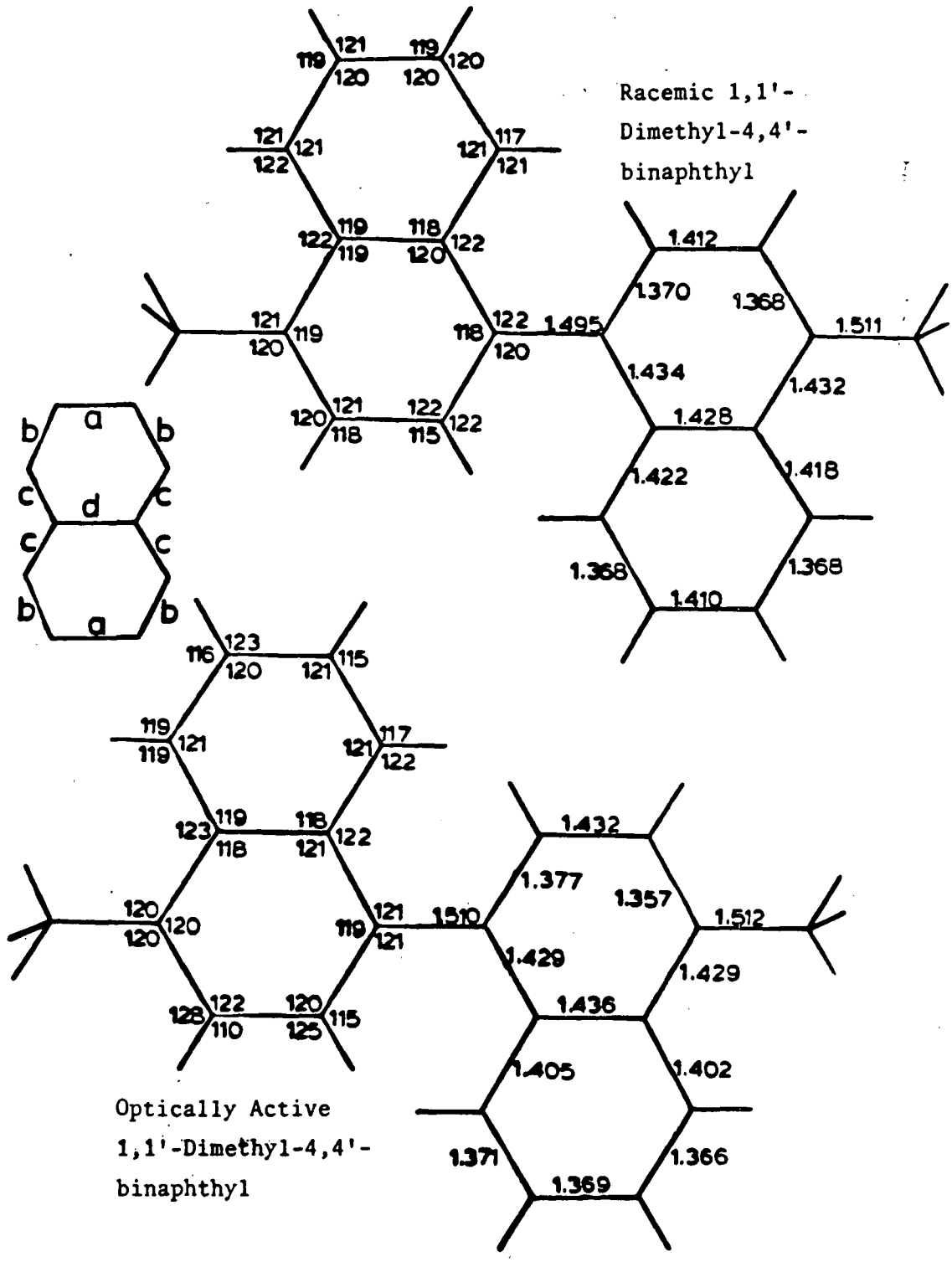

Figure 6. Bond Length Comparison.

TABLE XVI

Average Bond Length Comparison

Compound	Bond Lengths (Å) a	b	c	d
Racemic 4,4'-dimethyl-1,1'-binaphthyl:	1.410(3)	1.369(2)	1.426(3)	1.426(3)
Optically active 4,4'-dimethyl-1,1'-binaphthyl:	1.401(22)	1.368(4)	1.416(6)	1.436(6)
Average 4,4'-dimethyl-1,1'-binaphthyl:	1.405(12)	1.368(2)	1.421(4)	1.431(4)
1,1'-Binaphthyl:	1.404(3)	1.357(4)	1.418(4)	1.416(3)
Naphthalene:[17]	1.416(6)	1.357(4)	1.420(3)	1.405(6)

(errors are the maximum of the rms deviation from the mean and the rms standard deviation of the bond lengths)

hence the density is greater (1.246 g/cm³ as compared to 1.148 g/cm³). In other words, the racemate is packed slightly 'tighter' and for this reason its lattice will be of slightly higher lattice energy, hence somewhat more stable than that of the optically active form.

Packing Comparison.

The mode of packing is quite different for the two structures solved. By inspection of the packing diagram for the racemate (Fig. 7), we see that the structure essentially consists of layers parallel to the (001) planes, each layer containing molecules of one enantiomeric form, and so we have alternating layers of R and S molecules. The unit cell is a cross-section of two such layers, as is necessary for it to contain the entire repeating unit. Furthermore, within each layer all molecules have the same orientation with respect to the crystal axes, so there are only two orientations with which a molecule may add to the lattice during crystallization, and these two orientatons are enantiomeric. If we picture the molecule as two planes forming a wedge, the molecules are oriented such that the wedges are 'nested' along the shortest axis (c=10.8Å), an axial length apart (see Fig. 8).

In optically active 4,4'-dimethyl-1,1'-binaphthyl (Fig. 9) the molecules spiral around a 4-fold screw axis so there are four unique orientations. The shape of the unit-cell is (unlike the racemate) quite an elongated square prism. The square edge is only 8.3 Å long, which means that four identically oriented molecules surround each molecule at this distance. This at

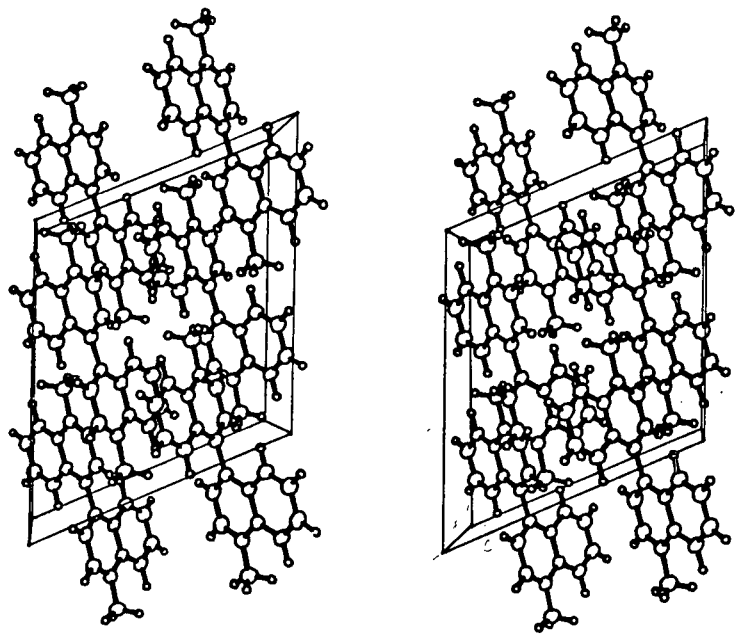

Figure 7. Packing Diagram for the Racemate.

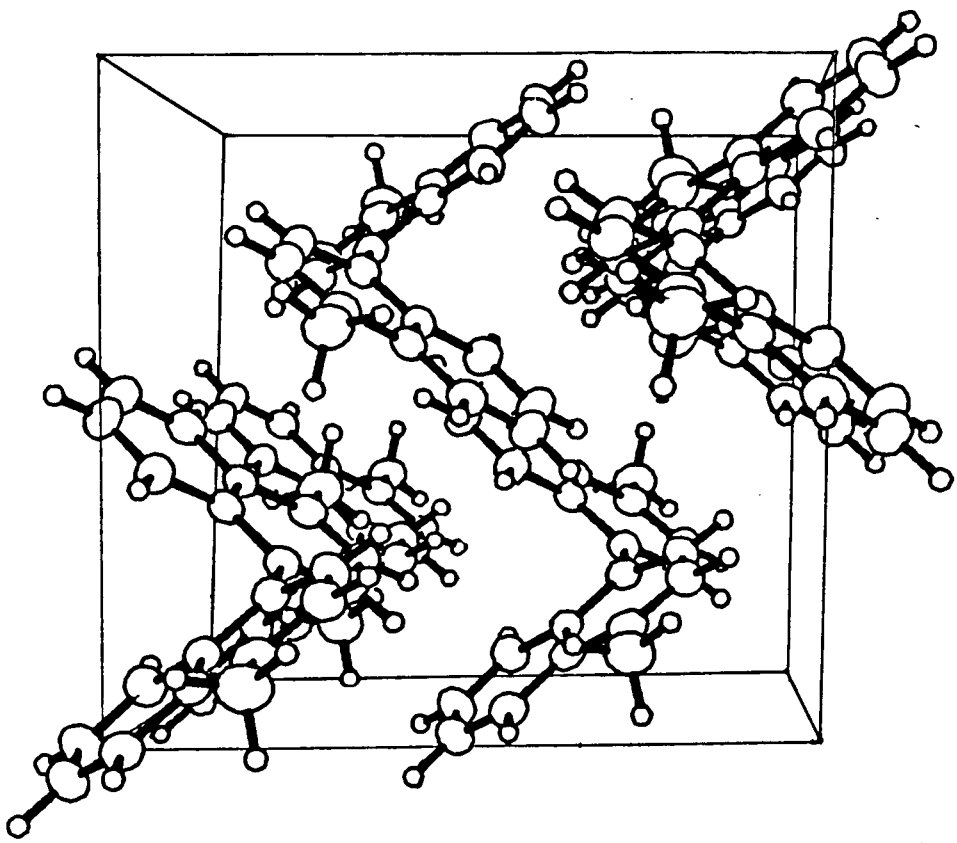

Figure 8. 'Wedge Nesting' in the Racemate Unit Cell

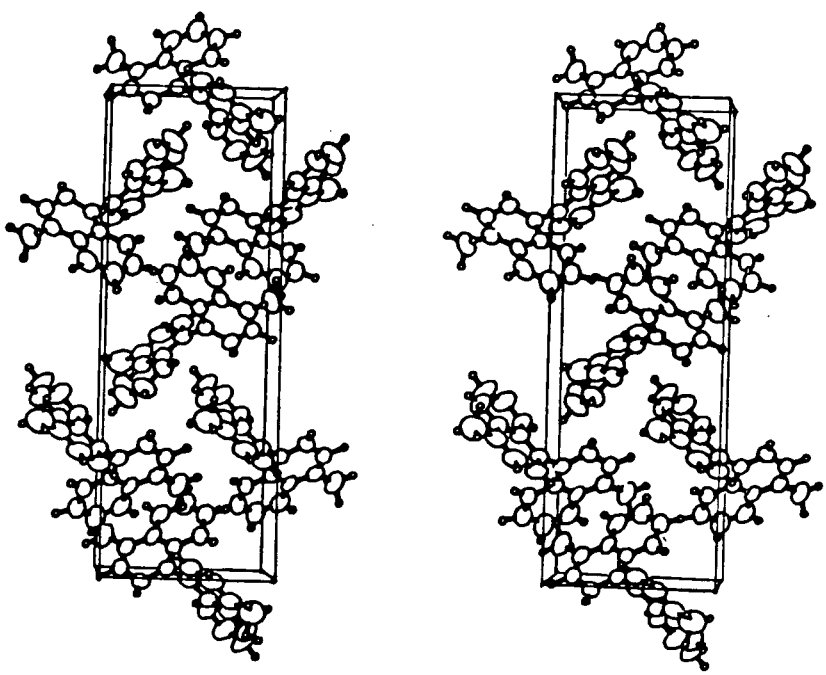

Figure 9. Packing Diagram for the Optically Active Form.

first might seem to be very dense packing, but in fact the 1-1' bond is oriented closer to the square diagonal than to the axes, which means that the direction of 'wedge-nesting' is along the diagonal, and the repeat distance here is approximately 11.7 Å, i.e. , not as close as for the racemate. Perhaps the complications of packing four molecular orientations account for the long c-axis and the slightly lower density of the optically active form.

Summary.

Any difference in stability in crystalline racemic and optically active 4,4'-dimethyl-1,1'-binaphthyl is reflected in the lattice energies of the two structures - a higher lattice energy is associated with greater stability of the lattice. The racemate is likely to have the greater lattice energy, as it is packed slightly tighter and with a little more strain in the molecules (lower dihedral angle between naphthalene residues). This would agree with the observation that the dimethyl-binaphthyl does not resolve spontaneously upon heating to the optically active lattice (as it is less stable) in contrast to 1,1'-binaphthyl, but this does not explain the difference in behaviour between these two species. It would be of interest to study the crystal structures of optically active 1,1'-binaphthyl and other related compounds for comparison, but even then the effect of increased temperature on the lattice is not always predictable.

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APPENDIX I.

STRUCTURE FACTOR TABLES FOR

RACEMIC 1,1'-DIMETHYL-4,4'-BINAPHTHYL

h	k	l	Fo	Fc
-6	0	12	4.61	4.56
-8	0	12	2.12	2.44
-5	-1	12	2.14	2.25
-2	-2	12	5.40	5.34
-4	-2	12	9.50	8.84
-6	-2	12	4.31	4.79
-3	-3	12	11.59	11.52
-5	-3	12	4.71	5.01
-7	-3	12	4.26	4.48
-9	-3	12	6.53	6.64
-6	-4	12	3.26	3.20
-8	-4	12	2.58	2.71
-1	-1	11	6.96	6.95
-7	-1	11	4.24	4.24
-11	-1	11	5.03	4.96
-2	-2	11	9.98	9.54
-4	-2	11	6.90	6.49
-10	-2	11	3.25	3.19
-3	-3	11	3.86	3.67
-5	-3	11	5.35	5.22
-9	-3	11	4.82	4.63
-2	-4	11	4.31	4.33
-4	-4	11	12.76	11.90
-8	-4	11	2.93	3.17
-3	-5	11	2.46	2.65
-4	-6	11	5.59	5.70
-2	0	10	2.27	2.27
-4	0	10	6.22	5.96
-6	0	10	11.78	10.59
-10	0	10	12.11	11.70
-12	0	10	24.23	22.68
-3	-1	10	4.17	3.81
-5	-1	10	5.37	5.31
-7	-1	10	9.19	8.40
-11	-1	10	6.09	5.77
-4	-2	10	4.61	4.44
-6	-2	10	5.62	5.38
-8	-2	10	2.19	2.25
-1	-3	10	3.66	3.23
-3	-3	10	10.84	10.40
-7	-3	10	3.73	3.52
-11	-3	10	2.66	2.96
-2	-4	10	12.05	11.46
-4	-4	10	10.46	9.38
-1	-5	10	5.10	5.19
-3	-5	10	14.71	13.76
-5	-5	10	2.86	2.85
-7	-5	10	4.21	4.31
-9	-5	10	11.67	11.65
-2	-6	10	5.48	5.65
-4	-6	10	5.82	5.73
-10	-6	10	2.13	2.40
-1	-7	10	3.12	3.21
-3	-7	10	17.55	17.41
-5	-7	10	6.06	5.99
-5	-1	9	10.53	10.25
-9	-1	9	3.15	3.39
-11	-1	9	15.60	14.85
-4	-2	9	2.39	2.33
-6	-2	9	2.41	2.29
-8	-2	9	3.29	3.26
-3	-3	9	5.21	5.01
-11	-3	9	4.81	5.04
-2	-4	9	16.12	15.85
-4	-4	9	3.80	3.93
-8	-4	9	2.17	2.59
-12	-4	9	3.65	3.78
-1	-5	9	8.51	8.27
-3	-5	9	10.48	10.09
-9	-5	9	4.58	4.68
-4	-6	9	10.18	9.57
-8	-6	9	9.35	9.74
-1	-7	9	3.03	3.39
-3	-7	9	7.45	7.51
-9	-7	9	2.03	2.67
-2	-8	9	6.06	6.36
-4	-8	9	3.38	3.59
-4	0	8	2.78	2.30
-6	0	8	13.10	12.89
-8	0	8	3.16	3.42
-10	0	8	15.51	15.59
-12	0	8	6.85	6.72
-1	-1	8	2.51	2.49
-5	-1	8	16.50	15.42
-7	-1	8	6.34	5.93
-9	-1	8	3.59	3.42
-11	-1	8	6.83	6.88
-13	-1	8	5.61	5.67
-2	-2	8	3.25	3.27
-4	-2	8	5.81	5.20
-10	-2	8	9.15	9.49
-12	-2	8	9.95	9.74
-1	-3	8	4.07	4.08
-3	-3	8	2.59	2.68
-7	-3	8	5.12	4.79
-11	-3	8	4.24	4.46
-2	-4	8	16.50	16.16
-4	-4	8	5.09	5.12
-6	-4	8	2.15	2.15
-8	-4	8	9.34	8.98
-10	-4	8	3.28	3.12
-1	-5	8	9.86	10.35
-3	-5	8	32.44	31.42
-5	-5	8	3.08	3.61
-7	-5	8	2.91	3.03
-9	-5	8	3.47	3.18
-2	-6	8	9.99	9.78
-8	-6	8	4.52	4.89
-10	-6	8	2.25	2.54
-12	-6	8	3.84	3.42
-3	-7	8	2.91	2.86
-5	-7	8	2.65	3.02
-6	-8	8	2.24	2.05
-1	-9	8	2.55	2.70
-3	-9	8	4.76	4.43
-5	-9	8	2.54	2.80

h	k	l	Fo	Fc
-3	-1	7	19.03	14.51
-5	-1	7	18.62	15.54
-7	-1	7	2.68	2.69
-9	-1	7	9.27	9.15
-11	-1	7	14.04	13.37
-2	-2	7	11.58	11.63
-4	-2	7	5.39	5.27
-10	-2	7	18.21	18.27
-12	-2	7	10.33	10.09
-1	-3	7	2.69	3.12
-3	-3	7	2.23	2.64
-9	-3	7	7.40	7.70
-11	-3	7	3.78	4.03
-13	-3	7	7.57	7.26
-2	-4	7	9.29	9.59
-4	-4	7	11.11	11.48
-8	-4	7	12.74	13.13
-1	-5	7	8.31	8.16
-3	-5	7	5.18	5.43
-5	-5	7	3.08	2.93
-7	-5	7	4.03	4.31
-9	-5	7	5.64	5.85
-11	-5	7	4.85	4.69
-2	-6	7	42.27	34.98
-4	-6	7	6.90	6.83
-8	-6	7	6.61	7.64
-10	-6	7	3.99	3.87
-1	-7	7	9.83	9.69
-7	-7	7	7.61	8.16
-9	-7	7	4.67	5.16
-6	-8	7	4.38	4.66
-8	-8	7	5.05	5.16
-10	-8	7	4.43	5.00
-1	-9	7	4.63	5.02
-3	-9	7	4.72	4.74
-4	-10	7	3.35	3.76
-2	0	6	11.64	9.73
-4	0	6	12.39	13.34
-6	0	6	9.40	8.83
-8	0	6	3.51	3.58
-12	0	6	3.18	3.39
-1	-1	6	5.89	5.85
-3	-1	6	11.26	12.05
-5	-1	6	10.28	11.10
-9	-1	6	5.44	5.53
-11	-1	6	8.27	8.63
-2	-2	6	5.33	5.01
-6	-2	6	12.69	13.51
-10	-2	6	18.04	17.20
-9	-3	6	9.54	9.24
-2	-4	6	15.72	15.20
-4	-4	6	8.78	9.24
-6	-4	6	8.06	8.44
-10	-4	6	3.15	3.12
-12	-4	6	4.75	5.04
-1	-5	6	15.96	17.59
-3	-5	6	4.95	5.70
-7	-5	6	4.26	4.16
-11	-5	6	2.65	3.10
-2	-6	6	3.04	3.38
-4	-6	6	2.27	2.53
-6	-6	6	3.47	3.70
-8	-6	6	3.56	3.93
-1	-7	6	6.01	5.92
-3	-7	6	12.46	12.14
-7	-7	6	4.77	5.20
-9	-7	6	2.55	2.83
-11	-7	6	3.66	3.85
-2	-8	6	10.92	10.90
-4	-8	6	4.30	4.63
-6	-8	6	3.74	3.89
-9	-9	6	4.60	5.01
-1	-1	5	8.76	8.91
-3	-1	5	7.72	8.31
-7	-1	5	10.47	10.31
-11	-1	5	4.71	4.98
-2	-2	5	3.41	2.92
-4	-2	5	27.45	25.20
-6	-2	5	4.05	4.38
-8	-2	5	17.99	16.53
-1	-3	5	55.32	42.93
-3	-3	5	42.76	35.39
-5	-3	5	12.93	12.39
-7	-3	5	13.32	13.71
-9	-3	5	17.77	17.45
-11	-3	5	13.73	13.39
-2	-4	5	49.20	39.69
-8	-4	5	4.11	4.69
-10	-4	5	22.67	22.10
-12	-4	5	2.85	3.00
-3	-5	5	3.78	3.98
-5	-5	5	4.19	4.35
-9	-5	5	3.15	2.91
-2	-6	5	3.60	3.64
-4	-6	5	9.18	8.59
-10	-6	5	7.25	7.06
-1	-7	5	13.80	14.18
-3	-7	5	10.47	10.78
-9	-7	5	4.80	5.21
-11	-7	5	6.76	6.38
-2	-8	5	15.56	14.97
-4	-8	5	9.75	9.15
-8	-8	5	5.32	5.89
-10	-8	5	7.82	8.39
-1	-9	5	3.98	4.15
-5	-9	5	8.51	8.75
-7	-9	5	8.96	9.20
-1	-11	5	5.75	5.37
-2	0	4	30.70	28.67
-6	0	4	11.26	10.64
-8	0	4	6.84	7.15
-10	0	4	5.54	5.28
12	0	-4	5.97	5.88
14	0	-4	6.54	6.44
-1	-1	4	14.57	12.79
-3	-1	4	8.55	9.00

h	k	l	Fo	Fc
-7	-1	4	5.77	5.45
-9	-1	4	8.52	7.58
13	1	-4	4.30	4.58
-2	-2	4	20.82	19.77
-4	-2	4	20.79	16.74
-6	-2	4	15.56	15.80
-8	-2	4	7.81	8.14
-10	-2	4	3.32	3.49
12	2	-4	8.19	8.34
14	2	-4	3.38	4.24
-1	-3	4	34.88	30.25
-3	-3	4	30.94	24.56
-5	-3	4	1.83	1.94
-7	-3	4	14.41	13.90
-9	-3	4	23.26	18.85
-11	-3	4	12.66	11.92
-6	-4	4	9.38	8.95
-8	-4	4	14.52	15.01
-10	-4	4	13.09	13.60
12	4	-4	2.90	2.92
-3	-5	4	4.55	5.21
-9	-5	4	4.70	4.96
-11	-5	4	5.62	5.50
-2	-6	4	2.59	2.58
-4	-6	4	7.63	7.72
-6	-6	4	3.93	3.67
-10	-6	4	2.69	2.68
12	6	-4	4.98	4.88
-1	-7	4	18.07	18.34
-7	-7	4	4.38	4.53
-11	-7	4	8.27	7.71
-2	-8	4	8.58	8.13
-4	-8	4	4.57	4.72
-6	-8	4	5.21	5.96
-8	-8	4	6.45	6.37
-10	-8	4	7.74	7.56
-3	-9	4	8.72	8.59
-7	-9	4	6.23	6.68
-2	-10	4	2.95	3.41
-1	-11	4	4.45	4.47
-3	-1	3	9.83	10.79
-5	-1	3	5.22	4.77
11	1	-3	3.51	3.18
-4	-2	3	8.73	8.61
-6	-2	3	4.13	4.13
-1	-3	3	9.94	10.29
-3	-3	3	2.32	2.32
-5	-3	3	4.32	4.13
9	3	-3	16.74	14.58
11	3	-3	3.07	3.44
-2	-4	3	12.38	13.36
-4	-4	3	24.82	22.20
-8	-4	3	10.26	10.36
-1	-5	3	9.34	9.34
-3	-5	3	9.66	8.73
-5	-5	3	7.77	7.23
11	5	-3	4.50	4.66
-4	-6	3	10.19	9.57
-8	-6	3	9.80	9.83
12	6	-3	3.70	3.46
-1	-7	3	3.20	3.25
-3	-7	3	7.27	7.14
-5	-7	3	10.04	9.83
11	7	-3	4.82	5.00
-2	-8	3	4.02	4.03
-4	-8	3	3.54	3.78
-6	-8	3	4.51	4.62
10	8	-3	3.04	3.17
-1	-9	3	18.60	18.62
-3	-9	3	4.52	4.32
-5	-9	3	4.68	4.74
-2	-10	3	2.88	3.19
-6	-10	3	3.35	3.87
-1	-11	3	2.37	2.97
-5	-11	3	3.72	3.95
-4	0	2	3.65	3.63
8	0	-2	8.31	8.16
10	0	-2	9.08	8.92
12	0	-2	13.26	15.38
14	0	-2	4.67	5.42
-3	-1	2	22.37	19.68
-5	-1	2	27.26	21.88
7	1	-2	25.84	20.37
-4	-2	2	15.91	17.21
6	2	-2	16.18	12.75
8	2	-2	10.47	9.77
10	2	-2	4.65	4.14
14	2	-2	3.30	3.46
-1	-3	2	17.44	17.08
-3	-3	2	23.03	19.37
-5	-3	2	6.86	6.85
7	3	-2	1.74	1.60
9	3	-2	6.36	6.77
-2	-4	2	14.90	16.39
-4	-4	2	22.01	18.25
6	4	-2	3.84	4.16
8	4	-2	4.28	3.95
10	4	-2	8.26	8.25
-1	-5	2	4.36	4.60
-3	-5	2	26.60	22.50
-5	-5	2	7.42	7.34
7	5	-2	9.03	9.08
9	5	-2	47.79	38.70
11	5	-2	7.07	7.06
-2	-6	2	5.19	5.03
-4	-6	2	11.03	11.89
8	6	-2	3.68	4.04
10	6	-2	6.92	6.90
-3	-7	2	17.15	17.29
-5	-7	2	5.74	5.49
9	7	-2	7.51	7.70
10	8	-2	3.13	3.50
-1	-9	2	17.52	15.51
9	9	-2	6.05	6.06
6	10	-2	7.12	7.75
-3	-11	2	2.59	2.69

h	k	l	Fo	Fc
3	1	-1	3.74	3.70
7	1	-1	1.63	1.76
11	1	-1	7.52	8.49
-2	-2	1	6.12	6.32
4	2	-1	4.19	4.07
6	2	-1	5.77	5.37
8	2	-1	7.74	7.33
-1	-3	1	12.02	13.21
3	3	-1	4.21	4.19
5	3	-1	3.98	4.44
7	3	-1	2.88	2.60
9	3	-1	4.42	4.74
11	3	-1	3.01	3.28
-2	-4	1	21.13	18.23
4	4	-1	5.29	5.53
6	4	-1	4.65	4.96
10	4	-1	8.52	7.98
12	4	-1	3.47	3.25
-1	-5	1	9.55	10.00
3	5	-1	24.05	19.15
5	5	-1	3.60	3.14
7	5	-1	6.27	6.71
9	5	-1	13.82	13.68
11	5	-1	5.13	4.77
4	6	-1	15.59	13.33
8	6	-1	27.40	22.72
10	6	-1	5.75	5.42
3	7	-1	8.85	8.22
5	7	-1	4.39	4.14
9	7	-1	9.00	8.91
11	7	-1	2.96	2.66
4	8	-1	3.40	3.55
-1	-9	1	3.24	3.43
6	10	-1	8.28	8.02
8	10	-1	2.35	2.45
-1	-11	1	25.51	21.02
3	11	-1	3.95	3.71
5	11	-1	4.52	4.37
4	0	0	9.10	9.24
12	0	0	3.91	4.08
1	1	0	24.26	21.19
3	1	0	7.85	7.12
5	1	0	39.48	30.11
9	1	0	5.00	4.99
13	1	0	3.20	3.80
0	2	0	16.86	15.03
2	2	0	20.24	17.37
4	2	0	6.72	6.72
6	2	0	26.96	21.18
8	2	0	3.41	3.30
12	2	0	5.94	7.07
1	3	0	21.34	18.37
3	3	0	2.94	2.51
5	3	0	9.28	9.40
7	3	0	9.06	9.42
9	3	0	11.75	11.52
11	3	0	4.32	4.63
13	3	0	2.84	2.92
2	4	0	15.87	15.67
4	4	0	6.77	6.41
6	4	0	5.79	5.39
8	4	0	17.51	17.24
10	4	0	14.48	14.29
1	5	0	13.49	11.90
3	5	0	45.60	35.17
5	5	0	7.73	7.25
7	5	0	3.89	4.10
9	5	0	9.54	9.30
0	6	0	4.42	4.27
2	6	0	11.98	11.34
4	6	0	2.24	2.20
6	6	0	2.19	2.15
8	6	0	9.28	8.79
10	6	0	8.13	7.54
1	7	0	6.91	6.55
3	7	0	7.36	6.16
7	7	0	11.11	11.86
9	7	0	15.92	18.35
0	8	0	7.27	7.25
2	8	0	3.04	3.75
8	8	0	5.00	5.82
3	9	0	2.02	2.32
5	9	0	2.00	2.51
0	10	0	16.45	17.37
4	10	0	2.40	2.45
6	10	0	6.37	7.45
1	11	0	3.03	3.34
0	12	0	11.50	11.96
1	1	1	1.11	1.15
3	1	1	9.50	10.57
7	1	1	6.32	6.43
9	1	1	5.73	6.08
2	2	1	5.01	5.64
4	2	1	6.37	6.79
8	2	1	3.63	3.90
10	2	1	11.11	12.45
12	2	1	2.62	3.51
1	3	1	15.66	17.39
3	3	1	7.00	7.94
7	3	1	6.86	7.02
0	4	1	8.07	9.16
2	4	1	15.27	13.91
4	4	1	8.66	9.01
6	4	1	8.23	9.06
8	4	1	25.62	27.84
10	4	1	3.52	3.93
1	5	1	3.41	3.30
3	5	1	8.13	7.11
9	5	1	7.91	8.01
0	6	1	2.44	2.58
2	6	1	29.21	24.50
4	6	1	8.08	7.07
8	6	1	14.87	13.97
1	7	1	6.61	6.72
7	7	1	15.28	15.05
9	7	1	10.72	10.59

h	k	l	Fo	Fc
0	8	1	5.38	4.86
4	8	1	6.27	6.22
8	8	1	9.62	8.31
1	9	1	13.05	12.24
5	9	1	2.57	3.03
0	10	1	9.60	8.52
2	10	1	3.62	3.80
4	10	1	2.94	3.56
6	10	1	2.65	3.16
1	11	1	7.05	6.66
5	11	1	5.94	6.17
0	12	1	2.42	2.34
2	0	2	20.15	19.21
6	0	2	5.81	5.67
10	0	2	3.65	4.00
3	1	2	18.38	17.65
5	1	2	9.00	8.74
2	2	2	7.98	8.24
4	2	2	7.25	7.34
6	2	2	8.45	7.43
8	2	2	17.35	17.83
1	3	2	19.62	18.10
3	3	2	29.65	24.76
5	3	2	19.68	18.06
7	3	2	7.37	7.08
9	3	2	12.97	12.84
2	4	2	10.18	10.32
4	4	2	7.01	6.76
6	4	2	7.47	7.82
8	4	2	4.24	4.31
3	5	2	6.35	6.31
0	6	2	9.91	10.13
2	6	2	7.07	7.32
4	6	2	2.73	2.80
6	6	2	3.15	3.22
8	6	2	7.66	7.45
1	7	2	2.05	2.31
3	7	2	11.55	10.46
7	7	2	10.15	9.60
0	8	2	5.06	5.06
2	8	2	11.01	10.69
6	8	2	9.25	9.36
8	8	2	4.38	4.32
1	9	2	12.91	12.82
5	9	2	3.53	4.29
7	9	2	11.08	10.66
0	10	2	15.04	13.93
2	10	2	4.62	4.64
1	1	3	9.26	8.70
3	1	3	3.99	3.91
5	1	3	10.42	10.44
7	1	3	11.13	11.22
9	1	3	3.22	3.32
2	2	3	4.91	4.78
4	2	3	6.24	6.07
6	2	3	25.19	23.98
8	2	3	19.75	19.40
1	3	3	41.46	33.86
3	3	3	3.01	2.66
5	3	3	20.75	20.41
7	3	3	11.88	11.60
9	3	3	16.13	15.55
11	3	3	3.62	3.66
0	4	3	1.90	2.21
2	4	3	25.69	21.02
4	4	3	8.91	8.16
10	4	3	8.85	8.79
1	5	3	4.67	4.57
3	5	3	2.16	1.99
0	6	3	9.73	9.29
2	6	3	5.53	5.29
6	6	3	4.66	4.39
8	6	3	6.83	6.76
1	7	3	11.94	10.80
3	7	3	3.94	4.07
5	7	3	2.23	1.96
7	7	3	2.91	3.18
9	7	3	3.90	3.56
0	8	3	2.88	3.03
2	8	3	7.63	6.98
4	8	3	4.69	5.58
6	8	3	10.31	9.97
8	8	3	12.01	11.71
5	9	3	2.98	3.73
7	9	3	11.45	11.20
0	10	3	9.18	8.86
4	10	3	2.63	2.69
1	11	3	2.79	2.73
0	0	4	14.56	15.72
2	0	4	4.10	4.02
4	0	4	2.68	2.68
6	0	4	9.50	9.89
8	0	4	17.44	14.80
1	1	4	9.55	9.17
3	1	4	10.64	10.88
7	1	4	5.37	5.18
9	1	4	11.33	11.28
0	2	4	1.86	2.04
2	2	4	15.94	16.39
4	2	4	11.60	11.31
6	2	4	11.17	11.29
8	2	4	8.17	8.31
3	3	4	13.32	11.52
5	3	4	4.58	4.68
7	3	4	11.99	12.81
0	4	4	15.26	15.53
2	4	4	4.39	4.35
4	4	4	4.91	4.79
6	4	4	3.39	3.08
10	4	4	3.45	3.62
3	5	4	7.64	7.31
5	5	4	5.24	5.35
7	5	4	4.09	4.05
9	5	4	2.52	2.70
4	6	4	4.80	5.23
6	6	4	4.41	4.94

h	k	l	Fo	Fc	h	k	l	Fo	Fc
3	7	4	3.45	3.21	0	6	6	11.06	10.75
5	7	4	4.51	5.06	4	6	6	5.26	5.36
0	8	4	22.91	22.87	5	7	6	5.18	5.40
2	8	4	4.09	4.42	1	9	6	4.86	4.90
4	8	4	4.62	4.94	0	10	6	7.14	6.80
6	8	4	8.05	8.31	1	1	7	13.52	12.45
1	9	4	2.14	2.57	7	1	7	9.16	8.93
5	9	4	6.84	7.28	6	2	7	6.37	6.49
2	10	4	4.04	3.91	1	3	7	4.59	4.63
1	1	5	24.74	25.86	0	4	7	2.08	1.91
5	1	5	2.36	2.25	4	4	7	4.07	4.33
7	1	5	21.66	21.90	3	5	7	5.53	5.84
9	1	5	5.59	5.55	5	5	7	4.53	4.70
0	2	5	21.37	18.78	0	6	7	11.21	11.26
2	2	5	13.60	10.94	4	6	7	8.96	9.42
4	2	5	3.03	2.71	3	7	7	7.01	7.24
8	2	5	3.09	3.26	0	0	8	33.27	28.41
3	3	5	5.71	5.68	2	0	8	11.47	10.01
5	3	5	5.85	5.47	4	0	8	2.46	2.65
7	3	5	3.39	3.40	6	0	8	16.44	16.34
0	4	5	24.27	20.36	5	1	8	11.47	11.20
4	4	5	12.16	11.80	0	2	8	4.00	3.99
6	4	5	10.22	9.63	2	2	8	5.69	5.64
8	4	5	3.98	4.13	4	2	8	2.44	2.42
1	5	5	5.35	4.92	6	2	8	14.32	14.20
3	5	5	7.07	6.62	2	4	8	5.74	5.31
5	5	5	7.72	7.72	3	5	8	12.10	11.49
2	6	5	2.86	2.60	5	5	8	5.76	5.89
4	6	5	7.56	8.19	0	6	8	2.51	2.76
6	6	5	3.26	3.49	2	6	8	6.26	6.07
3	7	5	3.72	3.69	4	6	8	3.02	3.14
5	7	5	6.81	7.49	3	7	8	3.95	3.99
0	8	5	15.31	14.36	0	8	8	3.35	3.22
1	9	5	7.70	7.22	5	1	9	11.07	10.90
3	9	5	4.70	4.61	0	2	9	6.74	6.31
5	9	5	4.03	3.92	4	2	9	4.02	4.04
0	10	5	4.04	3.75	3	3	9	5.98	6.11
2	10	5	3.22	3.17	5	3	9	5.25	5.21
2	0	6	13.74	12.51	0	4	9	2.93	2.83
6	0	6	32.00	27.72	2	6	9	5.95	6.14
8	0	6	8.26	8.15	1	7	9	2.41	2.48
1	1	6	14.08	13.18	0	0	10	3.12	2.85
5	1	6	8.71	8.51	1	1	10	4.97	4.81
7	1	6	19.61	18.86	0	2	10	13.42	11.38
9	1	6	5.52	4.99	2	2	10	2.85	3.11
0	2	6	13.30	12.20	3	3	10	5.98	6.26
2	2	6	14.79	13.85	1	5	10	3.86	3.65
4	2	6	3.50	3.95	0	2	11	3.32	2.98
6	2	6	2.43	2.85	0	4	11	2.74	2.82
8	2	6	2.40	2.38	-4	0	14	10.75	10.87
1	3	6	2.35	2.32	-6	0	14	3.05	2.83
5	3	6	2.17	2.38	-5	-1	14	2.55	2.90
0	4	6	4.61	4.64	-9	-1	14	1.97	2.29
2	4	6	7.96	7.61	-6	-2	14	4.23	4.21
4	4	6	12.05	12.12	-8	-2	14	2.24	2.90
8	4	6	3.09	3.31	-1	-1	13	1.83	1.97
1	5	6	3.28	3.26	-11	-1	13	3.01	3.10
3	5	6	6.17	6.00	-4	-2	13	9.42	8.97

h	k	l	Fo	Fc
-6	-2	13	6.59	6.15
-1	-3	13	4.54	4.70
-3	-3	13	7.74	7.91
-5	-3	13	4.28	3.87
-7	-3	13	3.73	3.77
-2	-4	13	3.60	3.42
-4	-4	13	2.55	2.62
-8	-4	13	2.99	2.89
-10	-4	13	3.10	3.40
-12	0	12	4.99	5.21
-11	-1	12	4.09	4.65
-13	-1	12	3.32	3.17
-12	-2	12	11.11	10.94
-2	-4	12	4.72	4.39
-10	-4	12	2.88	3.03
-3	-5	12	3.95	3.94
-4	-6	12	3.63	3.80
-12	-2	11	5.43	5.33
-8	-6	11	2.54	2.85
-10	-6	11	2.22	2.68
-3	-7	11	4.19	4.60
-5	-7	11	6.22	5.77
-2	-8	11	2.65	2.54
-6	-8	11	2.77	2.24
-8	-8	11	2.86	2.87
-14	0	10	4.06	3.88
-14	-2	10	1.85	1.71
-13	-3	10	2.07	2.51
-14	-4	10	3.22	3.16
-9	-7	10	3.73	3.67
-4	-8	10	2.27	2.65
-8	-8	10	2.05	2.35
-10	-8	10	2.05	2.38
-7	-9	10	2.20	1.94
-12	-6	9	4.01	3.79
-5	-9	9	2.22	2.22
-4	-10	9	4.92	4.16
-6	-10	9	5.69	4.86
-16	0	8	3.74	3.29
-15	-1	8	2.97	2.63
-15	-3	8	4.93	4.78
-13	-5	8	2.96	2.95
-14	-6	8	1.81	1.54
-13	-7	8	2.04	2.12
-12	-8	8	2.46	2.50
-6	-10	8	2.50	3.06
-8	-10	8	2.28	2.70
-16	-2	7	2.93	2.32
-14	-4	7	3.81	4.01
-13	-7	7	4.08	3.77
-12	-8	7	3.99	3.43
-9	-9	7	4.91	4.43
-11	-9	7	7.48	7.18
-6	-10	7	3.11	3.15
-5	-11	7	6.06	5.70
-7	-11	7	3.29	3.24
-15	-1	6	2.91	2.89
-16	-2	6	5.82	5.34
-15	-3	6	4.74	4.23
-15	-5	6	2.32	2.19
-13	-7	6	2.09	2.04
-12	-8	6	10.31	9.72
-11	-9	6	5.23	4.96
-10	-10	6	4.58	4.82
-4	-12	6	3.90	3.42
15	3	-5	2.67	2.53
15	5	-5	4.12	3.42
-14	-6	5	2.42	2.12
-12	-8	5	2.91	2.91
16	0	-4	2.29	1.81
15	3	-4	2.46	2.23
14	4	-4	4.90	4.32
15	5	-4	6.26	5.73
-11	-9	4	4.53	4.03
-10	-10	4	2.40	2.28
-2	-12	4	4.43	3.87
15	1	-3	2.59	2.87
13	7	-3	2.49	2.48
11	9	-3	3.53	3.24
10	10	-3	1.91	1.83
-7	-11	3	6.18	6.08
-2	-12	3	5.49	5.19
-6	-12	3	2.02	2.02
-1	-13	3	9.92	9.07
14	4	-2	5.20	4.19
14	6	-2	6.12	4.92
12	8	-2	2.39	2.36
6	12	-2	4.95	4.42
-1	-13	2	5.26	4.93
14	4	-1	2.93	2.47
13	7	-1	3.81	3.45
-2	-12	1	5.20	4.78
6	12	-1	4.71	4.74
-1	-13	1	3.20	3.21
14	0	0	2.11	1.74
14	2	0	4.69	4.34
13	5	0	3.92	3.17
11	9	0	3.63	3.25
2	12	0	3.12	2.88
6	12	0	4.94	5.12
3	13	0	1.95	2.05
14	2	1	2.41	2.17
13	3	1	2.56	2.19
13	5	1	2.05	1.91
12	6	1	2.86	2.68
10	8	1	3.14	2.74
8	10	1	2.86	2.96
4	12	1	4.92	5.03
1	13	1	4.77	4.78
13	1	2	4.48	4.17
13	3	2	6.06	6.07
11	7	2	3.63	3.46
10	8	2	3.29	3.45
9	9	2	7.50	7.15
0	12	2	8.36	7.84
13	1	3	5.41	5.07

h	k	l	Fo	Fc	h	k	l	Fo	Fc
12	4	3	3.23	3.43	4	4	10	5.84	6.02
10	6	3	2.73	2.64	3	5	10	2.88	2.51
10	8	3	2.22	1.92	0	8	10	3.20	3.01
9	9	3	4.86	5.19	4	2	11	5.32	5.45
6	10	3	2.78	3.23	3	3	11	6.43	6.58
0	12	3	5.15	4.76	2	4	11	4.01	4.47
11	1	4	2.17	2.27	0	0	12	2.85	2.74
12	2	4	2.53	2.58	1	1	12	3.91	3.75
11	3	4	4.60	4.68	2	2	12	2.56	2.57
11	5	4	3.71	4.15	1	3	12	4.25	4.41
9	7	4	3.93	3.39	0	4	12	2.34	2.55
8	8	4	6.58	6.25	-7	-7	8	3.90	4.13
7	9	4	9.09	9.16	-9	-7	8	4.33	4.85
6	10	4	3.15	3.24	-13	-1	5	4.27	5.22
5	11	4	1.81	1.88	-2	-4	4	24.57	21.64
2	12	4	2.56	2.31	-9	-7	4	16.30	14.74
7	9	5	2.50	2.50	-7	-1	3	28.41	24.30
6	10	5	5.26	5.72	10	6	-3	21.55	19.57
3	11	5	2.07	2.17	6	0	-2	60.59	44.24
0	12	5	2.46	2.14	7	1	0	23.95	17.97
9	3	6	4.31	4.30	11	1	1	7.24	7.45
10	4	6	2.97	2.78	0	0	6	2.19	1.87
9	5	6	9.97	9.27	-11	-3	13	3.98	4.28
7	7	6	3.20	3.10	-1	-1	1	46.54	48.09
6	8	6	3.43	3.74	-2	-2	2	67.27	67.06
9	3	7	2.42	2.46	-1	-1	2	42.78	37.02
6	6	7	2.19	2.70	-2	0	2	30.20	31.99
8	0	8	5.28	5.33	-2	-2	3	59.85	56.99
7	1	8	10.82	11.02	-1	-1	3	90.49	84.73
7	3	8	1.97	2.36	6	0	0	28.77	35.39
3	9	8	2.11	1.81	2	0	0	58.06	54.91
0	10	8	6.15	5.71	0	2	1	33.80	32.26
7	1	9	3.40	3.25	5	1	1	21.14	26.01
1	9	9	4.95	4.43	1	1	2	33.37	41.58
6	0	10	3.71	4.13	0	2	2	94.63	96.27
5	1	10	2.76	2.85	0	2	3	40.36	47.52
5	3	10	6.82	7.16	0	4	0	18.15	21.15

APPENDIX II.

STRUCTURE FACTOR TABLES FOR

OPTICALLY ACTIVE 1,1'-DIMETHYL-4,4'-BINAPHTHYL

h	k	l	Fo	Fc	h	k	l	Fo	Fc
1	1	0	42.94	80.47	0	5	2	6.74	12.68
0	2	0	17.65	29.09	1	5	2	8.06	9.35
1	2	0	37.15	65.46	2	5	2	9.42	4.60
2	2	0	6.52	10.89	3	5	2	5.94	11.43
1	3	0	15.11	24.18	4	5	2	4.76	1.45
2	3	0	8.69	14.11	5	5	2	4.48	8.80
3	3	0	29.68	50.56	1	6	2	5.22	8.68
0	4	0	33.08	57.09	2	6	2	19.23	23.95
1	4	0	6.26	8.99	3	6	2	8.33	4.41
2	4	0	13.99	23.20	4	6	2	5.95	7.32
3	4	0	4.13	7.56	5	6	2	6.91	10.17
4	4	0	3.65	5.84	1	7	2	9.92	12.96
1	5	0	6.25	8.82	2	7	2	4.87	2.32
2	5	0	10.28	16.83	3	7	2	5.09	6.44
3	5	0	3.99	6.31	1	8	2	5.20	7.44
4	5	0	2.79	3.03	0	2	3	77.60	85.59
0	6	0	3.66	5.72	1	2	3	31.26	46.43
1	6	0	9.21	15.66	0	3	3	2.61	4.06
2	6	0	9.39	16.37	1	3	3	14.58	22.61
4	6	0	4.85	8.39	2	3	3	35.63	13.35
1	7	0	5.92	8.96	0	4	3	6.97	8.29
2	7	0	5.33	7.95	1	4	3	28.39	33.31
3	7	0	6.60	11.19	2	4	3	9.11	7.63
0	8	0	9.81	17.29	3	4	3	10.43	14.29
2	8	0	3.24	5.17	0	5	3	5.06	6.23
0	1	1	11.64	11.51	1	5	3	6.96	4.92
0	2	1	3.58	2.58	2	5	3	18.97	18.32
1	2	1	17.06	27.40	3	5	3	4.12	5.47
1	3	1	25.87	40.22	4	5	3	3.77	1.34
2	3	1	12.49	13.37	0	6	3	6.26	6.32
0	4	1	5.84	6.34	1	6	3	4.75	1.55
1	4	1	26.80	4.88	2	6	3	3.52	2.54
2	4	1	19.06	15.85	3	6	3	13.02	4.67
3	4	1	9.02	7.71	4	6	3	6.24	11.97
1	5	1	15.57	2.19	5	6	3	3.88	6.62
2	5	1	16.45	10.77	0	7	3	7.06	7.99
3	5	1	10.74	10.75	1	7	3	10.50	17.97
1	6	1	12.02	17.95	2	7	3	8.81	14.38
2	6	1	8.03	13.11	4	7	3	3.63	2.45
3	6	1	13.35	21.87	1	8	3	8.29	12.12
4	6	1	4.35	4.17	0	0	4	5.37	12.79
5	6	1	3.04	4.41	1	1	4	56.02	97.28
0	7	1	5.16	5.82	0	2	4	23.60	38.38
1	7	1	4.22	5.08	1	2	4	37.81	52.83
2	7	1	11.34	15.69	2	2	4	14.76	23.38
3	7	1	5.32	8.88	1	3	4	23.04	11.24
0	8	1	7.71	8.86	2	3	4	13.64	17.61
1	8	1	2.96	4.73	3	3	4	15.76	26.98
1	2	2	4.51	3.10	0	4	4	21.53	33.39
2	2	2	19.86	35.26	1	4	4	18.43	29.02
0	3	2	2.94	5.09	2	4	4	6.12	0.71
1	3	2	11.29	15.31	3	4	4	2.90	5.60
2	3	2	23.05	29.50	4	4	4	8.25	12.54
3	3	2	20.53	34.22	1	5	4	8.36	3.11
1	4	2	7.47	4.24	2	5	4	2.41	1.33
2	4	2	38.43	34.42	3	5	4	13.49	12.41
3	4	2	7.18	10.74	4	5	4	5.00	4.25
4	4	2	2.58	4.82	5	5	4	4.73	8.24

h	k	l	Fo	Fc	h	k	l	Fo	Fc
0	6	4	3.27	5.12	1	7	6	7.75	8.16
1	6	4	6.40	6.81	2	7	6	2.88	1.09
2	6	4	11.17	12.29	0	1	7	5.14	8.63
3	6	4	4.42	3.27	0	2	7	19.98	25.15
4	6	4	6.50	10.85	1	2	7	45.89	40.75
5	6	4	4.01	5.39	2	2	7	45.75	0.01
1	7	4	15.66	0.18	0	3	7	21.59	25.26
2	7	4	3.61	4.26	1	3	7	26.80	39.76
4	7	4	3.51	2.04	2	3	7	11.26	19.84
1	8	4	3.49	3.16	0	4	7	10.30	11.62
0	2	5	56.76	65.52	1	4	7	24.97	10.19
1	2	5	66.29	96.92	2	4	7	6.15	3.78
0	3	5	9.36	13.37	3	4	7	3.88	3.58
1	3	5	26.63	35.84	0	5	7	3.93	3.25
2	3	5	24.95	3.37	1	5	7	4.75	4.37
0	4	5	8.79	11.74	2	5	7	6.55	10.38
1	4	5	5.44	5.70	3	5	7	6.53	6.81
2	4	5	10.62	7.00	4	5	7	18.99	16.41
3	4	5	2.50	5.10	0	6	7	10.25	10.86
0	5	5	4.41	6.49	1	6	7	11.02	17.82
1	5	5	2.94	2.79	2	6	7	2.88	3.90
2	5	5	3.45	6.26	3	6	7	3.77	6.14
3	5	5	6.53	9.29	4	6	7	6.42	10.65
4	5	5	8.85	14.61	0	7	7	23.05	25.67
0	6	5	2.10	1.84	2	7	7	3.25	4.25
1	6	5	8.08	10.76	3	7	7	5.68	7.47
2	6	5	5.23	0.41	0	0	8	31.97	38.98
3	6	5	5.75	0.51	1	1	8	63.25	104.86
5	6	5	10.62	9.97	0	2	8	18.98	31.91
0	7	5	3.46	2.62	1	2	8	9.07	10.51
1	7	5	14.62	23.98	2	2	8	17.22	21.50
2	7	5	3.45	2.31	1	3	8	39.21	59.37
4	7	5	3.46	5.94	2	3	8	32.82	36.02
0	8	5	9.82	11.42	1	4	8	10.81	9.89
1	8	5	3.60	3.45	2	4	8	11.31	14.30
0	1	6	34.45	54.62	4	4	8	5.10	5.98
1	1	6	2.30	1.85	1	5	8	8.03	11.92
1	2	6	16.49	25.26	2	5	8	3.74	4.79
2	2	6	6.26	10.75	3	5	8	7.50	12.76
0	3	6	51.02	79.98	4	5	8	8.69	13.17
1	3	6	37.78	35.02	5	5	8	2.67	3.18
2	3	6	19.81	18.52	1	6	8	4.64	7.49
3	3	6	6.30	9.54	2	6	8	7.30	9.13
1	4	6	11.28	8.53	3	6	8	3.91	5.68
2	4	6	6.13	5.61	4	6	8	10.51	15.91
3	4	6	3.70	2.10	1	7	8	7.60	11.56
0	5	6	9.92	14.64	2	7	8	3.06	2.36
1	5	6	6.42	11.29	3	7	8	3.52	0.63
2	5	6	12.80	16.57	0	1	9	11.11	7.28
3	5	6	6.34	9.77	0	2	9	8.91	12.82
4	5	6	8.15	14.66	1	2	9	33.28	16.94
5	5	6	12.06	21.23	1	3	9	21.44	28.80
1	6	6	8.08	5.00	2	3	9	16.77	13.39
2	6	6	8.37	3.71	0	4	9	17.59	18.63
3	6	6	12.15	17.49	1	4	9	5.09	8.26
4	6	6	4.98	0.35	2	4	9	7.48	9.48
5	6	6	6.85	2.84	3	4	9	4.87	1.48
0	7	6	9.57	16.41	0	5	9	8.72	11.83

h	k	l	Fo	Fc	h	k	l	Fo	Fc
1	5	9	5.66	9.45	2	2	12	7.98	3.22
2	5	9	9.89	17.53	1	3	12	7.27	5.88
3	5	9	10.00	12.99	2	3	12	22.81	35.99
4	5	9	8.20	2.96	3	3	12	3.49	4.80
0	6	9	9.48	10.82	0	4	12	4.13	14.46
1	6	9	10.65	16.17	1	4	12	11.14	15.25
2	6	9	4.34	2.03	2	4	12	7.34	4.19
3	6	9	3.77	5.38	3	4	12	12.04	12.59
4	6	9	5.80	1.60	4	4	12	11.57	15.99
0	7	9	14.18	16.70	1	5	12	8.13	9.22
1	7	9	3.42	1.69	2	5	12	8.41	12.81
2	7	9	4.43	5.85	3	5	12	9.27	1.03
0	1	10	19.54	27.54	4	5	12	4.34	4.80
1	1	10	10.06	19.70	5	5	12	3.77	6.56
1	2	10	14.58	13.16	0	6	12	2.68	1.77
2	2	10	34.65	59.72	1	6	12	6.04	2.36
0	3	10	4.12	11.46	2	6	12	9.21	3.18
1	3	10	29.03	31.56	3	6	12	3.87	6.82
2	3	10	9.93	11.76	1	7	12	7.40	0.05
3	3	10	2.35	0.29	0	1	13	17.69	24.57
1	4	10	22.52	19.09	0	2	13	18.53	20.77
2	4	10	7.21	1.61	1	2	13	7.95	4.60
3	4	10	4.68	7.09	1	3	13	9.37	10.40
4	4	10	4.24	8.54	2	3	13	16.79	12.98
1	5	10	6.03	9.75	0	4	13	12.85	12.95
2	5	10	10.93	8.96	1	4	13	3.76	7.11
3	5	10	10.02	9.33	2	4	13	19.94	22.42
5	5	10	13.26	22.76	3	4	13	7.52	5.12
1	6	10	6.87	9.27	0	5	13	6.05	8.97
2	6	10	4.48	6.81	1	5	13	6.80	1.41
3	6	10	7.28	8.58	2	5	13	11.68	2.17
4	6	10	5.47	7.04	3	5	13	3.61	0.97
1	7	10	3.57	4.73	2	6	13	3.75	4.16
2	7	10	5.13	4.37	3	6	13	2.36	2.98
0	1	11	27.52	31.71	0	1	14	18.38	22.31
0	2	11	12.67	15.68	1	1	14	7.79	10.47
1	2	11	17.69	6.34	1	2	14	6.20	5.50
0	3	11	20.53	21.00	2	2	14	7.36	12.29
1	3	11	18.68	14.48	0	3	14	2.36	6.46
2	3	11	4.40	4.52	1	3	14	8.45	8.40
0	4	11	3.43	3.65	2	3	14	7.62	13.96
1	4	11	23.70	34.70	3	3	14	3.60	2.38
2	4	11	10.66	1.71	1	4	14	5.35	8.00
3	4	11	9.60	4.41	2	4	14	19.31	12.20
1	5	11	7.44	11.46	3	4	14	2.45	0.80
2	5	11	4.32	0.95	0	5	14	3.25	3.55
3	5	11	9.56	2.57	1	5	14	4.18	2.35
4	5	11	5.43	8.71	2	5	14	15.35	3.88
0	6	11	3.44	3.85	3	5	14	6.51	11.29
1	6	11	4.72	4.35	2	6	14	4.54	5.17
2	6	11	2.88	2.79	0	1	15	6.22	5.15
3	6	11	6.23	7.87	0	2	15	3.06	3.46
4	6	11	3.79	2.72	0	3	15	5.78	3.95
1	7	11	5.01	2.38	1	3	15	6.23	7.95
2	7	11	2.74	4.00	0	4	15	10.56	12.31
1	1	12	13.47	22.41	1	4	15	3.70	4.42
0	2	12	12.84	23.77	2	4	15	21.65	3.49
1	2	12	3.90	3.52	3	4	15	5.53	0.74

h	k	l	Fo	Fc	h	k	l	Fo	Fc
0	5	15	8.57	10.03	2	3	21	5.09	3.47
2	5	15	5.33	7.03	1	1	22	3.89	4.90
3	5	15	4.58	3.29	1	2	22	4.00	9.48
1	6	15	2.82	0.48	2	2	22	3.50	5.90
2	6	15	3.77	4.51	6	6	0	11.56	19.47
0	0	16	60.59	80.80	5	7	0	2.20	3.26
1	1	16	5.99	9.32	6	7	0	5.39	8.73
2	2	16	3.68	15.63	1	9	0	3.17	3.64
1	3	16	2.36	3.52	2	9	0	4.07	6.46
2	3	16	4.49	1.12	5	7	1	2.83	3.02
3	3	16	5.99	9.31	6	7	1	3.74	2.99
0	4	16	5.45	13.56	3	8	1	4.40	4.52
1	4	16	16.42	1.91	4	8	1	2.26	2.00
2	4	16	9.45	5.35	0	9	1	2.50	2.59
3	4	16	7.36	5.36	6	6	2	7.02	11.71
1	5	16	6.87	2.92	5	7	2	2.62	0.88
2	5	16	6.67	0.24	6	7	2	4.50	6.14
3	5	16	7.63	4.72	1	9	2	4.17	7.08
0	1	17	4.09	6.53	5	7	3	2.94	3.57
1	2	17	2.70	0.78	6	7	3	3.57	5.64
0	3	17	6.94	11.21	3	8	3	2.63	3.85
1	3	17	6.50	2.97	4	8	3	2.95	3.48
2	3	17	6.32	6.51	2	9	3	2.69	3.14
0	4	17	10.16	13.49	5	7	4	3.28	1.52
1	4	17	7.41	9.99	2	8	4	4.15	5.17
2	4	17	6.86	2.42	2	9	4	2.94	2.69
3	4	17	3.06	2.07	2	8	5	3.55	3.53
0	5	17	3.59	3.96	3	8	5	2.38	1.49
2	5	17	4.90	5.50	0	9	5	3.36	4.40
0	1	18	4.36	2.56	6	6	6	2.14	2.43
1	1	18	11.62	17.86	1	8	6	2.53	1.71
1	2	18	3.66	9.94	2	8	6	3.69	2.55
1	3	18	8.84	11.58	3	8	6	2.49	2.73
2	3	18	3.92	2.91	4	8	6	2.50	3.29
3	3	18	10.18	19.33	4	7	7	3.08	4.72
1	4	18	7.44	1.45	0	8	7	5.86	6.47
3	4	18	3.70	6.87	1	8	7	3.20	0.79
0	5	18	2.60	5.48	2	8	7	2.38	3.67
1	5	18	4.76	2.37	5	6	8	3.76	4.87
0	1	19	2.17	5.25	6	6	8	2.27	5.35
0	3	19	2.44	1.96	5	7	8	2.34	0.71
1	3	19	10.11	18.54	0	8	8	10.75	17.39
2	3	19	13.33	8.14	1	8	8	4.32	7.13
0	4	19	4.05	4.54	3	8	8	3.11	0.58
1	4	19	4.45	3.04	0	8	9	3.21	3.91
2	4	19	4.79	6.02	1	8	9	6.65	1.96
0	0	20	6.00	0.22	2	8	9	2.48	4.30
0	2	20	2.88	4.27	3	8	9	2.66	2.81
1	2	20	5.31	4.91	5	6	10	4.45	2.40
1	3	20	3.76	5.70	6	6	10	2.13	3.24
2	3	20	9.66	17.26	4	7	10	2.12	1.18
3	3	20	4.24	5.91	1	8	10	5.96	3.65
1	4	20	8.91	5.95	2	8	10	5.13	5.91
0	1	21	6.69	8.20	3	8	10	2.88	3.83
0	2	21	6.33	7.09	5	6	11	5.04	2.51
1	2	21	5.09	9.08	4	7	11	3.54	0.98
0	3	21	6.97	10.03	0	8	11	5.06	5.74
1	3	21	6.77	8.07	1	8	11	4.74	5.27

h	k	l	Fo	Fc	h	k	l	Fo	Fc
2	8	11	3.90	4.90	0	2	25	4.76	5.90
4	6	12	4.29	6.12	0	3	25	5.10	7.63
2	7	12	3.88	5.93	1	3	25	5.90	1.19
3	7	12	2.96	2.31	7	7	0	3.32	6.19
1	8	12	4.41	6.59	0	10	0	4.92	8.93
2	8	12	3.93	0.86	5	8	1	2.40	0.82
4	6	13	2.83	3.91	4	9	1	2.56	3.01
0	7	13	4.00	4.49	7	7	2	4.04	6.88
1	7	13	4.36	6.83	6	8	2	2.29	1.90
3	7	13	4.24	4.89	2	10	2	2.44	0.90
4	7	13	2.86	0.75	5	8	3	2.42	0.76
1	8	13	3.27	5.07	3	9	3	2.85	4.95
5	6	14	4.56	6.79	4	9	3	2.22	2.84
0	7	14	2.22	1.21	6	8	4	2.57	0.20
1	7	14	2.97	3.14	3	9	5	2.45	2.23
2	7	14	3.09	2.56	1	10	5	2.43	0.24
3	7	14	2.40	0.65	7	7	6	2.24	2.68
4	5	15	2.31	2.04	4	9	6	2.90	3.34
3	6	15	4.20	7.61	6	7	8	2.47	3.17
4	6	15	3.68	5.33	3	9	8	2.62	1.27
2	7	15	4.66	6.58	4	9	8	2.81	0.54
2	6	16	3.24	1.02	0	9	11	3.26	4.48
4	6	16	3.28	0.55	2	9	11	2.35	0.63
1	6	17	2.80	3.49	3	8	12	2.93	3.06
2	6	17	4.29	8.69	1	9	12	3.23	3.38
2	5	18	8.87	0.53	2	8	13	3.24	1.73
3	5	18	6.03	8.58	4	8	13	3.05	3.91
4	5	18	2.80	4.08	1	8	14	4.12	4.74
1	6	18	2.89	1.38	3	8	14	2.79	4.20
2	6	18	3.81	7.51	5	6	15	2.34	1.88
3	6	18	4.45	0.53	4	7	15	2.83	1.65
2	5	19	2.77	0.07	5	7	15	2.70	4.30
2	6	19	2.54	2.46	0	8	15	2.40	0.46
2	4	20	3.25	5.96	2	8	15	2.82	4.39
3	4	20	3.11	4.69	6	6	16	2.93	4.03
1	5	20	3.58	1.49	3	7	16	2.46	3.30
2	5	20	4.76	2.57	4	7	16	3.00	1.72
3	5	20	3.76	7.10	0	8	16	2.72	2.79
0	4	21	2.66	3.66	3	8	16	4.06	1.69
1	4	21	6.52	11.05	3	7	17	2.65	3.00
2	4	21	4.85	5.18	0	8	17	3.42	3.00
3	4	21	3.24	2.04	2	8	17	2.13	1.59
1	5	21	3.10	2.77	5	5	18	2.37	1.71
2	5	21	3.68	4.91	5	6	18	2.27	1.63
0	3	22	7.95	9.69	2	7	18	2.28	2.96
2	3	22	5.17	5.79	3	6	19	3.08	2.31
1	4	22	5.28	9.06	1	7	19	3.10	4.05
2	4	22	5.95	0.67	2	7	19	3.42	3.98
3	4	22	2.25	0.37	5	5	20	2.16	2.85
0	2	23	2.92	1.05	4	6	20	2.13	1.58
0	3	23	5.62	4.02	4	5	21	2.52	0.99
1	3	23	6.81	12.09	0	7	21	2.29	0.98
2	3	23	3.58	3.30	1	7	21	2.74	4.05
0	4	23	6.91	9.17	3	5	22	2.39	0.47
0	0	24	3.97	1.54	4	5	22	2.62	4.94
1	2	24	3.44	4.39	1	6	22	2.20	3.11
1	3	24	3.32	0.54	0	5	23	2.50	0.97
2	3	24	4.24	1.62	3	5	23	2.52	1.83

h	k	l	Fo	Fc
1	4	24	4.53	3.05
2	4	24	3.36	1.15
3	4	24	2.22	0.16
2	5	24	2.96	2.77
2	3	25	3.18	0.16
0	4	25	2.43	2.18
0	5	25	2.30	1.62
2	2	26	5.16	8.96
1	4	26	3.67	1.25
2	4	26	2.21	0.43
0	1	27	3.26	6.16
1	2	27	2.13	0.95
0	3	27	2.43	0.92
1	3	27	3.49	0.84
1	1	28	2.07	3.59
0	2	28	2.19	2.39
1	2	28	2.13	1.77
2	2	28	1.90	3.75
5	9	0	2.19	3.57
5	9	4	1.99	0.76
2	10	5	2.02	2.72
1	10	8	2.00	0.76
6	8	9	1.69	0.16
6	7	13	1.97	3.01
2	9	14	1.99	2.96
4	8	15	2.15	2.96
3	6	17	2.70	3.73
3	8	17	2.17	2.10
6	6	18	2.53	4.41
2	8	19	2.28	2.51
5	6	20	2.48	4.06
3	7	20	2.43	3.62
5	5	22	1.85	3.64
4	6	22	2.29	2.41
0	7	22	2.35	3.19
4	5	24	1.83	0.63
0	5	26	2.62	3.67
1	5	26	2.67	3.34
1	4	27	3.58	5.12
2	3	28	2.70	3.71
0	1	30	2.82	5.07
0	1	2	82.13	138.03
1	1	2	96.26	165.71
0	1	3	83.49	94.62
1	1	3	104.62	0.01
0	1	5	127.71	144.25
1	1	5	133.33	0.01