X-RAY CRYSTALLOGRAPHIC STUDIES OF EIGHT ORGANIC COMPOUNDS

by

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ABSTRACT

Part one of this thesis contains the x-ray crystal structure analyses of six compounds related to natural product chemistry. The first three analyses were performed in order to identify two isomers, separated by chromatography, that were potential intermediates in the syntheses of stemodin and aphidicolin, and differed only in the orientation of a cyclobutyl moiety. The first eluted isomer was shown to be a β-cyclobutyl tricyclic enone ($C_{22}H_{32}O_3$, monoclinic, space group $P2_1/n$, a = 11.832(1), b = 11.423(1), c = 14.637(1) Å, β = 98.71(2)°, Z = 4, solved by direct methods and refined to R = 0.034 for 2052 observed reflections). The second eluted isomer was the α-cyclobutyl species ($C_{22}H_{32}O_3$, monoclinic, space group $P2_1/n$, a = 15.722(4), b = 7.463(2), c = 17.213(6) Å, β = 104.67(1)°, Z = 4, solved by direct methods and refined to R = 0.040 for 702 observed reflections). The third analysis was of the p-bromobenzoate derivative of the second eluted isomer, and confirmed the α-cyclobutyl structure ($C_{29}H_{37}BrO_4$, triclinic, space group $P\bar{1}$, a = 11.023(2), b = 11.877(1), c = 10.900(1) Å, α = 90.461(8), β = 111.57(1), γ = 80.51(1)°, Z = 2, solved by Patterson methods and refined to R = 0.032 for 2715 observed reflections).

The fourth structure was also a p-bromobenzoate derivative of a system involving a four-membered ring, and was undertaken

to verify the 1,4-homoenol structure of camphor-1,4-homoenol p-bromobenzoate ($C_{17}H_{19}BrO_2$, orthorhombic, space group $P2_12_12_1$, a = 6.875(1), b = 8.522(2), c = 26.658(6) Å, Z = 4, solved by both direct and Patterson methods and refined to R = 0.045 for 697 observed reflections).

The last two structures of this part proved to be crystallographically difficult. One was the previously unknown structure of raucubaine, an indole alkaloid isolated from the plant Rauwolfia salicifolia griseb. ($C_{20}H_{24}N_2O_3$, monoclinic, space group $P2_1$, a = 7.2179(3), b = 12.8169(7), c = 9.1996(2) Å, β = 93.040(3)°, Z = 2, solved by direct methods (with great difficulty) and refined to R = 0.046 for 1700 observed reflections). The other was a sugar that had remained unsolved for fourteen years ($C_{24}H_{24}Cl_2O_8$, monoclinic, space group $P2_1$, a = 5.752(3), b = 15.436(3), c = 13.698(3) Å, β = 93.74(3)°, Z = 2, solved by direct methods (with great difficulty) and refined to R = 0.042 for 898 observed reflections).

Part two contains two optically active structures as part of a project concerning spontaneous resolution in binaphthyl systems: the first being naphthidine ($C_{20}H_{16}N_2$, tetragonal, space group $P4_12_12$ or $P4_32_12$, a = 7.945(1), c = 24.264(5) Å, Z = 4, solved by direct methods and refined to R = 0.068 for 548 reflections) and the other 1,1'-binaphthyl ($C_{20}H_{14}$, tetragonal, space group $P4_12_12$ or $P4_32_12$, a = 7.164(2), c = 27.70(1) Å, Z = 4, solved by direct methods and refined to R = 0.030 for 562 observed reflections). These structures are compared to those of several related compounds.

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Something there is so appealing about a path
The way it is worn around the rocks
And through the grass.
Something in its neatness leads you on
The path is the smooth way,
The easy way along.
Off the path is up and down
Boulders and brambles little pain
Danger and frustration,
And rivers after rain.
Stay on the path, you will get somewhere
And you will never know all the places
That you miss around you as you go.
Get off the path, and you can have
More than a lifetime to spend
'Cause off the path
You don't ever have to reach an end.

'Path', by Michael Kennedy.

ACKNOWLEDGEMENT

I would very much like to thank Prof. James Trotter for all his diligent proofreading, help and guidance during the last five years. I am grateful to Drs. J.P. Kutney, T. Money, E. Piers and R.E. Pincock for supplying crystals and background information. I am indebted to my co-workers, from whom I have learned a great deal.

I would also like to thank all my friends for helping me maintain my sanity come 5.00 p.m.

DEDICATION

I would like to dedicate this thesis to my father, Gerry, for whom a major goal in life is fulfilled by this achievement. Cheers, Dad.

CHAPTER 1

INTRODUCTION

This thesis describes x-ray crystallographic investigations of the structures of eight organic compounds. It is divided into two parts: the first deals with the analyses of six compounds related to natural product chemistry and the second is concerned with two structures related to a project in spontaneous resolution of binaphthyl systems.

Each structure analysis is treated in a separate section which includes a general introduction to the compound and its preparation (it should perhaps be emphasized that no preparative work was carried out for this thesis; all crystals used were prepared by other research workers). In part one, the interest in the first four compounds lies in the nature of their four-membered ring systems. The next two compounds, both of space group $P2_1$, proved to be crystallographically difficult structures, and the main concern there lies in the methods of solution. Part two contains the work to date with respect to the spontaneous resolution project, followed by the structure analyses of two binaphthyl compounds. Comparisons of these structures with those of other binaphthyl systems and some general conclusions are presented in a final section.

The background theory of x-ray crystallography is readily available in various texts[1-5], and all nomenclature, symbols and conventions used in this thesis are consistent with those described in International Tables for X-ray Crystallography[6]. There are, however, various aspects of data collection and structure refinement that should be clarified here.

The data for all structures (except one in chapter 4) were collected on an Enraf-Nonius CAD-4 diffractometer. This machine

allows the user to select various data-collection parameters. The values chosen will appear in the experimental section of each structure, and a brief description of the more important parameters follows below.

The distance d(hkl) between sets of planes of indices hkl is related to the diffraction angle theta (the angle between the incident beam and the reflecting plane that will give rise to diffraction) by Bragg's law, $2d(hkl)\sin\theta = n\lambda$, where λ is the wavelength and n is an integer. The intensity I(hkl) of a beam of x-rays diffracted from a particular set of planes is measured by scanning across the intensity profile and measuring the x-ray count with a suitable detector. In front of the detector is an aperture which has a manually insertable slit of variable height (4mm has been found adequate for most structures) and a width which is varied during data collection to account for the widening of the reflection due to α_1-α_2 splitting in θ at higher angles. The intensity profile is scanned by moving either the crystal alone (ω-scan) or by moving the crystal and the detector (ω-θ scan). The ratio of crystal to detector movement determines the direction through which the intensity profile is scanned. This is important as reflections may streak out in a particular direction and a representative measurement of the background is desirable. The ω-scan angle must be large enough to ensure that the entire intensity profile is scanned -- this parameter depends on crystal mosaic spread and divergence of the primary beam. The ω-scan angle is also widened at higher angles during data collection, and is usually extended by 25% on each side for background measurements.

The estimated standard deviations (e.s.d.'s or σ's) of the intensities are :

$\sigma\{I(hkl)\} = (1/Lp)(S + 4B + (0.04S)^2)^{1/2}$, where S and B are the scan and background counts, (1/Lp) is the Lorentz - polarization correction factor, and 0.04 is an additional error factor included to allow for instrument instability. A reflection is normally considered observed if its intensity is greater than, say, three standard deviations ($I>3\sigma(I)$).

The speed with which a reflection is scanned may also be varied. As a compromise between speed and accuracy, the scan speed is calculated to give a desired value of $\sigma(I)/I$, normally in the range 0.01 to 0.05. In order to calculate the scan speed, a fast pre-scan is performed. The pre-scan speed is, however, chosen to be sufficiently slow such that the majority of reflections will already have the $\sigma(I)/I$ requirement satisfied. A pre-scan acceptance parameter is also chosen : if $\sigma(I)/I$ is greater than this parameter during the pre-scan, the reflection is flagged unobserved. A time limit of the order of 60 to 100 seconds is normally imposed on the final scan time.

As data collection often takes several days, a few reflections are chosen as standards and are checked at regular intervals for intensity and orientation. The reflections chosen for intensity control should be fairly strong. If their intensities vary systematically during data collection, the data are scaled accordingly. The reflections chosen for orientation control should ideally be strong and should preferably have scattering vectors that are as close to being mutually

perpendicular as possible. If during orientation control it is found that these scattering vectors differ from their calculated positions by more than a user-selected amount, reorientation occurs.

In general, the numeric values selected for data collection parameters will depend mainly on crystal size and quality. A few expressions used in the solution and refinement are defined in the following brief description.

From the collected intensities, the structure amplitudes may be derived, $|F(hkl)| = \{kI(hkl)/(Lp)\}^{1/2}$, where Lp is the geometric Lorentz - polarization correction and k is a proportionality constant. However, the sign (or phase) of these structure factors is unknown at this stage, and the direct methods procedure, which was used for most of the structures solved, begins here.

By comparing the structure to its square, Sayre[7] showed that

$$F(h,k,l) = \phi(h,k,l) \sum\sum\sum F(h',k',l') \cdot F(h-h',k-k',l-l')$$

where $\phi(h,k,l)$ is a calculable scaling term and the summations are over all h',k', and l'. A structure factor F(h,k,l) may hence be derived from the products of all the pairs of indices that add to give (h,k,l). This seems to imply that all F(h',k',l') and F(h-h',k-k',l-l') need be known before the phase of F(h,k,l) can be calculated, but in fact, should F(h,k,l) be large, the summations must tend strongly in one direction (+1 or -1, in the centrosymmetric case), and a reasonable approximation of the phase may be made by considering only the larger contributors.

One problem that arises is that the structure factors decrease considerably with an increase in θ (higher angle reflections will be weaker as reflections from different parts of the electron cloud will be increasingly out of phase), and relatively strong high-angled reflections would have structure factors that would hardly contribute to the above relation. For this reason the structure factors are normalized.

Unitary structure factors are calculated such that $U(hkl) = F(hkl)/F(000)$, where F(000) is the structure factor of the unobservable 0 0 0 reflection and corresponds to the number of electrons in the unit cell, and so $-1 \leq U \leq +1$. Karle and Hauptmann[8] introduced a normalized structure factor E such that $E^2 = U^2/U(\text{mean})^2$. There exist various numerical methods of scaling the E's, including K-curves and Wilson plots (see for example page 15). The E's have the same phase as their respective F's and U's, but their magnitudes are more representative of their relative reflection strength. Symmetry related intensity considerations are also taken into account in the determination of the E's.

Although theoretically independent of the size and content of the unit cell, the distribution of the E's does depend on the presence or absence of a centre of symmetry (see Table II, page 18). In practice, the presence of a centre of symmetry may be established by examination of the E-statistics. It is the larger E's that are expected to be the predominant contributors responsible for determining the phase of a reflection by Sayre's relationships.

A shift in unit cell origin is associated with a change of phase of reflections of parity depending on the direction of shift. The origin is therefore fixed by assigning phases to some reflections (three in general). Should these reflections have strong E's, they may be used to calculate the probable phases of other reflections using a relationship very similar in nature to that of Sayre:

$$\phi(h,k,l) = \phi(h',k',l') + \phi(h-h',k-k',l-l')$$

where $\phi(h,k,l)$ is the new phase to be calculated from the phases $\phi(h',k',l')$ and $\phi(h-h',k-k',l-l')$. This relationship is known as a Σ_2-relationship as it depends on the sum of two phases. Adding to this initial set of 'known' phases may be various phases determined from Σ_1-relationships (a special case of a Σ_2-relationship, where $\phi(2h,2k,2l) = \phi(h,k,l) + \phi(h,k,l)$).

From this starting set, the phasing may continue using Σ_2-relationships until further phasing is only made possible through the introduction of a few symbols for the unknown phases. These symbols, through their extensive use in Σ_2-relationships, will hopefully become involved in a number of equalities that will allow their evaluation. This is known as the symbolic addition procedure, and was first suggested by W.H. Zachariasen[9].

The determined phase values may be refined (before they are used for further phase determination) by the tangent formula[10];

$$\tan \phi(h,k,l) = \frac{\Sigma\, Q \sin\{\phi(h',k',l') + \phi(h-h',k-k',l-l')\}}{\Sigma\, Q \cos\{\phi(h',k',l') + \phi(h-h',k-k',l-l')\}}$$

where the sine and cosine portions are obtained from splitting the exponential form of Sayre's relation, the Q terms involve

the E magnitudes, and the summation is over all available terms. There are consistency parameters that describe the correctness of phases thus determined and enforce acceptance or rejection of these new phases.

The speed of the phasing procedure may be greatly enhanced by assigning initial phase values to the symbols, and carrying out multiple sets of phase determinations. There are computer programmmes available to perform this phasing, including TANS[11] (named after the tangent formula) and MULTAN[12] (from 'MULtiple TANgent refinement programmme).

These multiple phasings result in a number of possible sets of phases, and a Fourier synthesis using the E's as coefficients (an 'E-map') will produce electron density peaks corresponding to the positions of the larger atoms if the phases are correct.

This discussion is only intended to introduce some of the terms used in this thesis and to remove a little of the mystery often associated with direct methods for the uninitiated reader. For a more in-depth analysis of direct methods, the reader is referred to the more standard texts on the subject[13].

Patterson and Fourier methods were also successful in chapters 2 and 3 and will be briefly described therein.

The refinement was based on the minimization of the function $\Sigma(w(|Fo|-|scale\ x\ Fc|)^2)$, where Fo and Fc are the observed and calculated structure factors and w is a weighting factor. The structure amplitudes were corrected for thermal vibrations using the anisotropic temperature factors Uij in

$$f=f^0\exp\{-2\pi^2(U_{11}h^2a^{*2}+U_{22}k^2b^{*2}+U_{33}l^2c^{*2}+2U_{12}hka^*b^*+2U_{13}hla^*c^*+2U_{23}klb^*c^*)\}$$

where f^0 and f are the tabulated and corrected structure factors respectively. Isotropic thermal parameters have the form $f = f^0\exp\{-B(\sin\theta/\lambda)\}$ where $B = 8\pi^2U^2$ (U^2 is the mean-square displacement of the atom from its mean position). The scattering vectors used for the non-hydrogen atoms were obtained from reference 14, and those used for the hydrogen atoms were obtained from reference 15. Anomalous dispersion corrections, when used, were obtained from reference 16. In the temperature factor and all other tables, e.s.d.'s, if present, are given in parentheses and correspond to the least significant digit or digits.

The correctness of the final structure may be measured in terms of the agreement between the observed and the calculated structure factors. The agreement factors used here are the R and the weighted Rw in

$$R = \Sigma\{|Fo|-|\text{scale} \times Fc|\}/\Sigma|Fo|$$

and $$Rw = \{\Sigma[w(|Fo|-|\text{scale} \times Fc|)]^2/\Sigma w|Fo|^2\}^{1/2}.$$

More extensive details of data collection and refinement are discussed in the individual chapters.

Structure factor tables appear in the appendix.

PART ONE

CHAPTER 2

STEMODIN INTERMEDIATES

I. A β-CYCLOBUTYL TRICYCLIC ENONE

Introduction and preparation

Recent work by Dr. E. Piers and co-workers[17-18] directed towards the total synthesis of the tetracyclic diterpenoids aphidicolin (1) and stemodin (2) (see scheme 1) included the photochemically-induced cycloaddition of allene to the cyclic enone 3. Irradiation (low-pressure mercury lamp, 4.5 hours) of a cold solution of racemic 3 and allene in dry deoxygenated tetrahydrofuran gave a mixture of two isomeric photoadducts, presumably 4 and 5, which were separated by column chromatography on silica gel (elution with 10:5:2 cyclohexane-hexane-ethylacetate). The first eluted diastereomer (39% yield) exhibited m.p. 132-133°C, while the second (42% yield) had m.p. 134-135°C. It was thought that one of these isomers would be useful as an intermediate in the synthesis of stemodin, while the other might be useful in aphidicolin synthesis. In order to determine which adduct was which, an x-ray diffraction study was undertaken on the compound that yielded the better crystals: the first eluted isomer.

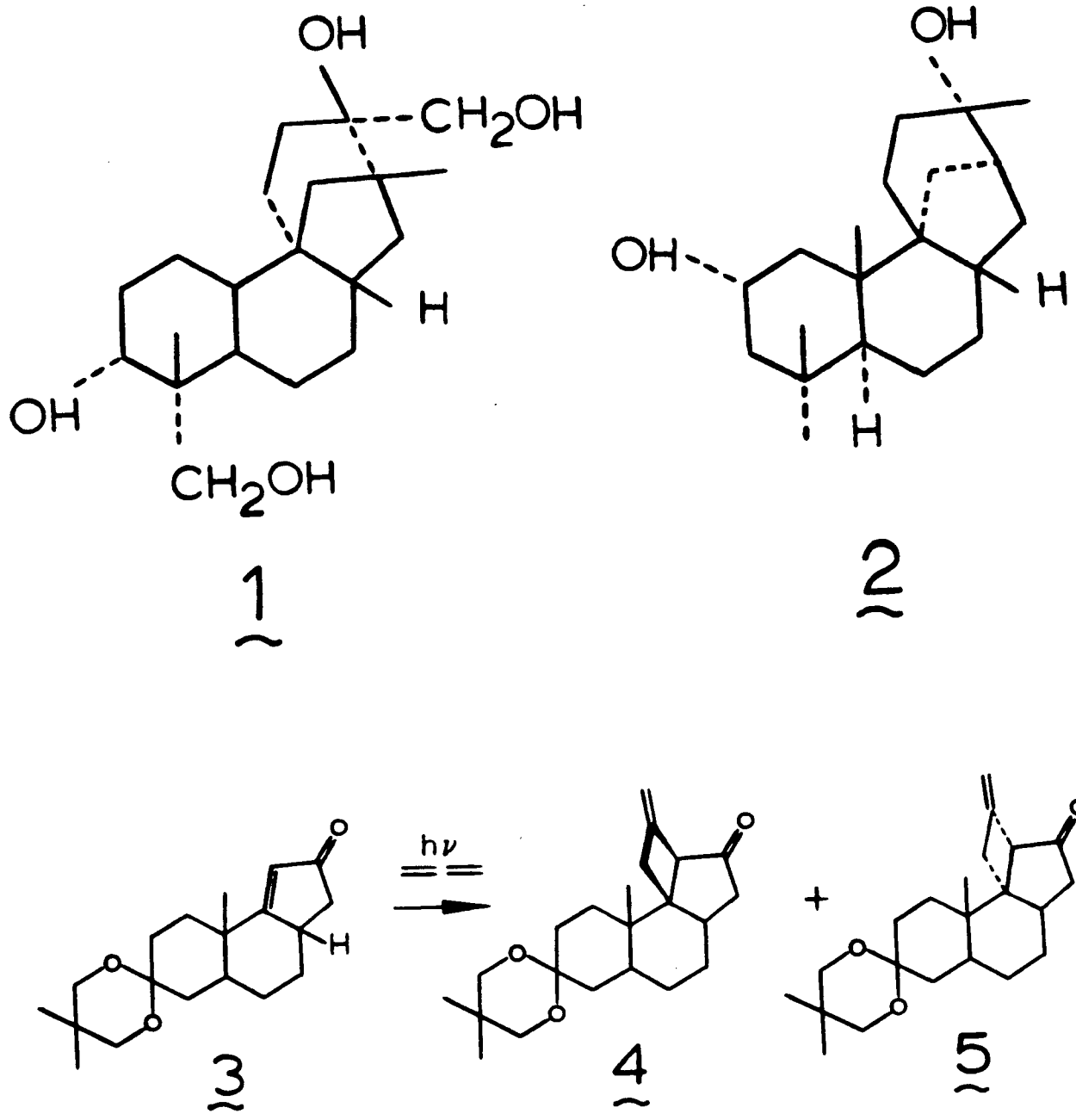


Figure 1. Scheme 1

Experimental

From preliminary photography, reflections of type 0k0 where k is odd and h0l where h+l is odd were found to be systematically absent, indicating a twofold screw axis parallel to b and a glide plane perpendicular to b of direction (a+c)/2 (i.e., [101]). The crystals are monoclinic and thus of space group $P2_1/n$. This space group is in fact equivalent to that most often found in organic crystals, $P2_1/c$, and may be converted to $P2_1/c$ by using [101] as the axial direction instead of [001].

The intensity data were collected using graphite-monochromatized MoKα radiation (λ = 0.71073 Å) and an ω-θ scan technique with an ω-scan angle of $(1.0 + 0.35 \tan\theta)^\circ$. The vertical and horizontal aperture widths were 4 mm and (2.5 + tan θ)mm, respectively. The intensities of three standard reflections (-2 5 -1, -1 6 -2, and -1 6 -3) were checked every one hour of x-ray exposure time and showed no significant variation. The same three reflections were checked for orientation every 100 reflections, and reorientation occurred if the difference between observed and calculated scattering vectors was greater than 0.05°. During the data collection, it was noticed that this difference was often between 0.05 and 0.06°, and so the reorientation condition was relaxed to 0.07°, after which reorientation occurred far less often (thereby saving considerable time).

In the range $0 < \theta \leq 25^\circ$, 2052 of the 3432 reflections collected (59.8%) had $I/\sigma(I) \geq 3.0$ and were considered observed. The cell parameters were refined by least-squares methods with

the $\sin\theta/\lambda$ values of 25 reflections within the limits $9 < \theta < 18^{\circ}$, and are listed with other crystal data in Table I.

****

TABLE I. CRYSTAL DATA FOR MOLECULE 4

$C_{22}H_{32}O_3$	f.w. = 344.5
Monoclinic	Z = 4
	space group = $P2_1/n$
a = 11.832(1)	
b = 11.423(1)	F(000) = 536
c = 14.637(1) Å	λ = 0.71073 Å
β = 98.71(2) $^{\circ}$	Dc = 1.17 g/cc
V = 1955.5 Å^3	μ = 0.7 cm^{-1}

****

Lorentz and polarization factors were applied as usual, and the crystal was sufficiently small to render absorption effects negligible.

The structure was solved by direct methods. Figure 2 shows the minimum profile of the K-curve that was used to place the the data on an absolute scale. As the relative intensities decrease with $\sin^2\theta/\lambda^2$, they are averaged within concentric shells in reciprocal space such that this variation is small. The ratio (K) of relative to absolute intensities is calculated within each shell and plotted against $\sin^2\theta/\lambda^2$ (s) to give the K-curve. The overall scale factor may be determined from the value of K when s = 0. A Wilson plot would also yield an overall temperature factor, however it assumes Gaussian-type temperature factors, which may not always be accurate. The E values are

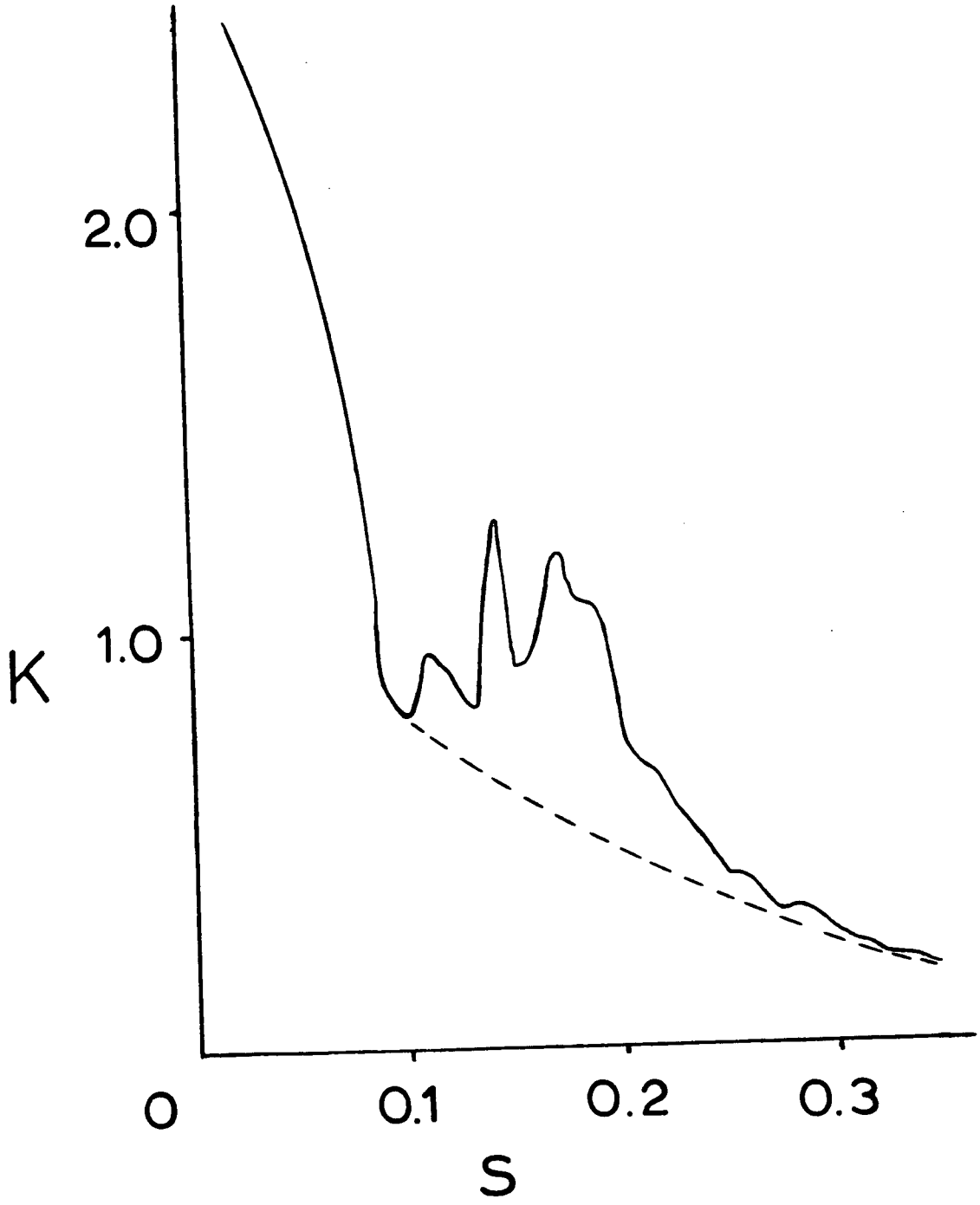


Figure 2. A K-curve showing the minimum profile

generally obtained from the minimum profile of the K-curve. This often produces $|E|^2$ values greater than 1.0, especially in the presence of a large 'hump' (as in figure 2) due to the abnormal intensity averages produced by non-random characteristics of a particular structure. E-statistics (Table II) indicate a centrosymmetric structure, agreeing with the chosen space group. The 500 highest E's were input into the MULTAN programme initially without success. MULTAN had produced two wrong sets of phased E's. However, it was noted that MULTAN had accepted the phases of three reflections determined through Σ_1-relations as their probabilities of being correct (determined from number of contributors) were greater than 0.95. A fourth reflection, 0 0 14, had a 0.949 probability of having phase π, and this phase was initially rejected, and later determined to have a phase of 0. This was unlikely to be correct, so the probability criterion of acceptance was relaxed to 0.94.

Now four phases were accepted as known from Σ_1-relations: 0 0 14 and 6 0 -10 (phase π), and 6 0 -8 and 4 0 -12 (phase 0). In order to fix the origin, the reflections 3 7 -5, 5 2 10 and 1 2 7 were assigned a phase of 0. The 2 8 -4 reflection, used as a symbol with initial phase assignments of 0 and π, completed the starting set of known phases, and MULTAN now produced two sets of phases (one for each value of the symbol), one of which was correct. The resulting E-map revealed the positions of all the non-hydrogen atoms.

After three isotropic and three anisotropic full-matrix least-squares refinement cycles, a difference map revealed the positions of 31 of the 32 hydrogens, and the remaining hydrogen

TABLE II. E-STATISTICS AFTER RENORMALIZATION

****

	OBSERVED	THEORETICAL	
		non-centro.	centro.
mean $\|E\|^2$	1.000	1.000	1.000
mean $\|E\|$	0.799	0.866	0.798
mean $\|\|E\|^2-1\|$	1.010	0.736	0.968
% reflections with:			
E > 1.5	12.38	10.54	13.40
E > 1.75	7.96	4.67	8.00
E > 2.0	4.92	1.83	4.56
E > 2.5	1.61	0.19	1.24

****

position was calculated geometrically. An additional two refinement cycles with isotropic temperature factors for the hydrogens and anisotropic temperature factors for the non-hydrogen atoms, with a polynomial weighting scheme[19] with coefficients that were updated after every cycle, resulted in the final R and Rw values of 0.034 and 0.046 respectively. After several more cycles the hydrogen atom parameters had converged. Final positional and thermal parameters are given in Tables III and IV. The hydrogen atoms are identified by three-digit numbers; the first two digits refer to the label of the atom to which they are bound, and the third refers to the number of the hydrogen on that atom. The final coefficients used in the polynomial weighting scheme were A = 0.2818, B = -0.00981, C = 0.000278, and D = 0.000024, where $w = 1/(A + B|Fo| + C|Fo|^2 + D|Fo|^3)$.

Results and discussion

The first eluted isomer has been shown to be the racemate of the β-cyclobutyl species 4, and has since been used as an intermediate in the projected synthesis of stemodin. A stereoview showing the molecular structure and the atomic labelling scheme is presented in Figure 3. Table V lists the results of some mean plane calculations of planes in the vicinity of the four-membered ring. A comparison of the mean planes to similar ones in the next structures will be presented at the end of this chapter (page 59). Bond lengths are listed in

TABLE III. ATOMIC POSITIONAL AND ISOTROPIC THERMAL PARAMETERS OF MOLECULE 4

(fractional x 10^4, H x 10^3, U x 10^3 Å^2)

Atom	x	y	z	Ueq/Uiso
O(1)	3520(2)	-3497(2)	8453(1)	103
O(2)	3527(1)	-579(1)	4657(1)	39
O(3)	2338(1)	-455(1)	3236(1)	45
C(1)	2995(2)	-3078(2)	4720(2)	45
C(2)	2851(2)	-2282(2)	3875(2)	48
C(3)	2542(2)	-1046(2)	4102(1)	37
C(4)	1500(2)	-1025(2)	4599(1)	40
C(5)	1635(2)	-1840(2)	5437(1)	36
C(6)	594(2)	-1791(2)	5940(2)	50
C(7)	803(2)	-2508(2)	6819(2)	55
C(8)	1179(2)	-3772(2)	6700(1)	47
C(9)	2139(2)	-3843(2)	6096(1)	42
C(10)	1925(2)	-3113(2)	5199(1)	38
C(11)	3270(2)	-3631(2)	6790(2)	52
C(12)	2928(2)	-3754(2)	7742(2)	64
C(13)	1726(2)	-4234(3)	7644(2)	63
C(14)	2574(2)	-5112(2)	5953(2)	62
C(15)	3716(2)	-4782(2)	6474(2)	65
C(16)	4758(3)	-5232(4)	6566(3)	101
C(17)	929(2)	-3683(3)	4554(2)	57
C(18)	3498(2)	659(2)	4782(1)	43
C(19)	3334(2)	1302(2)	3862(1)	45
C(20)	2259(2)	784(2)	3307(2)	46
C(21)	4358(2)	1101(3)	3362(2)	58
C(22)	3149(3)	2597(2)	4022(3)	74
H(011)	366(2)	-282(2)	515(1)	48(6)
H(012)	318(2)	-389(2)	455(2)	61(6)
H(021)	354(2)	-224(2)	360(1)	54(6)
H(022)	224(2)	-258(2)	340(2)	69(7)
H(041)	138(2)	-25(2)	483(1)	47(6)
H(042)	84(2)	-127(2)	416(2)	53(6)
H(051)	229(2)	-156(2)	587(1)	33(5)
H(061)	46(2)	-94(2)	607(2)	64(7)
H(062)	-8(2)	-212(2)	552(2)	62(7)
H(071)	146(2)	-210(2)	728(2)	76(8)
H(072)	13(2)	-255(2)	712(2)	70(7)
H(081)	53(2)	-423(2)	645(1)	53(6)
H(111)	374(2)	-293(2)	673(1)	54(6)
H(131)	179(2)	-515(3)	764(2)	83(8)
H(132)	134(2)	-394(2)	815(2)	73(8)
H(141)	218(2)	-573(2)	623(2)	78(8)
H(142)	257(2)	-535(2)	530(2)	75(8)

continued...

H(161)	542(4)	-478(4)	693(3)	157(18)
H(162)	473(3)	-603(4)	630(3)	122(14)
H(171)	23(2)	-384(2)	489(2)	77(8)
H(172)	67(2)	-320(2)	400(2)	83(8)
H(173)	119(3)	-438(3)	431(2)	94(10)
H(181)	289(2)	91(2)	513(1)	38(5)
H(182)	424(2)	85(2)	515(1)	51(6)
H(201)	217(2)	105(2)	267(2)	49(6)
H(202)	159(2)	102(2)	360(2)	55(6)
H(211)	429(2)	156(2)	281(2)	78(8)
H(212)	506(2)	133(2)	377(2)	78(8)
H(213)	443(2)	25(3)	321(2)	92(10)
H(221)	304(3)	305(3)	346(3)	115(12)
H(222)	246(3)	272(3)	434(2)	101(11)
H(223)	380(3)	290(3)	438(2)	106(11)

TABLE IV. ANISOTROPIC THERMAL PARAMETERS OF MOLECULE 4

(U_{ij} x 10^4 Å^2)

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1)	783(13)	1677(23)	547(11)	-209(14)	-171(10)	126(13)
O(2)	314(7)	467(8)	363(7)	-8(6)	-10(5)	30(6)
O(3)	494(8)	510(9)	324(7)	-85(6)	-21(6)	61(6)
C(1)	505(13)	393(12)	478(12)	63(10)	138(10)	-19(10)
C(2)	564(14)	478(13)	417(12)	-24(11)	159(11)	-30(10)
C(3)	350(10)	449(11)	301(9)	-12(8)	-9(8)	37(8)
C(4)	316(11)	427(12)	455(12)	9(9)	30(9)	58(10)
C(5)	297(10)	410(11)	379(10)	-1(8)	47(8)	6(9)
C(6)	406(12)	551(15)	587(14)	66(10)	169(11)	51(11)
C(7)	482(13)	681(15)	530(13)	6(11)	232(11)	27(12)
C(8)	410(12)	540(13)	460(12)	-94(10)	51(9)	70(10)
C(9)	399(11)	379(11)	468(11)	-32(9)	26(9)	28(9)
C(10)	384(10)	374(11)	372(10)	-33(8)	27(8)	-6(8)
C(11)	396(12)	562(14)	560(13)	-37(10)	-19(10)	172(11)
C(12)	551(14)	829(18)	509(14)	-34(13)	-32(12)	181(13)
C(13)	606(15)	771(19)	507(14)	-70(13)	80(12)	185(13)
C(14)	731(17)	379(13)	781(19)	36(12)	185(14)	106(13)
C(15)	544(14)	633(16)	802(17)	149(12)	169(12)	335(13)
C(16)	780(23)	1017(29)	1267(32)	344(22)	273(22)	472(26)
C(17)	624(16)	584(16)	467(13)	-177(12)	-17(12)	-45(12)
C(18)	404(12)	498(13)	368(11)	-45(10)	35(10)	-28(9)
C(19)	428(11)	471(12)	433(11)	-37(9)	45(9)	40(9)
C(20)	398(12)	548(15)	431(12)	-7(10)	16(10)	160(10)
C(21)	447(13)	794(19)	498(14)	-114(12)	68(11)	144(14)
C(22)	943(24)	493(16)	774(21)	-53(15)	63(19)	29(14)

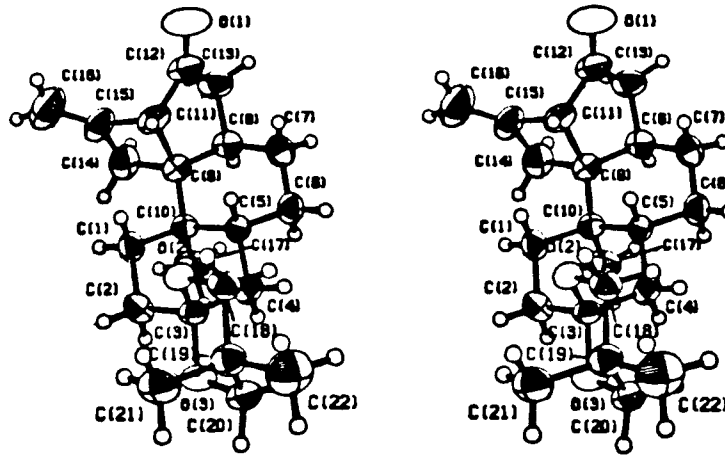


Figure 3. A stereoview of molecule 4

TABLE V. MEAN PLANES IN THE VICINITY OF THE CYCLOBUTYL RING IN MOLECULE 4

Equations of planes (lX+mY+nZ=p)

plane	l	m	n	p
1	0.3622	0.4486	-0.8170	-9.0314
2	0.5727	0.3236	-0.7532	-7.3883
3	-0.0488	0.9982	-0.0347	-4.6835
4	0.3664	-0.8221	-0.4358	-0.7156

Deviations from planes (Å)

atom	1	2	3	4
C(8)			0.051(2)*	0.000(2)*
C(9)	0.283(2)	0.000(2)*	-0.062(2)*	0.913(2)
C(11)	0.000(2)*	0.000(2)*	0.086(2)*	0.711(2)
C(12)			-0.071(3)*	0.000(3)*
C(13)			-0.545(3)	0.000(3)*
C(14)	0.000(3)*	0.000(3)*		
C(15)	0.000(3)*	0.261(3)		
C(16)	0.100(5)	0.689(5)		
O(1)			0.160(3)	-0.491(2)

*atoms included in plane calculations

Angles between normals to the planes

planes (1) and (2) : 14.5°
planes (2) and (3) : 108.7°
planes (3) and (4) : 34.6°

TABLE VI. BOND LENGTHS (Å) OF THE NON-HYDROGEN ATOMS IN MOLECULE 4

Bond	Distance	Bond	Distance
O(1)-C(12)	1.199(3)	C(8) -C(13)	1.528(3)
O(2)-C(3)	1.420(2)	C(9) -C(10)	1.544(3)
O(2)-C(18)	1.428(3)	C(9) -C(11)	1.571(3)
O(3)-C(3)	1.424(2)	C(9) -C(14)	1.563(3)
O(3)-C(20)	1.423(3)	C(10)-C(17)	1.538(3)
C(1)-C(2)	1.524(3)	C(11)-C(12)	1.515(4)
C(1)-C(10)	1.538(3)	C(11)-C(15)	1.515(4)
C(2)-C(3)	1.509(3)	C(12)-C(13)	1.511(4)
C(3)-C(4)	1.523(3)	C(14)-C(15)	1.496(4)
C(4)-C(5)	1.529(3)	C(15)-C(16)	1.324(4)
C(5)-C(6)	1.529(3)	C(18)-C(19)	1.521(3)
C(5)-C(10)	1.546(3)	C(19)-C(20)	1.521(3)
C(6)-C(7)	1.514(3)	C(19)-C(21)	1.525(3)
C(7)-C(8)	1.528(3)	C(19)-C(22)	1.518(4)
C(8)-C(9)	1.544(3)		

TABLE VII. BOND LENGTHS(Å) INVOLVING HYDROGEN ATOMS IN MOLECULE 4

Bond	Distance	Bond	Distance
C(1) -H(011)	0.98(2)	C(14)-H(142)	0.99(3)
C(1) -H(012)	0.99(2)	C(16)-H(161)	1.01(5)
C(2) -H(021)	0.96(2)	C(16)-H(162)	0.99(4)
C(2) -H(022)	0.99(3)	C(17)-H(171)	1.04(3)
C(4) -H(041)	0.96(2)	C(17)-H(172)	0.99(3)
C(4) -H(042)	0.97(2)	C(17)-H(173)	0.95(3)
C(5) -H(051)	0.98(2)	C(18)-H(181)	0.98(2)
C(6) -H(061)	1.01(2)	C(18)-H(182)	0.98(2)
C(6) -H(062)	1.00(2)	C(20)-H(201)	0.97(2)
C(7) -H(071)	1.06(3)	C(20)-H(202)	1.00(2)
C(7) -H(072)	0.97(3)	C(21)-H(211)	0.95(3)
C(8) -H(081)	0.96(2)	C(21)-H(212)	0.98(3)
C(11)-H(111)	0.98(2)	C(21)-H(213)	1.00(3)
C(13)-H(131)	1.05(3)	C(22)-H(221)	0.96(4)
C(13)-H(132)	0.99(3)	C(22)-H(222)	1.01(4)
C(14)-H(141)	0.97(3)	C(22)-H(223)	0.94(4)

Tables VI and VII and are representative of those expected for C-C, C=C, C-O, C=O and C-H bonds. Bond angles (Tables VIII and IX) are fairly close to values expected for 4-, 5-, and 6-membered rings, with some deviations due to strain in the polycyclic molecule. These angles and deviations, as well as those for the next two structures, will be examined more closely at the end of this chapter. A packing diagram is shown in Figure 4. There are no exceptionally short intermolecular distances; the crystal is held together by van der Waals forces.

TABLE VIII. BOND ANGLES(°) OF NON-HYDROGEN ATOMS IN MOLECULE 4

Bonds	Angle	Bonds	Angle
C(3) -O(2)-C(18)	114.3(1)	C(1) -C(10)-C(5)	107.7(2)
C(3) -O(3)-C(20)	114.3(2)	C(1) -C(10)-C(9)	111.1(2)
C(2) -C(1)-C(10)	112.8(2)	C(1) -C(10)-C(17)	109.7(2)
C(1) -C(2)-C(3)	112.4(2)	C(5) -C(10)-C(9)	109.3(2)
O(2) -C(3)-O(3)	110.2(1)	C(5) -C(10)-C(17)	111.2(2)
O(2) -C(3)-C(2)	105.8(2)	C(9) -C(10)-C(17)	107.8(2)
O(2) -C(3)-C(4)	112.0(2)	C(9) -C(11)-C(12)	105.1(2)
O(3) -C(3)-C(2)	105.1(2)	C(9) -C(11)-C(15)	88.3(2)
O(3) -C(3)-C(4)	112.2(2)	C(12)-C(11)-C(15)	110.8(2)
C(2) -C(3)-C(4)	111.2(2)	O(1) -C(12)-C(11)	125.0(2)
C(3) -C(4)-C(5)	112.3(2)	O(1) -C(12)-C(13)	126.1(2)
C(4) -C(5)-C(6)	111.7(2)	C(11)-C(12)-C(13)	108.9(2)
C(4) -C(5)-C(10)	113.1(2)	C(8) -C(13)-C(12)	103.0(2)
C(6) -C(5)-C(10)	111.4(2)	C(9) -C(14)-C(15)	89.3(2)
C(5) -C(6)-C(7)	110.5(2)	C(11)-C(15)-C(14)	92.6(2)
C(6) -C(7)-C(8)	115.3(2)	C(11)-C(15)-C(16)	131.9(4)
C(7) -C(8)-C(9)	111.5(2)	C(14)-C(15)-C(16)	135.2(4)
C(7) -C(8)-C(13)	108.3(2)	O(2) -C(18)-C(19)	111.6(2)
C(9) -C(8)-C(13)	104.6(2)	C(18)-C(19)-C(20)	105.6(2)
C(8) -C(9)-C(10)	114.7(2)	C(18)-C(19)-C(21)	110.3(2)
C(8) -C(9)-C(11)	104.5(2)	C(18)-C(19)-C(22)	109.6(2)
C(8) -C(9)-C(14)	114.3(2)	C(20)-C(19)-C(21)	110.3(2)
C(10)-C(9)-C(11)	118.8(2)	C(20)-C(19)-C(22)	109.5(2)
C(10)-C(9)-C(14)	113.7(2)	C(21)-C(19)-C(22)	111.4(2)
C(11)-C(9)-C(14)	88.0(2)	O(3) -C(20)-C(19)	111.5(2)

TABLE IX. BOND ANGLES(°) INVOLVING HYDROGEN ATOMS IN MOLECULE 4

Bonds	Angle	Bonds	Angle
C(2) -C(1) -H(011)	109(1)	H(131)-C(13)-H(132)	112(2)
C(2) -C(1) -H(012)	111(1)	C(9) -C(14)-H(141)	115(2)
C(10) -C(1) -H(011)	111(1)	C(9) -C(14)-H(142)	116(2)
C(10) -C(1) -H(012)	108(1)	C(15) -C(14)-H(141)	115(2)
H(011)-C(1) -H(012)	105(2)	C(15) -C(14)-H(142)	115(2)
C(1) -C(2) -H(021)	111(1)	H(141)-C(14)-H(142)	106(2)
C(1) -C(2) -H(022)	110(1)	C(15) -C(16)-H(161)	119(3)
C(3) -C(2) -H(021)	107(1)	C(15) -C(16)-H(162)	110(2)
C(3) -C(2) -H(022)	107(1)	H(161)-C(16)-H(162)	131(3)
H(021)-C(2) -H(022)	108(2)	C(10) -C(17)-H(171)	112(2)
C(3) -C(4) -H(041)	111(1)	C(10) -C(17)-H(172)	112(2)
C(3) -C(4) -H(042)	108(1)	C(10) -C(17)-H(173)	109(2)
C(5) -C(4) -H(041)	107(1)	H(171)-C(17)-H(172)	108(2)
C(5) -C(4) -H(042)	110(1)	H(171)-C(17)-H(173)	111(2)
H(041)-C(4) -H(042)	110(2)	H(172)-C(17)-H(173)	104(2)
C(4) -C(5) -H(051)	107(1)	O(2) -C(18)-H(181)	113(1)
C(6) -C(5) -H(051)	107(1)	O(2) -C(18)-H(182)	105(1)
C(10) -C(5) -H(051)	106(1)	C(19) -C(18)-H(181)	108(1)
C(5) -C(6) -H(061)	107(1)	C(19) -C(18)-H(182)	111(1)
C(5) -C(6) -H(062)	108(1)	H(181)-C(18)-H(182)	109(2)
C(7) -C(6) -H(061)	112(1)	O(3) -C(20)-H(201)	104(1)
C(7) -C(6) -H(062)	110(1)	O(3) -C(20)-H(202)	111(1)
H(061)-C(6) -H(062)	110(2)	C(19) -C(20)-H(201)	111(1)
C(6) -C(7) -H(071)	108(1)	C(19) -C(20)-H(202)	109(1)
C(6) -C(7) -H(072)	112(1)	H(201)-C(20)-H(202)	111(2)
C(8) -C(7) -H(071)	107(1)	C(19) -C(21)-H(211)	111(2)
C(8) -C(7) -H(072)	106(1)	C(19) -C(21)-H(212)	109(2)
H(071)-C(7) -H(072)	108(2)	C(19) -C(21)-H(213)	110(2)
C(7) -C(8) -H(081)	109(1)	H(211)-C(21)-H(212)	109(2)
C(9) -C(8) -H(081)	111(1)	H(211)-C(21)-H(213)	110(2)
C(13) -C(8) -H(081)	112(1)	H(212)-C(21)-H(213)	108(2)
C(9) -C(11)-H(111)	120(1)	C(19) -C(22)-H(221)	113(2)
C(12) -C(11)-H(111)	113(1)	C(19) -C(22)-H(222)	111(2)
C(15) -C(11)-H(111)	117(1)	C(19) -C(22)-H(223)	109(2)
C(8) -C(13)-H(131)	111(1)	H(221)-C(22)-H(222)	108(3)
C(8) -C(13)-H(132)	112(2)	H(221)-C(22)-H(223)	106(3)
C(12) -C(13)-H(131)	107(1)	H(222)-C(22)-H(223)	110(3)
C(12) -C(13)-H(132)	110(2)		

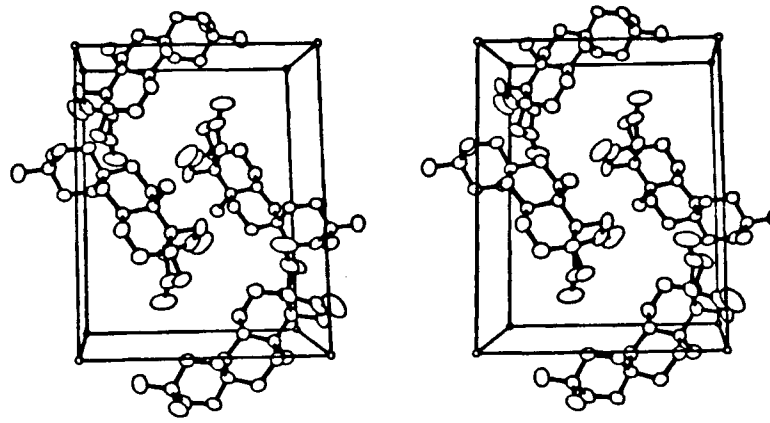


Figure 4. Stereo packing diagram for compound 4

II. AN α-CYCLOBUTYL TRICYCLIC ENONE

Introduction

The results of the last section indicated that the first eluted isomer was the cis-fused β-cyclobutyl adduct (4 in scheme 1). This allowed the assignment of the cis-fused α-cyclobutyl adduct 5 to the second eluted isomer, and the problem as initially stated was solved. However, some doubt was cast on the structure of 5 when the next step in the synthesis yielded identical products from both isomers[20].

Ozonolysis (O_3, CH_2Cl_2-MeOH, -78°C; Me_2S) (see Figure 5: scheme 2) of the adduct 4, followed by treatment of the resultant cyclobutanone with NaOMe/MeOH produced the expected keto ester 6. Surprisingly, subjection of the second photoadduct (expected to be 5) to the same sequence of reactions produced the same keto ester 6. Clearly, the expected product was the isomeric keto ester 7. This result seemed to imply that the only difference between the two adducts must lie in the orientation of the bond that was broken during the ring opening: C(11)-C(15). In other words, rather than possessing the cis-fused structure 5, it appeared that the second eluted isomer might have the more unlikely and highly strained trans-fused structure 8.

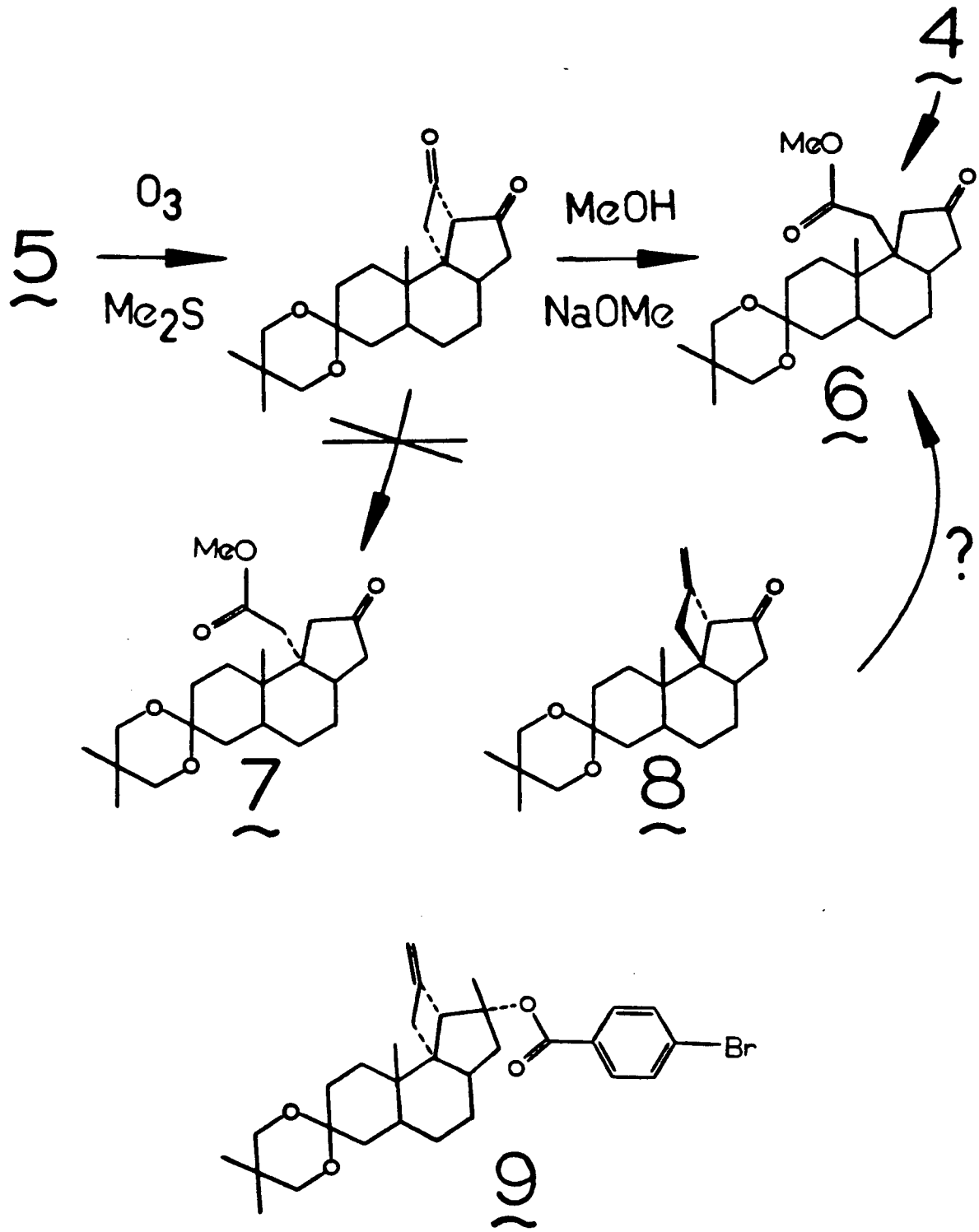


Figure 5. Scheme 2

In order to investigate this intrinsically fascinating possible trans-fused adduct and also to establish the identity of the second eluted isomer, an x-ray study was undertaken. Unfortunately, the material did not crystallize as easily as the first eluted isomer, and the quality of the crystals even after various recrystallizations was poor. Considerable effort was required in selecting a crystal which was suitable for analysis. Crystals that were apparently well-formed were shown to be multiple platelets by examination on a polarizing microscope, or if this failed, by photography. Eventually, by selecting a sufficiently thin platelet (0.05 x 0.42 x 0.35 mm^3), a single crystal was located.

Experimental

Preliminary photography showed the crystal to be monoclinic, but because of the poor crystal quality assignment of the axes and hence determination of systematic absences from the photographs was difficult. Cell parameters were determined on the CAD4 diffractometer, and systematic absences became apparent after the data collection and indicated the space group $P2_1/n$. The intensity data were collected using an ω-(1/3)θ scan technique and graphite-monochromatized MoKα radiation. The ω-scan angle was $(1.2 + 0.35 \tan \theta)^\circ$ and the aperture was (2.50 + tan θ)mm wide and 4 mm high. The intensities of three standard reflections (7 0 -5, 1 -1 4 and 5 0 -1) were measured every hour and showed no variation. The 7 0 -5, 1 1 -4, and

4 0 4 reflections were checked every 100 reflections for orientation, and reorientation occurred if the difference between observed and calculated scattering vectors was greater than 0.06°.

Of the 3836 reflections collected within the range $0 < \theta \leq 25°$, only 749 (19.5%) had $I/\sigma(I) \geq 3.0$, and so all reflections with a theta value greater than 19.5° were removed from the data set. Between 19.5 and 25°, only 47 of the 1865 (2.5%) reflections measured were observed, so by removing this data we are not making a great sacrifice in the amount of information available, but we are significantly reducing the amount of computer storage space needed to handle the problem. Of the remaining data, 702 out of 1784 (39%) reflections have $I/\sigma(I) \geq 3.0$ and were considered observed. The unit cell parameters were refined by least-squares methods with the $\sin\theta/\lambda$ values of 18 reflections in the θ range 9 to 18°. They appear together with other crystal data in Table X. It was thought that because of the anisotropic nature of the crystal shape (Figure 6), absorption effects could perhaps be significant. However, the calculated transmission factors varied from 0.971 to 0.997, indicating that absorption is negligible, and the corrections were not applied. Lorentz and polarization corrections were applied as usual.

The structure was solved by direct methods. 342 E's > 1.2 were obtained from a K-curve and, together with the 50 lowest E's, were input into the MULTAN programme. No Σ_1-relations were accepted, and the three largest E's (4 5 7, 8 2 -11 and 5 1 5) were assigned a phase of zero in order to fix the origin. Four

****

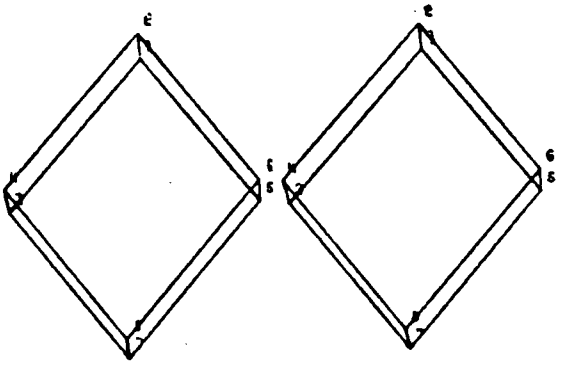
TABLE X. CRYSTAL DATA FOR MOLECULE 5

$C_{22}H_{32}O_3$	f.w. = 344.5
Monoclinic	Z = 4
	space group = $P2_1/n$
a = 15.722(4)	
b = 7.463(2)	F(000) = 536
c = 17.213(1) Å	λ = 0.71073 Å
β = 104.67(1) ^{0}	Dc = 1.17 g/cc
V = 1951.4 Å^3	μ = 0.7 cm^{-1}

****

symbols (2 0 2, 7 0 -5, 12 0 0, and 1 1 1) were assigned initial phases of 0 and π, thereby generating sixteen sets of phased E's. One set stood out as being correct, and from the E-map the positions of all 25 non-hydrogen atoms could be determined.

After three isotropic and seven anisotropic least-squares refinement cycles, all the hydrogen atoms could be located on a difference map. A single least-squares cycle including all the hydrogens fixed in their located positions and using sigma weights was run parallel with a cycle wherein the hydrogen atoms were in fixed calculated positions. The hydrogen atomic parameters were not refined as the amount of data was insufficient. The cycle using calculated hydrogen positions refined to R and Rw values of 0.046 and 0.052 respectively, whereas the cycle using located hydrogen positions refined to R and Rw values of 0.064 and 0.073, and so the hydrogen atoms were fixed in their calculated positions. Attempts to refine the



plane	vertices	distance from centre (mm)
0 0 1	1,2,3,4	0.175
0 0 -1	5,6,7,8	0.175
-2 2 1	1,2,5,6	0.225
2 -2 -1	3,4,7,8	0.200
-1 0 1	1,2,5,7	0.025
1 0 -1	2,4,6,8	0.025

crystal volume = 0.0100 mm^3

Figure 6. Stereoview of the examined crystal of 5

hydrogen thermal parameters proved unsuccessful, and each hydrogen was assigned a temperature factor 10% greater than that of the carbon to which it is bound (25% for methyl hydrogens). The structure converged after three more least-squares cycles using a Hughes' weighting scheme[21] where $(w)^{1/2} = 1.0$ for $|Fo| < F^*$ and $(w)^{1/2} = F^*/|Fo|$ for $|Fo| \geq F^*$, and $F^* = 21.0$. The weighting scheme could be slightly improved so three more cycles were run with $F^* = 20.0$. The final R values are R = 0.040 (0.169 including the unobserved reflections) and Rw = 0.053. Final positional and thermal parameters appear in Tables XI and XII.

Results and discussion

Contrary to the expectations from the ozonolysis results, the structure is that which was first assumed for the second eluted isomer: the cis-fused α-cyclobutyl species 5. A stereoview of the molecule is shown in Figure 7. Atomic labelling is the same as in the first eluted isomer. Mean planes, bond lengths and bond angles (Tables XIII, XIV, XV, XVI, and XVII) are very similar to those of the first eluted isomer, and will be compared at the end of this chapter. A packing diagram is presented in Figure 8; there are no unusually short intermolecular distances.

Confidence in the chemical evidence was sufficient to suggest that the crystal chosen for examination might have been one of an impurity - a minor third product. The validity of such a suggestion is difficult to establish. A structure

TABLE XI. ATOMIC POSITIONAL AND ISOTROPIC THERMAL PARAMETERS OF MOLECULE 5
(fractional x 10^4, H x 10^3, U x 10^3 Å^2)

Atom	x	y	z	Ueq/Uiso
O(1)	1489(4)	755(9)	2764(4)	82
O(2)	3008(3)	2381(8)	-1409(3)	53
O(3)	4525(3)	1907(8)	-1025(3)	56
C(1)	3037(5)	3008(12)	324(5)	54
C(2)	3814(5)	3193(12)	-42(5)	58
C(3)	3738(5)	1937(14)	-756(5)	53
C(4)	3586(6)	41(12)	-533(5)	55
C(5)	2813(5)	-154(11)	-153(5)	46
C(6)	2625(6)	-2089(13)	13(5)	65
C(7)	1812(6)	-2312(13)	342(5)	64
C(8)	1933(5)	-1105(12)	1072(5)	55
C(9)	2057(5)	847(12)	855(4)	44
C(10)	2909(5)	1083(12)	588(4)	46
C(11)	1874(5)	1946(12)	1578(5)	52
C(12)	1514(5)	561(12)	2062(6)	59
C(13)	1247(5)	-1075(14)	1567(5)	66
C(14)	1206(5)	1708(12)	300(5)	59
C(15)	1079(6)	2827(12)	1000(5)	59
C(16)	524(6)	4058(13)	1109(6)	81
C(17)	3708(5)	572(12)	1285(5)	66
C(18)	3112(5)	4039(14)	-1790(5)	61
C(19)	3949(5)	4064(14)	-2107(5)	60
C(20)	4691(5)	3521(13)	-1393(5)	61
C(21)	4092(6)	5927(15)	-2390(6)	85
C(22)	3880(6)	2690(15)	-2778(5)	85
H(011)	312	387	80	70
H(012)	251	345	-4	70
H(021)	437	302	36	65
H(022)	386	442	-25	65
H(041)	348	-81	-100	74
H(042)	410	-54	-15	74
H(051)	227	26	-55	65
H(061)	256	-283	-48	75
H(062)	314	-261	39	75
H(071)	129	-203	-4	81
H(072)	174	-360	49	81
H(081)	248	-156	145	72
H(111)	235	272	188	66
H(131)	65	-110	117	87
H(132)	124	-226	185	87
H(141)	132	235	-16	73
H(142)	75	85	6	73

continued...

H(161)	46	535	161	100
H(162)	-13	506	59	100
H(171)	366	-66	145	99
H(172)	425	71	115	99
H(173)	373	131	176	99
H(181)	259	444	-220	99
H(182)	317	511	-138	99
H(201)	475	453	-99	68
H(202)	525	351	-152	68
H(211)	418	677	-192	106
H(212)	459	607	-260	106
H(213)	358	639	-277	106
H(221)	372	153	-260	104
H(222)	339	301	-324	104
H(223)	439	257	-296	104

TABLE XII. ANISOTROPIC THERMAL PARAMETERS OF MOLECULE 5

(U_{ij} x 10^3 Å^2)

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1)	90(5)	111(6)	56(4)	5(4)	40(4)	0(4)
O(2)	38(3)	68(4)	51(3)	-3(3)	4(3)	0(4)
O(3)	40(3)	80(5)	54(4)	7(3)	20(3)	11(4)
C(1)	60(6)	53(7)	56(5)	-11(5)	28(5)	-13(6)
C(2)	57(6)	76(7)	43(5)	-21(5)	17(4)	-13(6)
C(3)	40(6)	76(9)	45(6)	-3(5)	16(5)	3(6)
C(4)	65(6)	53(7)	48(5)	8(6)	16(5)	-13(5)
C(5)	39(5)	59(7)	43(5)	6(5)	14(4)	0(6)
C(6)	82(7)	58(8)	61(6)	-1(6)	32(5)	-3(6)
C(7)	77(6)	58(8)	57(6)	-16(6)	19(5)	-5(6)
C(8)	61(6)	62(8)	44(5)	-11(5)	15(5)	-4(5)
C(9)	41(5)	53(7)	34(5)	3(5)	1(4)	2(5)
C(10)	39(5)	60(7)	37(5)	-7(5)	8(4)	-10(5)
C(11)	56(6)	58(6)	45(5)	-11(6)	20(5)	-5(5)
C(12)	51(6)	65(8)	63(7)	10(6)	19(5)	2(7)
C(13)	69(7)	70(8)	60(6)	-20(6)	17(5)	0(6)
C(14)	50(6)	73(7)	59(6)	-10(5)	20(5)	9(6)
C(15)	56(7)	57(7)	71(6)	13(6)	26(5)	24(7)
C(16)	73(7)	54(6)	129(9)	13(7)	48(6)	11(7)
C(17)	53(6)	92(7)	50(5)	1(6)	8(5)	-1(5)
C(18)	43(6)	79(8)	61(6)	4(5)	13(5)	9(6)
C(19)	55(6)	74(7)	52(6)	0(6)	16(5)	12(6)
C(20)	45(6)	87(8)	55(6)	-1(5)	19(5)	7(6)
C(21)	66(7)	96(8)	95(7)	-6(6)	25(6)	20(7)
C(22)	81(7)	127(9)	53(6)	2(7)	28(5)	-17(7)

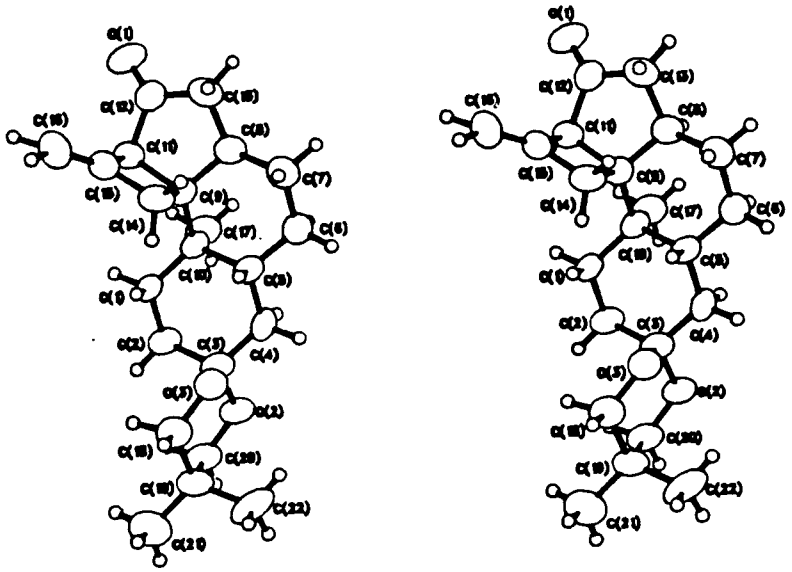


Figure 7. A stereoview of molecule 5

TABLE XIII. MEAN PLANES IN THE VICINITY OF THE CYCLOBUTYL RING IN MOLECULE 5

Equations of planes (lX+mY+nZ=p)

plane	l	m	n	p
1	0.6728	0.7079	-0.2151	1.9814
2	0.6183	0.7583	-0.2066	1.9536
3	-0.8246	0.0874	-0.5589	-3.1499
4	-0.3664	0.5520	-0.7079	-2.8512

Deviations from planes (Å)

atom	1	2	3	4
C(8)			-0.040(8)*	0.000(8)*
C(9)	0.084(8)	0.000(8)*	0.051(7)*	0.932(8)
C(11)	0.000(8)*	0.000(9)*	-0.054(8)*	0.797(8)
C(12)			0.047(9)*	0.00(1)*
C(13)			0.570(9)	0.000(9)*
C(14)	0.000(8)*	0.000(8)*		
C(15)	0.000(9)*	0.080(9)		
C(16)	-0.01(1)	0.17(1)		
O(1)			-0.309(6)	-0.596(6)

*atoms included in plane calculations

Angles between normals to the planes

planes (1) and (2) : 4.2°
planes (2) and (3) : 109.2°
planes (3) and (4) : 36.2°

TABLE XIV. BOND LENGTHS(Å) OF THE NON-HYDROGEN ATOMS IN MOLECULE 5

Bond	Distance	Bond	Distance
O(1)-C(12)	1.229(09)	C(8) -C(13)	1.534(11)
O(2)-C(3)	1.428(09)	C(9) -C(10)	1.532(10)
O(2)-C(18)	1.428(10)	C(9) -C(11)	1.577(10)
O(3)-C(3)	1.427(08)	C(9) -C(14)	1.571(10)
O(3)-C(20)	1.415(09)	C(10)-C(17)	1.549(10)
C(1)-C(2)	1.515(10)	C(11)-C(12)	1.523(10)
C(1)-C(10)	1.536(11)	C(11)-C(15)	1.534(11)
C(2)-C(3)	1.526(11)	C(12)-C(13)	1.487(12)
C(3)-C(4)	1.501(11)	C(14)-C(15)	1.521(11)
C(4)-C(5)	1.526(10)	C(15)-C(16)	1.312(11)
C(5)-C(6)	1.516(11)	C(18)-C(19)	1.546(10)
C(5)-C(10)	1.550(10)	C(19)-C(20)	1.520(11)
C(6)-C(7)	1.533(10)	C(19)-C(21)	1.510(13)
C(7)-C(8)	1.519(11)	C(19)-C(22)	1.529(12)
C(8)-C(9)	1.528(11)		

TABLE XV. BOND LENGTHS(Å) INVOLVING HYDROGEN ATOMS IN MOLECULE 5

Bond	Distance	Bond	Distance
C(1) -H(011)	1.02	C(14)-H(142)	0.98
C(1) -H(012)	0.96	C(16)-H(161)	1.32
C(2) -H(021)	0.98	C(16)-H(162)	1.40
C(2) -H(022)	0.99	C(17)-H(171)	0.97
C(4) -H(041)	1.01	C(17)-H(172)	0.94
C(4) -H(042)	1.00	C(17)-H(173)	0.98
C(5) -H(051)	1.00	C(18)-H(181)	0.98
C(6) -H(061)	0.99	C(18)-H(182)	1.05
C(6) -H(062)	0.98	C(20)-H(201)	1.02
C(7) -H(071)	0.94	C(20)-H(202)	0.96
C(7) -H(072)	1.01	C(21)-H(211)	1.00
C(8) -H(081)	1.00	C(21)-H(212)	0.94
C(11)-H(111)	0.99	C(21)-H(213)	0.97
C(13)-H(131)	1.01	C(22)-H(221)	0.97
C(13)-H(132)	1.01	C(22)-H(222)	0.99
C(14)-H(141)	0.99	C(22)-H(223)	0.93

TABLE XVI. BOND ANGLES(°) OF NON-HYDROGEN ATOMS IN MOLECULE 5

Bonds	Angle	Bonds	Angle
C(3) -O(2)-C(18)	113.6(6)	C(1) -C(10)-C(5)	107.8(6)
C(3) -O(3)-C(20)	113.9(7)	C(1) -C(10)-C(9)	112.8(7)
C(2) -C(1)-C(10)	112.9(8)	C(1) -C(10)-C(17)	108.6(7)
C(1) -C(2)-C(3)	111.8(7)	C(5) -C(10)-C(9)	105.6(6)
O(2) -C(3)-O(3)	109.7(6)	C(5) -C(10)-C(17)	112.4(6)
O(2) -C(3)-C(2)	112.0(7)	C(9) -C(10)-C(17)	109.8(6)
O(2) -C(3)-C(4)	105.8(7)	C(9) -C(11)-C(12)	104.2(7)
O(3) -C(3)-C(2)	112.0(7)	C(9) -C(11)-C(15)	89.6(6)
O(3) -C(3)-C(4)	106.0(8)	C(12)-C(11)-C(15)	106.9(7)
C(2) -C(3)-C(4)	111.0(7)	O(1) -C(12)-C(11)	124.5(9)
C(3) -C(4)-C(5)	113.3(7)	O(1) -C(12)-C(13)	125.9(8)
C(4) -C(5)-C(6)	112.7(7)	C(11)-C(12)-C(13)	109.5(7)
C(4) -C(5)-C(10)	112.0(6)	C(8) -C(13)-C(12)	101.1(7)
C(6) -C(5)-C(10)	113.3(6)	C(9) -C(14)-C(15)	90.3(6)
C(5) -C(6)-C(7)	113.2(8)	C(11)-C(15)-C(14)	91.7(6)
C(6) -C(7)-C(8)	107.5(7)	C(11)-C(15)-C(16)	132.0(9)
C(7) -C(8)-C(9)	111.1(7)	C(14)-C(15)-C(16)	136.3(9)
C(7) -C(8)-C(13)	120.7(8)	O(2) -C(18)-C(19)	112.2(8)
C(9) -C(8)-C(13)	105.9(7)	C(18)-C(19)-C(20)	105.1(7)
C(8) -C(9)-C(10)	111.0(7)	C(18)-C(19)-C(21)	109.6(9)
C(8) -C(9)-C(11)	104.0(6)	C(18)-C(19)-C(22)	110.8(8)
C(8) -C(9)-C(14)	113.4(7)	C(20)-C(19)-C(21)	111.1(8)
C(10)-C(9)-C(11)	121.7(7)	C(20)-C(19)-C(22)	109.0(8)
C(10)-C(9)-C(14)	116.4(7)	C(21)-C(19)-C(22)	111.2(8)
C(11)-C(9)-C(14)	88.3(6)	O(3) -C(20)-C(19)	113.4(7)

TABLE XVII. BOND ANGLES(°) INVOLVING HYDROGEN ATOMS IN MOLECULE 5

Bonds	Angle	Bonds	Angle
C(2) -C(1) -H(011)	109	H(131)-C(13)-H(132)	101
C(2) -C(1) -H(012)	111	C(9) -C(14)-H(141)	113
C(10) -C(1) -H(011)	110	C(9) -C(14)-H(142)	114
C(10) -C(1) -H(012)	111	C(15) -C(14)-H(141)	118
H(011)-C(1) -H(012)	103	C(15) -C(14)-H(142)	117
C(1) -C(2) -H(021)	111	H(141)-C(14)-H(142)	104
C(1) -C(2) -H(022)	112	C(15) -C(16)-H(161)	140
C(3) -C(2) -H(021)	111	C(15) -C(16)-H(162)	134
C(3) -C(2) -H(022)	106	H(161)-C(16)-H(162)	82
H(021)-C(2) -H(022)	104	C(10) -C(17)-H(171)	110
C(3) -C(4) -H(041)	113	C(10) -C(17)-H(172)	113
C(3) -C(4) -H(042)	115	C(10) -C(17)-H(173)	111
C(5) -C(4) -H(041)	107	H(171)-C(17)-H(172)	109
C(5) -C(4) -H(042)	106	H(171)-C(17)-H(173)	106
H(041)-C(4) -H(042)	101	H(172)-C(17)-H(173)	107
C(4) -C(5) -H(051)	108	O(2) -C(18)-H(181)	115
C(6) -C(5) -H(051)	104	O(2) -C(18)-H(182)	111
C(10) -C(5) -H(051)	105	C(19) -C(18)-H(181)	112
C(5) -C(6) -H(061)	111	C(19) -C(18)-H(182)	106
C(5) -C(6) -H(062)	109	H(181)-C(18)-H(182)	99
C(7) -C(6) -H(061)	110	O(3) -C(20)-H(201)	109
C(7) -C(6) -H(062)	110	O(3) -C(20)-H(202)	112
H(061)-C(6) -H(062)	104	C(19) -C(20)-H(201)	106
C(6) -C(7) -H(071)	112	C(19) -C(20)-H(202)	112
C(6) -C(7) -H(072)	111	H(201)-C(20)-H(202)	103
C(8) -C(7) -H(071)	111	C(19) -C(21)-H(211)	109
C(8) -C(7) -H(072)	110	C(19) -C(21)-H(212)	116
H(071)-C(7) -H(072)	105	C(19) -C(21)-H(213)	113
C(7) -C(8) -H(081)	104	H(211)-C(21)-H(212)	106
C(9) -C(8) -H(081)	110	H(211)-C(21)-H(213)	104
C(13) -C(8) -H(081)	105	H(212)-C(21)-H(213)	109
C(9) -C(11)-H(111)	117	C(19) -C(22)-H(221)	110
C(12) -C(11)-H(111)	117	C(19) -C(22)-H(222)	110
C(15) -C(11)-H(111)	119	C(19) -C(22)-H(223)	114
C(8) -C(13)-H(131)	107	H(221)-C(22)-H(222)	105
C(8) -C(13)-H(132)	110	H(221)-C(22)-H(223)	109
C(12) -C(13)-H(131)	119	H(222)-C(22)-H(223)	108
C(12) -C(13)-H(132)	119		

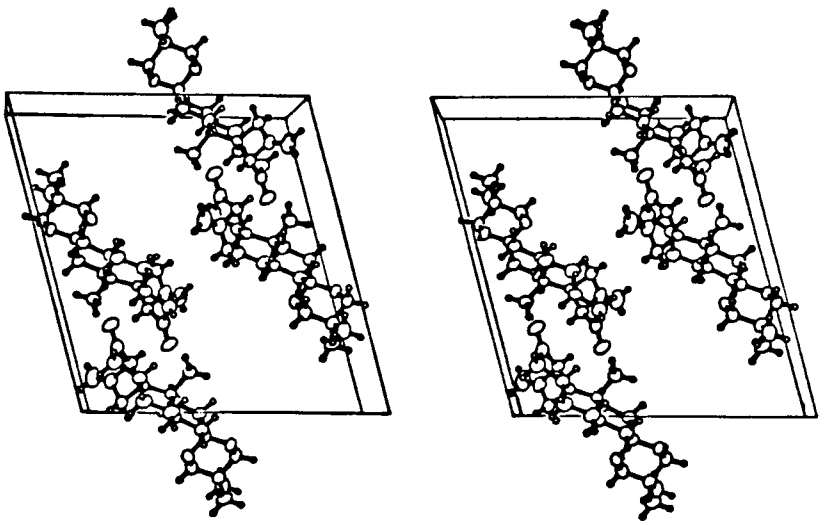


Figure 8. Stereo packing diagram for compound 5

determination on another crystal in the batch would be expensive, time consuming, and only conclusive if it produced different results. Also, the crystals were sufficiently poor that locating another single crystal of reasonable quality would have been a major task in itself.

An x-ray analysis of a derivative of the second eluted isomer that produces high quality crystals should solve this ambiguity.

III. THE P-BROMOBENZOATE DERIVATIVE OF THE SECOND ELUTED ISOMER

Introduction

The p-bromobenzoate derivative (9) (see scheme 2, page 32) of the second eluted isomer (5) was prepared by Dr. Piers and co-workers, and yielded high quality crystals. The x-ray analysis of this derivative should firmly and finally establish the second eluted isomer's structure.

Experimental

A single crystal of proportions 0.33 x 0.37 x 0.42 mm^3 was selected for study. The crystal's good quality and well-defined faces more than compensated for it's slightly large size. Preliminary photography showed that the crystal is triclinic (no systematic absences) and, as the derivative is a racemate, of the centrosymmetric space group $P\bar{1}$.

The data were collected using an ω-(4/3)θ scan technique and graphite-monochromatized MoKα radiation. The ω-scan angle was $(0.70 + 0.35 \tan \theta)^{\circ}$ and the aperture was $(2.50 + \tan \theta)$mm wide and 4 mm high. The intensities of three standard reflections (-2 -1 5, -1 -2 4 and -2 1 5) were measured every

one hour and showed a linear decay of 2% over the data collection. The data were scaled accordingly. Three reflections (-2 -1 5, -3 -6 0 and 6 2 1) were checked for orientation every 100 reflections, and reorientation occurred if the difference between observed and calculated scattering vectors was greater than 0.05°.

Of the 4549 reflections collected in the range $0 < \theta \leq 25°$, 2715 (59.7%) had $I/\sigma(I) \geq 3.0$ and were considered observed. The cell parameters were refined by least-squares methods with the $\sin\theta/\lambda$ values of 24 reflections in the θ range 15 to 19°, and appear together with other crystal data in Table XVIII. The

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TABLE XVIII. CRYSTAL DATA FOR DERIVATIVE 9

$C_{29}H_{37}BrO_4$	f.w. = 344.5
Triclinic	Z = 2
	space group = $P\bar{1}$
a = 11.832(1)	
b = 11.877(1) Å	F(000) = 536
c = 10.900(1) Å	λ = 0.71073 Å
α = 90.461(8)	Dc = 1.32 g/cc
β = 111.57(1)	V = 1306.8 Å^3
γ = 80.51(1) °	μ = 15.86 cm^{-1}

****

large value of the linear absorption coefficient μ (15.8 cm^{-1}), due mainly to the presence of bromine in the unit cell, indicates that an absorption correction is in order unless the crystal is extremely small. In order to accomplish this it is necessary to measure the distances from the indexed faces to an

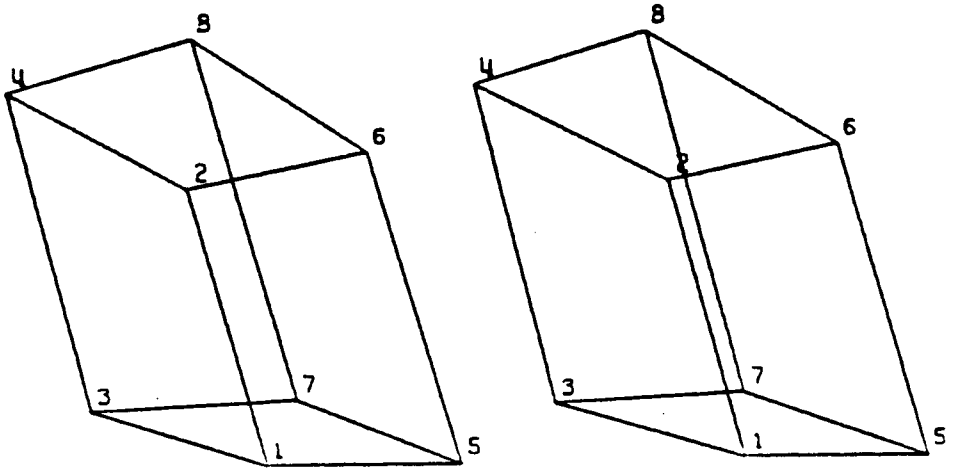
arbitrary centre of the crystal such that the shape of the crystal is defined. It then becomes possible to determine the x-ray path length through the crystal for any reflecting position, and the attenuation due to absorption may be calculated as a transmission factor. The transmission factor A is given by :

$$A = \int (1/V)\exp[-\mu(Ri+Rd)]dV$$

where V is the crystal volume, μ the linear absorption coefficient , and Ri and Rd the incident and diffracted path lengths. A is evaluated numerically by Gaussian integration. Sampling points are set up in a Gaussian grid to approximate the shape of the crystal - the number and spacing of these points are determined by experience; computing time per reflection increases sharply with the number of sampling points, but after a certain number no great increase in accuracy is obtained.

In this crystal the shape is determined by six faces (see Figure 9). 144 sampling points are used with an average spacing of 0.076 mm for a crystal volume of 0.0568 mm^3, and the resulting transmission factors varied from 0.569 for the -12 -4 4 reflection to 0.0631 for 5 -1 9. The intensities were thus corrected for absorption, as well as for Lorentz and polarization effects.

The structure was solved by Patterson techniques. A three-dimensional Patterson map is calculated from the structure amplitudes. The peaks in a Patterson map may be regarded as the heads of all the interatomic vectors grouped such that the tails are at a common origin. For n atoms in the unit cell, there will be n^2-n peaks in the Patterson plus a large peak at the origin representing the sum of all the vectors from each atom to



plane			vertices	distance from centre (mm)
0	-1	0	1,2,3,4	0.150
0	1	0	5,6,7,8	0.180
0	0	-1	1,2,5,6	0.220
0	0	1	3,4,7,8	0.150
-1	0	0	1,3,5,7	0.175
1	0	0	2,4,6,8	0.250

crystal volume = 0.0546 mm^3

Figure 9. Crystal shape of the p-bromobenzoate derivative

itself. The peak heights depend on the product of the number of electrons of each atom contributing to the vector. Thus a Br-Br peak should be larger than a C-C peak by a factor of $(35)^2/(6)^2$, or about 34. In practice these factors are slightly lower because of a smearing of the vector peak due to thermal motion. Patterson maps are thus more useful for structures containing heavy atoms.

In this structure, there are two bromines in the unit cell related by a centre of symmetry. If one bromine has fractional coordinates (x,y,z), the other will have coordinates (-x,-y,-z), and the interatomic vector is the difference between them, or (2x,2y,2z). The Patterson map contains one outstanding non-origin peak at (0.1542,0.3450,0.9215) which establishes the position of the bromines at ±(0.0771,0.1725,0.4608).

Three cycles of full-matrix least-squares refinement including only the bromine with isotropic temperature factors resulted in an R value of 0.46, at which stage a difference map revealed the positions of twelve other atoms. Three more refinement cycles with anisotropic temperature factors for the bromine and isotropic temperature factors for the other twelve atoms lowered R to 0.39 and the remaining non-hydrogen atoms could be located on a difference map. After three least-squares cycles with all atoms except the bromine having isotropic temperature factors, followed by two cycles with all atoms having anisotropic temperature factors, a difference map revealed the positions of all 37 hydrogen atoms. Four refinement cycles (hydrogen atoms with isotropic temperature factors, non-hydrogen atoms with anisotropic temperature factors) using a

polynomial weighting scheme followed by two cycles that included an anomalous dispersion correction for the bromine lowered R and Rw to their final values of 0.032 and 0.036 (0.083 and 0.076 including the unobserved reflections) respectively. The final coefficients used in the polynomial weighting scheme were A = 0.3074, B = 0.0464, C = -0.00511 and D = 0.000129. In the last cycle, the mean and maximum parameter shifts were 0.046 and 0.474 σ, respectively. A final difference map was calculated and showed two peaks of 0.4 electrons/Å^3. One is in the vicinity of the bromine and could perhaps be due to a lone pair interaction, but the other is near the ring containing the two oxygens for no obvious reason. Interestingly enough, bond lengths and bond angles involving O(2) and C(18) show some slight deviations (see comparison section). Final positional and thermal parameters are listed in Tables XIX and XX.

Results and discussion

A stereoview of the molecule is presented in Figure 10. With the exception of the p-bromobenzoate group, the atomic labelling is as before.

The p-bromobenzoate derivative contains the four-membered ring cis-fused to the cyclopentanone ring. Therefore the previous structure solved was not an impurity, but was indeed the second eluted isomer. The fact that both isomers give the same product upon ozonolysis followed by treatment with methoxide implies that they are both potential intermediates in

TABLE XIX. ATOMIC POSITIONAL AND ISOTROPIC THERMAL PARAMETERS FOR MOLECULE 9

(fractional x 10^4, Br x 10^5, H x 10^3, U x 10^3 Å^2)

Atom	x	y	z	Ueq/Uiso
Br	92418(5)	-17003(4)	-46120(4)	88
O(1)	7636(2)	653(2)	389(2)	54
O(2)	1527(2)	4669(2)	1602(2)	54
O(3)	2222(2)	5980(2)	3229(2)	52
O(4)	8288(2)	2182(2)	-211(2)	61
C(1)	4149(4)	4344(3)	1408(3)	47
C(2)	3501(4)	5345(3)	1976(4)	53
C(3)	2636(3)	4983(2)	2644(3)	47
C(4)	3389(4)	4000(3)	3666(3)	50
C(5)	4020(3)	2991(2)	3094(3)	45
C(6)	4697(4)	1968(3)	4103(3)	60
C(7)	5298(4)	935(3)	3533(4)	61
C(8)	6175(3)	1316(3)	2874(3)	52
C(9)	5397(3)	2278(2)	1814(3)	41
C(10)	4943(3)	3359(2)	2435(3)	41
C(11)	6229(3)	2327(3)	921(3)	43
C(12)	7333(4)	1283(3)	1419(3)	52
C(13)	6879(4)	485(3)	2181(4)	60
C(14)	4296(3)	1874(3)	601(3)	47
C(15)	5031(3)	2124(2)	-261(3)	46
C(16)	4722(5)	2227(3)	-1545(4)	66
C(17)	6161(4)	3777(4)	3434(4)	58
C(18)	407(3)	4606(3)	1909(4)	58
C(19)	-65(3)	5692(2)	2462(3)	48
C(20)	1131(3)	5914(3)	3622(3)	53
C(21)	-574(4)	6684(3)	1412(4)	66
C(22)	-1163(5)	5491(5)	2932(5)	79
C(23)	8092(3)	1209(3)	-366(3)	48
C(24)	8330(3)	492(2)	-1404(3)	46
C(25)	8391(4)	1023(3)	-2502(3)	62
C(26)	8636(4)	377(3)	-3464(4)	68
C(27)	8857(3)	-794(3)	-3307(3)	57
C(28)	8811(3)	-1334(3)	-2222(4)	59
C(29)	8527(3)	-684(3)	-1278(3)	53
H(011)	473(3)	462(2)	102(3)	54(8)
H(012)	349(3)	410(2)	71(3)	47(8)
H(021)	294(3)	593(3)	126(3)	55(8)
H(022)	416(3)	570(3)	259(3)	63(10)
H(041)	409(3)	430(2)	438(3)	52(8)
H(042)	287(3)	370(3)	404(3)	66(10)
H(051)	331(3)	275(2)	239(3)	35(7)
H(061)	545(3)	225(3)	491(3)	63(9)

continued...

H(062)	400(4)	176(3)	446(4)	84(11)
H(071)	460(3)	59(3)	293(3)	63(10)
H(072)	581(3)	36(3)	424(3)	69(10)
H(081)	688(3)	162(3)	351(3)	61(10)
H(111)	657(3)	302(2)	85(2)	42(7)
H(121)	806(3)	154(3)	192(3)	56(10)
H(131)	624(4)	4(3)	155(4)	75(11)
H(132)	757(4)	-1(3)	275(3)	65(10)
H(141)	426(3)	105(3)	73(3)	55(8)
H(142)	345(3)	228(2)	38(2)	35(7)
H(161)	534(3)	245(3)	-194(3)	69(10)
H(162)	386(4)	210(3)	-215(3)	71(10)
H(171)	653(4)	334(4)	426(5)	103(15)
H(172)	597(4)	459(4)	359(4)	92(12)
H(173)	684(4)	373(3)	312(3)	73(11)
H(181)	-33(3)	443(2)	108(3)	59(8)
H(182)	67(3)	390(3)	264(4)	83(11)
H(201)	133(3)	528(2)	432(3)	50(8)
H(202)	91(3)	666(3)	399(3)	68(9)
H(211)	-83(4)	481(4)	351(4)	89(14)
H(212)	-147(5)	619(4)	335(5)	119(16)
H(213)	-186(5)	522(4)	217(5)	109(15)
H(221)	19(4)	682(3)	113(3)	79(11)
H(222)	-88(3)	728(3)	184(3)	57(9)
H(223)	-133(4)	651(3)	62(4)	89(12)
H(251)	821(3)	182(3)	-256(3)	61(9)
H(261)	871(3)	73(3)	-419(4)	82(11)
H(281)	896(3)	-213(3)	-210(3)	68(10)
H(291)	854(3)	-106(2)	-52(3)	54(9)

TABLE XX. ANISOTROPIC THERMAL PARAMETERS IN MOLECULE 9

(U_{ij} x 10^4 Å^2)

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Br	1145(4)	873(3)	708(3)	-78(2)	476(2)	-205(2)
O(1)	557(13)	464(11)	681(13)	-27(10)	355(11)	-38(10)
O(2)	470(13)	641(13)	516(12)	-91(10)	184(11)	-211(10)
O(3)	487(12)	540(12)	548(12)	-60(10)	217(10)	-198(10)
O(4)	631(15)	480(13)	794(15)	-104(11)	358(12)	-89(11)
C(1)	476(19)	481(18)	496(18)	-59(15)	225(17)	-44(15)
C(2)	527(21)	471(18)	614(21)	-39(17)	266(18)	-47(17)
C(3)	446(18)	476(17)	489(17)	-74(14)	184(15)	-154(14)
C(4)	518(20)	575(19)	491(18)	-127(16)	258(17)	-116(16)
C(5)	425(18)	483(17)	451(17)	-77(14)	177(15)	-39(14)
C(6)	661(23)	632(21)	589(21)	-36(18)	341(20)	42(17)
C(7)	666(24)	532(20)	641(22)	15(19)	315(20)	124(19)
C(8)	497(20)	520(18)	530(18)	2(15)	223(17)	38(15)
C(9)	386(16)	426(16)	432(15)	-49(12)	165(13)	-18(12)
C(10)	350(16)	456(16)	424(15)	-70(12)	142(13)	-56(12)
C(11)	397(17)	412(16)	524(17)	-57(14)	221(14)	-26(13)
C(12)	460(20)	516(18)	588(19)	-16(16)	228(17)	-57(16)
C(13)	601(23)	515(19)	648(22)	103(19)	271(20)	106(18)
C(14)	390(19)	487(19)	524(18)	-84(15)	150(15)	-98(14)
C(15)	462(18)	419(16)	492(18)	-48(13)	183(15)	-30(13)
C(16)	696(27)	717(24)	554(22)	-171(20)	213(22)	-22(17)
C(17)	461(21)	694(24)	593(22)	-167(18)	177(18)	-200(19)
C(18)	493(21)	643(22)	604(21)	-126(17)	182(18)	-178(18)
C(19)	452(18)	529(18)	496(17)	-75(14)	209(15)	-80(14)
C(20)	505(21)	603(21)	508(19)	-33(16)	235(17)	-145(17)
C(21)	561(24)	601(23)	706(25)	34(19)	146(22)	-55(19)
C(22)	676(28)	979(35)	859(30)	-180(26)	423(26)	-82(28)
C(23)	368(17)	434(18)	613(19)	27(14)	183(15)	23(15)
C(24)	358(17)	446(17)	575(18)	-30(13)	183(14)	2(14)
C(25)	769(25)	456(20)	658(22)	-86(17)	297(19)	7(17)
C(26)	855(27)	647(24)	605(22)	-123(19)	337(20)	38(18)
C(27)	566(20)	625(21)	529(19)	-59(16)	239(16)	-113(16)
C(28)	633(22)	448(19)	736(23)	-42(16)	339(18)	-67(17)
C(29)	545(20)	463(18)	640(21)	-14(15)	312(17)	44(16)

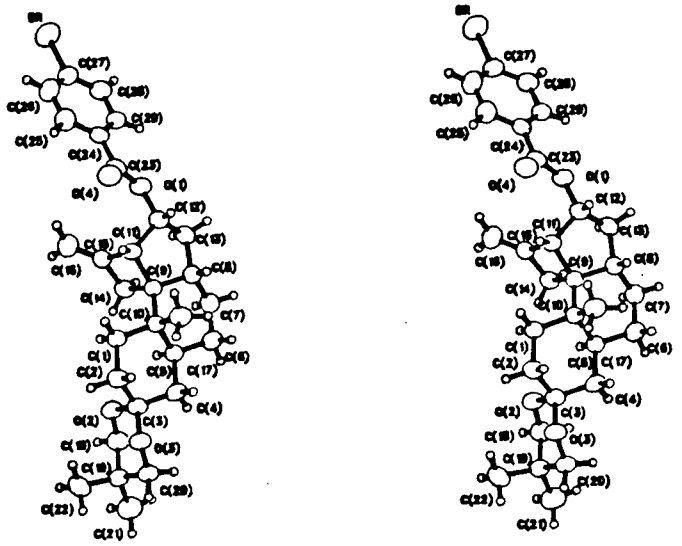


Figure 10. Stereoview of the p-bromobenzoate derivative

the synthesis of stemodin, and their separation was apparently unnecessary. Of interest now is the rearrangement mechanism that allows both isomers to yield the same compound, but this is beyond the scope of this thesis. Mean plane calculations, bond lengths and bond angles are listed in Tables XXI, XXII, XXIII, XXIV, and XXV. A packing diagram is shown in Figure 11. The crystal is held together by van der Waals forces.

Comparison of the three structures

Whenever the structures of three molecules as similar as those presented so far are solved, it is of interest to compare derived quantities, e.g. bond lengths, bond angles and molecular geometry.

Bond lengths for these three structures are compared in Table XXVI. Upon inspection of this table, it is immediately obvious that the estimated standard deviations (e.s.d.'s) in molecule 5 are about three times as great as those in molecules 4 and 9. This is not surprising; the crystal structure of 5 is the least accurate of the three, mainly as the crystal was so poor that only 20% of the collected reflections could be considered observed (as opposed to 60% in the other two structures). Consistent with this, the R values for the structure of molecule 5 are slightly larger than those for 4 and 9, especially when the unobserved reflections are included.

The bond lengths compare extremely well with each other. Only one pair shows a significant difference (i.e., has a

TABLE XXI. MEAN PLANES IN THE VICINITY OF THE CYCLOBUTYL RING IN MOLECULE 9

Equations of planes (lX+mY+nZ=p)

plane	l	m	n	p
1	-0.2436	0.9697	-0.0669	0.9328
2	-0.2024	0.9377	-0.2825	-0.9354
3	-0.4777	-0.6437	-0.5979	-5.6270
4	-0.6224	-0.0055	-0.7827	-5.9661

Deviations from planes (Å)

atom	1	2	3	4
C(8)			-0.036(3)*	0.000(4)*
C(9)	0.248(3)	0.000(3)*	0.036(3)*	0.985(3)
C(11)	0.000(3)*	0.000(3)*	-0.041(3)*	0.894(3)
C(12)			0.038(4)*	0.000(4)*
C(13)			-0.610(4)	0.000(4)*
C(14)	0.000(4)*	0.000(3)*		
C(15)	0.000(3)*	0.230(3)		
C(16)	0.084(4)	0.601(4)		

*atoms included in plane calculations

Angles between normals to the planes

planes (1) and (2) : 14.5°
planes (2) and (3) : 108.7°
planes (3) and (4) : 34.6°

TABLE XXII. BOND LENGTHS(Å) OF NON-HYDROGEN ATOMS IN MOLECULE 9

Bond	Distance	Bond	Distance
Br -C(27)	1.906(3)	C(9) -C(11)	1.571(4)
O(1)-C(12)	1.453(4)	C(9) -C(14)	1.566(4)
O(1)-C(23)	1.336(4)	C(10)-C(17)	1.540(4)
O(2)-C(3)	1.430(3)	C(11)-C(12)	1.529(4)
O(2)-C(18)	1.406(4)	C(11)-C(15)	1.520(4)
O(3)-C(3)	1.429(3)	C(12)-C(13)	1.521(5)
O(3)-C(20)	1.431(3)	C(14)-C(15)	1.507(4)
O(4)-C(23)	1.209(3)	C(15)-C(16)	1.315(5)
C(1)-C(2)	1.530(4)	C(18)-C(19)	1.515(4)
C(1)-C(10)	1.533(4)	C(19)-C(20)	1.513(4)
C(2)-C(3)	1.508(4)	C(19)-C(21)	1.531(5)
C(3)-C(4)	1.521(5)	C(19)-C(22)	1.529(5)
C(4)-C(5)	1.530(4)	C(23)-C(24)	1.484(4)
C(5)-C(6)	1.537(4)	C(24)-C(25)	1.386(4)
C(5)-C(10)	1.560(4)	C(24)-C(29)	1.377(4)
C(6)-C(7)	1.533(5)	C(25)-C(26)	1.373(5)
C(7)-C(8)	1.520(5)	C(26)-C(27)	1.374(5)
C(8)-C(9)	1.532(4)	C(27)-C(28)	1.370(5)
C(8)-C(13)	1.521(4)	C(28)-C(29)	1.378(4)
C(9)-C(10)	1.542(4)		

TABLE XXIII. BOND ANGLES(°) OF NON-HYDROGEN ATOMS IN MOLECULE 9

Bonds	Angle	Bonds	Angle
C(12)-O(1) -C(23)	117.0(2)	C(9) -C(11)-C(12)	104.1(2)
C(3) -O(2) -C(18)	116.1(2)	C(9) -C(11)-C(15)	88.6(2)
C(3) -O(3) -C(20)	114.6(2)	C(12)-C(11)-C(15)	114.3(3)
C(2) -C(1) -C(10)	113.2(3)	O(1) -C(12)-C(11)	114.9(3)
C(1) -C(2) -C(3)	112.6(3)	O(1) -C(12)-C(13)	108.3(3)
O(2) -C(3) -O(3)	110.3(2)	C(11)-C(12)-C(13)	106.7(3)
O(2) -C(3) -C(2)	105.4(2)	C(8) -C(13)-C(12)	102.4(3)
O(2) -C(3) -C(4)	112.2(2)	C(9) -C(14)-C(15)	89.2(2)
O(3) -C(3) -C(2)	105.5(2)	C(11)-C(15)-C(14)	92.5(2)
O(3) -C(3) -C(4)	111.9(2)	C(11)-C(15)-C(16)	133.2(3)
C(2) -C(3) -C(4)	111.1(3)	C(14)-C(15)-C(16)	134.1(3)
C(3) -C(4) -C(5)	112.6(2)	O(2) -C(18)-C(19)	112.8(3)
C(4) -C(5) -C(6)	112.4(2)	C(18)-C(19)-C(20)	105.4(3)
C(4) -C(5) -C(10)	112.0(2)	C(18)-C(19)-C(21)	110.5(3)
C(6) -C(5) -C(10)	113.0(3)	C(18)-C(19)-C(22)	109.0(3)
C(5) -C(6) -C(7)	113.1(3)	C(20)-C(19)-C(21)	111.2(3)
C(6) -C(7) -C(8)	109.5(3)	C(20)-C(19)-C(22)	110.2(3)
C(7) -C(8) -C(9)	110.9(3)	C(21)-C(19)-C(22)	110.3(3)
C(7) -C(8) -C(13)	121.8(3)	O(3) -C(20)-C(19)	111.2(2)
C(9) -C(8) -C(13)	103.5(2)	O(1) -C(23)-O(4)	123.5(3)
C(8) -C(9) -C(10)	111.0(2)	O(1) -C(23)-C(24)	112.1(3)
C(8) -C(9) -C(11)	105.9(2)	O(4) -C(23)-C(24)	124.3(3)
C(8) -C(9) -C(14)	112.9(2)	C(23)-C(24)-C(25)	118.9(3)
C(10)-C(9) -C(11)	121.1(2)	C(23)-C(24)-C(29)	121.4(3)
C(10)-C(9) -C(14)	115.7(2)	C(25)-C(24)-C(29)	119.7(3)
C(11)-C(9) -C(14)	88.3(2)	C(24)-C(25)-C(26)	120.0(3)
C(1) -C(10)-C(5)	108.4(2)	C(25)-C(26)-C(27)	119.4(3)
C(1) -C(10)-C(9)	112.8(2)	Br -C(27)-C(26)	119.8(3)
C(1) -C(10)-C(17)	108.0(3)	Br -C(27)-C(28)	118.8(2)
C(5) -C(10)-C(9)	105.3(2)	C(26)-C(27)-C(28)	121.3(3)
C(5) -C(10)-C(17)	112.7(3)	C(27)-C(28)-C(29)	119.1(3)
C(9) -C(10)-C(17)	109.6(2)	C(24)-C(29)-C(28)	120.4(3)

TABLE XXIV. BOND LENGTHS(Å) OF HYDROGEN ATOMS IN MOLECULE 9

Bond	Distance	Bond	Distance
C(1) -H(011)	0.98(3)	C(16)-H(162)	0.97(4)
C(1) -H(012)	0.92(3)	C(17)-H(171)	0.95(4)
C(2) -H(021)	0.98(3)	C(17)-H(172)	0.98(4)
C(2) -H(022)	0.94(3)	C(17)-H(173)	0.92(4)
C(4) -H(041)	0.98(3)	C(18)-H(181)	1.01(3)
C(4) -H(042)	0.92(3)	C(18)-H(182)	1.08(4)
C(5) -H(051)	0.95(3)	C(20)-H(201)	1.01(3)
C(6) -H(061)	1.05(3)	C(20)-H(202)	1.00(3)
C(6) -H(062)	1.04(4)	C(21)-H(211)	1.03(4)
C(7) -H(071)	0.96(4)	C(21)-H(212)	0.93(3)
C(7) -H(072)	0.97(4)	C(21)-H(213)	1.00(4)
C(8) -H(081)	0.95(3)	C(22)-H(221)	0.96(4)
C(11)-H(111)	0.97(3)	C(22)-H(222)	1.01(5)
C(12)-H(121)	0.88(3)	C(22)-H(223)	0.99(5)
C(13)-H(131)	1.00(4)	C(25)-H(251)	0.93(3)
C(13)-H(132)	0.90(4)	C(26)-H(261)	0.93(4)
C(14)-H(141)	0.99(3)	C(28)-H(281)	0.93(3)
C(14)-H(142)	0.92(3)	C(29)-H(291)	0.93(3)
C(16)-H(161)	0.99(3)		

TABLE XXV. BOND ANGLES(°) INVOLVING HYDROGEN ATOMS IN MOLECULE 9

Bonds	Angle	Bonds	Angle
C(2) -C(1) -H(011)	109(2)	C(9) -C(14)-H(142)	117(2)
C(2) -C(1) -H(012)	108(2)	C(15) -C(14)-H(141)	115(2)
C(10) -C(1) -H(011)	110(2)	C(15) -C(14)-H(142)	117(2)
C(10) -C(1) -H(012)	111(2)	H(141)-C(14)-H(142)	107(2)
H(011)-C(1) -H(012)	106(2)	C(15) -C(16)-H(161)	122(2)
C(1) -C(2) -H(021)	110(2)	C(15) -C(16)-H(162)	120(2)
C(1) -C(2) -H(022)	110(2)	H(161)-C(16)-H(162)	118(3)
C(3) -C(2) -H(021)	108(2)	C(10) -C(17)-H(171)	113(3)
C(3) -C(2) -H(022)	109(2)	C(10) -C(17)-H(172)	112(2)
H(021)-C(2) -H(022)	107(3)	C(10) -C(17)-H(173)	112(2)
C(3) -C(4) -H(041)	107(2)	H(171)-C(17)-H(172)	109(3)
C(3) -C(4) -H(042)	114(2)	H(171)-C(17)-H(173)	106(3)
C(5) -C(4) -H(041)	109(2)	H(172)-C(17)-H(173)	104(3)
C(5) -C(4) -H(042)	106(2)	O(2) -C(18)-H(181)	109(2)
H(041)-C(4) -H(042)	108(3)	O(2) -C(18)-H(182)	108(2)
C(4) -C(5) -H(051)	106(1)	C(19) -C(18)-H(181)	110(2)
C(6) -C(5) -H(051)	108(1)	C(19) -C(18)-H(182)	109(2)
C(10) -C(5) -H(051)	105(1)	H(181)-C(18)-H(182)	109(3)
C(5) -C(6) -H(061)	107(2)	O(3) -C(20)-H(201)	114(2)
C(5) -C(6) -H(062)	107(2)	O(3) -C(20)-H(202)	107(2)
C(7) -C(6) -H(061)	110(2)	C(19) -C(20)-H(201)	107(2)
C(7) -C(6) -H(062)	112(2)	C(19) -C(20)-H(202)	110(2)
H(061)-C(6) -H(062)	107(3)	H(201)-C(20)-H(202)	108(2)
C(6) -C(7) -H(071)	109(2)	C(19) -C(21)-H(211)	109(2)
C(6) -C(7) -H(072)	109(2)	C(19) -C(21)-H(212)	103(2)
C(8) -C(7) -H(071)	112(2)	C(19) -C(21)-H(213)	111(2)
C(8) -C(7) -H(072)	110(2)	H(211)-C(21)-H(212)	115(3)
H(071)-C(7) -H(072)	106(3)	H(211)-C(21)-H(213)	110(3)
C(7) -C(8) -H(081)	110(2)	H(212)-C(21)-H(213)	110(3)
C(9) -C(8) -H(081)	106(2)	C(19) -C(22)-H(221)	107(2)
C(13) -C(8) -H(081)	103(2)	C(19) -C(22)-H(222)	111(3)
C(9) -C(11)-H(111)	120(2)	C(19) -C(22)-H(223)	107(3)
C(12) -C(11)-H(111)	112(2)	H(221)-C(22)-H(222)	114(3)
C(15) -C(11)-H(111)	116(2)	H(221)-C(22)-H(223)	101(3)
O(1) -C(12)-H(121)	107(2)	H(222)-C(22)-H(223)	116(3)
C(11) -C(12)-H(121)	107(2)	C(24) -C(25)-H(251)	117(2)
C(13) -C(12)-H(121)	113(2)	C(26) -C(25)-H(251)	123(2)
C(8) -C(13)-H(131)	110(2)	C(25) -C(26)-H(261)	120(2)
C(8) -C(13)-H(132)	113(2)	C(27) -C(26)-H(261)	120(2)
C(12) -C(13)-H(131)	110(2)	C(27) -C(28)-H(281)	122(2)
C(12) -C(13)-H(132)	112(2)	C(29) -C(28)-H(281)	119(2)
H(131)-C(13)-H(132)	109(3)	C(24) -C(29)-H(291)	121(2)
C(9) -C(14)-H(141)	111(2)	C(28) -C(29)-H(291)	118(2)

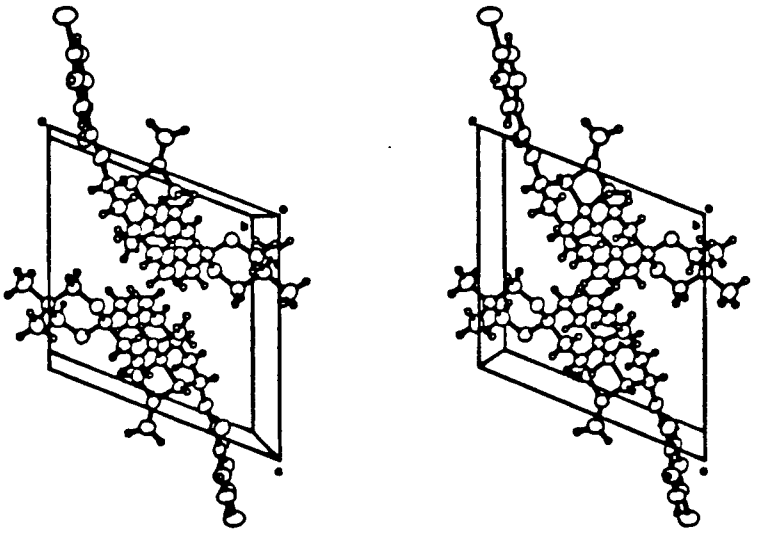


Figure 11. Packing diagram of the p-bromobenzoate derivative

TABLE XXVI. COMPARISONS OF EQUIVALENT BOND LENGTHS(Å) IN MOLECULES 4 , 5 , AND 9

Bond	4	5	9
O(1)-C(12)	1.199(3)	1.229(09)	-
O(2)-C(3)	1.420(2)	1.428(09)	1.430(3)
O(2)-C(18)	1.428(3)	1.428(10)	1.406(4)
O(3)-C(3)	1.424(2)	1.427(08)	1.429(3)
O(3)-C(20)	1.423(3)	1.415(09)	1.431(3)
C(1)-C(2)	1.524(3)	1.515(10)	1.530(4)
C(1)-C(10)	1.538(3)	1.536(11)	1.533(4)
C(2)-C(3)	1.509(3)	1.526(11)	1.508(4)
C(3)-C(4)	1.523(3)	1.501(11)	1.521(5)
C(4)-C(5)	1.529(3)	1.526(10)	1.530(4)
C(5)-C(6)	1.529(3)	1.516(11)	1.537(4)
C(5)-C(10)	1.546(3)	1.550(10)	1.560(4)
C(6)-C(7)	1.514(3)	1.533(10)	1.533(5)
C(7)-C(8)	1.528(3)	1.519(11)	1.520(5)
C(8)-C(9)	1.544(3)	1.528(11)	1.532(4)
C(8)-C(13)	1.528(3)	1.538(11)	1.521(4)
C(9)-C(10)	1.544(3)	1.532(10)	1.542(4)
C(9)-C(11)	1.571(3)	1.577(10)	1.571(4)
C(9)-C(14)	1.563(3)	1.571(10)	1.566(4)
C(10)-C(17)	1.538(3)	1.549(10)	1.540(4)
C(11)-C(12)	1.515(4)	1.523(10)	1.529(4)
C(11)-C(15)	1.515(4)	1.534(11)	1.520(4)
C(12)-C(13)	1.511(4)	1.487(12)	1.521(5)
C(14)-C(15)	1.496(4)	1.521(11)	1.507(4)
C(15)-C(16)	1.324(4)	1.312(11)	1.315(5)
C(18)-C(19)	1.521(3)	1.546(10)	1.515(4)
C(19)-C(20)	1.521(3)	1.520(11)	1.513(4)
C(19)-C(21)	1.525(3)	1.510(13)	1.531(5)
C(19)-C(22)	1.518(4)	1.529(12)	1.529(5)

difference greater than 3.0 σ): O(2)-C(18) has lengths 1.428(3) (in 4) and 1.406(4) Å (in 9), a difference of only 0.022 Å (about 4.5σ) and so of little importance. The largest absolute difference is for C(18)-C(19), which has lengths 1.546(10) (in 5) and 1.515(4) Å (in 9), a difference of 0.031 Å, but this only corresponds to about 3.0σ.

More interesting trends may be found by comparing the bond angles (Table XXVII). Because of the larger e.s.d.'s in 5, the closest match is most often found between the values from 4 and 9. However, bond angles involving O(2) and C(18) in 9 again show significant but most probably unimportant deviations. This might be related to the slight electron density (0.4 electrons/Å^3) located near this ring. Important deviations do occur when we examine bond angles in the vicinity of the four-membered ring. Angles involving atoms in the six-membered ring closest to the cyclobutyl ring are very similar in 5 and 9 but very different in 4. It seems very likely that the magnitudes of these angles are affected by the orientation of the four-membered ring. This effect is especially pronounced in the angles marked with an asterisk (*) in Table XXVII. In examining the angles in the five-membered ring, one notices that this time the odd angles seem to come from the p-bromobenzoate derivative, 9. This is not too surprising as the hybridization of C(12) is different in 9. The angles that are affected by this are marked with a plus sign (+) in Table XXVII. The cyclobutyl angles are all fairly similar and do not deviate greatly from the free cyclobutyl angle[22], 89.3°.

Mean plane calculations have been presented for planes in

TABLE XXVII. COMPARISON OF BOND ANGLES(°) IN MOLECULES 4 ,5 AND 9

Bonds	4	5	9
C(3) -O(2)-C(18)	114.3(1)	113.6(6)	116.1(2)
C(3) -O(3)-C(20)	114.3(2)	113.9(7)	114.6(2)
C(2) -C(1)-C(10)	112.8(2)	112.9(8)	113.2(3)
C(1) -C(2)-C(3)	112.4(2)	111.8(7)	112.6(3)
O(2) -C(3)-O(3)	110.2(1)	109.7(6)	110.3(2)
O(2) -C(3)-C(2)	105.8(2)	112.0(7)	105.4(2)
O(2) -C(3)-C(4)	112.0(2)	105.8(7)	112.2(2)
O(3) -C(3)-C(2)	105.1(2)	112.0(7)	105.5(2)
O(3) -C(3)-C(4)	112.2(2)	106.0(8)	111.9(2)
C(2) -C(3)-C(4)	111.2(2)	111.0(7)	111.1(3)
C(3) -C(4)-C(5)	112.3(2)	113.3(7)	112.6(2)
C(4) -C(5)-C(6)	111.7(2)	112.7(7)	112.4(2)
C(4) -C(5)-C(10)	113.1(2)*	112.0(6)	112.0(2)
C(6) -C(5)-C(10)	111.4(2)*	113.3(6)	113.0(3)
C(5) -C(6)-C(7)	110.5(2)*	113.2(8)	113.1(3)
C(6) -C(7)-C(8)	115.3(2)*	107.5(7)	109.5(3)
C(7) -C(8)-C(9)	111.5(2)	111.1(7)	110.9(3)
C(7) -C(8)-C(13)	108.3(2)*	120.7(8)	121.8(3)
C(9) -C(8)-C(13)	104.6(2)	105.9(7)	103.5(2)+
C(8) -C(9)-C(10)	114.7(2)*	111.0(7)	111.0(2)
C(8) -C(9)-C(11)	104.5(2)	104.0(6)	105.9(2)+
C(8) -C(9)-C(14)	114.3(2)	113.4(7)	112.9(2)
C(10)-C(9)-C(11)	118.8(2)*	121.7(7)	121.1(2)
C(10)-C(9)-C(14)	113.7(2)*	116.4(7)	115.7(2)
C(11)-C(9)-C(14)	88.0(2)	88.3(6)	88.3(2)
C(1) -C(10)-C(5)	107.7(2)	107.8(6)	108.4(2)
C(1) -C(10)-C(9)	111.1(2)*	112.8(7)	112.8(2)
C(1) -C(10)-C(17)	109.7(2)	108.6(7)	108.0(3)
C(5) -C(10)-C(9)	109.3(2)*	105.6(6)	105.3(2)
C(5) -C(10)-C(17)	111.2(2)*	112.4(6)	112.7(3)
C(9) -C(10)-C(17)	107.8(2)*	109.8(6)	109.6(2)
C(9) -C(11)-C(12)	105.1(2)*	104.2(7)	104.1(2)
C(9) -C(11)-C(15)	88.3(2)	89.6(6)	88.6(2)
C(12)-C(11)-C(15)	110.8(2)	106.9(7)	114.3(3)
O(1) -C(12)-C(11)	125.0(2)	124.5(9)	-
O(1) -C(12)-C(13)	126.1(2)	125.9(8)	-
C(11)-C(12)-C(13)	108.9(2)	109.5(7)	106.7(3)+
C(8) -C(13)-C(12)	103.0(2)	101.1(7)	102.4(3)
C(9) -C(14)-C(15)	89.3(2)	90.3(6)	89.2(2)
C(11)-C(15)-C(14)	92.6(2)	91.7(6)	92.5(2)
C(11)-C(15)-C(16)	131.9(4)	132.0(9)	133.2(3)

Continued...

C(14)-C(15)-C(16)	135.2(4)	136.3(9)	134.1(3)
O(2) -C(18)-C(19)	111.6(2)	113.4(7)	112.8(3)
C(18)-C(19)-C(20)	105.6(2)	105.1(7)	105.4(3)
C(18)-C(19)-C(21)	110.3(2)	109.6(9)	110.5(3)
C(18)-C(19)-C(22)	109.6(2)	110.8(8)	109.0(3)
C(20)-C(19)-C(21)	110.3(2)	111.1(8)	111.2(3)
C(20)-C(19)-C(22)	109.5(2)	109.0(8)	110.2(3)
C(21)-C(19)-C(22)	111.4(2)	111.2(8)	110.3(3)
O(3) -C(20)-C(19)	111.5(2)	112.2(8)	111.2(2)

the vicinity of the cyclobutyl ring for all three structures. The envelope tip of the 5-membered ring is always oriented in the same direction as the cyclobutyl ring. The change of hybridization at C(12) in the p-bromobenzoate derivative affects the geometry of the 5-membered ring only slightly - the envelope tip is bent down 35 and 36° in 4 and 5, and 39° in 9. When O(1) is doubly bound to C(12) it is on the opposite side of plane 3 (the 5-membered ring) to the envelope tip. The cyclobutyl ring is folded away from the envelope tip along the C(11)...C(14) axis in every structure, although the degree of folding varies - 14°, 4.3° and 12.7° in 4, 5, and 9 respectively. The angle between the cyclobutyl and 5-membered rings is about 109° for all three structures. For the p-bromobenzoate derivative, the phenyl group is oriented at 22° to the carboxyl moiety (Table XXVIII). The bromine lies in the phenyl plane.

With the determination of these three crystal structures, the identities of 4 and 5 have been firmly established. After three structures the conclusion was essentially the same as after one, but the value of investigating the possible trans-fused adduct should not be underestimated. As well as determining the structure of the second eluted isomer, the last two structures also confirmed the existence of an unusual rearrangement mechanism which is still under investigation.

TABLE XXVIII. MEAN PLANES OF THE P-BROMOBENZOATE MOIETY IN MOLECULE 9

Equations of planes (lX+mY+nZ=p)

plane	l	m	n	p
1	-0.8726	-0.0174	-0.4881	-7.8953
2	-0.7206	0.3002	-0.6250	-6.0556

Deviations from planes (Å)

atom	1	2
O(1)	0.368(2)	0.000(2)*
O(4)	-0.464(2)	0.000(2)*
C(23)	-0.067(3)	0.001(3)*
C(24)	-0.007(3)*	0.000(4)*
C(25)	-0.009(4)*	0.421(4)
C(26)	0.018(4)*	0.405(4)
C(27)	0.007(4)*	-0.069(4)
C(28)	-0.002(4)*	-0.500(4)
C(29)	0.009(4)*	-0.446(4)
Br	-0.0002(5)*	-0.1398(5)

*atoms included in plane calculations

Angles between normals to the planes

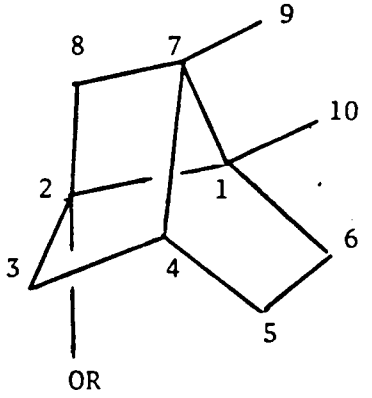
planes (1) and (2) : 21.8°

CHAPTER 3

THE CRYSTAL STRUCTURE OF CAMPHOR-1,4-HOMOENOL P-BROMOBENZOATE

Introduction and preparation

In investigations of homoenolization of bicyclic ketones[23], it was noted that the C(3), C(6), C(8) and C(10) hydrogen atoms in camphor underwent exchange (prolonged treatment with KOBu/HOBu, 185-250°C, sealed tube), and the intermediacy of a highly strained 1,4-homoenol was proposed to account for the exchange at the C(8) position. During recent attempts to extend the use of camphor in monoterpenoid[24-26] and sesquiterpenoid[27] synthesis, a new compound was obtained (treatment of 8-bromocamphor with $Ca/NH_3/CH_3OH$, -78°C, yield 45%, camphor 33%) which had spectral properties[28] indicative of the 1,4-homoenol structure (1, R = H). In order to verify this structure, an x-ray analysis was performed on the p-bromobenzoate derivative, camphor-1,4-homoenol p-bromobenzoate (1, R = COC_6H_4Br).



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The p-bromobenzoate derivative was prepared[28] by stirring a solution of camphor-1,4-homoenol (1, R = H, 250 mg) and p-bromobenzoyl chloride (1.0 g) in 2 mls of dry methylphosphoramide (under N_2, 20°C, 24 hrs). After dilution with water, extraction with ether, and washing of the extract with sodium bicarbonate solution, drying and evaporation

produced a yellow semi-solid. Purification of this by chromatography over alumina yielded a colorless oil which crystallized from petroleum ether to give the desired product. Spectral and analytical properties of this derivative agreed with the proposed structure[28].

Experimental

X-ray photography showed that along the h00, 0k0, and 001 axes reflections were systematically absent when h, k, and l were respectively odd, indicating the presence of three mutually perpendicular twofold screw axes, and hence that the crystal must be of the orthorhombic space group $P2_12_12_1$.

The intensity data were collected using graphite-monochromatized MoKα radiation and an ω-(4/3)θ scan technique with an ω-scan angle of $(0.8 + 0.35 \tan \theta)^\circ$. The vertical and horizontal aperture widths were 4 mm and $(2.5 + \tan \theta)$mm, respectively. The intensities of three standard reflections (2 0 -7, -1 1 -7, and 2 0 -3) were measured every one hour of x-ray exposure time and were used to scale the data although variations were small. The orientations of another three reflections (2 1 -10, 0 4 -8, and 2 0 -8) were checked every 100 reflections, and reorientation occurred if the difference between observed and calculated scattering vectors was greater than 0.05°. In the range $0 < \theta \leq 25^\circ$, 700 out of 1645 reflections collected had $I/\sigma(I) \geq 3.0$ and were considered observed. In the refinement, only the reflections in the range

$0 < \theta \leq 23^{\circ}$ were used, where 697 out of 1300 (53.6%) were observed. The cell parameters were refined by least-squares methods using the $\sin\theta/\lambda$ values of 25 reflections in the range $6 < \theta < 14^{\circ}$, and appear with other crystal data in Table XXIX.

The linear absorption coefficient μ is fairly large and an

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TABLE XXIX. CRYSTAL DATA FOR CAMPHOR-P-BROMOBENZOATE

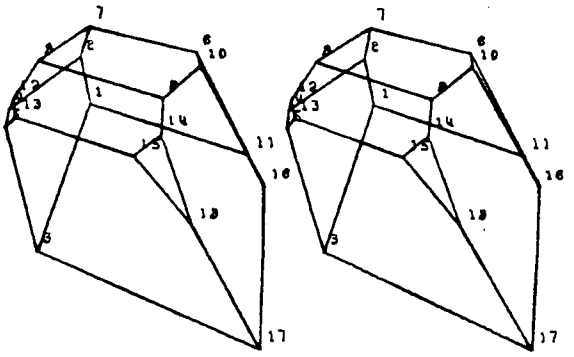
$C_{17}H_{19}BrO_2$	f.w. = 344.5
Orthorhombic	Z = 4
	space group = $P2_12_12_1$
a = 6.875(1)	F(000) = 688
b = 8.522(2)	λ = 0.71073 Å
c = 26.658(6) Å	μ = 25.5 cm^{-1}
V = 1562 $Å^3$	Dc = 1.32 g/cc

****

absorption correction is in order. The crystal shape was approximated by 11 faces (see figure 12). 184 sampling points were used with an average spacing of 5.0×10^{-3}cm in a crystal of volume 0.0205 mm^3 and the resulting transmission factors ranged from 0.450 to 0.633. The intensities were corrected for absorption, as well as for Lorentz and polarization effects.

The structure was solved by Patterson and direct methods. Direct methods were used because of an eagerness to elucidate the structure, and the Patterson map was initially miscalculated. Later a correct Patterson map was used to arrive at the same set of bromine positions.

The 500 highest E's obtained by following the minimum



plane	vertices	distance from centre (mm)
1 0 0	1,2,3,4,5	0.200
0 1 0	6,7,8,9,10	0.150
0 0 1	1,2,6,7,11	0.100
0 1 -1	8,9,12,13,14,15	0.140
0 -1 1	1,3,11,16 17	0.100
-2 1 2	6,10,11,16	0.175
1 1 -2	4,5,12,13	0.200
2 1 0	2,4,7,8,12	0.210
-1 1 0	14,15,18	0.175
-2 1 0	8,10,14,16,17,18	0.175
0 0 -1	3,5,13,15,17,18	0.125

Figure 12. Crystal shape of camphor homoenol p-bromobenzoate

profile of a K-curve were input into the MULTAN programme. Four phases were accepted as known from their Σ_1-relationships (4 0 10 and 4 0 0 had phase π, 4 4 0 and 0 8 0 had phase 0). The origin determining phase assignments were $\pi/2$ for 0 5 18, $\pi/2$ for 0 7 5 and $\pi/4$ for 3 5 2. The 3 5 2 phase assignment also fixed the enantiomorph. The highest E (2 7 0) was used as a symbol and was assigned initial phases of 0 and π. Two sets of phases were generated and both proved to be correct. E-maps were calculated for each set, and one high peak appeared in both cases and could be assigned to the bromine. The positions of the peaks in the second set were related to those in the first by the symmetry operation (1/2-x,y,z), i.e., the two sets are enantiomorphic. The restriction on the phase assignment of (3 5 2) failed to fix the enantiomorph. The first set was arbitrarily chosen placing the bromine at the fractional coordinates (0.3886, 0.6046, 0.0100).

If we have a bromine at (x,y,z) in the space group $P2_12_12_1$, then we will also have bromines at :

(1/2-x,-y,1/2+z)

(1/2+x,1/2-y,-z)

(-x,1/2+y,1/2-y).

The bromine interatomic vectors we would expect to find on a Patterson map are then :

(1/2,1/2±2y,±2z)

(±2x,1/2,1/2±2z)

(1/2±2x,±2y,1/2).

These are known as the Harker sections at x=1/2, y=1/2 and z=1/2 respectively. As an interatomic vector may originate at either

of the two atoms it connects, a Patterson map is always centrosymmetric. If the original space group is not centrosymmetric, the Patterson then has an additional symmetry element, and the unique volume will be half that of the original space group. In $P2_12_12_1$ the unique volume is a quarter of the unit cell and so only an eighth of the Patterson need be examined.

In this eighth, there were three large outstanding peaks, one on each Harker section, due to the bromine-bromine interatomic vectors : (0.2209,0.5000,0.4939), corresponding to the Harker section -2x,1/2,1/2-2z and solving to x = 0.3895 and z = 0.0030; (0.5000,0.2840,0.0186), corresponding to the Harker section 1/2,1/2-2y,+2z and solving to y = 0.6080 and z = 0.0093; and (0.2692,0.2141,0.5000), corresponding to the Harker section 1/2+2x,+2y,1/2 and solving to x = 0.3846 and y = 0.6070. These average to give the one out of four solutions (the other three are symmetry related) that places the bromine at (0.3870,0.6075,0.0062) in agreement with the direct methods result.

Obtaining the same solution from both methods is always reassuring. It is also of interest to see that direct methods, a statistical technique based on the assumption that the electron density is distributed randomly throughout the unit cell, can also work for heavy-atom structures.

A difference map calculated after refining the bromine for three least-squares cycles revealed the positions of nine other atoms, and nine more could be located on a map calculated after refining the first 10 atoms for three cycles. A third difference

map, calculated after three more refinement cycles, revealed the positions of the remaining two non-hydrogen atoms. All twenty non-hydrogen atoms were refined anisotropically for three cycles, lowering R to 0.062. All nineteen hydrogens could now be located on a difference map, but they would not refine, and better results were obtained by keeping the hydrogens fixed in calculated positions with fixed isotropic temperature factors ($U = 0.057\ A^2$). Hughes' weighting scheme was introduced ($(w)^{1/2} = 1$ for $|Fo| < F^*$ and $(w)^{1/2} = F^*/|Fo|$ for $|Fo| \geq F^*$, $F^* = 23.5$) and the structure was refined for four more cycles to an R value of 0.049.

Anomalous dispersion corrections have different effects on the magnitudes of the structure factors F(hkl) and F(-(hkl)), and if these differences are sufficiently large it is possible to determine the absolute configuration of a molecule by determining which observed set of data, Fo(hkl) or Fo(-(hkl)), gives better agreement with the calculated structure factors. Very often the F(-(hkl)) data are not collected, so alternatively it may be determined which enantiomorph gives rise to the calculated strucure factors that have the best agreement with the Fo(hkl) data (with anomalous dispersion corrections applied).

Anomalous dispersion corrections were applied to the bromine, carbon, and oxygen atoms, and the two enantiomers were each refined in three equivalent least-squares cycles. For the enantiomer originally chosen, the three cycles refined to R = 0.056 and Rw = 0.070; for the enantiomer obtained by changing the signs of x, y, and z, the three cycles refined to

R = 0.046 and Rw = 0.056. The latter is therefore the correct enantiomer (better agreement with the observed data) and was used in further refinement. The structure was refined for four cycles before the weighting scheme was modified such that F* was now equal to 18.0. The weighting scheme needed further modification after four more least-squares refinement cycles; F* was now 17.8 and a factor G* was introduced such that when $|Fo| < G^*$, $(w)^{1/2} = |Fo|/G^*$ and G* = 11.5. A final least-squares cycle refined the structure to convergence with R and Rw values of 0.045 and 0.054 (0.123 and 0.054 including unobserved reflections) respectively. Positional and thermal parameters are given in Tables XXX and XXXI.

Results and discussion

Figure 13 shows a stereoview of the structure with the atomic labelling. The 1,4-homoenol structure is verified, and all bond lengths (Tables XXXII and XXXIII) agree fairly well with expected values. The C(1)-C(2) and C(1)-C(7) bond lengths (1.569 and 1.558 Å) are slightly longer than normal sp^3-sp^3 carbon-carbon bonds (1.53 Å)[29], but this is not surprising considering the strained nature of their environment. Mean plane calculations (Table XXXIV) show that the p-bromobenzoate group is not far from planar - there is an angle of 5° between the bromophenyl and carboxyl planes, as opposed to 21° in the p-bromobenzoate derivative in the last chapter. Bond angles (Table XXXV) in the p-bromobenzoate group are normal.

TABLE XXX. POSITIONAL AND ISOTROPIC THERMAL PARAMETERS OF CAMPHOR-1,4-HOMOENOL P-BROMOBENZOATE
(fractional x 10^4, H x 10^3, U x 10^3 $Å^2$)

Atom	x	y	z	Ueq/Uiso
Br	-3868(3)	-6066(2)	-94(1)	84
O(1)	893(12)	-268(9)	-1329(3)	56
O(2)	3485(13)	-1335(11)	-954(4)	77
C(1)	931(17)	2325(12)	-1793(4)	49
C(2)	2068(17)	907(15)	-1558(4)	52
C(3)	3478(20)	436(13)	-1966(4)	64
C(4)	3889(19)	2120(16)	-2165(4)	65
C(5)	2471(25)	2396(16)	-2601(5)	79
C(6)	509(20)	2183(17)	-2346(4)	65
C(7)	2988(20)	3074(14)	-1739(4)	53
C(8)	3288(17)	2100(16)	-1259(4)	63
C(9)	3309(26)	4843(17)	-1747(6)	87
C(10)	-706(20)	2914(17)	-1480(6)	78
C(11)	1762(19)	-1316(16)	-1019(5)	61
C(12)	339(17)	-2411(13)	-794(4)	47
C(13)	-1595(17)	-2434(14)	-923(4)	53
C(14)	-2878(21)	-3488(15)	-719(5)	56
C(15)	-2136(21)	-4566(13)	-373(5)	56
C(16)	-228(22)	-4572(14)	-236(4)	60
C(17)	1055(21)	-3482(13)	-445(4)	59
H(031)	273	-29	-225	57
H(032)	470	-17	-183	57
H(04)	539	233	-225	57
H(051)	265	352	-275	57
H(052)	271	150	-289	57
H(061)	-51	306	-248	57
H(062)	-12	102	-244	57
H(081)	260	262	-93	57
H(082)	476	183	-118	57
H(091)	234	541	-146	57
H(092)	477	518	-166	57
H(093)	289	536	-211	57
H(101)	-187	213	-142	57
H(102)	-118	392	-167	57
H(103)	-19	334	-110	57
H(13)	-216	-154	-117	57
H(14)	-443	-353	-85	57
H(16)	33	-549	0	57
H(17)	261	-347	-33	57

TABLE XXXI. ANISOTROPIC THERMAL PARAMETERS OF CAMPHOR P-BROMOBENZOATE
($U_{ij} \times 10^3$ Å^2)

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Br	112(1)	64(1)	77(1)	-26(1)	-6(1)	15(1)
O(1)	47(5)	58(4)	64(5)	-7(5)	-3(5)	11(4)
O(2)	52(6)	76(6)	103(7)	15(6)	-10(5)	29(5)
C(1)	45(7)	42(6)	58(7)	8(6)	1(7)	3(5)
C(2)	39(6)	61(7)	55(7)	-4(6)	2(6)	2(7)
C(3)	65(10)	58(7)	69(8)	13(7)	4(7)	-4(6)
C(4)	54(8)	86(9)	56(8)	-10(8)	9(8)	8(7)
C(5)	114(13)	67(9)	55(8)	-13(9)	-1(9)	4(7)
C(6)	68(10)	72(8)	55(8)	-9(8)	-19(6)	5(7)
C(7)	68(9)	47(7)	45(8)	-12(7)	3(7)	3(6)
C(8)	50(9)	85(9)	55(8)	0(7)	-6(6)	-7(7)
C(9)	98(13)	69(9)	93(10)	-28(9)	5(9)	-8(8)
C(10)	55(9)	80(8)	99(11)	12(8)	18(8)	27(9)
C(11)	55(10)	66(9)	63(8)	27(8)	-6(7)	-11(7)
C(12)	42(8)	42(6)	58(7)	-3(6)	0(5)	0(6)
C(13)	59(10)	54(7)	46(7)	0(7)	0(6)	2(6)
C(14)	66(9)	52(7)	52(8)	5(7)	-10(6)	10(6)
C(15)	60(10)	54(8)	55(7)	-12(6)	11(7)	-18(7)
C(16)	73(10)	51(7)	55(9)	5(7)	-3(7)	12(6)
C(17)	64(8)	58(7)	54(7)	7(8)	-6(8)	3(6)

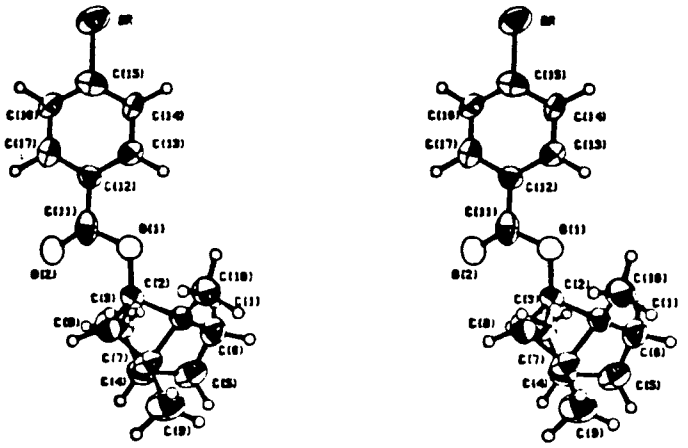


Figure 13. The camphor homoenol p-bromobenzoate molecule

TABLE XXXII. BOND LENGTHS (Å) OF THE NON-HYDROGEN ATOMS IN CAMPHOR HOMOENOL P-BROMOBENZOATE

Bond	Distance	Bond	Distance
Br -C(15)	1.898(12)	C(4) -C(7)	1.528(17)
O(1)-C(2)	1.424(14)	C(5) -C(6)	1.522(21)
O(1)-C(11)	1.354(15)	C(7) -C(8)	1.541(16)
O(2)-C(11)	1.197(13)	C(7) -C(9)	1.523(17)
C(1)-C(2)	1.569(16)	C(11)-C(12)	1.479(18)
C(1)-C(6)	1.509(16)	C(12)-C(13)	1.373(16)
C(1)-C(7)	1.558(17)	C(12)-C(17)	1.394(16)
C(1)-C(10)	1.487(17)	C(13)-C(14)	1.371(17)
C(2)-C(3)	1.511(16)	C(14)-C(15)	1.400(17)
C(2)-C(8)	1.541(16)	C(15)-C(16)	1.361(18)
C(3)-C(4)	1.556(17)	C(16)-C(17)	1.397(17)
C(4)-C(5)	1.536(19)		

TABLE XXXIII. BOND LENGTHS (Å) INVOLVING HYDROGEN ATOMS IN CAMPHOR HOMOENOL P-BROMOBENZOATE

Bond	Distance	Bond	Distance
C(3)-H(031)	1.11	C(9) -H(092)	1.07
C(3)-H(032)	1.05	C(9) -H(093)	1.09
C(4)-H(04)	1.07	C(10)-H(101)	1.05
C(5)-H(051)	1.05	C(10)-H(102)	1.04
C(5)-H(052)	1.09	C(10)-H(103)	1.14
C(6)-H(061)	1.09	C(13)-H(13)	1.08
C(6)-H(062)	1.11	C(14)-H(14)	1.12
C(8)-H(081)	1.09	C(16)-H(16)	1.09
C(8)-H(082)	1.06	C(17)-H(17)	1.11
C(9)-H(091)	1.12		

TABLE XXXIV. MEAN PLANES OF THE P-BROMOBENZOATE MOIETY IN CAMPHOR HOMOENOL P-BROMOBENZOATE

Equations of planes (lX+mY+nZ=p)

plane	l	m	n	p
1	0.2074	-0.6334	-0.7455	2.9216
2	0.1325	-0.6015	-0.7878	3.0019

Deviations from planes (Å)

atom	1	2
C(2)	-0.02(1)	-0.01(1)*
O(1)	-0.009(8)	0.007(8)*
O(4)	0.19(1)	0.006(9)*
C(11)	-0.07(1)	-0.02(1)*
C(12)	0.01(1)*	-0.06(1)
C(13)	0.00(1)*	0.04(1)
C(14)	-0.01(1)*	0.04(1)
C(15)	-0.01(1)*	-0.07(1)
C(16)	-0.01(1)*	-0.17(1)
C(17)	0.00(1)*	-0.18(1)
Br	0.000(1)*	-0.036(1)

*atoms included in plane calculations

Angles between normals to the planes

planes (1) and (2) : 5.3°

TABLE XXXV. BOND ANGLES (°) OF NON-HYDROGEN ATOMS IN CAMPHOR HOMOENOL P-BROMOBENZOATE

Bonds	Angle	Bonds	Angle
C(2)-O(1)-C(11)	118.3(0.9)	C(1) -C(7) -C(8)	88.7(0.9)
C(2)-C(1)-C(6)	115.0(1.0)	C(1) -C(7) -C(9)	122.4(1.2)
C(2)-C(1)-C(7)	80.0(0.8)	C(4) -C(7) -C(8)	106.0(1.0)
C(2)-C(1)-C(10)	114.5(0.9)	C(4) -C(7) -C(9)	117.3(1.2)
C(6)-C(1)-C(7)	107.3(0.9)	C(8) -C(7) -C(9)	121.7(1.2)
C(6)-C(1)-C(10)	115.4(1.1)	C(2) -C(8) -C(7)	81.5(0.8)
C(7)-C(1)-C(10)	119.8(1.0)	O(1) -C(11)-O(2)	122.3(1.3)
O(1)-C(2)-C(1)	115.5(0.9)	O(1) -C(11)-C(12)	111.8(1.1)
O(1)-C(2)-C(3)	119.1(1.0)	O(2) -C(11)-C(12)	125.9(1.3)
O(1)-C(2)-C(8)	123.3(0.9)	C(11)-C(12)-C(13)	123.2(1.1)
C(1)-C(2)-C(3)	103.7(0.9)	C(11)-C(12)-C(17)	116.8(1.1)
C(1)-C(2)-C(8)	88.2(0.9)	C(13)-C(12)-C(17)	120.0(1.2)
C(3)-C(2)-C(8)	101.5(0.9)	C(12)-C(13)-C(14)	122.3(1.2)
C(2)-C(3)-C(4)	96.7(0.9)	C(13)-C(14)-C(15)	117.1(1.3)
C(3)-C(4)-C(5)	106.5(1.1)	Br -C(15)-C(14)	118.1(1.1)
C(3)-C(4)-C(7)	99.5(0.8)	Br -C(15)-C(16)	119.8(1.1)
C(5)-C(4)-C(7)	103.0(1.2)	C(14)-C(15)-C(16)	122.0(1.2)
C(4)-C(5)-C(6)	101.9(0.9)	C(15)-C(16)-C(17)	120.0(1.2)
C(1)-C(6)-C(5)	104.9(1.0)	C(12)-C(17)-C(16)	118.6(1.2)
C(1)-C(7)-C(4)	94.7(0.9)		

From Table XXXV it can be seen that there is evidence for considerable strain in the vicinity of the cyclobutyl ring. The C-C-C angles in the four-membered ring are 80, 88, 81, and 89°, i.e., two opposite angles are significantly smaller than the free cyclobutyl angle[22], 89.3°. These two angles, C(2)-C(1)-C(7) and C(2)-C(8)-C(7), also form the envelope tips of two five-membered rings whose conformations are adapted to allow for the cyclobutyl ring. The C-C-C angles in the five-membered ring are lower than normal (97,99,101,81,106 and 97,99,104,95,80°) and the dihedral angles between the envelope flaps and the remainder of the ring are 117 and 110° (see figure 14), i.e., quite small (plane C(2)-C(3)-C(4)-C(7) has equation -0.7953X + 0.0525Y - 0.6039Z = 1.3643, with atom displacements C(2) 0.05, C(3) -0.08, C(4) 0.09, C(7) -0.06, C(1) 1.12, and C(8) -1.04(1) Å). The envelope tip of the C(1)C(2)C(3)C(4)C(7) ring has shifted from C(7) in the free camphor to C(1) in the 1,4-homoenol derivative. The cyclobutyl ring is fairly tightly folded; the dihedral angles (133 and 129°, see figure 14) are significantly smaller than the free cyclobutyl angle[22] of 162.6°. Deviations from ideal bond angles are less pronounced further away from the four-membered ring.

Bond angles involving hydrogen atoms in their fixed calculated positions are listed in Table XXXVI. Figure 15 presents a stereo packing diagram - there are no unusually short intermolecular contacts and the crystal is held together by van der Waals forces.

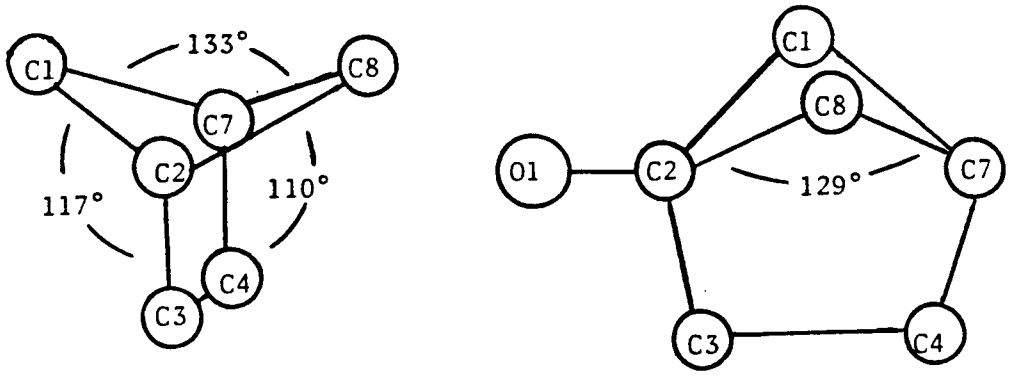


Figure 14. Dihedral angles in the cyclobutyl moiety

TABLE XXXVI. BOND ANGLES (°) INVOLVING HYDROGEN ATOMS IN CAMPHOR HOMOENOL P-BROMOBENZOATE

Bonds	Angle	Bonds	Angle
C(2) -C(3)-H(031)	110	H(081)-C(8) -H(082)	110
C(2) -C(3)-H(032)	114	C(7) -C(9) -H(091)	109
C(4) -C(3)-H(031)	111	C(7) -C(9) -H(092)	113
C(4) -C(3)-H(032)	115	C(7) -C(9) -H(093)	112
H(031)-C(3)-H(032)	109	H(091)-C(9) -H(092)	107
C(3) -C(4)-H(04)	114	H(091)-C(9) -H(093)	106
C(5) -C(4)-H(04)	115	H(092)-C(9) -H(093)	109
C(7) -C(4)-H(04)	118	C(1) -C(10)-H(101)	117
C(4) -C(5)-H(051)	111	C(1) -C(10)-H(102)	104
C(4) -C(5)-H(052)	109	C(1) -C(10)-H(103)	112
C(6) -C(5)-H(051)	112	H(101)-C(10)-H(102)	111
C(6) -C(5)-H(052)	111	H(101)-C(10)-H(103)	107
H(051)-C(5)-H(052)	111	H(102)-C(10)-H(103)	105
C(1) -C(6)-H(061)	113	C(12) -C(13)-H(13)	119
C(1) -C(6)-H(062)	111	C(14) -C(13)-H(13)	118
C(5) -C(6)-H(061)	110	C(13) -C(14)-H(14)	121
C(5) -C(6)-H(062)	111	C(15) -C(14)-H(14)	122
H(061)-C(6)-H(062)	107	C(15) -C(16)-H(16)	120
C(2) -C(8)-H(081)	117	C(17) -C(16)-H(16)	119
C(2) -C(8)-H(082)	119	C(12) -C(17)-H(17)	121
C(7) -C(8)-H(081)	113	C(16) -C(17)-H(17)	120
C(7) -C(8)-H(082)	114		

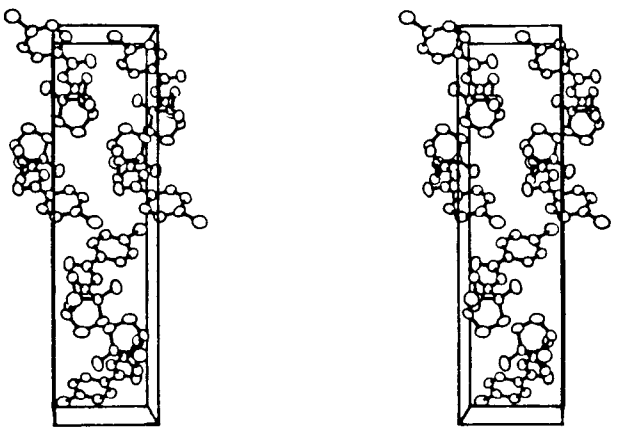


Figure 15. Packing diagram for camphor homoenol p-bromobenzoate

CHAPTER 4

DIFFICULT $P2_1$ STRUCTURES

I. CRYSTAL STRUCTURE OF RAUCUBAINE

Introduction.

The leaves of the plant Rauwolfia salicifolia griseb., a species endemic to Cuba, yield a new alkaloid, raucubaine. The plant was collected in Baracoa, a zone in the Guantanamo province. Spectroscopic data[30] suggested structures of various fragments, but did not allow complete structural elucidation of the alkaloid. In order to obtain the complete structure the compound was subjected to x-ray diffraction analysis.

Data collection.

Preliminary photography showed the crystal to be monoclinic with the 0 k 0 reflections systematically absent when k is odd, indicating the presence of a twofold screw axis and establishing the space group as $P2_1$ (or $P2_1/m$, which could be disregarded as the material is optically active).

The intensity data were collected using nickel-filtered CuKα radiation (λ = 1.54188 Å) and an ω-2θ scan with an ω scan angle of $(0.9 + 0.15 \tan \theta)^\circ$. The horizontal and vertical aperture widths were $(2.0 + 0.5 \tan \theta)$ mm and 4 mm respectively. The intensities of three standard reflections (-5 -2 6, -6 -3 4,

and -2 -3 7) were measured every one hour of x-ray exposure time and were used to scale the data. The same three reflections were checked for orientation every 100 reflections, and reorientation occurred if the difference between observed and calculated scattering vectors was greater than 0.05°. Reorientation occurred only once during the data collection.

Of the 1822 reflections measured in the range $2 \leq \theta \leq 75°$, 1700 (93.3%) had $I/\sigma(I) \geq 3.0$ and were considered observed. The cell parameters were refined by least-squares methods using the $\sin\theta/\lambda$ values of 25 reflections in the range $39 < \theta < 49°$ and are presented with other crystal data in Table XXXVII. Lorentz and polarization corrections were applied.

****

TABLE XXXVII. CRYSTAL DATA FOR RAUCUBAINE

$C_{20}H_{24}N_2O_3$	f.w. = 340.4
Monoclinic	Z = 2
Space group = $P2_1$	F(000) = 364
a = 7.2179(3)	λ = 1.54188 Å
b = 12.8169(3)	V = 849.87 Å^3
c = 9.1996(3) Å	Dc = 1.33 gcm^{-1}
β = 93.040(3) °	μ(Mo radn.) = 0.85 cm^{-1}
	μ(Cu radn.) = 6.8 cm^{-1}

****

Solution

Attempts to solve the crystal structure of raucubaine spanned well over a year. Probably the simplest way of recounting these attempts is in a chronological fashion.

All solution attempts involved the use of direct methods, as the lack of heavy atoms in a molecule this size would render the Patterson map virtually uninterpretable. In the space group $P2_1$, the conventional origin choice involves the assignment of phase 0 or π to two reflections in the h 0 l zone, thereby limiting the origin to one of the four unique screw axes parallel to b (at 0 y 0, 0.5 y 0, 0 y 0.5, or 0.5 y 0.5). A third general reflection preferably of type h 1 l is assigned any phase in order to fix the origin along the chosen screw axis. The origin determining reflections, or any additive combination of the origin determining reflections, may not have parity ggg (e.g. a parity permissible origin set could be u 0 g, u 0 u, and g 1 u). The enantiomorph is fixed by limiting the phase of an additional reflection to the range 0 to π. Further considerations in origin choice should be that the origin determining reflections have strong E's and be involved in as many Σ_2-relationships with other strong E's as possible. The probability of a phase $\phi(h,k,l)$ being determined correctly from Σ_2-relationships depends on the number of contributing Σ_2-relationships, and on the strength of the (h',k',l') and (h-h',k-k',l-l') contributors within each relationship. In order for the symbolic addition routine to phase other reflections correctly, it is especially important that the initial Σ_2-

relationships used (i.e. those involving the origin and symbols) hold true.

Unfortunately, it is not always possible to find two strong h 0 l reflections that are involved in many Σ_2-relationships, as is required in $P2_1$, and the elucidation could present immediate difficulties.

In this structure the first reflections chosen for the origin were 1 0 6, 7 0 -3 and 4 1 5 (origin 1 in Table XXXVIII). If the E's are ranked according to magnitude, these reflections have ranks 44, 38 and 2, respectively. The strengths of the zonal reflections are somewhat lower than desirable; however these were the strongest h 0 l reflections available. This origin was discarded upon discovering that it was not involved in any of the 77 Σ_2-relationships derived from the 50 strongest E's.

Based on the 168 Σ_2-relationships from the 65 strongest E's, the next origin chosen was origin 2 in Table XXXVIII. Working through the Σ_2-relationships by hand, as many reflections were phased from the origin as possible before the first symbol assignment was made to the reflection with the most Σ_2-contributors of reasonable strength. Phasing was continued manually and the second and third symbols were selected in a similar manner to give a starting symbol set of 1 9 -6 (rank 15), 1 2 -7 (rank 18), and 3 10 3 (rank 13) (symbol set 'a' in Table XXXVIII). Using two values for each of the symbols, and 9545 Σ_2-relationships derived from the 261 strongest E's, eight sets of phases were generated which were in fact two enantiomorphic groups of four sets each, the enantiomorph being

TABLE XXXVIII. ORIGIN AND SYMBOL SETS FOR RAUCUBAINE.

Origins

	hkl	rank	hkl	rank	hkl	rank
1	1 0 6	44	7 0 -3	38	4 1 5	2
2	1 0 6	44	1 0 -9	60	6 1 -3	4
3	1 0 6	44	1 0 -9	60	4 1 5	2
4	1 0 6	44	1 0 -9	60	5 1 4	30
5	2 9 3	15	1 7 5	29	3 10 -3	40
6	-3 -13 -5	2	1 -10 3	146	0 6 -5	152
7	4 1 5	1	3 9 2	53	2 2 -3	100
8	5 7 5	43	2 9 3	58	3 10 -3	155

Symbols

	hkl	rank	hkl	rank	hkl	rank
a	1 9 -6	15	1 2 -7	18	3 10 3	13
b	1 9 9	7	6 3 -4	8	3 10 3	13
c	3 10 3	13	1 2 -7	18	0 7 6	6
d	6 1 -3	4	1 2 -7	18	0 7 6	6
e	6 3 -4	8	1 2 -7	18	0 7 6	6
f	1 9 -6	15	1 2 -7	18	0 7 6	6
g	4 1 5	2	1 2 -7	18	0 7 6	6
h	3 13 -5	1	4 0 0	21		
i	3 13 -5 2 9 3	1 30	2 -3 8	14	2 -9 3	29
j	3 13 -5	1	6 0 -4	10		
k	2 9 3	15	4 0 0	21		
m	3 13 -5 2 3 -8	1 145	1 10 -4	14	4 0 0	21

determined by the starting phase of the 1 9 -6 reflection.

For each set of phases various criteria exist that allow discrimination of the correct set from the incorrect sets, including average consistencies, various R factors, the number of phases determined, and the number of Σ_2-relationships actually used. For the correct set, the values for most if not all of these criteria should stand out significantly above those for the incorrect sets (significantly below for the R factors). The number of phases determined and one of the R factors ('R-Karle') are the more sensitive indicators. Of the eight sets of phases determined, none were obviously correct. In an attempt to improve the discrimination between the sets, the symbols were given four starting values rather than two (except for 1 9 -6, which was limited to two starting values within the range 0 to π to determine the enantiomorph), and 32 sets of phases were generated, the best of which did not yield any recognizable features on an E-map.

At this stage it was thought that perhaps an insufficient number of E's (and hence an insufficient number of Σ_2-relationships) was being used to determine the structure correctly. With the same origin and symbols, 380 E's greater than 1.25 were used to generate eight starting sets, but the E-map from the best set showed no improvement.

The phasing procedure in the programme TANS involves incrementing the number of E's included in the various phase determining cycles. As it was noticed that the initial phasing was very slow (i.e. few phases were succesfully assigned in the first ten of fifteen cycles) a 'lump refinement' was attempted

in which all 380 E's were introduced at the first cycle. Although convergence was more rapid, the final results were no better. As attempted earlier, the number of sets generated was increased to 32, but still no significant discrimination between the sets could be observed. A few E-maps were calculated from the slightly better sets without success. Before rejecting this origin, it was tried with a different symbol set (set b in Table XXXVIII) but there were still no useful results.

To check on the origin choice, a Σ_2-listing involving the highest 129 E's was inspected, using $\Sigma\{E(h,k,l)E(h',k',l')E(h-h',k-k',l-l')\}$ as a parameter to indicate the strength and Σ_2-involvement of each reflection. It was decided that the 4 1 5 reflection was as good an origin choice as 6 1 -3, and it was reused in origin/symbol set 3a (see Table XXXVIII) to generate 48 starting sets, none of which refined well.

The 4 0 0 reflection could be assigned phase π from various Σ_1-relationships and several attempts at solution including 4 0 0 in the starting set (starting sets 3c, 3d, 3e, 3f and 4g) gave no useful results.

The above calculations were performed in May and June of 1978. In July, 1978, our laboratory group started using K-curves (see chapter two, page 15) as an alternate method of calculating E's, and in the hope that it might make a difference, the raucubaine data were renormalized using a K-curve. Further attempts at solution were still in vain, but later an error in the multiplicity of zonal reflections was discovered in the renormalization procedure. This was corrected in January, 1979. At this time our laboratory had also started using the MULTAN

programme for direct methods, which features automatic origin selection and is in general more powerful with more sensitive correctness criteria than some other available programmes.

410 E's greater than 1.20 obtained from the K-curve were input into the MULTAN programme. The origin MULTAN selected was unconventional: it assigned phase 0 to 2 9 3 (rank 15), starting phases $\pm \pi/4$ to 1 7 5 (rank 29) and starting phase $\pi/4$ to 3 10 -3 (rank 40), with symbols 3 13 -5 and 4 0 0 (ranks 1 and 21), i.e., starting set 5h in Table XXXVIII. Presumably if the zonal reflections would have been sufficiently strong they would have appeared in the MULTAN starting set. The rationale for being able to use general reflections as origin determining reflections lies in the following: a phase may lie in one of four quadrants: quadrant 1, 2, 3, or 4. A shift in origin (e.g. from one twofold screw axis to another) would result in the shifting of a phase of a general reflection from quadrant 1 to quadrant 3 or from quadrant 4 to quadrant 2. Restricting the phase of this general reflection to quadrants 1 and 4 then renders it origin determining. However, the phase may move from, say, quadrant 1 to quadrant 4 upon changing the enantiomorph. Restricting the phase to one quadrant therefore also determines the enantiomorph. This argument may also be extended to other space groups.

In this origin selected by MULTAN, the two zonal reflections have been replaced by 1 7 5, which is restricted to quadrants 1 and 2, and 3 10 -3, which is restricted to quadrant one and hence is also enantiomorph determining. The assignment of zero phase to a third reflection of proper parity (2 9 3)

completes the origin and enantiomorph definition.

E-maps were calculated from the apparently most correct sets of phases generated, but each had only a single large peak and offered no structural information.

A crystallographic device sometimes used to solve difficult structures is the reduction of the symmetry of the system in which the structure is being solved, i.e., essentially to ignore one of the symmetry elements present. This is most often used when it is the symmetry element that is the source of the difficulty (e.g. large parts of the structure lying near a mirror plane, pseudo centre of symmetry, etc.) and hence its removal might eliminate the confusion. It is doubtful that the $P2_1$ axis was related to the source of trouble in raucubaine, but at this stage (February 1979) it seemed that trying to solve the structure in a lower symmetry space group might be worthwhile.

The only symmetry element present in $P2_1$ is the twofold screw axis, and its removal places the structure in the lowest symmetry space group, P1. P1 is triclinic with one asymmetric unit, which would hopefully incorporate two molecules of raucubaine related by a twofold screw. The (h,-k,l) reflections (which were not collected for the monoclinic system) were added to the data set with F(h,-k,l) = F(h,k,l). The highest 500 E's calculated by following the minimum profile of a K-curve were input into the MULTAN programme. The origin was selected by assigning zero phase to three reflections: 3 -13 -5 (rank 2), 1 -10 3 (rank 146) and 0 6 -5 (rank 152); and four symbols were used: 3 13 -5, 2 -3 8, 2 -9 3 and 2 9 3 (origin/symbol set 6i). All of the resultant sets of phases showed identical values for

the correctness criteria (i.e., no discrimination) and an E-map from one of the sets produced only a single large peak.

The ranks of the origin determining reflections were very high, so an origin and symbol set was chosen manually by examining a Σ_2-listing (-3 -13 5, 0 -7 6 and 2 -9 3, with symbols 3 10 3, 4 0 0 and 2 -3 8), and with this origin set eight sets of phases were generated using the TANS programme, but once more the E-maps from the best two sets revealed nothing but single large peaks.

With the lack of success in P1, it was hard to pinpoint the area of difficulty. There could perhaps have been something inherently wrong with the E's, or with their renormalization. For this reason, a few attempts were made using unrenormalized E's. The K-curve was redone without the renormalization, and the 500 highest E's were input into MULTAN, which selected origin/symbol set 5j and accepted the 4 0 0 Σ_1-relationship as having phase π, but the sets of phases produced showed poor values for the correctness criteria, and the E-maps calculated produced single large peaks. Using this same origin, TANS produced no better results.

The highest E is 3 13 -5 and it has value of about 3.8, which is considerably larger than the next few E's. If there is some peculiarity among the E's, this strongest E may well be affected, and were it initially incorrectly phased, it would in turn affect the phasing of the remaining E's. This was tested by omitting the strongest E in the direct methods procedure. Without the 3 13 -5 reflection, MULTAN chose origin/symbol set 7k, but no useful results were obtained. A TANS run with the

same origin was also unsuccesful.

In continued efforts to investigate alternate E distributions, a set of E's was also generated by following the K-curve instead of using its minimum profile, and was input into MULTAN. MULTAN chose origin/symbol set 8h, but there was no outstanding solution.

The correctness criteria that MULTAN uses are a figure of merit (FOM) (which should approach unity for the correct set of phases), an R-factor, and a $\Psi(0)$ parameter (read 'psi-zero'), which is essentially an R-factor based on the closeness of fit of the lowest E's, and is very sensitive to the correct solution. Up to this stage (July 1979) the $\Psi(0)$ test had not been included.

Incorporating the $\Psi(0)$ test, 500 renormalized E's were input into the MULTAN programme, which accepted phase π for the 4 0 0 Σ_1-relationship, and chose origin/symbol set 5j.

The resulting set of phases with the lowest $\Psi(0)$ value was also the set with the highest R and lowest FOM, and so was unlikely to be correct. The run was repeated with fifty $\Psi(0)$ reflections being the fifty absolute lowest E's rather than the lowest fifty of the 500 E's input, and purposely not accepting the 4 0 0 reflection as phased. This time, using origin/symbol set 5h, a set of phases was generated that had not only the lowest $\Psi(0)$ value, but also the lowest R, yet the discrimination was not very good. In an attempt to improve this discrimination, still using 500 E's and the fifty lowest E's for the $\Psi(0)$ test, an imposition on MULTAN forced it to choose four rather than two symbols. The resulting origin/symbol set was 5m in Table

XXXVIII, and this time the lowest $\Psi(0)$ value still corresponded to the lowest R and both stood out a little better than in the previous MULTAN run. An E-map calculated from this set revealed the chemically-reasonable positions of 25 non-hydrogen atoms, and the structure was finally solved.

Post-solution analysis.

Once the structure was solved, the correct set of phases was examined with the hope of finding possible trouble spots that rendered the solution so difficult. The first oddity is that the 3 10 -3 reflection, which is supposedly enantiomorph determining, changed sign (or enantiomorph) from +45 to -83° in the refinement of phases. The significance of this is not immediately obvious, however the results were affected. In the previous MULTAN run, the numeric values for the starting set corresponding to those that produced the correct solution, produced a set of phases in which the enantiomorph determining reflection (still 3 10 -3) did not flip - however this particular set of phases was incorrect. Also in this previous MULTAN run, the best set of phases (lowest $\Psi(0)$ and R) did not correspond to the correct set.

It is possible that the 3 10 -3 reflection is involved in some misleading Σ_2-relationships early in the phase determining procedure, and that by forcing numerical values on extra symbols, these misleading relationships are overcome. From the numerical values of the origin/symbol set that led to the

correct solution (set 5m), the phases for other reflections were calculated manually from their Σ_2-relationships. It was found that two of the symbols were not involved in many Σ_2-relationships and that the phasing proceeded rather slowly. The first reflection to be assigned a phase completely different to that in the final correct set was 2 3 8, and its phase was directly derived from that of 3 10 -3. The next few inconsistent phases were those of 1 10 3, also derived from 3 10 -3, and 4 6 -5, derived from 1 10 3. These are all relatively strong reflections, and their being incorrectly phased would definitely decrease the probability of solving the structure.

MULTAN uses a convergence tree (or map) from which it determines the order in which the reflections are phased. The phasings of 25 reflections at the root of the convergence tree were examined using both the starting and flipped value of the 3 10 -3 phase. A sensitive measure of consistency is the parameter $\cos(\phi_1 + \phi_2 - \phi_3)$, where ϕ_1, ϕ_2, and ϕ_3 are the phases involved in the Σ_2-relationship. This parameter should be 1.0 for a correctly phased reflection. It was found that for the phases that were dependent on the 3 10 -3 phase, this consistency was generally greater than 0.95 if the flipped value of the 3 10 -3 phase was used (-83°) and around 0.5 if the starting value (45°) was used.

The difficulties arising from this enantiomorph determining reflection may not be the only reasons for the difficulties encountered with this structure; there may have been more reflections that were involved in poor Σ_2-relationships, or perhaps there were not sufficient Σ_2-relationships to allow

straightforward solution.

In conclusion, the circumstances that led to success in the solution of raucubaine were the use of a sensitive discriminator (the $\Psi(0)$ test), the use of four symbols in MULTAN's symbolic addition routine (thereby essentially increasing computing power and discrimination), and perseverance.

Refinement

All 25 non-hydrogen atoms were initially assigned carbon scattering factors, and after three full-matrix least-squares refinement cycles, the atoms could easily be identified as carbon, nitrogen, or oxygen from their temperature factors. Three additional isotropic least-squares cycles and a difference map confirmed the correctness of the atom-type assignment. After an anisotropic refinement cycle a difference map revealed all the hydrogen positions, and several more cycles with unit weights (hydrogens with isotropic temperature factors, non-hydrogens with anisotropic temperature factors) lowered R to 0.067. For several intense low-theta reflections the observed structure factors were consistently less than the calculated structure factors. When a least-squares cycle with a polynomial weighting scheme failed to change this situation, the need for an extinction correction became apparent.

There are two types of extinction, primary and secondary, both of which attenuate the diffracted x-ray beam when the crystal is set at the Bragg angle for a reflection. Primary

extinction is due to attenuation of the incident beam inside the crystal as a result of destructive interference from multiply reflected (and hence perfectly out of phase) incident rays. Primary extinction effects are significant in crystals that are very close to ideally perfect, and are negligible in most cases. Secondary extinction is more common in crystals of high quality (not ideally perfect), and occurs when a strongly reflecting plane reflects a substantial portion of the incident beam from the first few lattice planes, thereby denying the rest of the crystal the full incident intensity, and thus the diffracted beam is attenuated. Secondary extinction is more pronounced at low angle reflections where the intensities are inherently greater. Often only a few reflections are seriously affected, and in practice it is quite common simply to exclude these from the refinement. Isotropic extinction may be measured in terms of g, the secondary extinction coefficient, where $Ic/Io = 1 + 2gIc$, and g is characteristic of the crystal for a given radiation. Anisotropic extinction is more difficult to handle, but in raucubaine there is no evidence for the extinction being anisotropic.

Four least-squares refinement cycles refining only g and the scale factor lowered R from 0.054 to 0.049, showing that the extinction effect is definitely appreciable.

The structure was refined until convergence using a polynomial weighting scheme with coefficients that were updated after every cycle. The final coefficients used are A = -0.0493, B = 0.06254, C = -0.004685, and D = 0.000210. The final R and Rw are 0.046 and 0.066 respectively (0.051 and 0.066 including the

unobserved reflections). The final g value is 1.627×10^3.

Efforts to determine the absolute configuration were unsuccessful, as might well be expected as the anomalous dispersion corrections for the atoms carbon, nitrogen, oxygen, and hydrogen are very small. Parallel refinements of both enantiomorphs with anomalous dispersion corrections applied, using the Fo data collected, yielded no significant differences in R values. Structure factors were calculated for both the enantiomorphs (including the anomalous dispersion corrections) and the twenty reflections that produced the greatest differences in Fc between the two enantiomorphs were recorded. The intensities of the Friedel pairs of these twenty reflections were recollected very accurately. The signs of the differences between Fo(hkl) and Fo(-(hkl)) were compared to the signs of the differences between Fc(hkl) and Fc(-(hkl)) for these twenty reflections. Had the signs matched for each Friedel pair, the original enantiomorph chosen would probably have been correct. Had the signs been consistently opposite for each Friedel pair, the correct enantiomer would have been enantiomorphic to that originally chosen. Unfortunately, in several attempts, about 50% of the Friedel pairs had matched signs, and the remaining Friedel pairs had opposite signs, offering no conclusion.

Final atomic and thermal parameters for the original enantiomorph chosen appear in Tables XXXIX and XL.

TABLE XXXIX. POSITIONAL AND ISOTROPIC THERMAL PARAMETERS FOR RAUCUBAINE

(fractional x 10^4, H x 10^3, U x 10^3 Å^2)

Atom	x	y	z	Ueq/Uiso
O(1)	1116(4)	4770	5140(3)	38
O(2)	-1156(4)	5339(3)	6484(3)	46
C(1)	-1066(6)	4300(5)	10212(5)	49
C(2)	-1807(8)	4045(7)	11552(6)	67
C(3)	-1200(8)	3173(7)	12299(6)	68
C(4)	202(8)	2559(5)	11811(5)	60
C(5)	972(6)	2813(4)	10507(4)	42
C(6)	289(6)	3677(4)	9696(4)	39
C(7)	1326(5)	3681(4)	8302(4)	34
C(8)	3134(5)	3189(4)	8898(4)	38
C(9)	3906(9)	1854(5)	10774(6)	59
N(1)	2470(5)	2341(4)	9816(4)	46
C(10)	1795(5)	4769(4)	7657(4)	34
C(11)	3664(5)	4822(4)	6884(4)	38
C(12)	5235(6)	4104(5)	7421(5)	45
C(13)	4449(5)	3015(4)	7658(4)	42
N(2)	3488(5)	2647(4)	6267(4)	42
C(14)	1704(7)	2160(4)	6513(5)	43
C(15)	334(6)	2897(4)	7205(4)	39
C(16)	3473(6)	3382(4)	5020(5)	42
C(17)	3089(5)	4547(4)	5306(4)	39
C(18)	409(5)	5021(4)	6415(4)	36

continued...

C(19)	4037(6)	5244(4)	4204(5)	46
O(3)	3684(6)	6299(4)	4579(4)	55
C(20)	3340(9)	5037(6)	2640(6)	59
H(1)	-146(7)	493(5)	965(6)	48(13)
H(2)	-274(12)	466(8)	1190(9)	93(24)
H(3)	160(10)	293(7)	1328(8)	76(21)
H(4)	67(9)	193(6)	1230(7)	64(17)
H(8)	374(6)	372(4)	955(5)	33(11)
H(91)	455(10)	249(6)	1137(8)	75(21)
H(92)	352(10)	142(7)	1150(8)	76(21)
H(93)	481(11)	150(7)	1020(8)	80(22)
H(10)	170(5)	529(3)	844(4)	21(9)
H(11)	407(6)	555(4)	691(4)	25(9)
H(121)	573(8)	444(5)	828(6)	50(14)
H(122)	628(9)	410(6)	670(7)	68(18)
H(13)	534(8)	248(5)	788(6)	47(14)
H(141)	119(7)	188(5)	560(6)	47(14)
H(142)	191(7)	159(5)	713(5)	39(12)
H(151)	-30(8)	322(5)	648(7)	50(14)
H(152)	-51(7)	245(4)	772(6)	42(13)
H(161)	269(7)	310(5)	429(6)	42(12)
H(162)	469(9)	338(5)	450(7)	50(15)
H(19)	536(8)	513(5)	443(6)	55(15)
H(201)	396(8)	549(5)	189(6)	56(15)
H(202)	359(13)	428(8)	248(9)	90(25)
H(203)	207(10)	516(6)	250(6)	61(17)
H(O)	469(14)	665(9)	436(10)	103(30)

TABLE XL. ANISOTROPIC THERMAL PARAMETERS FOR RAUCUBAINE

(U_{ij} x 10^3 Å^2)

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1)	37(1)	40(1)	35(1)	-2(1)	-1(1)	0(1)
O(2)	38(1)	50(2)	51(2)	7(1)	2(1)	6(1)
C(1)	41(2)	63(3)	43(2)	1(2)	2(2)	-1(2)
C(2)	54(3)	99(5)	49(3)	0(3)	10(2)	-8(3)
C(3)	62(3)	99(5)	45(2)	-9(3)	9(2)	8(3)
C(4)	72(3)	64(3)	42(2)	-21(3)	-8(2)	14(2)
C(5)	49(2)	37(2)	39(2)	-11(2)	-6(1)	4(2)
C(6)	42(2)	42(2)	34(2)	-6(2)	-1(1)	0(1)
C(7)	36(2)	30(2)	34(2)	1(1)	-3(1)	0(1)
C(8)	41(2)	34(2)	38(2)	4(2)	-6(1)	-1(2)
C(9)	74(3)	48(3)	54(3)	7(3)	-16(2)	13(2)
N(1)	57(2)	39(2)	40(2)	2(2)	-11(1)	7(1)
C(10)	41(2)	29(2)	33(2)	-5(1)	0(1)	-5(1)
C(11)	40(2)	32(2)	41(2)	-5(2)	-2(1)	0(2)
C(12)	34(2)	48(2)	52(2)	-4(2)	-4(2)	-5(2)
C(13)	38(2)	44(2)	44(2)	9(2)	-3(1)	1(2)
N(2)	48(2)	35(2)	42(2)	4(1)	1(1)	-5(1)
C(14)	56(2)	28(2)	45(2)	-9(2)	-2(2)	-5(2)
C(15)	44(2)	36(2)	38(2)	-10(2)	-5(1)	0(2)
C(16)	48(2)	37(2)	40(2)	1(2)	1(2)	-7(2)
C(17)	35(2)	43(2)	38(2)	-1(2)	4(1)	-4(2)
C(18)	41(2)	27(2)	39(2)	-2(1)	3(1)	1(1)
C(19)	45(2)	45(2)	49(2)	-12(2)	10(2)	3(2)
O(3)	67(2)	34(2)	65(2)	-10(2)	18(2)	4(1)
C(20)	71(3)	61(3)	47(2)	-11(3)	11(2)	6(2)

Discussion.

The molecular structure, shown in Figure 16 with the atomic labelling scheme, is a new type of indole alkaloid with interesting biosynthetic aspects that will not be discussed here. Figure 17 shows a stereoview of the molecule illustrating a cage-like framework. Excluding the dihydroindole group, the central cage consists of three six-membered rings and a five-membered ring so connected as to form a 'bowl' of which the open face is the nine-membered ring C(7)-C(15)-C(14)-N(2)-C(16)-C(17)-O(1)-C(18)-C(10). The C(10)-C(11)-C(12)-C(13)-C(8)-C(7) ring has a slightly distorted chair conformation, while the other two six-membered rings have slightly skewed boat conformations.

Mean plane calculations (Table XLI) show that the five-membered ring has a skew conformation: C(10) and C(11) are both 0.24 Å away from the mean plane of the ring including O(2), but on opposite sides. The dihydroindole group is planar if C(8) and C(9) are not included. In this way the greatest deviation of any atom in the plane C(1)-C(7),N(1) from the mean plane is 0.057 Å for C(4), whereas C(8) and C(9) are 0.66 and 0.73 Å away from the mean plane on the same side; i.e, the envelope tip of the dihydroindole five-membered ring is C(8).

Bond distances are listed in Tables XLII and XLIII and have reasonable values. The C(7)-C(10) and C(7)-C(15) bonds have lengths that are significantly greater (1.559(5) and 1.570(5) Å, respectively) than normal sp^3-sp^3 carbon-carbon bonds, but occurences such as these are not uncommon in strained systems.

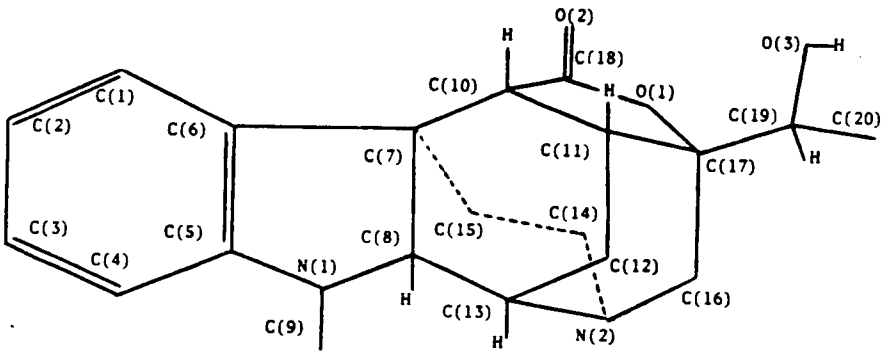


Figure 16. Raucubaine: structure and atomic labelling scheme

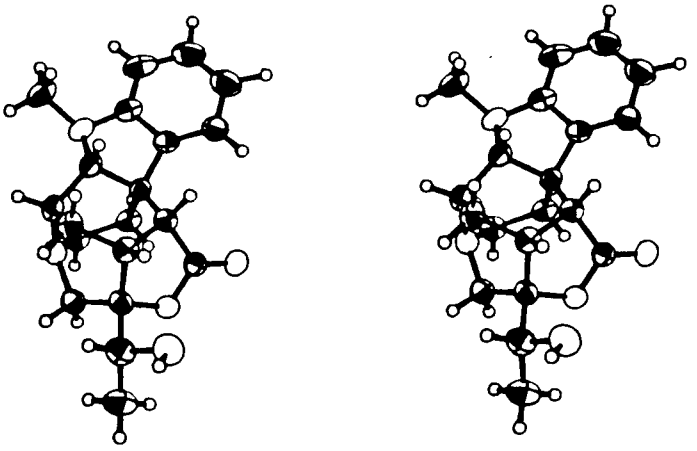


Figure 17. Stereoview of the raucubaine molecule

TABLE XLI. MEAN PLANE CALCULATIONS IN RAUCUBAINE

Equation of plane: $(lX+mY+nZ=p)$

plane	l	m	n	p
1	-0.6517	-0.5997	-0.4643	-6.7818
2	0.2347	0.9641	-0.1241	5.4737

Deviations from plane (Å)

plane 1		plane 2	
O(1)*	-0.036(1)	C(1)*	-0.053(5)
O(2)*	0.115(4)	C(2)*	-0.039(7)
C(10)*	-0.238(5)	C(3)*	0.052(7)
C(11)*	0.242(5)	C(4)*	0.057(6)
C(17)*	0.003(5)	C(5)*	0.014(5)
C(18)*	-0.005(5)	C(6)*	-0.008(5)
		C(7)*	0.051(4)
		C(8)	-0.656(4)
		C(9)	-0.734(6)
		N(1)	-0.054(4)

*atoms included in mean plane calculation

TABLE XLII. BOND LENGTHS OF NON-HYDROGEN ATOMS IN RAUCUBAINE

Bond	Length(Å)	Bond	Length(Å)
O(1)-C(17)	1.453(4)	C(8)-C(13)	1.538(6)
O(1)-C(18)	1.343(5)	C(9)-N(1)	1.464(6)
O(2)-C(18)	1.206(5)	C(10)-C(11)	1.559(5)
C(1)-C(2)	1.408(7)	C(10)-C(18)	1.513(5)
C(1)-C(6)	1.366(7)	C(11)-C(12)	1.522(6)
C(2)-C(3)	1.372(11)	C(11)-C(17)	1.530(5)
C(3)-C(4)	1.376(10)	C(12)-C(13)	1.527(7)
C(4)-C(5)	1.387(7)	C(13)-N(2)	1.499(5)
C(5)-C(6)	1.410(6)	N(2)-C(14)	1.460(6)
C(5)-N(1)	1.419(6)	N(2)-C(16)	1.484(6)
C(6)-C(7)	1.519(5)	C(14)-C(15)	1.531(6)
C(7)-C(8)	1.526(5)	C(16)-C(17)	1.544(6)
C(7)-C(10)	1.559(5)	C(17)-C(19)	1.539(6)
C(7)-C(15)	1.570(5)	C(19)-O(3))	1.422(6)
C(8)-N(1)	1.472(6)	C(19)-C(20)	1.521(7)

TABLE XLIII. BOND LENGTHS INVOLVING HYDROGEN ATOMS IN RAUCUBAINE

Bond	Length(Å)	Bond	Length(Å)
C(1)-H(1)	0.99(6)	C(13)-H(13)	0.96(6)
C(2)-H(2)	1.10(9)	C(14)-H(141)	0.97(6)
C(3)-H(3)	1.01(8)	C(14)-H(142)	0.94(6)
C(4)-H(4)	0.97(8)	C(15)-H(151)	0.89(6)
C(8)-H(8)	0.99(5)	C(15)-H(152)	0.98(6)
C(9)-H(91)	1.08(8)	C(16)-H(161)	0.93(6)
C(9)-H(92)	0.92(8)	C(16)-H(162)	1.02(6)
C(9)-H(93)	0.97(8)	C(19)-H(19)	0.98(6)
C(10)-H(10)	0.99(4)	O(3))-H(O)	0.89(11)
C(11)-H(11)	0.98(5)	C(20)-H(201)	1.03(6)
C(12)-H(121)	0.96(6)	C(20)-H(202)	1.00(10)
C(12)-H(122)	1.03(7)	C(20)-H(203)	0.93(7)

Bond angles (Tables XLIV and XLV) are as reasonable as could be expected given the constraints of the cage.

A stereo packing diagram is presented in Figure 18. The molecules are strung together along the twofold screw axis (b) by O(3)-H...N(2) hydrogen bonds. The hydrogen bond length O(3)...N(2) is 2.815(5) Å.

The structure is consistent with previous spectral data[30].

TABLE XLIV. BOND ANGLES OF THE NON-HYDROGEN ATOMS IN RAUCUBAINE

Bonds	Angle(deg)	Bonds	Angle(deg)
C(17)-O(1)-C(18)	111.8(3)	C(10)-C(11)-C(12)	118.2(3)
C(2)-C(1)-C(6)	118.7(5)	C(10)-C(11)-C(17)	103.0(3)
C(1)-C(2)-C(3)	120.1(6)	C(12)-C(11)-C(17)	109.2(3)
C(2)-C(3)-C(4)	121.6(5)	C(11)-C(12)-C(13)	108.8(3)
C(3)-C(4)-C(5)	118.9(5)	C(8)-C(13)-C(12)	102.8(4)
C(4)-C(5)-C(6)	119.8(5)	C(8)-C(13)-N(2)	113.8(3)
C(4)-C(5)-N(1)	129.6(4)	C(12)-C(13)-N(2)	109.0(3)
C(6)-C(5)-N(1)	110.6(4)	C(13)-N(2)-C(14)	112.0(3)
C(1)-C(6)-C(5)	120.8(4)	C(13)-N(2)-C(16)	116.3(3)
C(1)-C(6)-C(7)	133.2(4)	C(14)-N(2)-C(16)	114.9(3)
C(5)-C(6)-C(7)	106.0(4)	N(2)-C(14)-C(15)	113.3(3)
C(6)-C(7)-C(8)	98.4(3)	C(7)-C(15)-C(14)	112.3(3)
C(6)-C(7)-C(10)	116.8(3)	N(2)-C(16)-C(17)	118.5(3)
C(6)-C(7)-C(15)	108.2(3)	O(1)-C(17)-C(11)	105.6(3)
C(8)-C(7)-C(10)	107.9(3)	O(1)-C(17)-C(16)	110.9(3)
C(8)-C(7)-C(15)	108.6(3)	O(1)-C(17)-C(19)	106.5(3)
C(10)-C(7)-C(15)	115.4(3)	C(11)-C(17)-C(16)	110.0(3)
C(7)-C(8)-N(1)	102.3(3)	C(11)-C(17)-C(19)	112.6(3)
C(7)-C(8)-C(13)	110.0(3)	C(16)-C(17)-C(19)	111.0(3)
N(1)-C(8)-C(13)	123.5(4)	O(1)-C(18)-O(2)	121.8(3)
C(5)-N(1)-C(8)	102.8(3)	O(1)-C(18)-C(10)	110.0(3)
C(5)-N(1)-C(9)	116.4(4)	O(2)-C(18)-C(10)	128.1(4)
C(8)-N(1)-C(9)	114.7(4)	C(17)-C(19)-O(3))	107.5(4)
C(7)-C(10)-C(11)	115.1(3)	C(17)-C(19)-C(20)	112.7(4)
C(7)-C(10)-C(18)	109.2(3)	O(3))-C(19)-C(20)	109.9(4)
C(11)-C(10)-C(18)	101.5(3)		

TABLE XLV. BOND ANGLES INVOLVING HYDROGEN ATOMS IN RAUCUBAINE

Bonds	Angle(deg)
C(2)-C(1)-H(1)	122(3)
C(6)-C(1)-H(1)	119(3)
C(1)-C(2)-H(2)	111(5)
C(3)-C(2)-H(2)	129(4)
C(2)-C(3)-H(3)	127(5)
C(4)-C(3)-H(3)	111(5)
C(3)-C(4)-H(4)	124(4)
C(5)-C(4)-H(4)	117(4)
C(7)-C(8)-H(8)	106(3)
N(1)-C(8)-H(8)	108(3)
C(13)-C(8)-H(8)	106(3)
N(1)-C(9)-H(91)	105(4)
N(1)-C(9)-H(92)	118(5)
N(1)-C(9)-H(93)	110(5)
H(91)-C(9)-H(92)	103(6)
H(91)-C(9)-H(93)	110(7)
H(92)-C(9)-H(93)	111(7)
C(7)-C(10)-H(10)	107(2)
C(11)-C(10)-H(10)	114(2)
C(18)-C(10)-H(10)	110(2)
C(10)-C(11)-H(11)	107(2)
C(12)-C(11)-H(11)	111(2)
C(17)-C(11)-H(11)	108(2)
C(11)-C(12)-H(121)	103(3)
C(11)-C(12)-H(122)	111(4)
C(13)-C(12)-H(121)	115(4)
C(13)-C(12)-H(122)	112(4)
H(121)-C(12)-H(122)	106(5)

Bonds	Angle(deg)
C(8)-C(13)-H(13)	112(3)
C(12)-C(13)-H(13)	116(3)
N(2)-C(13)-H(13)	103(3)
N(2)-C(14)-H(141)	109(3)
N(2)-C(14)-H(142)	108(3)
C(15)-C(14)-H(141)	111(3)
C(15)-C(14)-H(142)	109(3)
H(141)-C(14)-H(142)	106(5)
C(7)-C(15)-H(151)	112(4)
C(7)-C(15)-H(152)	110(3)
C(14)-C(15)-H(151)	107(4)
C(14)-C(15)-H(152)	106(3)
H(151)-C(15)-H(152)	109(5)
N(2)-C(16)-H(161)	107(4)
N(2)-C(16)-H(162)	113(3)
C(17)-C(16)-H(161)	113(4)
C(17)-C(16)-H(162)	104(4)
H(161)-C(16)-H(162)	100(5)
C(17)-C(19)-H(19)	104(4)
O(3))-C(19)-H(19)	106(4)
C(20)-C(19)-H(19)	116(3)
C(19)-O(3))-H(O)	105(7)
C(19)-C(20)-H(201)	114(3)
C(19)-C(20)-H(202)	105(5)
C(19)-C(20)-H(203)	112(4)
H(201)-C(20)-H(202)	111(6)
H(201)-C(20)-H(203)	106(5)
H(202)-C(20)-H(203)	109(7)

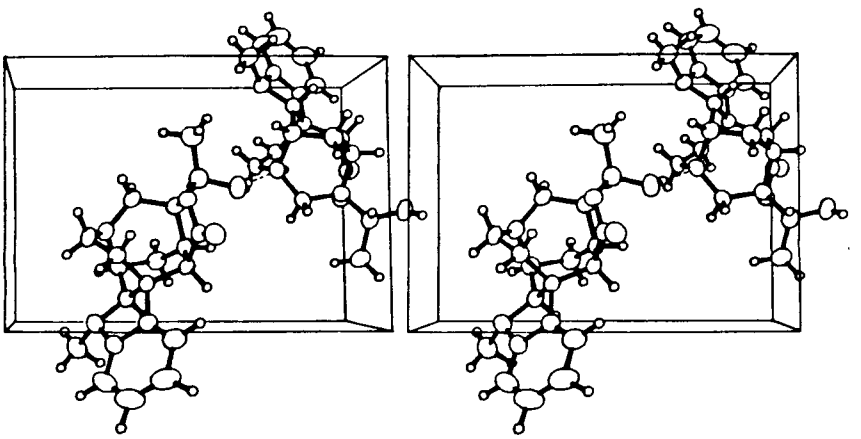
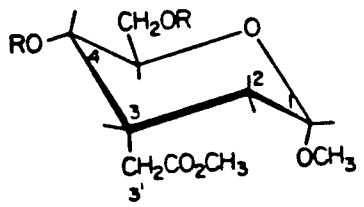


Figure 18. Stereo packing diagram for raucubaine

II. CRYSTAL STRUCTURE OF METHYL 3-C-(CARBOMETHOXYMETHYL)-4,6-DI-O-p-CHLOROBENZOYL-2,3-DIDEOXY-α-D-RIBO-HEXOPYRANOSIDE

Introduction and previous attempts at solution

After the success with raucubaine, it seemed worthwhile to apply any freshly gained experience to another difficult $P2_1$ structure - that of the title compound (1, R = ClC_6H_4CO-).



1

Following initial investigations by Dr. David Hughes[31] in 1967, this structure had remained unsolved for fourteen years. Considering the advances in computational methods over the last decade, the problem was approached optimistically, however the solution was not straightforward.

The compound was prepared by Rosenthal and Catsoulacos[32]. Branched chain sugars of this type were of great interest after some antibiotics were found to have branched chain sugar substituent groups. The x-ray structure analysis was attempted in order to confirm structural assignments obtained from

N.M.R. data[32].

Details of elaborate attempts at structural elucidation, using both Patterson and direct methods, are found in Dr. Hughes' Ph.D. thesis[31]. His Patterson attempts were extensive, involving various methods of sharpening the Patterson map, and various structural postulations that placed the planar p-chlorobenzoate groups of the molecule in the more strongly reflecting planes of the crystal lattice. Although he often obtained not unreasonable positions for the chlorine atoms, the rest of the molecule never appeared in a difference map. Attempts at solution involving the Patterson function have been fairly well exhausted, and there was little point in repeating Dr. Hughes' efforts.

The direct methods attempts by Dr. Hughes produced no outstanding sets of phases, and the E-maps that he calculated did not produce recognizable fragments of the molecule. Some peaks were persistent from one map to the other; however difference Fourier maps based on these peaks plus postulated attached molecular fragments produced no useful results.

Hopefully the more powerful techniques available today in direct methods would facilitate the solution. These techniques include the use of K-curves and the $\Psi(0)$ test, and due to improvements in computing power large numbers of E's and symbols may be used (the programme Dr. Hughes was using was limited to only 200 E's and three symbols).

Experimental

The data were collected by Dr. Hughes on a G.E. XRD-6 automated diffractometer using a θ-2θ scan technique and nickel-filtered CuKα radiation. The scan speed was 1°/minute with 40 second background measurements before and after the scan. Of the 1028 reflections collected in the range $0 < \theta \leq 45°$, 898 had $I/\sigma(I) \geq 2.0$ and were considered observed, where $\sigma(I)$ was defined $\sigma^2(I) = S + B + (0.02S)^2$ and S and B are the scan and background counts over the same range.

The crystal examined was very small (0.25 x 0.05 x 0.04 mm^3) and no absorption correction was necessary. Lorentz and polarization corrections were applied and the structure amplitudes calculated. The cell dimensions were obtained by a least-squares refinement based on the $\sin\theta/\lambda$ values of 30 reflections, and are listed together with other crystal data in Table XLVI. A list of the 1028 Fo's is presented in Dr. Hughes' thesis, and it is with these data that the present investigation commenced.

Solution

The 238 highest E's obtained from a K-curve together with the 45 lowest E's for use in the $\Psi(0)$ test were input into the MULTAN programme. The origin selected by MULTAN was a conventional $P2_1$ origin: the 1 0 3, 1 0 6, and 1 1 3 reflections (ranks 1, 6, and 86, respectively) were assigned phases of zero. Unlike the raucubaine structure, there was no problem finding

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TABLE XLVI. CRYSTAL DATA FOR THE PYRANOSIDE

$C_{24}H_{24}Cl_2O_8$	f.w. = 511.26
Monoclinic	Z = 2
Space group = $P2_1$	F(000) = 532
a = 5.752(3)	λ = 1.54188 Å
b = 15.436(3)	V = 849.87 Å^3
c = 13.698(3) Å	Dm = 1.43 gcm^{-1}
β = 93.74(3) ^{0}	Dc = 1.40 gcm^{-1}
	μ(Cu radn.) = 28.1 cm^{-1}

****

two strong zonal reflections. The 2 0 6 reflection could be assigned phase zero from several Σ_1-relationships. Three symbols were chosen: 1 6 -4, 0 2 3, and 2 3 9 of ranks 2, 3, and 15, respectively, and ten sets of phases were generated. The best set of phases had the lowest R and $\Psi(0)$ values, but also had the lowest FOM, and the discrimination was not very good. The E-map of this set revealed some unrecognizable fragments of a molecule, and attempts to refine these fragments failed.

Because of the statistical nature of direct methods, it is not always necessarily the set of phases with the best correctness criteria that leads to the correct solution, especially when there are several sets that seem reasonable. With this in mind, an E-map was calculated from the second best set, and a fragment including 22 atoms but no p-chlorobenzoate groups was revealed. Attempts to refine this fragment also failed.

The MULTAN programme was rerun with the 500 highest and 50 lowest E's obtained from a Wilson plot, and with four rather than three symbols in an attempt to improve the discrimination between sets. The same origin was selected, and the symbols were now 1 6 -4, 0 2 3, 1 7 -2, and 1 2 0, of ranks 2, 3, 11 and 26 respectively. The 2 0 6 reflection was assigned phase 0 from Σ_1-relationships. This phase had changed to π in 5 of the 16 sets of phases generated. The set with the lowest $\Psi(0)$ value had a very high R value, and vice-versa, lending little confidence to the correctness of any set. The E-maps that were calculated mostly showed a single large peak, although the E-map from the set with the lowest $\Psi(0)$ value produced two high peaks some distance apart: quite possibly the chlorine atoms. Attempts to refine these atoms and to obtain additional information from subsequent difference maps failed.

The MULTAN programme contains a feature which involves the calculation of spherically averaged group scattering factors for sections of the molecule of known geometry but of random orientation. This is useful in the scaling in the renormalization procedure which otherwise assumes a random distribution of the unit cell contents throughout the unit cell. A third MULTAN run included the stereochemical information of the four p-chlorobenzoate groups in the unit cell. The 500 largest and 50 smallest E's used were obtained from a K-curve. MULTAN selected the same origin as before with symbols 1 6 -4, 0 2 3, 1 3 6, and 2 3 9 (ranks 2, 3, 5 and 15). As the 2 0 6 reflection changed phase several times in the previous MULTAN run, it was not permitted to accept the phase of zero as

suggested by the Σ_1-relationships, but was allowed to refine. The phase did, however, refine to phase zero in each of the 16 sets of phases generated. The set with the lowest $\Psi(0)$ value also had the highest R, and was unlikely to be correct. No recognizable molecular fragments could be located on the few E-maps that were calculated.

In an attempt to improve discrimination, five symbols were used in a fourth MULTAN run. As before, the maximum number of E's (obtained from a K-curve) and the stereochemical information of the p-chlorobenzoate groups were used. The 2 0 6 reflection was again allowed to accept phase zero from Σ_1-relationships, and once again MULTAN selected the same origin, this time with symbols 1 6 -4, 0 2 3, 1 3 6, 2 3 9 and 1 10 8 of ranks 2, 3, 5, 15, and 69 respectively. 25 sets of phases were generated of which none stood out significantly above the rest.

MULTAN was run a fifth time, where the only difference from the fourth run was that only 400 E's were used. This can sometimes force MULTAN to choose a slightly different starting set, and with luck this could lead to the solution. The same origin was selected with symbols 1 6 -4, 0 2 3, 1 3 6, 2 3 9, and 1 12 5, of ranks 2, 3, 5, 15, and 61, but the results were no more useful.

A final attempt was made including the 200 lowest E's for use in the $\Psi(0)$ test (still trying for better discrimination between generated sets of phases) but the set with the lowest $\Psi(0)$ value had quite a high R value, and the E-map from this set produced no structural information.

At this stage (January 1981) the problem was given lower

priority than other matters, and it was left aside until discussions with Dr. E. Subramanian, visiting professor from the University of Madras, India, renewed interest in this difficult structure. Dr. Subramanian recalled that in a recent problem structure that he had solved, he surmounted the difficulty by reducing the number of E's used to an absolute minimum rather than by using as many E's as possible.

Traditionally[33], the optimum number of E's to be used in the direct methods procedure is approximately ten times the number of non-hydrogen atoms in the asymmetric unit. For problem structures, the trend has been to increase the number of E's in use, the philosophy being that the greater the number of its Σ_2-contributors, the greater probability a certain phase has of being calculated correctly. Dr. Subramanian suggested that for problem structures the E's to atoms ratio should be much lower, approximately five to one. The general philosophy here is that this should prevent weaker erroneous E's from contributing to the phase determining procedure. He also suggested that this would force MULTAN to choose a stronger E for the third origin determining reflection. Throughout the above attempts, the 1 1 3 reflection (rank 86) had been used, which is indeed a little weak.

By this time (July 1981) the laboratory group had started using the most recent version of the MULTAN programme, MULTAN-80[34]. One of the differences between this and the previous version, MULTAN-78, is that a user specified number of reflections that are least likely to be phased accurately may be eliminated from the phase-determining procedure - i.e., their

phases may be determined, but may not be used for the determination of other phases. The worst Σ_2-relationships may similarly be rejected. This should prevent a lot of spurious phasing.

The 170 highest E's obtained from a K-curve were input into MULTAN, 50 of which were rejected from the phase-determining process. The 43 lowest E's were used for the $\Psi(0)$ test. The origin included the two zonal reflections 1 0 3 and 1 0 6 as before but the third origin determining reflection was 1 3 6 (rank 5), which had been previously used as a symbol. Four symbols were used: 0 2 3, 1 6 -4, 1 2 3, and 1 0 -3 (ranks 2, 3, 7, and 125). The 2 0 6 reflection was assigned phase zero. 18 sets of phases were generated, and the set that had the lowest $\Psi(0)$ value also had the lowest R value, and both values stood out significantly below those of other sets. From the E-map 27 non-hydrogen atoms could be located, and the structure was solved.

Refinement

The 27 non-hydrogen atoms could easily be identified as carbon, chlorine or oxygen from their molecular geometry. Three isotropic least-squares refinement cycles followed by a difference map revealed the positions of 7 more non-hydrogen atoms. Three more isotropic refinement cycles and a difference map proved the position of one -O-Me group to be faulty, and this group could be relocated correctly on a difference map

following two additional least-squares cycles. With all non-hydrogen atoms included, R dropped to 0.124 after two more refinement cycles.

Following two cycles with anisotropic temperature factors for the chlorine atoms which lowered R to 0.094, a difference map revealed the positions of 18 of the 24 hydrogen atoms. The hydrogen atoms were included in calculated positions, but were not refined because of insufficient data. After two cycles with anisotropic temperature factors for the chlorine and oxygen atoms a weighting scheme was introduced where $(w)^{1/2} = |Fo|/G^*$ for $|Fo| < G^*$, $(w)^{1/2} = 1$ for $G^* \leq |Fo| < F^*$, and $(w)^{1/2} = F^*/|Fo|$ for $|Fo| \geq F^*$, and $F^* = 25.0$ and $G^* = 5.0$. After five cycles with anisotropic temperature factors for all non-hydrogen atoms, the refinement had converged R = 0.043 and Rw = 0.048.

In order to determine the correct enantiomorph, a parallel refinement of both enantiomorphs with anomalous dispersion corrections applied to all non-hydrogen atoms was undertaken. After three refinement cycles, for the enantiomorph originally chosen, R and Rw had increased to 0.044 and 0.050, whereas in the enantiomorph obtained by changing the signs of x, y, and z, R and Rw had decreased to 0.042 and 0.048, respectively. Using Hamilton's ratio test[35], it may be said with 99.5% confidence that the latter is therefore the correct isomer. This assignment is consistent with the chemically-known configuration. Two more least-squares cycles were sufficient to refine the correct enantiomorph to convergence with R and Rw equal to 0.042 and 0.048 (0.053 and 0.048 including the unobserved reflections)

respectively. Final atomic positional and thermal parameters appear in Tables XLVII and XLVIII.

Post-solution analysis

The structure was not solved using a large number of E's and a great amount of computing power, so in retrospect, of the experience gained in solving raucubaine only the $\Psi(0)$ test (and perseverence) were of use here. Credit must be given to the experienced insight of Dr. Subramanian for drastically reducing the number of E's to 170. Clearly part of the difficulty was that the lower E's were given too much weight in the phase-determining procedure. This is reflected in the first origin choice, which included one low E which persisted through six MULTAN runs but failed to produce the solution. The new reflection weighting feature of MULTAN-80 must also have served to correct this problem.

It might also be mentioned that this one to five ratio of atoms to E's as suggested by Dr. Subramanian has since also been succesful in the solution of a phosphazene[36] that had remained unsolved for several years.

A possible reason for the difficulty Dr. Hughes experienced in obtaining the chlorine positions from the Patterson map is that there exists considerable motion at the chlorine termini of the p-chlorobenzoate side chains. This is especially evident in the thermal parameters of Cl(1) where $\underline{U}_{1,1}$ = 0.311 Å^2 (see Table XLVIII) corresponding to the very large root mean square

TABLE XLVII. POSITIONAL AND ISOTROPIC THERMAL PARAMETERS FOR THE PYRANOSIDE

(fractional x 10^4, H x 10^3, U x 10^3 Å^2)

Atom	x	y	z	Ueq/Uiso
Cl(1)	408(11)	-2322	6160(3)	185
Cl(2)	5271(7)	6067(4)	4487(3)	126
O(1)	-2207(11)	3526(5)	8974(5)	67
O(2)	1147(14)	3580(6)	9998(5)	68
O(3)	100(11)	1122(7)	11837(5)	90
O(4)	3668(13)	1133(7)	11349(5)	97
O(5)	-2011(12)	1215(6)	8498(5)	69
O(6)	-5397(15)	571(6)	8705(6)	95
O(7)	-1419(13)	3467(6)	6936(6)	74
O(8)	1313(15)	2517(8)	6618(6)	95
C(1)	-1282(22)	3566(8)	9950(9)	68
C(2)	-2107(18)	2811(10)	10556(8)	69
C(3)	-1724(15)	1941(8)	10085(7)	53
C(4)	-2643(17)	1989(8)	9029(8)	61
C(5)	-1641(14)	2745(8)	8487(7)	56
C(6)	2094(23)	4285(8)	9489(10)	88
C(7)	818(17)	1635(7)	10214(8)	59
C(8)	1396(19)	1276(7)	11226(8)	58
C(9)	4443(20)	751(11)	12262(10)	108
C(10)	-3509(22)	565(9)	8375(8)	64
C(11)	-2518(23)	-135(8)	7832(8)	62
C(12)	-3657(22)	-926(11)	7777(9)	81
C(13)	-2745(37)	-1600(9)	7235(11)	104
C(14)	-731(38)	-1477(13)	6801(10)	109
C(15)	374(29)	-711(14)	6861(10)	106
C(16)	-472(24)	-36(9)	7375(9)	81
C(17)	-2635(18)	2821(10)	7454(8)	75
C(18)	529(24)	3234(11)	6550(8)	70
C(19)	1624(20)	3960(10)	6037(8)	62
C(20)	3640(22)	3794(8)	5558(9)	74
C(21)	4721(24)	4450(12)	5095(10)	91
C(22)	3818(24)	5255(10)	5100(9)	79
C(23)	1851(25)	5443(9)	5558(12)	99
C(24)	782(21)	4780(11)	6024(10)	85

continued...

H(011)	-194	409	1027	82
H(021)	-375	285	1066	73
H(022)	-128	281	1118	73
H(031)	-264	153	1042	61
H(041)	-438	204	899	69
H(051)	5	266	847	62
H(061)	216	413	879	77
H(062)	359	444	973	77
H(063)	107	478	949	77
H(071)	186	208	1004	57
H(072)	104	117	972	57
H(091)	528	21	1220	105
H(092)	325	65	1269	105
H(093)	559	114	1261	105
H(121)	-520	-102	810	79
H(131)	-366	-218	717	102
H(151)	185	-67	647	117
H(161)	31	50	747	81
H(171)	-427	298	746	68
H(172)	-259	227	711	68
H(201)	430	319	553	79
H(211)	618	432	477	83
H(231)	119	605	547	103
H(241)	-53	490	639	91

TABLE XLVIII. ANISOTROPIC TEMPERATURE FACTORS OF THE PYRANOSIDE

(U_{ij} x 10^3 Å^2)

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Cl(1)	311(7)	117(3)	123(3)	107(4)	-7(4)	-33(3)
Cl(2)	124(3)	117(3)	135(3)	-39(3)	-4(2)	35(3)
O(1)	60(4)	69(5)	72(6)	14(4)	-2(4)	-1(5)
O(2)	59(6)	60(5)	84(6)	-5(4)	-6(4)	-6(4)
O(3)	61(4)	143(7)	69(5)	0(5)	30(4)	31(6)
O(4)	52(5)	152(8)	86(6)	14(6)	8(4)	52(6)
O(5)	57(4)	70(5)	81(5)	-12(5)	21(4)	-24(5)
O(6)	68(5)	104(6)	115(6)	-25(5)	30(5)	-31(5)
O(7)	55(4)	97(6)	70(5)	6(5)	11(4)	8(5)
O(8)	108(7)	78(7)	102(6)	14(6)	42(5)	9(5)
C(1)	74(11)	69(8)	62(9)	13(7)	6(7)	-14(7)
C(2)	56(7)	86(9)	67(7)	10(7)	19(6)	-22(8)
C(3)	41(6)	67(8)	52(7)	-4(5)	10(5)	-5(6)
C(4)	43(6)	65(8)	76(8)	-4(6)	17(6)	-3(8)
C(5)	40(6)	69(8)	60(7)	5(6)	12(5)	-20(7)
C(6)	81(9)	59(9)	123(12)	-17(7)	-10(8)	-2(9)
C(7)	47(7)	64(7)	67(8)	-1(5)	18(5)	2(6)
C(8)	51(8)	50(7)	72(9)	11(6)	1(6)	0(6)
C(9)	66(8)	161(15)	97(10)	20(8)	0(7)	39(10)
C(10)	53(8)	68(9)	72(8)	-10(8)	11(7)	-9(7)
C(11)	84(9)	54(9)	48(7)	-8(8)	-5(7)	0(7)
C(12)	93(9)	85(10)	64(9)	-8(9)	11(7)	10(8)
C(13)	185(17)	59(11)	65(10)	-2(11)	-20(10)	-7(9)
C(14)	172(18)	94(14)	59(10)	59(15)	-6(11)	-6(10)
C(15)	116(13)	122(14)	81(12)	11(11)	19(9)	-12(11)
C(16)	90(10)	89(10)	64(8)	19(8)	8(7)	-11(8)
C(17)	52(7)	117(10)	56(7)	-15(8)	-4(6)	2(8)
C(18)	77(10)	83(11)	49(8)	7(9)	1(7)	-2(8)
C(19)	56(8)	81(11)	49(7)	0(8)	-7(6)	-21(7)
C(20)	73(9)	76(9)	72(8)	16(8)	2(7)	4(7)
C(21)	80(9)	106(12)	88(10)	6(10)	16(7)	23(9)
C(22)	79(10)	84(11)	72(9)	-11(9)	-10(7)	9(8)
C(23)	77(10)	71(10)	147(14)	8(9)	-7(9)	-22(10)
C(24)	75(9)	74(10)	110(11)	-5(9)	20(8)	-17(9)

displacement of 0.558 Å.

Discussion

A stereoview of the pyranoside molecule with its atomic labelling scheme is shown in Figure 19. The sugar has the six-membered pyranose ring in a slightly flattened chair conformation. Intraannular torsion angles for the six-membered ring are listed in Table XLIX.

The two bulky p-chlorobenzoyl side chains both have equatorial orientations with the shorter -O-Me and C-COOMe side chains in axial positions.

Bond lengths and angles appear in Tables L, LI, LII, and LIII, and are normal.

A packing diagram is shown in Figure 20. There are no strong intermolecular attractions; the crystal is held together by van der Waals forces. The four p-chlorobenzoyl groups in the unit cell do not lie in but are very close to the (1 0 3) planes (see Figure 21), which is consistent with the strength of the 1 0 3 reflection, as expected by Dr. Hughes.

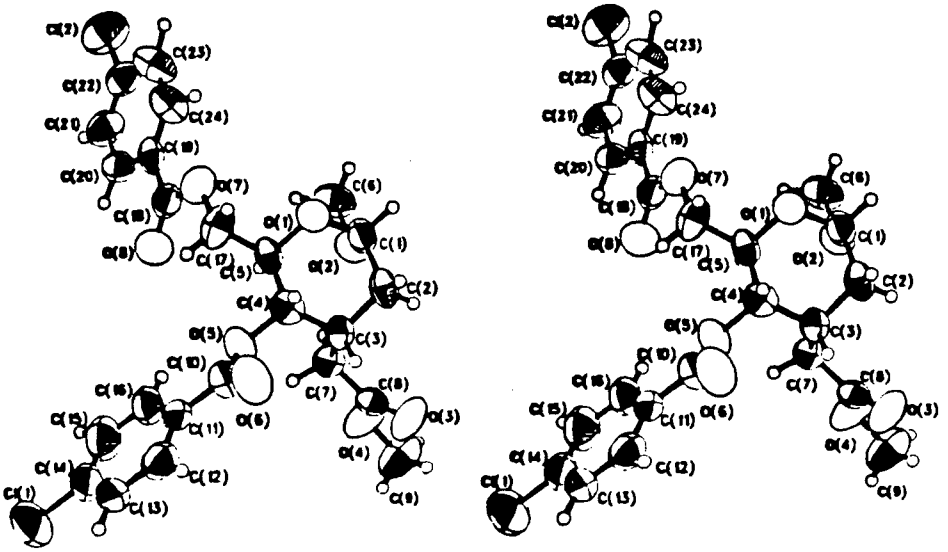

Figure 19. The pyranoside molecule

TABLE XLIX. INTRAANNULAR TORSION ANGLES FOR THE SUGAR RING

Atoms	Value(deg)
C(5)-O(1)-C(1)-C(2)	-58.0(11)
C(1)-O(1)-C(5)-C(4)	60.8(10)
O(1)-C(1)-C(2)-C(3)	51.6(12)
C(1)-C(2)-C(3)-C(4)	-48.6(11)
C(2)-C(3)-C(4)-C(5)	53.2(10)
C(3)-C(4)-C(5)-O(1)	-58.8(10)

TABLE L. NON-HYDROGEN BOND LENGTHS OF THE PYRANOSIDE

Bond	Length(Å)	Bond	Length(Å)
Cl(1)-C(14)	1.725(15)	C(3)-C(7)	1.536(14)
Cl(2)-C(22)	1.751(13)	C(4)-C(5)	1.517(14)
O(1)-C(1)	1.407(13)	C(5)-C(17)	1.495(13)
O(1)-C(5)	1.427(11)	C(7)-C(8)	1.509(13)
O(2)-C(1)	1.395(11)	C(10)-C(11)	1.450(15)
O(2)-C(6)	1.419(13)	C(11)-C(12)	1.39(2)
O(3)-C(8)	1.181(11)	C(11)-C(16)	1.377(15)
O(4)-C(8)	1.326(10)	C(12)-C(13)	1.40(2)
O(4)-C(9)	1.427(13)	C(13)-C(14)	1.35(2)
O(5)-C(4)	1.457(12)	C(14)-C(15)	1.34(2)
O(5)-C(10)	1.326(12)	C(15)-C(16)	1.36(2)
O(6)-C(10)	1.203(11)	C(18)-C(19)	1.48(2)
O(7)-C(17)	1.433(14)	C(19)-C(20)	1.393(15)
O(7)-C(18)	1.320(14)	C(19)-C(24)	1.35(2)
O(8)-C(18)	1.197(14)	C(20)-C(21)	1.37(2)
C(1)-C(2)	1.52(2)	C(21)-C(22)	1.35(2)
C(2)-C(3)	1.512(15)	C(22)-C(23)	1.36(2)
C(3)-C(4)	1.508(14)	C(23)-C(24)	1.37(2)

TABLE LI. NON-HYDROGEN BOND ANGLES OF THE PYRANOSIDE

Bonds	Angle(deg)	Bonds	Angle(deg)
C(1)-O(1)-C(5)	113.4(8)	C(10)-C(11)-C(12)	119.0(12)
C(1)-O(2)-C(6)	113.9(9)	C(10)-C(11)-C(16)	121.8(11)
C(8)-O(4)-C(9)	115.5(8)	C(12)-C(11)-C(16)	119.2(12)
C(4)-O(5)-C(10)	120.1(7)	C(11)-C(12)-C(13)	119.5(12)
C(17)-O(7)-C(18)	117.7(10)	C(12)-C(13)-C(14)	119.4(14)
O(1)-C(1)-O(2)	111.2(8)	Cl(1)-C(14)-C(13)	119(2)
O(1)-C(1)-C(2)	111.9(10)	Cl(1)-C(14)-C(15)	120(2)
O(2)-C(1)-C(2)	109.4(9)	C(13)-C(14)-C(15)	120.9(15)
C(1)-C(2)-C(3)	112.7(8)	C(14)-C(15)-C(16)	121.4(15)
C(2)-C(3)-C(4)	108.4(9)	C(11)-C(16)-C(15)	119.6(13)
C(2)-C(3)-C(7)	112.8(9)	O(7)-C(17)-C(5)	110.8(9)
C(4)-C(3)-C(7)	113.4(8)	O(7)-C(18)-O(8)	123.1(13)
O(5)-C(4)-C(3)	110.8(8)	O(7)-C(18)-C(19)	112.4(12)
O(5)-C(4)-C(5)	105.7(7)	O(8)-C(18)-C(19)	124.5(12)
C(3)-C(4)-C(5)	112.7(8)	C(18)-C(19)-C(20)	118.4(13)
O(1)-C(5)-C(4)	108.4(7)	C(18)-C(19)-C(24)	123.3(12)
O(1)-C(5)-C(17)	106.9(9)	C(20)-C(19)-C(24)	118.3(12)
C(4)-C(5)-C(17)	112.9(9)	C(19)-C(20)-C(21)	120.0(11)
C(3)-C(7)-C(8)	111.7(8)	C(20)-C(21)-C(22)	119.6(12)
O(3)-C(8)-O(4)	122.5(9)	Cl(2)-C(22)-C(21)	117.5(13)
O(3)-C(8)-C(7)	127.9(9)	Cl(2)-C(22)-C(23)	120.4(13)
O(4)-C(8)-C(7)	109.6(9)	C(21)-C(22)-C(23)	122.1(13)
O(5)-C(10)-O(6)	122.8(11)	C(22)-C(23)-C(24)	117.9(12)
O(5)-C(10)-C(11)	110.8(10)	C(19)-C(24)-C(23)	122.1(12)
O(6)-C(10)-C(11)	126.4(12)		

TABLE LII. BOND LENGTHS OF THE HYDROGEN ATOMS OF THE PYRANOSIDE

Bond	Length(Å)	Bond	Length(Å)
C(1)-H(011)	1.01	C(9)-H(092)	0.95
C(2)-H(021)	0.96	C(9)-H(093)	0.99
C(2)-H(022)	0.95	C(12)-H(121)	1.03
C(3)-H(031)	0.97	C(13)-H(131)	1.04
C(4)-H(041)	1.00	C(15)-H(151)	1.04
C(5)-H(051)	0.98	C(16)-H(161)	0.95
C(6)-H(061)	0.99	C(17)-H(171)	0.97
C(6)-H(062)	0.93	C(17)-H(172)	0.97
C(6)-H(063)	0.96	C(20)-H(201)	1.01
C(7)-H(071)	0.96	C(21)-H(211)	0.99
C(7)-H(072)	1.01	C(23)-H(231)	1.01
C(9)-H(091)	0.98	C(24)-H(241)	0.95

TABLE LIII. BOND ANGLES INVOLVING HYDROGEN ATOMS IN THE PYRANOSIDE

Bonds	Angle(deg)
O(1)-C(1)-H(011)	109
O(2)-C(1)-H(011)	112
C(2)-C(1)-H(011)	104
C(1)-C(2)-H(021)	112
C(1)-C(2)-H(022)	110
C(3)-C(2)-H(021)	107
C(3)-C(2)-H(022)	107
H(021)-C(2)-H(022)	108
C(2)-C(3)-H(031)	106
C(4)-C(3)-H(031)	109
C(7)-C(3)-H(031)	107
O(5)-C(4)-H(041)	108
C(3)-C(4)-H(041)	110
C(5)-C(4)-H(041)	109
O(1)-C(5)-H(051)	112
C(4)-C(5)-H(051)	109
C(17)-C(5)-H(051)	108
O(2)-C(6)-H(061)	109
O(2)-C(6)-H(062)	114
O(2)-C(6)-H(063)	111
H(061)-C(6)-H(062)	108
H(061)-C(6)-H(063)	105
H(062)-C(6)-H(063)	110
C(3)-C(7)-H(071)	111
C(3)-C(7)-H(072)	108
C(8)-C(7)-H(071)	113
C(8)-C(7)-H(072)	109
H(071)-C(7)-H(072)	104

Bonds	Angle(deg)
O(4)-C(9)-H(091)	114
O(4)-C(9)-H(092)	114
O(4)-C(9)-H(093)	109
H(091)-C(9)-H(092)	108
H(091)-C(9)-H(093)	104
H(092)-C(9)-H(093)	107
C(11)-C(12)-H(121)	121
C(13)-C(12)-H(121)	119
C(12)-C(13)-H(131)	118
C(14)-C(13)-H(131)	122
C(14)-C(15)-H(151)	115
C(16)-C(15)-H(151)	124
C(11)-C(16)-H(161)	117
C(15)-C(16)-H(161)	124
O(7)-C(17)-H(171)	110
O(7)-C(17)-H(172)	110
C(5)-C(17)-H(171)	109
C(5)-C(17)-H(172)	112
H(171)-C(17)-H(172)	106
C(19)-C(20)-H(201)	121
C(21)-C(20)-H(201)	119
C(20)-C(21)-H(211)	119
C(22)-C(21)-H(211)	121
C(22)-C(23)-H(231)	117
C(24)-C(23)-H(231)	125
C(19)-C(24)-H(241)	118
C(23)-C(24)-H(241)	119

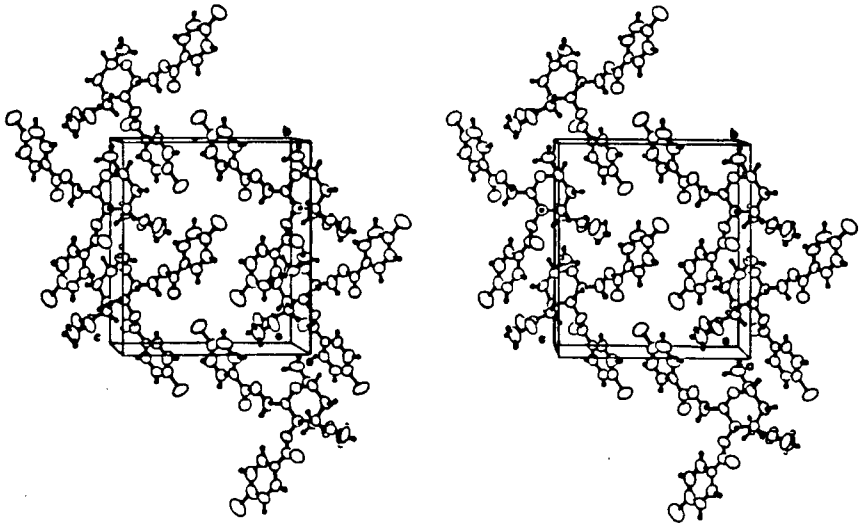

Figure 20. Stereo packing diagram for the pyranoside

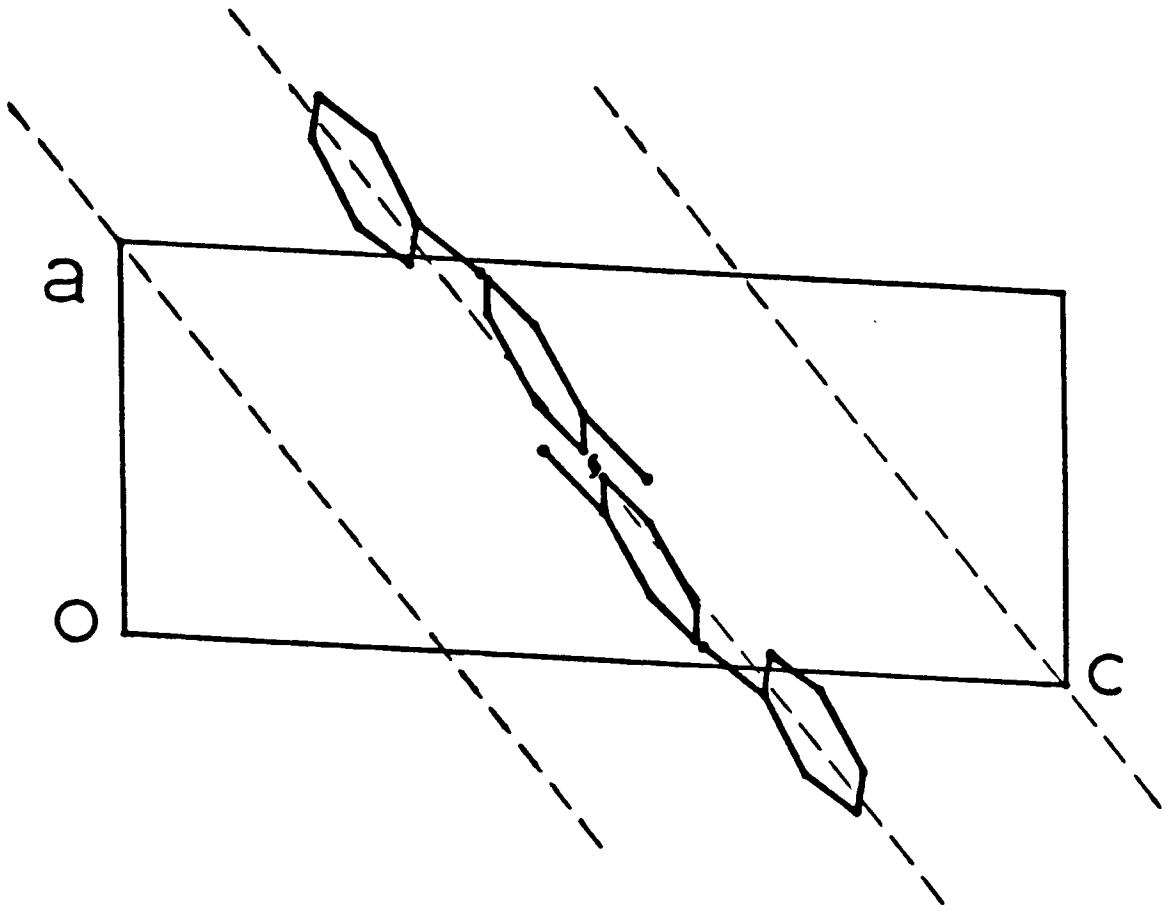

Figure 21. The unit cell viewed down b, showing the proximity of the p-chlorobenzene groups to the 103 planes (dashed)

PART TWO

CHAPTER 5

SPONTANEOUS RESOLUTION IN BINAPHTHYL SYSTEMS

Introduction

1,1'-binaphthyl may convert into its enantiomer by a rotation along the 1,1' bond. Two crystalline forms of 1,1'-binaphthyl are known: a low melting form (m.p. = 145°C) which has been shown by x-ray crystallography to be the racemate[37] with two R and two S molecules per unit cell, and an optically active high-melting form (m.p. = 158°C). Upon heating from room temperature to just below the melting point, racemic 1,1'-binaphthyl undergoes spontaneous resolution to optically active binaphthyl[38-41]. This effect is not observed in the 4,4'-dimethyl or 4,4'-diamino derivatives[42]. Optically active forms of either derivative may be obtained by seeding the racemic melt with optically active naphthidine[43]. In an attempt to understand these observations, the x-ray crystal structures of racemic and optically active 4,4'-dimethyl-1,1'-binaphthyl were determined[44-45]. As a continuation of this project, x-ray analyses of optically active 4,4'-diamino-1,1'-binaphthyl (naphthidine) and optically active 1,1'-binaphthyl (both prepared by Dr. R.E. Pincock) were undertaken. Hopefully this would present a clear overall view of any solid state properties that might give rise to the above differences in behaviour.

Experimental

A. Optically active 4,4'-diamino-1,1'-binaphthyl

Preliminary photography showed the crystal to be tetragonal

and of one of the enantiomeric space groups $P4_12_12$ or $P4_32_12$ as the 001 reflections were only present for l = 4n (indicating presence of a fourfold screw axis) and the h00 (or 0k0, as the system is tetragonal) reflections were only present for h=2n (indicating a twofold screw axis). The crystals were well-formed brown tetragonal bipyramids, but they were weakly diffracting and had a tendency to decompose (become opaque, often with liquified surfaces) after some time. It has been reported[43] that naphthidine slowly loses optical activity over a period of months.

Initial data collection was attempted using MoKα radiation, but only 226 out of 869 reflections (26.0%) in the range $0 < \theta \leq 25^{\circ}$ had $I/\sigma(I) \geq 3.0$ and were considered observed. Accurate cell parameters were obtained with this radiation by least-squares refinement of $\sin\theta/\lambda$ values of 25 reflections in the range $7.5 < \theta < 12^{\circ}$, and they are listed together with other crystal data in Table LIV.

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TABLE LIV. CRYSTAL DATA FOR OPTICALLY ACTIVE NAPHTHIDINE

$C_{20}H_{16}N_2$	f.w. = 254.4
Tetragonal	Z = 4
space group = $P4_12_12$ or $P4_32_12$	
	μ = 5.3 cm^{-1}
a = 7.945(1) Å	F(000) = 600
c = 24.265(5) Å	λ = 1.5418 Å
V = 1532 $Å^3$	Dc = 1.23 gcm^{-1}

****

In order to obtain greater intensities and more observed reflections, the data were recollected using CuKα radiation and this time 316 out of 430 reflections (73.5%) in the range $0 < \theta \leq 45^{\circ}$ had $I/\sigma(I) \geq 3.0$. The data collection used an ω-(4/3)θ scan technique with an ω-scan angle of $(0.60 + 0.35 \tan \theta)^{\circ}$, and the aperture was (2.50 + tan θ)mm wide and 4 mm high. The intensities of three standard reflections (2 2 7, 1 1 8, and 3 0 6) were measured every one hour and were used to scale the data. The same three reflections were used for orientation control; reorientation occurred if the difference between observed and calculated scattering vectors was greater than 0.065°.

The stucture was solved by direct methods. The 350 highest E's, obtained by use of the minimum profile of a K-curve, were input into the MULTAN programme. The origin and enantiomorph were fixed by assigning phase $\pi/2$ to the 3 3 17 reflection and phase $\pi/4$ to the 5 2 4 reflection respectively, and using three symbols, 40 sets of phases were generated, one of which stood out as being correct. The E-map from this set of phases revealed the positions of all non-hydrogen atoms. After six isotropic and three anisotropic least-squares refinement cycles including only the non-hydrogen atoms, a difference map failed to locate all the hydrogens. When the hydrogen atoms were inserted in calculated positions there were some difficulties with the refinement, and so the data were recollected once again in an attempt to improve data quantity and quality.

This time an ω-(2/3)θ scan was used with an ω-scan angle of $(0.70 + 0.14 \tan \theta)^{\circ}$ (CuKα radiation) and 548 out of 834

reflections (65.7%) in the range $0 < \theta \leq 65^{\circ}$ were considered observed (i.e., had $I/\sigma(I) \geq 3.0$). Three reflections (1 1 -1, 1 2 -1, and 1 2 4) were used for intensity control and data scaling, but no orientation control was used.

The scale factor was refined to fit the new data, but attempts to locate and refine the hydrogen atoms were still unsuccessful. All except the amino hydrogens (as their geometry is not uniquely defined) were placed in calculated positions and assigned an arbitrary isotropic temperature factors of 4.5 $Å^2$. After six least-squares cycles (unit weights, non-hydrogen atoms anisotropic, hydrogen atoms not refined), a difference map was calculated to try and locate the two amino hydrogens. The map yielded no outstanding peaks, but after calculating bond distances and angles of the highest six peaks in the vicinity of the nitrogen, only two were chemically reasonable, and these two positions were used for the amino hydrogens.

With all hydrogens fixed, six cycles of least-squares refinement with Hughes' weighting scheme ($(w)^{1/2} = 1.0$ for $|Fo| < F^*$ and $(w)^{1/2} = F^*/|Fo|$ for $|Fo| \geq F^*$; $F^* = 20.0$) led to convergence with R = 0.087 and Rw = 0.095 for 548 reflections. At this stage, the hydrogen temperature factors were allowed to refine (F^* now equal to 19.0), and after six cycles R and Rw had dropped to their final values of 0.068 and 0.075 respectively (0.113 and 0.075 including the unobserved reflections). Final positional and thermal parameters are shown in Tables LV and LVI.

TABLE LV. POSITIONAL AND ISOTROPIC THERMAL PARAMETERS OF OPTICALLY ACTIVE NAPHTHIDINE

(fractional x 10^4, H x 10^3, U x 10^3 Å^2)

Atom	x	y	z	Ueq/Uiso
C(1)	1070(6)	193(7)	2412(2)	68
C(2)	2654(8)	250(10)	2640(2)	95
C(3)	3852(8)	1496(12)	2452(3)	113
C(4)	3430(10)	2615(9)	2053(3)	108
C(5)	1381(12)	3705(9)	1369(4)	116
C(6)	-133(13)	3595(12)	1125(4)	136
C(7)	-1340(12)	2458(11)	1312(3)	117
C(8)	-954(8)	1363(8)	1734(2)	80
C(9)	653(7)	1385(7)	1992(2)	68
C(10)	1846(8)	2616(8)	1803(3)	87
N	4680(9)	3775(11)	1878(3)	166
H(2)	300	-64	295	87(17)
H(3)	509	150	264	108(19)
H(5)	228	461	123	217(37)
H(6)	-37	436	77	198(38)
H(7)	-255	246	114	247(52)
H(8)	-189	50	188	169(31)
H(N1)	601	342	202	220(42)
H(N2)	442	501	196	281(60)

TABLE LVI. ANISOTROPIC THERMAL PARAMETERS FOR OPTICALLY ACTIVE NAPHTHIDINE

(U_{ij} x 10^3 Å^2)

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	62(3)	74(4)	68(3)	-20(3)	5(3)	-3(3)
C(2)	69(4)	138(6)	79(4)	-12(4)	-5(3)	-11(4)
C(3)	66(4)	177(8)	95(4)	-41(5)	8(4)	-38(5)
C(4)	116(7)	105(6)	102(5)	-39(5)	39(5)	-20(5)
C(5)	134(7)	71(4)	142(6)	-2(5)	51(6)	33(5)
C(6)	144(8)	129(7)	135(7)	30(8)	40(7)	51(6)
C(7)	122(7)	124(6)	104(5)	17(5)	17(5)	40(5)
C(8)	89(5)	70(4)	82(3)	-3(4)	5(4)	12(3)
C(9)	72(4)	59(3)	72(3)	-12(3)	18(3)	-7(3)
C(10)	79(4)	81(4)	100(4)	-13(4)	28(4)	-14(4)
N	125(6)	142(7)	232(8)	-80(5)	75(6)	-20(6)

B. Optically active 1,1'-binaphthyl

From x-ray photography, the crystals were shown to give rise to the same systematic absences, and hence could be assigned the same space groups, as naphthidine. The cell parameters were refined from the $\sin\theta/\lambda$ values of 21 reflections in the range $11 < \theta < 22^{\circ}$ and appear with other crystal data in Table LVII. The intensity data were collected with use of an ω-θ

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TABLE LVII. CRYSTAL DATA FOR OPTICALLY ACTIVE 1,1'-BINAPHTHYL

$C_{20}H_{14}$	f.w. = 254.4
Tetragonal	Z = 4
space group = $P4_12_12$ or $P4_32_12$	
	μ = 0.6 cm^{-1}
a = 7.164(2) Å	F(000) = 536
c = 27.70(1) Å	λ = 0.71073 Å
V = 1422 $Å^3$	Dc = 1.19 gcm^{-1}

****

scan technique and graphite-monochromatized MoKα radiation. The ω-scan angle was $(1.00 + 0.35 \tan\theta)^{\circ}$, and the aperture was $(2.75 + \tan\theta)$mm wide and 4 mm high. The intensities of three check reflections (0 -4 0, -1 0 -17, and 0 -1 -17) were measured every one hour and were used to scale the data. The same three reflections were used for orientation control; reorientation occurred if the difference between observed and calculated scattering vectors was greater than 0.050°. Of the 812 unique reflections collected in the range $1 \leq \theta \leq 25^{\circ}$, 562 (69.2%) had

$I/\sigma(I) \geq 3.0$ and were considered observed.

The structure was solved by direct methods. 305 E's greater than 1.20 were obtained from a K-curve and input into the MULTAN programme. Seven reflections had known phases determined from Σ_1-relationships (8 0 0, 0 0 16, and 6 2 0 phase 0; 2 2 30, 6 0 0, 4 2 0, and 5 1 0 phase π) and the origin was fixed by assigning phase $3\pi/4$ to the strongest E, 7 0 5. Three symbols, one of which had restricted values to determine the enantiomorph, were used to generate 12 sets of phases, and an E-map off the correct set enabled the location of all ten carbons in the asymmetric unit.

After three isotropic and three anisotropic full-matrix least-squares refinement cycles, a difference map showed all the hydrogen atoms. After several more least-squares cycles with a polynomial weighting scheme with coefficients that were updated after every cycle, the refinement converged to a final R of 0.030 and Rw of 0.037 (0.060 and 0.037 including the unobserved reflections). In the last cycle, the coefficients of the polynomial weighting scheme were A = -0.0201, B = 0.03722, C = -0.004211, and D = 0.000169. Final positional and thermal parameters are listed in Tables LVIII and LIX.

Results

For each structure the asymmetric unit is one half the molecule; the other half is generated by rotation about a twofold axis intersecting the C(1)-C(1') bond. Molecular views

TABLE LVIII. POSITIONAL AND ISOTROPIC THERMAL PARAMETERS OF OPTICALLY ACTIVE 1,1'-BINAPHTHYL

(fractional x 10^4, H x 10^3, U x 10^3 Å^2)

Atom	x	y	z	Ueq/Uiso
C(1)	4852(3)	3743(3)	2582(1)	45
C(2)	4660(4)	2074(3)	2347(1)	60
C(3)	3353(4)	740(4)	2493(1)	69
C(4)	2253(4)	1070(3)	2878(1)	63
C(5)	1299(4)	3116(4)	3556(1)	64
C(6)	1483(4)	4729(4)	3807(1)	71
C(7)	2744(4)	6096(4)	3652(1)	65
C(8)	3831(3)	5813(3)	3254(1)	50
C(9)	3702(3)	4117(3)	2989(1)	42
C(10)	2397(3)	2755(3)	3141(1)	49
H(2)	551(3)	186(3)	207(1)	66(6)
H(3)	327(4)	-51(4)	231(1)	91(8)
H(4)	138(3)	20(4)	300(1)	71(7)
H(5)	33(4)	208(4)	364(1)	95(9)
H(6)	69(4)	497(4)	409(1)	83(8)
H(7)	290(4)	725(4)	383(1)	79(8)
H(8)	466(3)	678(3)	314(1)	51(6)

TABLE LIX. ANISOTROPIC THERMAL PARAMETERS OF OPTICALLY ACTIVE 1,1'-BINAPHTHYL

(U_{ij} x 10^3 Å^2)

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	50(1)	46(1)	40(1)	0(1)	-5(1)	1(1)
C(2)	73(2)	56(1)	52(1)	-3(1)	3(1)	-10(1)
C(3)	88(2)	49(1)	70(1)	-12(1)	-8(2)	-12(1)
C(4)	67(2)	50(1)	72(1)	-20(1)	-7(1)	6(1)
C(5)	59(1)	68(2)	67(1)	-4(1)	10(1)	11(1)
C(6)	67(2)	79(2)	67(1)	4(2)	22(1)	-1(1)
C(7)	71(2)	62(2)	62(1)	4(2)	9(1)	-13(1)
C(8)	53(1)	44(1)	52(1)	1(1)	1(1)	-2(1)
C(9)	40(1)	44(1)	42(1)	1(1)	-6(1)	5(1)
C(10)	47(1)	47(1)	52(1)	-2(1)	-3(1)	10(1)

showing the labelling scheme and 50% thermal ellipsoids of both molecules are shown in Figure 22. The naphthalene residues are approximately at right angles to each other in both cases, although in the naphthidine the arrangement is slightly cis (dihedral angle of 87°) while in binaphthyl it is slightly trans (101.5°). Bond lengths and angles are shown in Tables LX and LXI.

There are four types of bonds in the naphthalene unit: the C(2)-C(3) type (a), the C(1)-C(2) type (b), the C(1)-C(9) type (c) and the C(9)-C(10) type (d). For each structure the bond lengths have been averaged within each bond type and the results are compared to other structures containing the naphthalene unit in Table LXII. The structure of the naphthalene unit is reasonably invariant in these compounds. Bond angles are all very close to the expected sp^2 angle of 120°. Mean plane calculations (Tables LXIII and LXIV) show a slight bend in the asymmetric unit along the C(9)-C(10) bond of 2.54° for naphthidine and 1.6° for 1,1'-binaphthyl. The amino hydrogens are not coplanar with the rest of the asymmetric unit; rather the NH_2 plane (plane 4, Table LXIII) is about 60° away from the naphthalene plane. The position of the amino hydrogens, however, should not be given too much weight considering the difficulty in locating them.

In both structures the molecules spiral around the fourfold axis in the c-direction and the crystals are held together by van der Waals forces. Figures 23 and 24 display packing diagrams of these two structures. In naphthidine the closest intermolecular N...N distance is 3.48(2) Å, which is

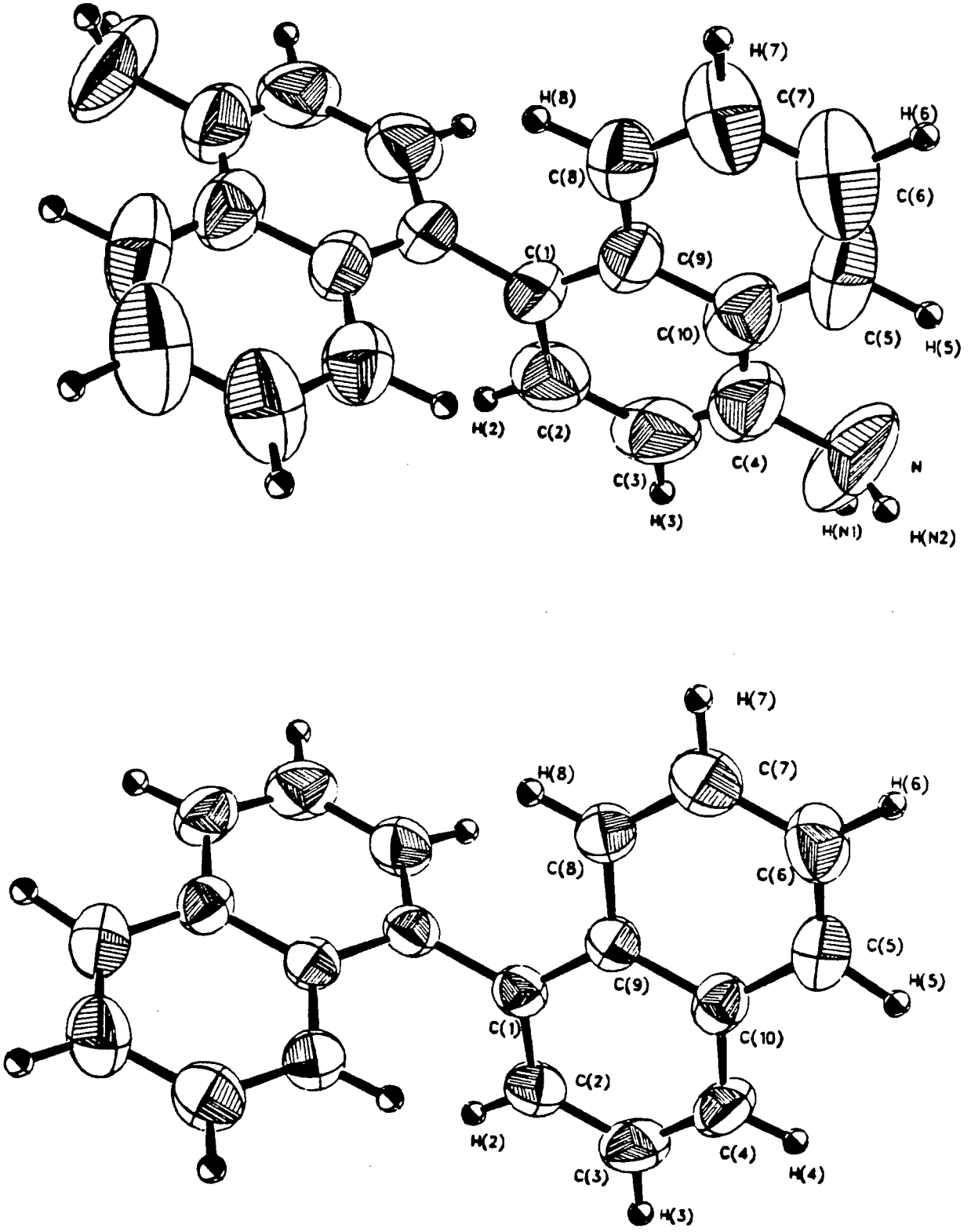

Figure 22. Molecular views of optically active naphthidine (above) and 1,1'-binaphthyl (below)

TABLE LX. BOND LENGTHS AND ANGLES IN OPTICALLY ACTIVE NAPHTHIDINE

bond	length(Å)	bond	length(Å)
C(1)-C(2)	1.375(7)	C(5)-C(6)	1.344(11)
C(1)-C(9)	1.430(7)	C(5)-C(10)	1.411(9)
C(1)-C(1)'	1.481(9)	C(6)-C(7)	1.394(11)
C(2)-C(3)	1.447(9)	C(7)-C(8)	1.378(9)
C(3)-C(4)	1.357(10)	C(8)-C(9)	1.423(7)
C(4)-C(10)	1.396(9)	C(9)-C(10)	1.438(8)
C(4)-N	1.420(8)		
C(2)-H(2)	1.078(7)	C(7)-H(7)	1.049(10)
C(3)-H(3)	1.084(7)	C(8)-H(8)	1.072(6)
C(5)-H(5)	1.075(8)	N-H(N1)	1.147(9)
C(6)-H(6)	1.063(9)	N-H(N2)	1.028(9)

bonds	angle(deg)	bonds	angle(deg)
C(2)-C(1)-C(9)	118.4(5)	C(5)-C(6)-C(7)	120.9(8)
C(2)-C(1)-C(1)'	121.8(5)	C(6)-C(7)-C(8)	119.9(9)
C(9)-C(1)-C(1)'	119.7(4)	C(7)-C(8)-C(9)	121.3(7)
C(1)-C(2)-C(3)	119.9(6)	C(1)-C(9)-C(8)	120.9(5)
C(2)-C(3)-C(4)	120.7(7)	C(1)-C(9)-C(10)	121.7(5)
C(3)-C(4)-C(10)	122.2(7)	C(8)-C(9)-C(10)	117.3(6)
C(3)-C(4)-N	117.7(9)	C(4)-C(10)-C(5)	124.1(7)
C(10)-C(4)-N	120.0(8)	C(4)-C(10)-C(9)	117.0(6)
C(6)-C(5)-C(10)	121.6(7)	C(5)-C(10)-C(9)	118.9(7)
C(1)-C(2)-H(2)	119.5(6)	C(6)-C(7)-H(7)	119.6(8)
C(3)-C(2)-H(2)	120.6(6)	C(8)-C(7)-H(7)	120.5(8)
C(2)-C(3)-H(3)	118.1(8)	C(7)-C(8)-H(8)	120.2(7)
C(4)-C(3)-H(3)	121.2(8)	C(9)-C(8)-H(8)	118.5(5)
C(6)-C(5)-H(5)	119.9(9)	C(4)-N-H(N1)	113.3(8)
C(10)-C(5)-H(5)	118.5(9)	C(4)-N-H(N2)	114.9(7)
C(5)-C(6)-H(6)	118.3(10)	H(N1)-N-H(N2)	111.4(6)
C(7)-C(6)-H(6)	120.7(10)		

TABLE LXI. BOND LENGTHS AND ANGLES IN OPTICALLY ACTIVE 1,1'-BINAPHTHYL

bond	length(Å)	bond	length(Å)
C(1)-C(2)	1.369(3)	C(5)-C(6)	1.354(4)
C(1)-C(9)	1.421(3)	C(5)-C(10)	1.417(3)
C(1)-C(1)'	1.494(4)	C(6)-C(7)	1.400(4)
C(2)-C(3)	1.398(4)	C(7)-C(8)	1.364(3)
C(3)-C(4)	1.347(3)	C(8)-C(9)	1.424(3)
C(4)-C(10)	1.415(3)	C(9)-C(10)	1.416(3)
C(2)-H(2)	0.99(2)	C(6)-H(6)	0.98(2)
C(3)-H(3)	1.02(3)	C(7)-H(7)	0.96(3)
C(4)-H(4)	0.94(2)	C(8)-H(8)	0.97(2)
C(5)-H(5)	1.04(3)		

bonds	angle(deg)	bonds	angle(deg)
C(2)-C(1)-C(9)	119.0(2)	C(6)-C(7)-C(8)	120.8(2)
C(2)-C(1)-C(1)'	120.7(2)	C(7)-C(8)-C(9)	120.5(2)
C(9)-C(1)-C(1)'	120.4(2)	C(1)-C(9)-C(8)	122.2(2)
C(1)-C(2)-C(3)	121.8(2)	C(1)-C(9)-C(10)	119.3(2)
C(2)-C(3)-C(4)	120.1(2)	C(8)-C(9)-C(10)	118.5(2)
C(3)-C(4)-C(10)	121.0(2)	C(4)-C(10)-C(5)	122.3(2)
C(6)-C(5)-C(10)	121.2(3)	C(4)-C(10)-C(9)	118.8(2)
C(5)-C(6)-C(7)	120.2(2)	C(5)-C(10)-C(9)	118.9(2)
C(1)-C(2)-H(2)	116.2(14)	C(10)-C(5)-H(5)	115.2(13)
C(3)-C(2)-H(2)	122.0(14)	C(5)-C(6)-H(6)	120.0(15)
C(2)-C(3)-H(3)	119.9(15)	C(7)-C(6)-H(6)	120(2)
C(4)-C(3)-H(3)	120.0(15)	C(6)-C(7)-H(7)	121.6(15)
C(3)-C(4)-H(4)	123.7(15)	C(8)-C(7)-H(7)	117.7(15)
C(10)-C(4)-H(4)	115.2(15)	C(7)-C(8)-H(8)	120.6(11)
C(6)-C(5)-H(5)	123.6(13)	C(9)-C(8)-H(8)	118.9(11)

TABLE LXII. NAPHTHALENE UNIT BOND LENGTH COMPARISON (Å)

	bond type			
structure	a	b	c	d
opt. active naphthidine	1.421(27)	1.364(13)	1.415(13)	1.438(8)
opt. active 1,1'-binaphthyl	1.399(4)	1.395(9)	1.419(4)	1.416(3)
racemic 1,1'-binaphthyl	1.404(3)	1.357(4)	1.418(4)	1.416(3)
opt. active 4,4'-dimethyl-1,1'-binaphthyl	1.401(22)	1.368(4)	1.416(6)	1.436(6)
racemic 4,4'-dimethyl-1,1'-binaphthyl	1.410(3)	1.369(2)	1.426(3)	1.426(3)
naphthalene	1.416(6)	1.357(4)	1.420(3)	1.405(3)

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

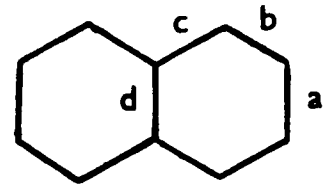

TABLE LXIII. MEAN PLANES IN OPTICALLY ACTIVE NAPHTHIDINE

Equations of planes (lX+mY+nZ=p)

plane	l	m	n	p
1	0.3352	-0.6499	-0.6821	-3.8157
2	0.3284	-0.6382	-0.6963	-3.8953
3	0.3461	-0.6628	-0.6641	-3.7679
4	0.3607	0.2622	-0.8951	-1.9504

Deviations from planes (Å)

atom	1	2	3	4
C(1)	0.008(5)*	0.000(5)*	0.073(5)	-2.94
C(2)	0.025(7)*	0.002(7)*	0.113(7)	-2.97
C(3)	0.012(8)*	-0.001(8)*	0.089(8)	-1.95
C(4)	-0.018(8)*	-0.004(8)*	0.027(8)	-0.98
C(5)	0.005(8)*	0.064(8)	-0.009(8)*	0.14
C(6)	0.063(10)*	0.138(10)	0.026(10)*	0.21
C(7)	0.017(8)*	0.083(8)	-0.008(8)*	-0.77
C(8)	-0.012(6)*	0.026(6)	-0.006(6)*	-1.80
C(9)	-0.023(5)*	-0.003(5)*	0.008(5)*	-1.90
C(10)	-0.027(6)*	0.005(6)*	0.007(6)*	-0.89
N	0.005(8)*	0.030(8)	0.041(8)	0.00*
H(N1)	0.309	0.319	0.367	0.00*
H(N2)	-0.844	-0.810	-0.819	0.00*

*atoms included in plane calculations

Angles between normals to the planes

planes (1) and (2):	1.13°	planes (2) and (3):	2.54°
planes (1) and (3):	1.41°	planes (2) and (4):	55.0°
planes (1) and (4):	55.9°	planes (3) and (4):	57.0°

TABLE LXIV. MEAN PLANES IN OPTICALLY ACTIVE 1,1'-BINAPHTHYL

Equations of planes (lX+mY+nZ=p)

plane	l	m	n	p
1	-0.6879	0.4111	-0.5981	-5.5682
2	-0.7050	0.4143	-0.5762	-5.4144
3	-0.6960	0.4121	-0.5880	-5.5119

Deviations from planes (Å)

atom	1	2	3
C(1)	0.000(2)*	-0.050(2)	-0.009(2)*
C(2)	-0.006(2)*	-0.071(3)	-0.022(3)*
C(3)	0.003(3)*	-0.039(3)	-0.002(3)*
C(4)	0.005(3)*	0.000(3)	0.017(3)*
C(5)	-0.047(3)	0.004(3)*	-0.009(3)*
C(6)	-0.076(3)	-0.011(3)*	-0.033(3)*
C(7)	-0.040(3)	0.004(3)*	-0.006(3)*
C(8)	0.000(2)	0.006(2)*	0.016(2)*
C(9)	0.004(2)*	-0.007(2)*	0.013(2)*
C(10)	-0.007(2)*	0.005(2)*	0.013(2)*
H(2)	-0.03(2)	-0.13(2)	-0.06(2)
H(3)	-0.02(3)	-0.08(3)	-0.04(3)
H(4)	-0.02(2)	-0.01(2)	-0.00(2)
H(5)	-0.02(3)	0.05(3)	0.03(3)
H(6)	-0.08(2)	0.01(2)	-0.03(2)
H(7)	-0.07(3)	-0.01(3)	-0.03(3)
H(8)	0.06(2)	0.06(2)	0.07(2)

*atoms included in plane calculations

Angles between normals to the planes

planes (1) and (2) : 1.60°
planes (1) and (3) : 0.74°
planes (2) and (3) : 0.85°

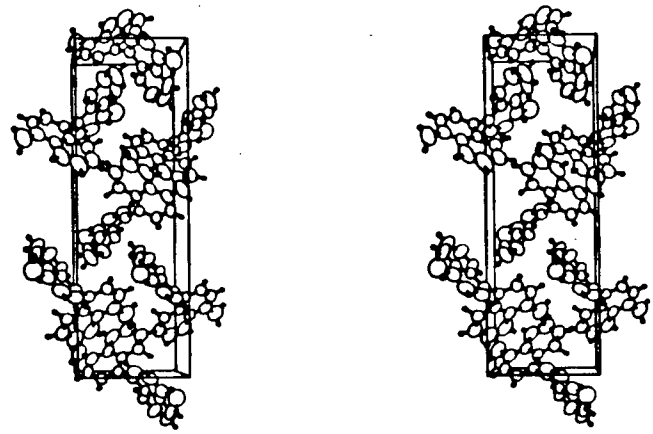

Figure 23. Packing diagram for optically active naphthidine

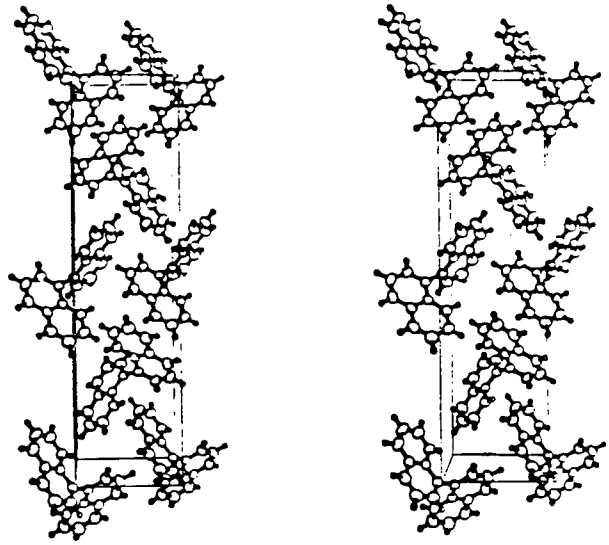

Figure 24. Packing diagram for optically active 1,1'-binaphthyl

considerably greater than the N...N range of 2.88 to 3.38 Å normally associated with N-H...N type hydrogen bonding[46].

Discussion

Table LXV compares the two structures described in this chapter to some other related compounds with known crystal structure. We are looking for some unique aspect of the crystal structures of the racemic/optically active 1,1'-binaphthyl pair that might help explain why only racemic 1,1'-binaphthyl undergoes spontaneous resolution whereas the substituted binaphthyls do not.

There are five structures in question: those of racemic[37] and optically active 1,1'-binaphthyl, racemic and optically active 4,4'-dimethyl-1,1'-binaphthyl[44], and optically active naphthidine. Of these, the two racemic structures crystallize in the monoclinic space group C2/c, and the three optically active structures crystallize in the tetragonal space group $P4_12_12$ (or $P4_32_12$). This already seems more than mere coincidence, although it may not appear immediately significant.

The dihedral angles between the naphthalene residues of the racemic structures are virtually identical (68.4 and 68.6°), whereas those of the optically active structures vary between 80 and 101°. This agrees with the calculated densities - because of the smaller dihedral angle, the racemic molecules are more compact and hence pack more densely (1.28 vs 1.19 gcm^{-3} for binaphthyl and 1.25 vs 1.15 gcm^{-3} for dimethylbinaphthyl). The

TABLE LXV. COMPARISON OF BINAPHTHYL-TYPE STRUCTURES

structure	space group	cell param. (Å,deg.)	V (Å^3)	Dc (g/cc)	p,q* (deg.)
racemic 1,1'-binaphthyl	C2/c	a=21.126 b= 6.342 c=10.218 β=105.19	1302	1.279	p=68.6 q=1.0
optically active 1,1'-binaphthyl	$P4_12_12$	a= 7.164 c=27.70	1422	1.19	p=101.5 q=1.6
racemic 4,4'-dimethyl-1,1'-binaphthyl	C2/c	a=13.225 b=10.768 c=11.572 β=114.04	1505	1.246	p=68.4 q=3.0
opt. active 4,4'-dimethyl-1,1'-binaphthyl	$P4_12_12$	a=8.3031 c=23.706	1634	1.148	p=80 q=2.7
opt. active naphthidine	$P4_12_12$	a= 7.945 c=24.264	1532	1.23	p=87 q=2.5

*p = the dihedral angle between molecular halves
q = the bending angle along C(9)-C(10)

racemates, because they are packed slightly more efficiently, will have a slightly higher lattice energy and thus are slightly more stable, at least at room temperature. Unfortunately as this is true for both 1,1'-binaphthyl and the dimethyl derivative, room-temperature lattice energies offer no explanation for the observed differences in behaviour.

The unit cell volume is somewhat greater for dimethylbinaphthyl and naphthidine than for binaphthyl in both racemic and optically active cases, as could be expected from the extra bulk due to the 4,4' substituents.

It has been suggested[37] that the 'bending angle' between the two rings of the naphthalene residue is a function of intramolecular close contacts, which in turn depend on the dihedral angle between the molecular halves, but the figures shown in Table LXV do not support this suggestion. If anything, the bending angles seem to depend on the substituent: 1.0 and 1.6° for binaphthyl, 2.5° for naphthidine, and 2.7 and 3.0° for dimethylbinaphthyl; however all these bendings are small and the differences between them are probably insignificant.

The shapes of the unit cells are worth some discussion: the optically active structures all have unit cells of similar dimensions, approximately an 8 Å base and elongated along the fourfold axis (c-direction) to about 24 Å. This could be the reason why optically active naphthidine so readily acts as a seed[43] in the resolution of dimethylbinaphthyl. It is interesting to note that the binaphthyl racemate, which does undergo spontaneous resolution to the optically active form, also has an elongated unit cell of similar dimensions

(21 x 6 x 10 Å^3, β = 105°), whereas the dimethylbinaphthyl racemate, which does not undergo spontaneous resolution, has a unit cell of quite a different shape (13 x 11 x 12 Å^3, β = 114°). Intuitively one might expect that the solid state conversion from the racemate to the optically active form would proceed more easily if the unit cells were of similar shape than if they were very different. Although it would not be conclusive, it would still be of great interest to see if this also holds true for naphthidine, i.e., to see if the racemic naphthidine unit cell shape is also very different to that of the optically active naphthidine.

Recently, I.C. Paul and co-workers[47] have studied the spontaneous resolution process visually under a microscope and have observed the nucleation and subsequent migration of an opaque front through the crystals, and also the growth of optically active crystals on the racemic crystal surfaces. They suggest that, at least at their experimental conditions, the conversion from racemic to optically active 1,1'-binaphthyl involves a sublimation process rather than a solid-solid transformation. If this is the case, room temperature investigations into the solid state properties of the materials involved may not present immediate results. Perhaps the systems should be studied at elevated temperatures to determine the temperature effects on the relative energies of racemic and optically active 1,1'-binaphthyl. These effects might then be able to explain why optically active binaphthyl becomes more stable at higher temperature.

Half-normal probability plots

The probability of two (or more) simultaneous (or nearly simultaneous) independent determinations of a given crystal structure has greatly increased since automated diffractometers and more powerful techniques have become widely available. In such cases it is of interest to perform a statistical comparison of the derived parameters, thereby either increasing or decreasing confidence in the results.

The optically active 1,1'-binaphthyl structure presented in this chapter has recently been independently determined by I.C. Paul et al[47] in Urbana, Illinois (a = 7.181(2), c = 27.68(1) Å, R = 0.043 for 978 reflections with $I/\sigma(I) \geq 2.0$), and by S.F. Mason et al[48] in London, U.K. (a = 7.2126(9), c = 27.510(5), R = 0.045 for 731 reflections with $I/\sigma(I) \geq 3.0$). These sets of results (set L from London, set U from Urbana, set V from Vancouver) may be compared using normal probability plots[49]. The ranked deviates are plotted against those expected for a particular distribution, and if the assumed distribution is correct, the plot should be linear with unit slope and pass through the origin. Deviations from linearity indicate that the assumed distribution may not be totally correct; deviations from unit slope indicate that the standard deviations have been overestimated (if slope < 1) or underestimated (if slope > 1); and deviation from zero intercept indicates either systematic error or some scaling problem.

In the following plots, the magnitudes used are the positional parameters of the ten carbons, and their deviates are

plotted against those expected for a normal distribution. The positional parameters of the hydrogen atoms could not be included in the U-V comparison as they were not refined in set U but they were in set V. For the sake of conformity they were also excluded in the L-V comparison. For positional parameters the signs of the deviates are redundant (as it is equally valid to use transformed sets of coordinates where the signs of the deviates would be different) and a half-normal probability plot is used. The coordinates for set L are related to those of sets U and V by the symmetry operation (1/2-y,1/2-x,1/2-z).

The deviates $\delta(Pi)$ of the i'th positional parameter are calculated as

$$\delta(Pi)=|Pi(U)-Pi(V)| \;/\; \{\sigma^2[Pi(U)]+\sigma^2[Pi(V)]\}^{1/2}$$

and $$\delta(Pi)=|Pi(L)-Pi(V)| \;/\; \{\sigma^2[Pi(L)]+\sigma^2[Pi(V)]\}^{1/2}$$

and ranked according to magnitude. The expected ranked deviates are readily available from Table 4.3.2.D of International Tables for X-ray Crystallography[6], Vol. IV, and the resulting plots appear in Figure 25. The apparent departures from linearity are without significance; the linearity of the plot compares reasonably well to previous half-normal plots[49]. The intercepts are sufficiently close to zero to indicate no systematic errors of importance. For the U-V comparison, the slope is slightly less than unity (0.7) indicating that the pooled standard deviations are only slightly overestimated (by about 30%). For the L-V comparison, the slope is around 1.5, indicating that the pooled standard deviations are underestimated by about 50%.

In other words, the results of I. Paul add confidence to the results obtained in this chapter, whereas the results of

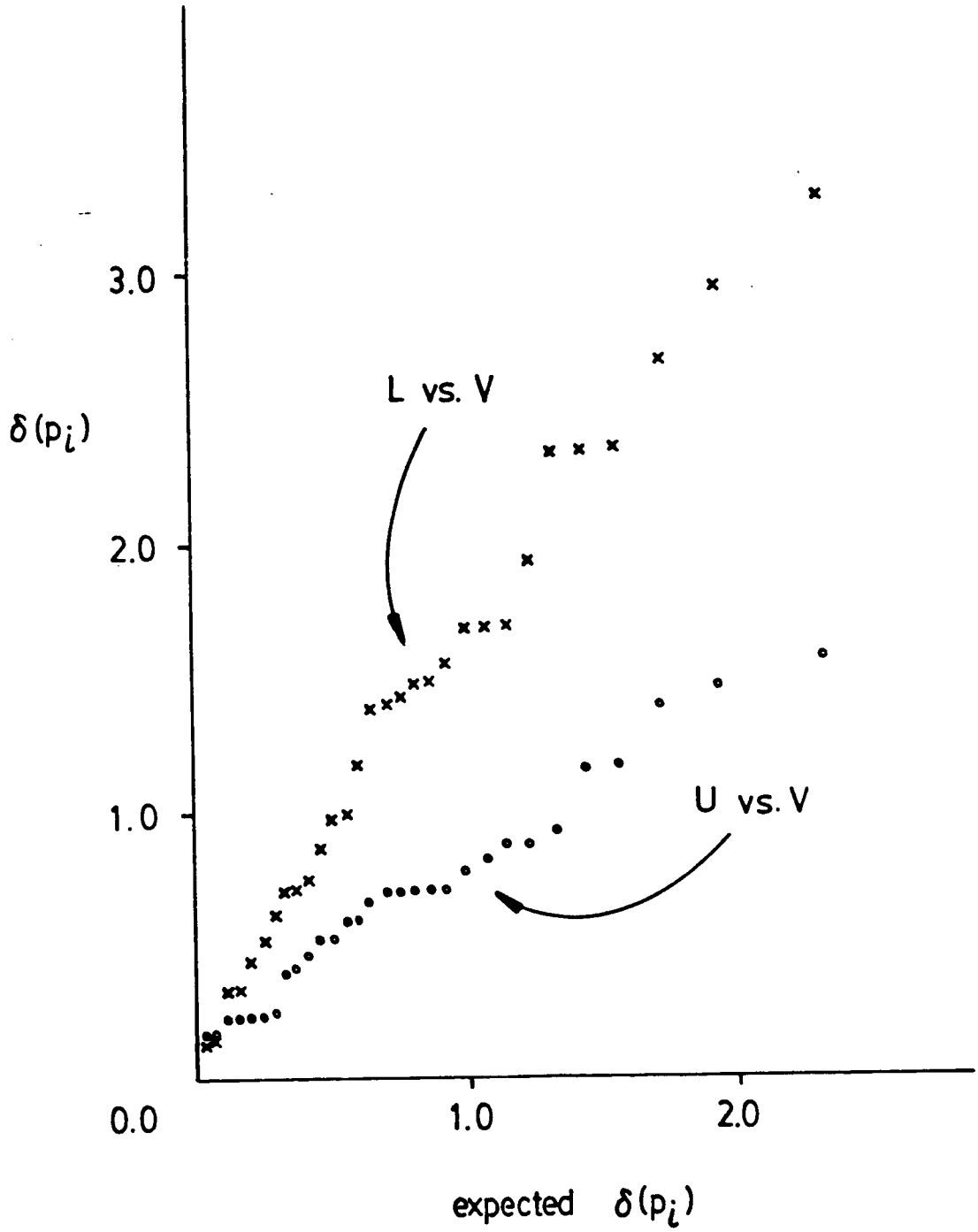

Figure 25. A half-normal probability plot

S.F. Mason are slightly different. To account for this difference, the pooled standard deviations between sets L and V should be slightly increased. From the R values and standard deviations of the three structure determinations, it might be inferred that the work in set L is the least accurate of the three, and perhaps it is the standard deviations in set L that should be slightly increased. It must be pointed out, however, that although the analysis in this chapter does have the lowest R value, it is based on the least amount of observed data. One might argue that it is easier to create a model to fit 562 reflections well than one to fit 731 or 978 reflections, and that the R value might well be lower for fewer reflections. Nevertheless, the standard deviations in the analysis in this chapter are lower than in the other two sets, and these have also traditionally been taken as a measure of accurate determination. The results of the U-V comparison suggest that these standard deviations may even be slightly reduced.

In conclusion, it may be said that the positional parameters from sets U and V are indeed representative of the true structure of 1,1'-binaphthyl, and that the positional parameters from set L would likely be more representative of the structure were their standard deviations increased by roughly 50%.

SUMMARY

This thesis has presented the successful structure determinations of eight organic compounds in a variety of applications. In chapter two, the concern was the identification of two separated isomers that would have been difficult to characterize by other means. Some chemical evidence inspired additional analyses to investigate the possibility of one of the isomers having a highly strained trans-fused four and five membered ring system. The existence of this strained ring system was disproved, leading, with true scientific spirit, to further organic mechanistic research.

Chapter three contains a more typical project: confirmation of a postulated structure by x-ray analysis. A strained intermediate had been proposed for a hydrogen exchange reaction, a compound had been isolated with spectral properties suggesting this intermediate, and the structure was confirmed by x-ray crystallography.

Chapter four contains two analyses that should dispel the myth that x-ray structure determinations have become routine. These analyses required far more effort than is obvious from their descriptions, and their ultimate solution was sufficient reward. Raucubaine, the first of these two structures, is an indole alkaloid which could not be completely characterized by

other means, i.e., its structure was previously unknown and hence the elucidation was more significant. The second analysis was meant to confirm the N.M.R. structural assignments of a sugar, but was undertaken here more as a challenge than out of chemical interest (because it was still there...).

Chapter five shows that x-ray analyses may be used to lend insight into problems that are not purely of a structural nature. In this case, packing effects are inspected in order to try and account for differences in stability between two enantiomeric forms of 1,1'-binaphthyl. It seems now, however, that this investigation would probably be more meaningful were it carried out at elevated temperatures.

It is hoped that the utility of x-ray crystallography in chemical research is demonstrated.

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APPENDIX: STRUCTURE FACTOR TABLES

STRUCTURE FACTOR TABLES FOR A β-CYCLOBUTYL TRICYCLIC ENONE

h	k	l	Fo	Fc
-2	-2	-16	4.66	3.77
-1	-1	-16	4.21	3.95
-2	0	-16	5.52	6.11
-1	0	-15	5.91	5.83
-2	-1	-15	3.36	3.54
-1	-1	-15	7.18	7.39
-4	-3	-15	7.99	7.36
-1	-6	-15	6.11	5.83
-3	-6	-14	4.78	4.11
-2	-6	-14	4.57	4.34
-1	-6	-14	8.69	7.99
-5	-4	-14	6.97	7.12
-3	-4	-14	7.62	6.96
-2	-4	-14	7.29	6.94
-1	-3	-14	4.27	4.18
-2	-3	-14	5.63	4.57
-4	-3	-14	3.96	3.39
-6	-2	-14	5.32	5.07
-5	-2	-14	3.82	3.68
-3	-2	-14	5.29	5.46
-2	-2	-14	6.11	6.02
-1	-2	-14	4.73	4.70
-1	-1	-14	11.80	12.22
-2	-1	-14	4.39	4.59
-3	-1	-14	5.23	5.45
-2	0	-14	6.17	6.18
-1	0	-13	8.29	8.50
-3	0	-13	21.51	21.91
-5	0	-13	10.68	10.66
-7	-1	-13	10.07	10.27
-4	-1	-13	4.01	3.44
-1	-1	-13	14.12	14.34
-1	-2	-13	7.51	7.89
-4	-2	-13	11.16	11.12
-6	-3	-13	3.99	3.89
-5	-3	-13	3.45	3.69
-4	-3	-13	5.01	4.48
-1	-3	-13	9.88	9.54
-2	-4	-13	9.36	9.59
-3	-4	-13	4.97	4.78
-4	-4	-13	10.96	11.16
-5	-4	-13	7.75	8.04
-6	-5	-13	5.90	5.70
-4	-5	-13	11.89	11.51
-2	-5	-13	9.73	9.21
-1	-5	-13	4.46	3.69
-1	-6	-13	6.14	7.19
-2	-6	-13	6.75	6.54
-4	-6	-13	9.08	8.84
-1	-8	-13	8.79	8.40
-4	-8	-12	6.15	6.49
-1	-8	-12	6.38	6.00
-2	-7	-12	7.35	7.04
-4	-6	-12	8.23	8.14
-3	-6	-12	5.89	5.19
-2	-6	-12	10.34	9.64
-1	-6	-12	5.51	3.55
-1	-5	-12	4.84	5.67
-2	-5	-12	18.81	19.32
-3	-5	-12	6.16	5.48
-4	-5	-12	7.57	8.16
-7	-4	-12	4.57	4.66
-5	-4	-12	5.00	5.29
-3	-4	-12	4.45	4.82
-2	-4	-12	5.88	4.98
-1	-4	-12	3.80	4.85
-1	-3	-12	5.72	6.02
-2	-3	-12	6.76	6.51
-5	-3	-12	9.75	10.31
-6	-3	-12	4.19	3.69
-8	-2	-12	8.13	8.02
-6	-2	-12	6.98	6.78
-5	-2	-12	10.88	11.37
-4	-2	-12	9.92	9.82
-2	-2	-12	11.22	11.00
-1	-2	-12	5.52	6.03
-1	-1	-12	29.64	30.17
-2	-1	-12	4.90	4.17
-3	-1	-12	16.96	16.51
-6	-1	-12	11.12	11.49
-8	-1	-12	11.21	11.19
-6	0	-12	17.48	18.20
-4	0	-12	21.42	20.78
-2	0	-12	19.51	20.14
-1	0	-11	16.98	16.60
-3	0	-11	18.36	18.24
-5	0	-11	30.69	31.31
-9	0	-11	15.31	14.65
-7	-1	-11	9.28	9.53
-6	-1	-11	15.67	15.20
-5	-1	-11	16.57	17.00
-4	-1	-11	21.37	21.24
-3	-1	-11	10.52	10.39
-2	-1	-11	3.44	3.13
-1	-1	-11	12.01	12.10
-2	-2	-11	4.55	4.91
-4	-2	-11	6.05	6.09
-5	-2	-11	4.76	4.81
-6	-2	-11	10.91	11.14
-7	-2	-11	8.19	8.76
-6	-3	-11	5.73	5.64
-4	-3	-11	5.69	5.30
-3	-3	-11	11.10	10.52
-2	-3	-11	17.61	17.15
-1	-3	-11	7.32	7.44
-1	-4	-11	8.50	9.14
-3	-4	-11	12.19	11.91
-5	-4	-11	9.45	9.79
-6	-5	-11	4.92	5.22
-5	-5	-11	3.93	3.99
-4	-5	-11	13.30	14.01
-2	-5	-11	7.41	8.35
-1	-5	-11	6.70	6.28
-1	-6	-11	5.58	6.15
-3	-6	-11	3.44	3.68
-4	-6	-11	6.87	7.34
-6	-6	-11	8.75	8.71
-4	-7	-11	11.63	11.51
-3	-7	-11	5.84	5.90
-2	-7	-11	9.49	9.89
-1	-8	-11	4.95	4.00
-5	-8	-11	5.14	5.56
-4	-9	-11	5.32	5.45
-2	-9	-11	6.08	5.90
-1	-9	-11	4.01	3.10
-1	-10	-11	7.44	6.93
-2	-10	-10	3.63	3.95
-1	-10	-10	6.44	6.14
-3	-8	-10	4.08	3.40
-1	-7	-10	3.29	3.19
-4	-7	-10	4.30	4.35
-5	-7	-10	5.73	5.60
-6	-7	-10	6.79	5.92
-7	-7	-10	8.67	8.43
-5	-6	-10	8.49	8.44
-3	-6	-10	14.04	13.87
-1	-5	-10	2.86	2.51
-2	-5	-10	4.14	3.79
-4	-5	-10	6.37	6.55
-5	-5	-10	11.34	11.72
-6	-5	-10	9.79	10.25
-7	-5	-10	6.00	6.28
-7	-4	-10	5.51	5.45
-6	-4	-10	3.32	3.89
-5	-4	-10	6.65	6.75
-4	-4	-10	13.40	14.21
-3	-4	-10	13.39	13.09
-1	-4	-10	7.64	8.34
-2	-3	-10	5.23	5.69
-3	-3	-10	7.42	7.97
-6	-3	-10	8.33	8.47
-7	-3	-10	6.48	6.11
-8	-2	-10	3.58	3.23
-7	-2	-10	5.26	4.81
-5	-2	-10	4.16	4.42
-3	-2	-10	15.97	16.42
-2	-2	-10	5.90	5.93
-1	-2	-10	6.54	5.99
-1	-1	-10	13.18	13.09
-3	-1	-10	24.35	24.15
-5	-1	-10	30.60	30.23
-6	-1	-10	7.30	6.94
-7	-1	-10	3.83	3.97
-6	0	-10	30.12	30.14
-4	0	-10	65.58	66.11
-2	0	-10	25.66	25.76
-1	0	-9	11.42	11.17
-3	0	-9	47.94	47.73
-5	0	-9	8.83	8.35
-7	0	-9	3.87	4.29
-8	-1	-9	7.29	7.77
-6	-1	-9	17.49	18.38
-5	-1	-9	32.08	32.77
-4	-1	-9	11.92	11.89
-3	-1	-9	20.77	21.09
-1	-1	-9	14.73	14.85
-1	-2	-9	11.60	11.10
-4	-2	-9	18.53	19.16
-5	-2	-9	20.49	20.24
-6	-2	-9	24.59	25.05
-7	-2	-9	10.32	9.71
-8	-2	-9	7.27	7.38
-9	-3	-9	4.41	4.21
-6	-3	-9	3.96	3.94
-5	-3	-9	11.08	11.38
-3	-3	-9	9.54	9.71
-1	-3	-9	17.76	17.38
-2	-4	-9	7.12	7.16
-3	-4	-9	8.33	9.01
-5	-4	-9	4.98	5.03
-6	-4	-9	7.65	8.11
-7	-4	-9	13.66	13.37
-8	-4	-9	3.29	4.16
-9	-5	-9	4.68	3.91
-8	-5	-9	4.64	4.66
-4	-5	-9	6.49	6.74
-2	-5	-9	9.85	9.57
-1	-5	-9	3.32	2.61
-1	-6	-9	3.92	3.89
-2	-6	-9	4.67	4.54
-3	-6	-9	14.22	14.69
-4	-6	-9	4.14	4.50
-5	-6	-9	20.25	20.86
-6	-6	-9	8.84	8.92
-7	-6	-9	6.79	7.13
-7	-7	-9	3.49	4.44
-4	-7	-9	5.29	5.65
-1	-7	-9	3.87	3.34
-2	-8	-9	4.47	5.55
-5	-8	-9	12.40	12.38
-6	-8	-9	5.63	5.16
-3	-9	-9	5.08	5.66
-1	-9	-9	13.88	12.70
-4	-10	-9	5.90	6.15
-2	-11	-9	10.44	9.84
-1	-11	-8	4.22	4.13
-2	-11	-8	4.85	4.30
-2	-10	-8	4.78	4.32
-1	-9	-8	8.53	8.16
-3	-9	-8	5.44	5.35
-4	-9	-8	5.64	5.42
-5	-9	-8	7.67	7.91
-7	-9	-8	5.14	4.68
-8	-8	-8	8.28	8.14
-7	-8	-8	7.34	6.84
-5	-8	-8	4.76	5.15
-4	-8	-8	15.06	14.89
-3	-8	-8	4.66	4.88
-2	-8	-8	8.13	8.71
-1	-8	-8	3.79	3.21
-1	-7	-8	10.14	10.78
-3	-7	-8	5.21	5.29
-5	-7	-8	4.79	4.37
-6	-7	-8	7.25	7.43
-8	-6	-8	8.56	8.95
-7	-6	-8	17.74	17.75
-6	-6	-8	13.74	14.28
-5	-6	-8	14.73	14.38
-4	-6	-8	15.40	16.10
-3	-6	-8	3.08	2.72
-1	-6	-8	6.07	6.18
-1	-5	-8	12.96	12.90
-2	-5	-8	4.15	3.66
-5	-5	-8	12.90	13.34
-6	-5	-8	10.94	11.91
-7	-5	-8	3.24	2.53
-8	-4	-8	7.19	7.96
-6	-4	-8	5.37	5.75
-4	-4	-8	3.56	3.83
-3	-4	-8	4.02	3.53
-1	-4	-8	5.37	5.40
-1	-3	-8	23.64	23.18
-2	-3	-8	6.06	6.42
-3	-3	-8	13.21	12.11
-5	-3	-8	5.42	4.97
-7	-3	-8	4.81	4.44
-11	-2	-8	6.65	6.49
-8	-2	-8	9.15	8.14
-7	-2	-8	9.82	9.97
-6	-2	-8	9.35	9.23
-5	-2	-8	9.23	9.74
-4	-2	-8	17.88	18.33
-3	-2	-8	17.26	17.13
-2	-2	-8	14.99	14.48
-1	-2	-8	34.46	33.26
-1	-1	-8	19.72	20.88
-2	-1	-8	13.21	13.10
-3	-1	-8	33.96	34.05
-4	-1	-8	4.37	4.08
-5	-1	-8	33.44	33.22
-7	-1	-8	10.30	10.32
-8	-1	-8	6.79	6.41
-9	-1	-8	7.84	8.04
-10	0	-8	13.18	13.54
-8	0	-8	26.63	26.54
-6	0	-8	36.82	37.08
-4	0	-8	9.18	8.48
-2	0	-8	23.62	23.42
-1	0	-7	5.06	5.58
-3	0	-7	21.59	20.24
-5	0	-7	25.32	24.29
-7	0	-7	5.01	5.43
-9	0	-7	8.89	9.33
-11	-1	-7	15.48	15.06
-10	-1	-7	5.19	5.41
-9	-1	-7	7.76	8.11
-8	-1	-7	16.20	15.80
-7	-1	-7	5.14	4.79
-6	-1	-7	12.67	12.30
-5	-1	-7	5.48	5.63

h	k	l	Fo	Fc
-4	-1	-7	8.01	8.65
-3	-1	-7	24.08	24.51
-2	-1	-7	17.28	17.00
-1	-2	-7	59.99	58.82
-2	-2	-7	16.00	15.66
-3	-2	-7	7.51	7.38
-4	-2	-7	31.94	30.84
-5	-2	-7	6.79	7.01
-6	-2	-7	14.99	14.51
-7	-2	-7	5.92	5.51
-8	-2	-7	12.69	12.83
-9	-2	-7	5.19	5.14
-10	-2	-7	8.21	7.94
-11	-2	-7	7.14	6.89
-9	-3	-7	3.91	2.91
-8	-3	-7	7.99	7.91
-7	-3	-7	6.59	6.82
-6	-3	-7	4.79	4.94
-5	-3	-7	7.49	7.45
-4	-3	-7	14.76	14.50
-3	-3	-7	14.12	13.18
-2	-3	-7	24.46	22.80
-1	-3	-7	33.60	34.24
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-3 0	9		7.24	8.37
-5 0	9		9.20	9.31
-7 0	9		7.02	7.67
-9 0	9		4.26	3.40
-11 0	9		5.90	6.31
-12 -1	9		4.60	4.25
-10 -1	9		13.73	12.78
-4 -1	9		15.28	16.41
-3 -1	9		5.72	5 50
-2 -1	9		31.31	30.75
-1 -1	9		4.53	5.08

h	k	l	Fo	Fc
0	-1	9	4.51	4.31
0	-2	9	4.48	4.14
-1	-2	9	7.98	8.29
-2	-2	9	15.05	15.56
-5	-2	9	19.85	20.21
-6	-2	9	9.09	9.57
-9	-2	9	8.61	8.90
-11	-2	9	6.98	6.52
-8	-3	9	21.12	22.08
-5	-3	9	22.39	24.00
-4	-3	9	14.94	15.49
-3	-3	9	7.97	8.48
-2	-3	9	21.90	21.55
-1	-3	9	18.99	19.88
0	-3	9	13.16	12.45
-1	-4	9	6.87	7.42
-3	-4	9	7.03	6.24
-4	-4	9	27.04	27.60
-5	-4	9	25.27	26.63
-6	-4	9	11.50	12.76
-7	-4	9	30.88	30.90
-11	-5	9	8.64	7.60
-8	-5	9	6.13	5.94
-6	-5	9	17.04	17.41
-5	-5	9	4.03	4.75
-4	-5	9	6.32	6.66
-2	-5	9	6.07	6.32
-1	-5	9	10.11	9.74
0	-5	9	4.50	4.63
0	-6	9	9.43	9.74
-1	-6	9	5.27	5.08
-2	-6	9	5.56	5.37
-3	-6	9	8.34	8.37
-5	-6	9	13.18	12.82
-9	-6	9	6.05	5.93
-7	-7	9	7.19	6.91
-6	-7	9	4.02	4.51
-5	-7	9	16.11	15.97
-3	-7	9	21.82	22.37
-1	-7	9	10.14	10.56
0	-7	9	11.89	10.86
-1	-8	9	6.03	6.25
-3	-8	9	3.85	3.82
-4	-8	9	4.76	4.03
-5	-8	9	4.93	4.98
-9	-8	9	5.08	4.89
-5	-9	9	4.69	3.89
-4	-9	9	4.28	4.52
-3	-9	9	5.03	4.75
-2	-9	9	9.00	9.29
-1	-9	9	5.74	5.88
0	-9	9	6.06	6.02
-1	-10	9	4.26	4.18
-2	-10	9	4.24	4.56
-3	-10	9	5.45	5.19
-4	-10	9	5.37	5.15
-6	-10	9	8.90	7.75
-7	-10	9	8.35	7.24
-4	-11	9	5.18	5.14
0	-11	9	9.45	8.90
-4	-10	10	5.72	5.34
-2	-10	10	3.76	4.17
-2	-9	10	6.58	6.18
-3	-9	10	12.41	12.16
-4	-9	10	10.42	10.59
-5	-9	10	7.53	7.60
-6	-8	10	9.69	9.53
-5	-8	10	4.20	4.88
-4	-8	10	5.63	5.88
-3	-8	10	7.85	8.03
-2	-8	10	24.53	24.64
0	-8	10	7.50	7.66
0	-7	10	11.73	12.52
-2	-7	10	7.31	7.53
-3	-7	10	10.44	9.66
-4	-7	10	7.90	7.80
-5	-7	10	7.31	7.44
-7	-7	10	5.47	5.20
-10	-6	10	11.74	11.02
-5	-6	10	10.88	11.02
-2	-6	10	5.69	5.51
-1	-5	10	10.95	10.64
-2	-5	10	8.43	8.58
-5	-5	10	5.66	5.30
-11	-5	10	6.08	5.29
-10	-4	10	7.06	7.19
-6	-4	10	7.55	7.52
-5	-4	10	8.91	9.95
-1	-4	10	4.26	3.18
0	-4	10	10.51	10.51
-2	-3	10	4.87	4.64
-3	-3	10	28.90	29.91
-4	-3	10	24.13	24.73
-5	-3	10	24.55	25.50
-6	-3	10	11.78	11.93
-7	-3	10	4.80	5.49
-10	-3	10	5.32	6.20
-8	-2	10	4.75	5.06
-7	-2	10	16.19	16.92
-6	-2	10	9.89	9.69
-5	-2	10	37.30	38.60
-4	-2	10	15.43	16.08
-3	-2	10	6.98	6.33
-1	-2	10	4.14	4.32
0	-2	10	15.15	14.27
0	-1	10	17.32	18.16
-1	-1	10	10.08	10.34
-2	-1	10	8.39	7.88
-3	-1	10	5.45	5.84
-4	-1	10	6.25	6.72
-5	-1	10	16.81	16.48
-7	-1	10	15.28	15.12
-8	-1	10	6.19	6.52
-9	-1	10	8.32	7.75
-11	-1	10	6.26	5.43
-10	0	10	10.51	11.39
-8	0	10	7.69	7.96
-4	0	10	14.44	14.55
0	0	10	9.63	9.42
-1	0	11	11.89	11.59
-3	0	11	7.94	7.78
-5	0	11	25.91	25.89
-7	0	11	15.32	15.38
-11	0	11	4.76	4.30
-9	-1	11	5.83	6.63
-8	-1	11	7.34	8.04
-7	-1	11	7.84	8.21
-6	-1	11	27.18	28.13
-5	-1	11	4.85	4.07
-4	-1	11	8.14	8.54
-3	-1	11	6.53	6.88
-2	-1	11	7.74	7.84
0	-2	11	5.23	4.79
-1	-2	11	4.13	3.84
-3	-2	11	28.97	29.48
-4	-2	11	21.34	21.71
-5	-2	11	23.12	23.80
-7	-2	11	4.20	4.57
-8	-2	11	5.68	6.77
-6	-3	11	17.61	18.32
-5	-3	11	10.70	11.30
-4	-3	11	16.34	16.90
-3	-3	11	27.92	29.38
-1	-3	11	10.63	10.83
0	-3	11	10.35	10.33
-1	-4	11	6.88	6.89
-2	-4	11	6.81	6.61
-4	-4	11	5.69	6.13
-5	-4	11	14.81	15.16
-9	-5	11	7.09	6.71
-8	-5	11	6.51	7.09
-7	-5	11	4.96	5.89
-6	-5	11	10.15	10.68
-5	-5	11	9.30	9.14
-1	-5	11	4.60	3.96
-3	-6	11	4.69	5.00
-5	-6	11	8.91	9.09
-10	-6	11	6.23	5.37
-6	-7	11	5.88	6.15
-4	-7	11	8.13	8.43
-3	-7	11	12.54	12.91
-1	-7	11	8.45	8.88
0	-8	11	8.25	7.69
-2	-8	11	11.64	11.32
-3	-8	11	6.03	5.70
-4	-8	11	7.84	8.15
-5	-8	11	6.76	7.21
-6	-8	11	5.53	6.00
-7	-8	11	4.71	4.28
-1	-9	11	4.83	5.25
0	-7	12	6.84	6.33
-4	-7	12	8.08	7.82
-5	-6	12	6.67	6.73
0	-5	12	9.98	10.99
-1	-5	12	10.68	10.73
-3	-5	12	6.85	6.93
-4	-5	12	7.07	7.35
-5	-5	12	7.70	7.94
-8	-5	12	5.83	5.53
-10	-5	12	4.29	3.37
-3	-4	12	10.30	10.01
-2	-4	12	6.17	5.95
-1	-3	12	8.22	8.16
-4	-3	12	5.32	5.79
-5	-3	12	6.72	6.69
-6	-3	12	14.75	15.45
-7	-3	12	6.69	6.98
-8	-3	12	8.12	8.08
-8	-2	12	15.70	16.22
-7	-2	12	4.95	4.15
-6	-2	12	19.22	19.53
-5	-2	12	21.92	21.67
-4	-2	12	13.84	14.65
-3	-2	12	19.91	20.13
-2	-2	12	11.56	11.21
-1	-2	12	6.94	6.31
0	-2	12	8.76	9.30
0	-1	12	15.56	16.42
-1	-1	12	24.36	24.63
-3	-1	12	8.30	8.13
-4	-1	12	16.49	16.68
-5	-1	12	11.04	11.09
-10	0	12	6.09	5.09
-6	0	12	15.65	16.13
-4	0	12	6.39	6.66
-2	0	12	19.31	18.78
0	0	12	10.09	9.37
-1	0	13	12.56	11.90
-3	0	13	6.15	6.32
-5	0	13	6.43	6.13
-9	0	13	7.07	6.00
-7	-1	13	7.07	7.12
-6	-1	13	12.34	12.83
-4	-1	13	15.04	15.24
-1	-1	13	24.44	24.62
0	-2	13	9.38	9.30
-1	-2	13	7.94	7.85
-2	-2	13	13.13	13.46
-4	-2	13	9.67	9.97
-5	-2	13	11.53	11.26
-7	-2	13	8.34	8.01
-9	-2	13	6.63	6.29
-9	-3	13	9.45	8.69
-7	-3	13	5.92	6.15
-6	-3	13	4.43	4.26
-4	-3	13	7.20	7.35
-2	-3	13	5.66	5.24
-1	-3	13	4.48	4.30
0	-3	13	11.20	11.43
-1	-4	13	3.92	3.88
-2	-4	13	7.92	7.90
-5	-4	13	10.98	10.39
-6	-4	13	8.14	8.87
-7	-4	13	6.66	6.43
-8	-4	13	7.29	7.52
0	-5	13	8.36	7.78
-1	-6	13	8.89	8.82
-4	-6	13	4.32	4.09
-5	-6	13	6.02	5.90
-6	-7	13	5.72	5.28
-4	-7	13	6.31	6.27
0	-7	13	7.29	7.00
-1	-8	13	4.79	3.52
-2	-7	14	5.13	5.16
-5	-6	14	9.76	9.85
-3	-6	14	4.21	4.45
-1	-6	14	11.45	11.52
0	-6	14	8.00	7.93
-2	-5	14	10.85	10.38
-5	-5	14	6.07	6.13
-6	-5	14	6.38	6.61
-8	-4	14	6.18	5.61
-6	-4	14	5.94	5.87
-5	-4	14	5.03	5.14
-4	-4	14	12.23	12.54
-3	-4	14	9.94	10.12
-2	-4	14	4.73	5.30
0	-4	14	4.62	4.60
-2	-3	14	11.44	11.05
-4	-3	14	13.48	13.06
-9	-3	14	10.54	8.66
-9	-2	14	5.07	5.14
-8	-2	14	5.92	5.14
-6	-2	14	6.19	6.51
-1	-2	14	6.57	7.12
-2	-1	14	15.40	14.83
-3	-1	14	4.62	4.62
-5	-1	14	9.32	9.95
-6	-1	14	6.78	6.84
-7	-1	14	12.63	12.66
-8	0	14	4.41	4.07
-4	0	14	5.02	4.67
-2	0	14	27.77	28.45
0	0	14	38.29	38.14
-1	0	15	5.25	4.91
-3	0	15	4.84	4.56
-5	0	15	4.80	4.51
-2	-1	15	9.83	9.70
-1	-1	15	5.51	5.01
0	-1	15	13.48	14.34
-8	-2	15	7.52	6.84
-7	-3	15	8.30	7.99
-5	-3	15	4.93	4.50
-4	-3	15	6.59	6.00
-2	-3	15	9.93	9.36
0	-4	15	7.92	7.25
-1	-4	15	6.20	5.62
-2	-4	15	6.09	6.28
-3	-4	15	12.77	12.84
-5	-4	15	6.88	6.97

h	k	l	Fo	Fc
-6	-4	15	5.07	5.15
-4	-5	15	12.31	11.42
-2	-5	15	9.09	9.25
-1	-5	15	5.83	5.11
0	-5	15	7.44	7.35
0	-6	15	5.24	4.53
-3	-6	15	4.43	4.73
-1	-5	16	7.98	7.25
-2	-5	16	9.56	9.37
-3	-4	16	5.11	5.19
-1	-4	16	4.52	4.75
0	-4	16	6.62	6.44
-3	-3	16	7.76	7.10
-5	-3	16	5.74	5.53
-1	-1	16	5.66	5.42
-3	-1	16	4.25	4.70
-4	-1	16	3.86	3.53
-2	0	16	5.45	4.80
0	0	16	17.74	17.48
-4	-1	17	4.13	4.11
-2	-1	17	5.29	5.44
-2	-2	17	4.22	4.11

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STRUCTURE FACTOR TABLES FOR AN α-CYCLOBUTYL TRICYCLIC ENONE

h	k	l	Fo	Fc
-14	0	0	5.88*	5.41
-12	0	0	48.47	49.81
-10	0	0	24.41	23.58
-8	0	0	0.0 *	1.88
-6	0	0	16.19	17.05
-4	0	0	117.98	114.70
-2	0	0	121.46	119.49
2	0	0	121.95	119.49
4	0	0	117.93	114.70
6	0	0	16.12	17.05
8	0	0	0.0 *	1.88
10	0	0	24.03	23.58
12	0	0	48.17	49.81
14	0	0	10.29*	5.41
14	1	0	21.58	21.32
13	1	0	10.47*	8.72
12	1	0	15.75	15.80
11	1	0	23.08	23.40
10	1	0	9.90*	10.86
9	1	0	0.0 *	0.63
8	1	0	0.0 *	2.24
7	1	0	9.91	9.69
6	1	0	31.55	31.53
5	1	0	2.95*	4.37
4	1	0	25.65	23.66
3	1	0	63.56	63.43
2	1	0	82.56	87.03
1	1	0	50.26	51.24
-1	1	0	52.99	51.23
-2	1	0	88.03	87.03
-3	1	0	62.79	63.43
-4	1	0	24.79	23.66
-5	1	0	5.39*	4.37
-6	1	0	31.19	31.53
-7	1	0	10.53	9.69
-8	1	0	3.86*	2.25
-9	1	0	0.0 *	0.63
-10	1	0	9.45*	10.86
-11	1	0	23.22	23.40
-12	1	0	13.45	15.80
-13	1	0	8.93*	8.72
-14	1	0	20.66	21.32
-13	2	0	10.71*	13.37
-12	2	0	6.01*	9.25
-11	2	0	8.00*	6.56
-10	2	0	18.06	16.67
-9	2	0	4.60*	2.70
-8	2	0	10.96	11.01
-7	2	0	6.45*	3.50
-6	2	0	10.24	10.42
-5	2	0	21.87	21.23
-4	2	0	7.03*	8.16
-3	2	0	5.65*	7.43
-2	2	0	10.94	11.54
-1	2	0	4.17*	2.58
0	2	0	53.27	53.76
1	2	0	4.61*	2.58
2	2	0	10.33	11.54
3	2	0	5.71*	7.43
4	2	0	9.13	8.16
5	2	0	21.92	21.23
6	2	0	7.53*	10.42
7	2	0	0.0 *	3.50
8	2	0	10.16	11.01
9	2	0	2.22*	2.70
10	2	0	16.59	16.67
11	2	0	4.74*	6.56
12	2	0	10.28*	9.25
13	2	0	12.83	13.37
12	3	0	0.0 *	9.60
11	3	0	0.0 *	4.94
10	3	0	4.87*	7.65
9	3	0	6.88*	11.91
8	3	0	12.25	11.82
7	3	0	1.15*	5.95
6	3	0	15.90	16.60
5	3	0	5.01*	7.02
4	3	0	0.0 *	2.91
3	3	0	40.41	37.40
2	3	0	24.62	24.84
1	3	0	29.74	31.12
-1	3	0	30.56	31.12
-2	3	0	25.60	24.84
-3	3	0	42.46	37.40
-4	3	0	3.01*	2.91
-5	3	0	1.35*	7.02
-6	3	0	16.40	16.60
-7	3	0	7.69*	5.95
-8	3	0	8.90*	11.82
-9	3	0	12.55	11.91
-10	3	0	0.0 *	7.65
-11	3	0	6.14*	4.94
-12	3	0	0.0 *	9.60
-11	4	0	8.32*	1.51
-10	4	0	7.11*	0.39
-9	4	0	4.46*	5.16
-8	4	0	0.0 *	3.24
-7	4	0	8.60*	10.08
-6	4	0	0.0 *	1.91
-5	4	0	14.40	13.41
-4	4	0	0.0 *	1.17
-3	4	0	11.16	10.06
-2	4	0	18.68	18.59
-1	4	0	18.19	17.62
0	4	0	4.76*	2.26
1	4	0	18.34	17.62
2	4	0	17.78	18.59
3	4	0	9.95	10.06
4	4	0	0.0 *	1.17
5	4	0	14.04	13.41
6	4	0	0.0 *	1.91
7	4	0	10.05*	10.08
8	4	0	0.0 *	3.24
9	4	0	0.0 *	5.16
10	4	0	0.0 *	0.39
11	4	0	10.24*	1.51
10	5	0	0.0 *	3.77
9	5	0	4.08*	7.24
8	5	0	8.95*	3.68
7	5	0	6.79*	0.34
6	5	0	0.0 *	2.43
5	5	0	0.0 *	3.71
4	5	0	5.49*	3.84
3	5	0	12.62	12.32
2	5	0	6.58*	5.03
1	5	0	16.88	17.45
-1	5	0	18.37	17.45
-2	5	0	7.13*	5.03
-3	5	0	12.29	12.32
-4	5	0	4.37*	3.84
-5	5	0	5.62*	3.71
-6	5	0	4.58*	2.43
-7	5	0	7.80*	0.34
-8	5	0	7.40*	3.68
-9	5	0	7.07*	7.24
-10	5	0	2.98*	3.77
-7	6	0	5.11*	9.83
-6	6	0	6.13*	1.14
-5	6	0	7.63*	6.02
-4	6	0	0.0 *	3.03
-3	6	0	7.00*	9.54
-2	6	0	6.52*	7.81
-1	6	0	0.0 *	1.12
0	6	0	13.82	15.34
1	6	0	5.23*	1.11
2	6	0	8.49*	7.81
3	6	0	9.96*	9.54
4	6	0	0.0 *	3.03
5	6	0	6.96*	6.02
6	6	0	0.0 *	1.14
7	6	0	10.43*	9.83
1	7	0	0.0 *	0.88
-1	7	0	0.0 *	0.88
-1	7	1	0.0 *	1.16
0	7	1	8.16*	1.06
7	6	1	0.0 *	5.58
6	6	1	9.97*	11.93
5	6	1	5.70*	2.34
4	6	1	11.65	13.33
3	6	1	6.21*	6.94
2	6	1	9.26*	2.06
1	6	1	8.99*	7.42
0	6	1	0.0 *	1.86
-1	6	1	13.80	13.20
-2	6	1	0.0 *	2.59
-3	6	1	9.71*	1.02
-4	6	1	0.0 *	3.55
-5	6	1	5.09*	0.55
-6	6	1	0.0 *	5.20
-7	6	1	8.19*	1.50
-10	5	1	17.94	17.31
-9	5	1	0.0 *	6.10
-8	5	1	5.39*	1.02
-7	5	1	0.0 *	0.58
-6	5	1	10.99	10.16
-5	5	1	6.68*	5.87
-4	5	1	7.12*	4.88
-3	5	1	16.19	16.44
-2	5	1	14.00	13.75
-1	5	1	7.48*	7.04
0	5	1	12.57	12.57
1	5	1	5.83*	2.11
2	5	1	3.47*	6.19
3	5	1	14.68	16.05
4	5	1	4.91*	2.93
5	5	1	0.0 *	0.21
6	5	1	10.76*	10.07
7	5	1	0.0 *	0.20
8	5	1	0.0 *	3.83
9	5	1	6.34*	4.20
11	4	1	0.0 *	11.00
10	4	1	11.35*	6.69
9	4	1	0.0 *	3.03
8	4	1	8.47*	7.04
7	4	1	9.59*	9.50
6	4	1	12.31	11.87
5	4	1	10.48	10.60
4	4	1	9.27*	7.74
3	4	1	0.0 *	3.60
2	4	1	14.76	14.18
1	4	1	5.58*	8.70
0	4	1	27.94	31.16
-1	4	1	22.34	21.75
-2	4	1	9.52	8.08
-3	4	1	25.01	23.45
-4	4	1	20.41	17.87
-5	4	1	0.0 *	4.73
-6	4	1	0.0 *	3.03
-7	4	1	0.0 *	0.43
-8	4	1	0.0 *	1.70
-9	4	1	8.22*	2.74
-10	4	1	6.93*	2.44
-11	4	1	9.43*	5.10
-13	3	1	8.74*	10.05
-12	3	1	0.0 *	0.29
-11	3	1	8.61*	2.09
-10	3	1	0.0 *	6.22
-9	3	1	17.43	18.22
-8	3	1	5.71*	6.15
-7	3	1	7.54*	10.00
-6	3	1	5.95*	9.27
-5	3	1	2.94*	2.69
-4	3	1	7.12*	5.03
-3	3	1	22.71	19.57
-2	3	1	6.41*	5.50
-1	3	1	17.42	19.54
0	3	1	3.89*	1.15
1	3	1	2.45*	0.86
2	3	1	0.0 *	0.05
3	3	1	10.46	11.15
4	3	1	8.25*	10.63
5	3	1	18.92	19.80
6	3	1	11.19	11.11
7	3	1	15.66	16.37
8	3	1	7.08*	3.94
9	3	1	12.26	11.07
10	3	1	2.66*	0.01
11	3	1	7.97*	6.64
12	3	1	9.82*	1.10
13	2	1	0.0 *	4.98
12	2	1	5.75*	6.64
11	2	1	8.83*	5.86
10	2	1	0.0 *	4.88
9	2	1	17.43	17.55
8	2	1	0.0 *	3.45
7	2	1	29.32	29.50
6	2	1	26.04	26.04
5	2	1	27.24	27.07
4	2	1	22.88	23.26
3	2	1	11.00	12.07
2	2	1	32.18	32.26
1	2	1	27.73	28.75
0	2	1	24.71	27.82
-1	2	1	22.63	24.82
-2	2	1	25.44	24.86
-3	2	1	57.38	58.95
-4	2	1	12.33	12.50
-5	2	1	12.64	12.75
-6	2	1	33.50	32.31
-7	2	1	12.61	11.59
-8	2	1	13.33	11.43
-9	2	1	2.40*	5.38
-10	2	1	8.49*	1.23
-11	2	1	0.0 *	1.43
-12	2	1	9.17*	6.89
-13	2	1	18.14	18.54
-14	1	1	9.62*	11.64
-13	1	1	7.79*	0.01
-12	1	1	26.82	28.28
-11	1	1	33.52	34.46
-10	1	1	9.03*	3.99
-9	1	1	4.65*	2.86
-8	1	1	31.04	30.77
-7	1	1	12.16	12.00
-6	1	1	11.35	11.52
-5	1	1	16.35	14.35
-4	1	1	74.78	73.24
-3	1	1	100.31	96.99
-2	1	1	65.52	67.40
-1	1	1	3.30*	3.83
0	1	1	84.34	93.60
1	1	1	129.22	146.46
2	1	1	72.07	73.12
3	1	1	32.74	30.23
4	1	1	32.99	32.23
5	1	1	16.84	15.68
6	1	1	9.67	10.40
7	1	1	0.0 *	2.64
8	1	1	14.33	13.40
9	1	1	1.50*	4.40

h	k	l	Fo	Fc
10	1	1	2.13*	2.69
11	1	1	24.68	26.14
12	1	1	4.52*	2.62
13	1	1	26.37	27.76
13	0	1	9.62*	10.73
11	0	1	34.91	34.76
9	0	1	24.95	26.66
7	0	1	4.06*	5.64
5	0	1	27.45	26.95
3	0	1	51.82	53.71
1	0	1	79.98	79.21
-1	0	1	13.35	13.89
-3	0	1	11.12	10.23
-5	0	1	87.67	83.35
-7	0	1	20.57	20.99
-9	0	1	0.0 *	0.30
-11	0	1	10.81	11.18
-13	0	1	3.40*	7.84
-14	0	2	14.87	14.93
-12	0	2	0.0 *	4.77
-10	0	2	14.23	13.61
-8	0	2	0.0 *	4.84
-6	0	2	46.65	45.30
-4	0	2	41.60	42.68
-2	0	2	80.54	86.43
0	0	2	66.56	66.92
2	0	2	211.08	215.78
4	0	2	65.26	64.36
6	0	2	63.73	62.75
8	0	2	0.0 *	1.50
10	0	2	6.31*	0.37
12	0	2	33.95	34.95
13	1	2	9.62*	7.24
12	1	2	10.43*	10.72
11	1	2	11.75	10.85
10	1	2	15.04	16.01
9	1	2	0.0 *	8.50
8	1	2	7.57*	2.21
7	1	2	6.71*	4.36
6	1	2	31.57	32.01
5	1	2	17.92	18.85
4	1	2	2.05*	7.07
3	1	2	64.46	63.29
2	1	2	31.27	32.48
1	1	2	126.96	127.84
0	1	2	31.31	37.30
-1	1	2	8.75*	3.58
-2	1	2	7.86	7.02
-3	1	2	43.60	42.38
-4	1	2	50.59	49.90
-5	1	2	22.28	21.10
-6	1	2	0.0 *	2.98
-7	1	2	7.50*	6.71
-8	1	2	29.05	29.17
-9	1	2	13.74	14.10
-10	1	2	6.29*	5.27
-11	1	2	27.44	27.61
-12	1	2	8.36*	10.80

h	k	l	Fo	Fc
-13	1	2	2.02*	7.33
-14	1	2	0.0 *	0.61
-14	2	2	18.48	18.27
-13	2	2	8.59*	9.84
-12	2	2	9.30*	11.96
-11	2	2	14.11	14.08
-10	2	2	0.0 *	1.48
-9	2	2	0.0 *	1.51
-8	2	2	5.42*	4.06
-7	2	2	2.77*	0.56
-6	2	2	12.64	13.00
-5	2	2	9.44	7.35
-4	2	2	30.51	28.75
-3	2	2	9.69	11.27
-2	2	2	20.68	21.27
-1	2	2	21.58	19.53
0	2	2	44.92	49.30
1	2	2	49.02	53.98
2	2	2	34.94	38.48
3	2	2	2.61*	3.02
4	2	2	22.36	21.50
5	2	2	13.08	10.79
6	2	2	0.0 *	3.82
7	2	2	9.75	10.09
8	2	2	7.36*	7.96
9	2	2	8.84*	9.87
10	2	2	0.0 *	10.71
11	2	2	13.56	13.20
12	2	2	10.01*	3.91
13	2	2	2.09*	0.05
12	3	2	0.0 *	2.52
11	3	2	7.18*	6.21
10	3	2	0.0 *	6.29
9	3	2	5.00*	8.54
8	3	2	15.22	14.92
7	3	2	4.72*	5.19
6	3	2	13.88	14.59
5	3	2	7.62*	5.16
4	3	2	6.79*	13.63
3	3	2	8.29*	8.02
2	3	2	15.00	17.36
1	3	2	3.13*	2.33
0	3	2	11.81	10.74
-1	3	2	18.30	18.83
-2	3	2	0.0 *	3.06
-3	3	2	48.52	49.13
-4	3	2	20.24	18.32
-5	3	2	6.85*	5.06
-6	3	2	14.26	14.34
-7	3	2	10.45	11.19
-8	3	2	11.48	9.92
-9	3	2	0.0 *	1.60
-10	3	2	0.0 *	3.10
-11	3	2	0.0 *	1.81
-12	3	2	19.97	20.34
-13	3	2	4.42*	4.95
-12	4	2	0.0 *	1.84
-11	4	2	7.84*	4.81

h	k	l	Fo	Fc
-10	4	2	0.0 *	1.17
-9	4	2	18.41	19.12
-8	4	2	0.0 *	2.87
-7	4	2	6.06*	5.31
-6	4	2	0.0 *	1.28
-5	4	2	13.36	11.12
-4	4	2	19.33	20.16
-3	4	2	38.59	37.56
-2	4	2	9.17	8.06
-1	4	2	37.51	37.46
0	4	2	26.87	27.29
1	4	2	18.86	18.29
2	4	2	2.21*	4.36
3	4	2	0.0 *	6.55
4	4	2	8.29*	6.87
5	4	2	6.82*	6.52
6	4	2	6.97*	1.69
7	4	2	3.13*	6.34
8	4	2	9.73*	6.48
9	4	2	6.30*	8.62
10	4	2	4.03*	0.92
11	4	2	8.89*	10.17
9	5	2	0.0 *	3.05
8	5	2	4.94*	5.28
7	5	2	0.0 *	1.86
6	5	2	3.01*	3.82
5	5	2	5.72*	8.47
4	5	2	7.36*	1.90
3	5	2	0.0 *	4.07
2	5	2	9.59*	7.37
1	5	2	35.77	35.78
0	5	2	8.88*	8.16
-1	5	2	18.13	17.90
-2	5	2	9.50*	12.27
-3	5	2	14.53	13.26
-4	5	2	0.0 *	5.67
-5	5	2	6.92*	0.62
-6	5	2	8.62*	9.41
-7	5	2	6.88*	2.88
-8	5	2	17.78	16.45
-9	5	2	0.0 *	2.78
-10	5	2	10.75	10.46
-7	6	2	0.0 *	2.20
-6	6	2	7.69*	9.38
-5	6	2	7.89*	8.93
-4	6	2	9.33*	7.09
-3	6	2	13.82	13.57
-2	6	2	0.0 *	4.51
-1	6	2	26.69	26.80
0	6	2	18.33	17.42
1	6	2	5.94*	5.51
2	6	2	6.59*	2.74
3	6	2	6.95*	0.74
4	6	2	19.77	18.24
5	6	2	9.74*	11.92
6	6	2	0.0 *	5.41
6	6	3	9.25*	4.66
5	6	3	6.33*	3.15

h	k	l	Fo	Fc
4	6	3	4.03*	4.32
3	6	3	20.50	20.11
2	6	3	4.80*	3.48
1	6	3	0.0 *	11.49
0	6	3	15.07	12.90
-1	6	3	14.68	14.57
-2	6	3	0.0 *	2.16
-3	6	3	5.17*	7.19
-4	6	3	1.40*	3.18
-5	6	3	12.61	12.94
-6	6	3	6.76*	5.98
-7	6	3	8.58*	2.95
-10	5	3	8.72*	3.45
-9	5	3	4.74*	1.67
-8	5	3	30.01	30.14
-7	5	3	14.98	15.10
-6	5	3	6.85*	0.57
-5	5	3	0.0 *	1.36
-4	5	3	0.0 *	1.57
-3	5	3	0.0 *	3.06
-2	5	3	4.63*	6.87
-1	5	3	14.06	14.21
0	5	3	0.0 *	5.10
1	5	3	28.90	28.88
2	5	3	14.41	13.61
3	5	3	6.54*	8.34
4	5	3	4.87*	1.04
5	5	3	12.44	11.76
6	5	3	10.66*	8.30
7	5	3	0.0 *	7.62
8	5	3	10.92*	2.88
9	5	3	5.77*	5.29
10	4	3	4.58*	3.40
9	4	3	5.41*	7.47
8	4	3	5.22*	2.25
7	4	3	0.0 *	1.52
6	4	3	3.08*	5.65
5	4	3	9.77*	10.35
4	4	3	8.34*	6.04
3	4	3	11.78	9.89
2	4	3	10.02	10.72
1	4	3	18.46	18.34
0	4	3	18.78	19.18
-1	4	3	37.64	35.22
-2	4	3	7.63*	11.86
-3	4	3	2.73*	2.47
-4	4	3	0.0 *	3.55
-5	4	3	6.80*	3.33
-6	4	3	13.22	11.92
-7	4	3	7.66*	7.92
-8	4	3	13.43	12.30
-9	4	3	0.0 *	1.75
-10	4	3	13.42	13.86
-11	4	3	0.0 *	3.97
-12	4	3	7.85*	7.57
-13	3	3	0.0 *	4.29
-12	3	3	8.14*	7.36
-11	3	3	3.58*	3.94

h	k	l	Fo	Fc
-10	3	3	7.88*	7.69
-9	3	3	6.90*	1.85
-8	3	3	16.30	15.83
-7	3	3	19.80	19.90
-6	3	3	3.62*	1.36
-5	3	3	1.54*	3.32
-4	3	3	0.0 *	1.30
-3	3	3	68.01	65.76
-2	3	3	48.79	50.89
-1	3	3	34.77	36.55
0	3	3	21.63	22.06
1	3	3	0.0 *	2.80
2	3	3	1.40*	3.92
3	3	3	8.45*	8.75
4	3	3	4.76*	0.78
5	3	3	3.50*	0.83
6	3	3	0.0 *	5.34
7	3	3	0.0 *	4.15
8	3	3	9.60*	8.45
9	3	3	1.86*	9.57
10	3	3	5.99*	4.39
11	3	3	0.0 *	5.22
12	2	3	0.0 *	7.34
11	2	3	6.16*	0.79
10	2	3	21.42	22.81
9	2	3	8.99*	8.84
8	2	3	0.0 *	2.62
7	2	3	4.38*	0.39
6	2	3	29.75	29.59
5	2	3	51.76	50.63
4	2	3	35.01	33.05
3	2	3	2.57*	0.95
2	2	3	33.47	31.94
1	2	3	14.51	15.70
0	2	3	31.93	34.34
-1	2	3	18.82	19.93
-2	2	3	44.24	47.84
-3	2	3	6.71*	10.13
-4	2	3	0.0 *	3.93
-5	2	3	23.05	19.97
-6	2	3	15.46	16.46
-7	2	3	17.55	17.64
-8	2	3	12.53	13.93
-9	2	3	3.70*	0.78
-10	2	3	0.0 *	2.12
-11	2	3	13.28	12.74
-12	2	3	8.48*	10.16
-13	2	3	8.82*	9.85
-14	2	3	14.24	14.60
-14	1	3	0.0 *	4.76
-13	1	3	6.64*	3.37
-12	1	3	9.10*	11.20
-11	1	3	16.68	16.46
-10	1	3	0.0 *	1.51
-9	1	3	12.60	12.39
-8	1	3	35.91	35.54
-7	1	3	40.57	36.33
-6	1	3	6.05*	1.38

h	k	l	Fo	Fc
-5	1	3	7.22*	8.63
-4	1	3	26.79	27.07
-3	1	3	0.0 *	5.78
-2	1	3	18.40	19.44
-1	1	3	62.64	69.05
0	1	3	32.60	33.17
1	1	3	0.0 *	4.29
2	1	3	25.60	26.04
3	1	3	84.16	82.00
4	1	3	30.98	28.59
5	1	3	42.59	41.59
6	1	3	17.71	17.50
7	1	3	38.62	40.75
8	1	3	10.63	11.75
9	1	3	12.55	11.74
10	1	3	9.21*	6.00
11	1	3	0.0 *	8.47
12	1	3	6.76*	8.29
13	1	3	12.16	13.05
13	0	3	15.07	14.05
11	0	3	12.11	11.87
9	0	3	30.72	29.35
7	0	3	28.43	28.41
5	0	3	5.22*	3.79
3	0	3	24.28	23.71
1	0	3	64.55	64.29
-1	0	3	80.14	77.25
-3	0	3	49.43	47.75
-5	0	3	17.08	16.55
-7	0	3	12.54	13.88
-9	0	3	76.16	76.80
-11	0	3	39.67	40.03
-13	0	3	9.00*	2.44
-14	0	4	0.0 *	2.04
-12	0	4	8.27*	9.29
-10	0	4	21.07	18.96
-8	0	4	76.21	71.32
-6	0	4	79.02	75.26
-4	0	4	15.11	16.16
-2	0	4	14.74	14.98
0	0	4	0.0 *	1.67
2	0	4	68.67	68.80
4	0	4	64.15	63.29
6	0	4	6.79*	7.97
8	0	4	19.88	20.29
10	0	4	13.30	13.75
12	0	4	25.26	26.37
12	1	4	13.33	12.62
11	1	4	0.0 *	7.12
10	1	4	20.52	21.18
9	1	4	3.94*	2.93
8	1	4	10.09	9.84
7	1	4	16.21	15.03
6	1	4	18.58	18.50
5	1	4	65.57	64.09
4	1	4	10.64	10.97
3	1	4	19.07	18.91
2	1	4	0.0 *	5.29
1	1	4	83.37	80.15
0	1	4	18.63	17.63
-1	1	4	14.68	14.99
-2	1	4	29.98	31.94
-3	1	4	47.98	48.08
-4	1	4	8.78	8.60
-5	1	4	50.50	47.91
-6	1	4	34.18	34.73
-7	1	4	32.18	31.61
-8	1	4	14.22	13.76
-9	1	4	20.76	19.41
-10	1	4	6.00*	6.43
-11	1	4	0.0 *	3.47
-12	1	4	4.17*	3.42
-13	1	4	0.0 *	1.45
-14	1	4	0.0 *	4.84
-14	2	4	0.0 *	5.48
-13	2	4	14.68	14.71
-12	2	4	9.19*	8.64
-11	2	4	0.0 *	2.12
-10	2	4	0.0 *	3.55
-9	2	4	0.0 *	0.37
-8	2	4	16.74	16.97
-7	2	4	0.0 *	2.88
-6	2	4	29.88	27.62
-5	2	4	6.90*	2.50
-4	2	4	18.34	18.56
-3	2	4	42.30	44.73
-2	2	4	4.31*	11.39
-1	2	4	22.99	24.95
0	2	4	8.50*	7.58
1	2	4	30.00	29.63
2	2	4	16.96	16.57
3	2	4	7.57*	9.48
4	2	4	35.25	35.72
5	2	4	17.82	17.29
6	2	4	13.39	13.26
7	2	4	18.04	16.48
8	2	4	11.68	11.91
9	2	4	0.0 *	8.26
10	2	4	3.01*	4.31
11	2	4	0.0 *	6.38
12	2	4	0.0 *	0.11
11	3	4	0.0 *	0.98
10	3	4	9.19*	4.44
9	3	4	0.0 *	3.63
8	3	4	7.30*	0.92
7	3	4	3.03*	2.12
6	3	4	20.66	20.46
5	3	4	0.0 *	3.19
4	3	4	8.84*	6.38
3	3	4	8.60*	1.33
2	3	4	21.93	24.46
1	3	4	17.20	17.92
0	3	4	46.19	49.23
-1	3	4	23.09	24.95
-2	3	4	52.13	53.44
-3	3	4	25.41	24.92
-4	3	4	2.31*	1.05
-5	3	4	26.84	26.78
-6	3	4	34.52	33.92
-7	3	4	17.38	17.32
-8	3	4	10.07	9.84
-9	3	4	14.03	13.38
-10	3	4	6.05*	6.50
-11	3	4	6.91*	9.89
-12	3	4	15.50	16.69
-13	3	4	9.54	9.82
-12	4	4	1.48*	1.86
-11	4	4	17.65	18.12
-10	4	4	0.0 *	0.06
-9	4	4	4.62*	6.13
-8	4	4	0.0 *	7.56
-7	4	4	17.42	17.92
-6	4	4	5.69*	5.67
-5	4	4	2.64*	1.05
-4	4	4	4.16*	14.19
-3	4	4	14.65	14.23
-2	4	4	8.76*	12.40
-1	4	4	30.99	31.65
0	4	4	14.62	15.45
1	4	4	19.19	18.70
2	4	4	20.60	20.48
3	4	4	0.0 *	5.94
4	4	4	7.91*	10.90
5	4	4	2.15*	4.88
6	4	4	9.51*	7.85
7	4	4	10.77*	9.61
8	4	4	0.0 *	1.42
9	4	4	0.0 *	0.66
10	4	4	9.14*	8.21
8	5	4	9.63*	5.92
7	5	4	7.13*	6.30
6	5	4	7.60*	4.77
5	5	4	6.20*	1.02
4	5	4	1.87*	8.01
3	5	4	10.54*	14.04
2	5	4	4.23*	1.56
1	5	4	18.72	20.10
0	5	4	8.02*	1.13
-1	5	4	0.0 *	7.36
-2	5	4	8.50*	5.21
-3	5	4	0.0 *	0.50
-4	5	4	12.91	13.38
-5	5	4	0.0 *	1.12
-6	5	4	0.0 *	3.37
-7	5	4	4.35*	5.86
-8	5	4	3.95*	1.59
-9	5	4	0.0 *	4.32
-10	5	4	0.0 *	3.92
-7	6	4	5.33*	2.37
-6	6	4	3.24*	8.36
-5	6	4	0.0 *	5.88
-4	6	4	7.72*	0.11
-3	6	4	0.0 *	0.70
-2	6	4	5.55*	1.00
-1	6	4	17.28	16.63
0	6	4	10.09*	8.54
1	6	4	19.15	18.39
2	6	4	15.21	15.79
3	6	4	7.17*	7.47
4	6	4	0.0 *	0.56
5	6	4	8.46*	2.41
4	6	5	0.0 *	5.33
3	6	5	9.59*	10.26
2	6	5	13.46	15.36
1	6	5	18.65	18.45
0	6	5	3.14*	9.93
-1	6	5	8.87*	4.09
-2	6	5	5.62*	2.14
-3	6	5	13.98	13.25
-4	6	5	8.89*	1.56
-5	6	5	0.0 *	3.97
-6	6	5	0.0 *	0.49
-7	6	5	11.34	10.85
-10	5	5	21.39	22.58
-9	5	5	0.0 *	3.74
-8	5	5	3.44*	2.76
-7	5	5	0.0 *	0.08
-6	5	5	8.80*	9.91
-5	5	5	0.0 *	6.32
-4	5	5	7.45*	0.63
-3	5	5	0.0 *	5.97
-2	5	5	8.45*	8.67
-1	5	5	17.43	17.22
0	5	5	16.48	15.81
1	5	5	10.68*	11.20
2	5	5	29.84	33.50
3	5	5	8.09*	8.74
4	5	5	0.0 *	0.35
5	5	5	0.0 *	2.24
6	5	5	8.37*	6.58
7	5	5	11.07*	8.15
9	4	5	0.0 *	3.91
8	4	5	0.0 *	2.84
7	4	5	7.30*	1.19
6	4	5	4.73*	6.86
5	4	5	0.0 *	5.57
4	4	5	6.31*	1.81
3	4	5	13.32	11.86
2	4	5	17.91	18.85
1	4	5	12.55	12.38
0	4	5	19.48	19.61
-1	4	5	11.92	12.77
-2	4	5	7.61*	0.93
-3	4	5	9.62*	10.87
-4	4	5	4.70*	5.54
-5	4	5	7.09*	2.75
-6	4	5	13.18	13.29
-7	4	5	4.55*	0.13
-8	4	5	12.75	11.66
-9	4	5	20.03	19.27
-10	4	5	10.76	11.03
-11	4	5	0.0 *	4.73
-12	4	5	0.0 *	1.08
-13	3	5	15.76	14.32
-12	3	5	14.85	15.46
-11	3	5	0.0 *	1.56
-10	3	5	0.0 *	0.23
-9	3	5	6.29*	0.16
-8	3	5	15.69	15.64
-7	3	5	13.18	13.60
-6	3	5	0.0 *	0.48
-5	3	5	22.16	22.51
-4	3	5	7.18*	8.36
-3	3	5	39.11	41.12
-2	3	5	8.94*	10.89
-1	3	5	8.78*	10.60
0	3	5	20.21	20.90
1	3	5	24.92	25.65
2	3	5	13.06	12.74
3	3	5	23.38	24.16
4	3	5	17.24	18.04
5	3	5	8.23*	6.41
6	3	5	0.0 *	1.22
7	3	5	8.43*	2.38
8	3	5	7.25*	5.79
9	3	5	7.79*	4.10
10	3	5	0.0 *	1.62
11	3	5	8.69*	1.78
11	2	5	0.0 *	8.49
10	2	5	7.55*	5.77
9	2	5	1.32*	2.26
8	2	5	0.0 *	3.20
7	2	5	18.49	19.25
6	2	5	0.0 *	2.30
5	2	5	12.99	12.71
4	2	5	6.19*	4.83
3	2	5	5.84*	7.96
2	2	5	32.75	32.08
1	2	5	8.67	9.42
0	2	5	8.76*	8.27
-1	2	5	51.67	58.71
-2	2	5	69.16	74.55
-3	2	5	6.54*	7.89
-4	2	5	18.68	20.70
-5	2	5	18.71	19.63
-6	2	5	14.48	14.83
-7	2	5	7.87*	6.07
-8	2	5	18.18	18.44
-9	2	5	13.55	12.92
-10	2	5	9.95	9.37
-11	2	5	0.0 *	2.94
-12	2	5	9.65	10.87
-13	2	5	13.50	12.67
-14	2	5	21.17	22.54
-14	1	5	2.51*	0.59
-13	1	5	0.0 *	1.83
-12	1	5	3.51*	2.95
-11	1	5	20.46	19.99
-10	1	5	5.51*	3.89
-9	1	5	35.74	33.29

h	k	l	Fo	Fc
-8	1	5	35.16	33.91
-7	1	5	54.87	50.83
-6	1	5	15.74	15.42
-5	1	5	57.19	54.31
-4	1	5	4.27*	5.36
-3	1	5	36.46	37.93
-2	1	5	54.76	53.96
-1	1	5	24.58	22.91
0	1	5	40.80	39.60
1	1	5	35.96	35.85
2	1	5	14.89	14.31
3	1	5	11.11	12.39
4	1	5	32.09	30.22
5	1	5	86.63	84.26
6	1	5	8.59*	2.23
7	1	5	31.58	30.41
8	1	5	11.55	11.06
9	1	5	3.39*	2.87
10	1	5	17.01	17.24
11	1	5	14.12	13.50
12	1	5	11.16	11.22
11	0	5	20.76	20.97
9	0	5	7.07*	3.46
7	0	5	18.10	16.89
5	0	5	25.81	23.25
3	0	5	85.33	84.67
1	0	5	0.0 *	3.20
-1	0	5	13.77	13.33
-3	0	5	43.84	42.37
-5	0	5	11.43	10.21
-7	0	5	117.90	112.38
-9	0	5	60.12	55.91
-11	0	5	7.18*	8.19
-13	0	5	0.0 *	1.90
-14	0	6	10.24*	6.41
-12	0	6	0.0 *	5.14
-10	0	6	11.98	13.28
-8	0	6	3.77*	0.49
-6	0	6	76.27	70.91
-4	0	6	23.71	23.27
-2	0	6	55.33	55.31
0	0	6	10.48	10.54
2	0	6	34.78	33.36
4	0	6	10.29	10.38
6	0	6	59.34	59.44
8	0	6	6.95*	2.37
10	0	6	0.0 *	1.84
11	1	6	0.0 *	0.98
10	1	6	0.0 *	5.87
9	1	6	0.0 *	3.90
8	1	6	6.56*	4.53
7	1	6	16.15	16.05
6	1	6	3.63*	2.10
5	1	6	23.64	23.71
4	1	6	27.52	27.07
3	1	6	5.23*	7.40
2	1	6	14.35	14.13
1	1	6	33.09	31.55
0	1	6	40.70	40.28
-1	1	6	1.75*	0.24
-2	1	6	15.66	14.70
-3	1	6	11.78	11.73
-4	1	6	20.35	19.83
-5	1	6	4.98*	9.91
-6	1	6	19.74	19.61
-7	1	6	12.85	12.36
-8	1	6	17.23	17.43
-9	1	6	21.14	21.14
-10	1	6	7.89*	4.99
-11	1	6	6.12*	5.14
-12	1	6	8.23*	6.24
-13	1	6	9.77*	8.14
-14	1	6	7.99*	2.80
-14	2	6	0.0 *	6.15
-13	2	6	11.73	12.24
-12	2	6	0.0 *	3.22
-11	2	6	6.57*	4.57
-10	2	6	7.44*	4.97
-9	2	6	7.71*	10.53
-8	2	6	0.0 *	0.12
-7	2	6	0.0 *	0.54
-6	2	6	8.60	8.73
-5	2	6	8.07*	10.16
-4	2	6	16.23	17.31
-3	2	6	13.70	13.00
-2	2	6	64.99	73.51
-1	2	6	66.98	64.41
0	2	6	26.56	25.93
1	2	6	26.09	25.22
2	2	6	9.89	8.88
3	2	6	7.51*	2.24
4	2	6	5.36*	1.55
5	2	6	10.73	9.22
6	2	6	14.30	13.46
7	2	6	12.99	12.83
8	2	6	3.67*	11.04
9	2	6	1.36*	3.40
10	2	6	10.01*	11.06
11	2	6	8.51*	10.12
10	3	6	0.0 *	3.48
9	3	6	0.0 *	1.95
8	3	6	7.80*	6.46
7	3	6	0.0 *	7.00
6	3	6	7.67*	7.07
5	3	6	3.44*	1.45
4	3	6	3.62*	2.58
3	3	6	22.32	22.48
2	3	6	20.14	20.82
1	3	6	15.88	17.10
0	3	6	17.77	19.28
-1	3	6	45.02	48.69
-2	3	6	14.45	14.15
-3	3	6	6.95*	1.00
-4	3	6	14.71	15.13
-5	3	6	2.49*	3.97
-6	3	6	0.0 *	4.19
-7	3	6	11.65	11.30
-8	3	6	6.61*	6.32
-9	3	6	8.54*	11.81
-10	3	6	6.99*	5.25
-11	3	6	9.00*	8.17
-12	3	6	4.94*	3.11
-13	3	6	20.82	20.18
-11	4	6	8.93*	2.49
-10	4	6	11.03	11.48
-9	4	6	32.47	33.65
-8	4	6	5.43*	4.20
-7	4	6	0.0 *	1.98
-6	4	6	8.19*	6.55
-5	4	6	7.33*	7.92
-4	4	6	9.63*	10.78
-3	4	6	7.50*	0.48
-2	4	6	16.68	15.82
-1	4	6	7.44*	5.87
0	4	6	10.45*	11.26
1	4	6	0.0 *	6.58
2	4	6	20.89	21.34
3	4	6	10.48*	12.31
4	4	6	20.89	19.48
5	4	6	7.85*	10.77
6	4	6	5.87*	7.13
7	4	6	5.20*	0.52
8	4	6	17.94	18.10
9	4	6	0.0 *	1.82
7	5	6	0.0 *	2.67
6	5	6	0.0 *	2.48
5	5	6	9.46*	4.37
4	5	6	8.66*	6.50
3	5	6	11.32*	10.37
2	5	6	8.31*	10.16
1	5	6	18.67	19.58
0	5	6	10.75*	9.87
-1	5	6	5.35*	6.61
-2	5	6	0.0 *	1.18
-3	5	6	0.0 *	5.98
-4	5	6	0.0 *	1.28
-5	5	6	9.45*	8.24
-6	5	6	4.04*	3.99
-7	5	6	10.47*	5.84
-8	5	6	8.51*	6.91
-9	5	6	5.25*	3.30
-6	6	6	7.96*	4.41
-5	6	6	2.52*	0.79
-4	6	6	0.0 *	2.59
-3	6	6	4.96*	6.51
-2	6	6	5.14*	1.28
-1	6	6	6.64*	3.61
0	6	6	10.89*	10.17
1	6	6	7.19*	1.79
2	6	6	8.60*	7.88
3	6	6	0.0 *	4.23
2	6	7	11.89	11.29
1	6	7	0.0 *	3.91
0	6	7	4.49*	2.28
-1	6	7	0.0 *	1.62
-2	6	7	0.0 *	1.42
-3	6	7	0.0 *	2.48
-4	6	7	0.0 *	4.11
-5	6	7	9.69*	6.42
-9	5	7	0.0 *	1.14
-8	5	7	21.94	22.51
-7	5	7	0.0 *	1.26
-6	5	7	5.69*	4.87
-5	5	7	0.0 *	1.16
-4	5	7	8.50*	3.32
-3	5	7	0.0 *	7.86
-2	5	7	8.61*	2.36
-1	5	7	4.20*	1.44
0	5	7	5.34*	2.06
1	5	7	0.0 *	2.97
2	5	7	27.68	28.08
3	5	7	8.08*	3.82
4	5	7	43.44	44.10
5	5	7	6.93*	5.34
6	5	7	8.63*	3.11
8	4	7	7.51*	7.04
7	4	7	6.54*	3.28
6	4	7	7.95*	9.09
5	4	7	0.0 *	1.24
4	4	7	5.40*	6.29
3	4	7	23.52	22.96
2	4	7	28.40	28.83
1	4	7	12.73	12.11
0	4	7	7.83*	6.08
-1	4	7	0.0 *	4.55
-2	4	7	14.66	13.72
-3	4	7	8.70*	9.12
-4	4	7	11.54	11.79
-5	4	7	0.0 *	2.12
-6	4	7	0.0 *	5.62
-7	4	7	33.35	32.63
-8	4	7	15.33	15.97
-9	4	7	26.42	25.92
-10	4	7	16.95	16.12
-11	4	7	8.65*	2.74
-12	3	7	22.99	23.89
-11	3	7	31.56	31.27
-10	3	7	9.56*	5.89
-9	3	7	23.10	21.39
-8	3	7	11.74	12.20
-7	3	7	3.63*	1.03
-6	3	7	6.45*	7.01
-5	3	7	3.04*	5.44
-4	3	7	6.76*	5.49
-3	3	7	26.88	28.02
-2	3	7	15.21	15.38
-1	3	7	40.91	39.99
0	3	7	5.64*	5.45
1	3	7	6.78*	8.27
2	3	7	9.79	10.10
3	3	7	6.59*	4.49
4	3	7	2.44*	0.19
5	3	7	6.66*	1.15
6	3	7	10.17*	11.09
7	3	7	0.0 *	3.94
8	3	7	0.0 *	2.53
9	3	7	12.60	11.76
10	2	7	3.56*	8.21
9	2	7	3.95*	2.15
8	2	7	5.69*	8.18
7	2	7	10.00*	11.19
6	2	7	0.0 *	2.32
5	2	7	8.66*	7.19
4	2	7	0.0 *	0.90
3	2	7	5.06*	7.70
2	2	7	14.89	13.32
1	2	7	56.33	54.56
0	2	7	42.68	42.56
-1	2	7	36.20	34.01
-2	2	7	5.53*	5.33
-3	2	7	8.80*	2.56
-4	2	7	0.0 *	8.04
-5	2	7	3.68*	3.76
-6	2	7	14.91	13.84
-7	2	7	27.98	27.74
-8	2	7	21.68	19.79
-9	2	7	11.39	11.48
-10	2	7	0.0 *	3.64
-11	2	7	0.0 *	2.11
-12	2	7	4.15*	7.63
-13	2	7	6.28*	5.47
-14	1	7	0.0 *	1.01
-13	1	7	9.34*	2.88
-12	1	7	2.70*	0.80
-11	1	7	7.86*	1.82
-10	1	7	13.33	13.75
-9	1	7	16.14	18.46
-8	1	7	7.93*	10.95
-7	1	7	7.75*	7.79
-6	1	7	37.93	34.83
-5	1	7	8.99	7.94
-4	1	7	0.0 *	5.23
-3	1	7	18.64	17.61
-2	1	7	11.33	11.04
-1	1	7	34.51	34.32
0	1	7	11.93	10.89
1	1	7	17.74	17.09
2	1	7	11.92	13.18
3	1	7	7.65*	6.10
4	1	7	28.21	27.83
5	1	7	10.47	10.66
6	1	7	1.20*	7.53
7	1	7	20.18	18.71
8	1	7	9.23*	5.14
9	1	7	6.40*	4.35
10	1	7	0.0 *	4.27
11	1	7	11.49	10.97
11	0	7	0.0 *	3.41
9	0	7	10.78	10.19
7	0	7	0.0 *	5.58

h	k	l	Fo	Fc
5	0	7	33.07	31.39
3	0	7	15.19	15.14
1	0	7	6.20*	4.98
-1	0	7	6.68*	1.73
-3	0	7	7.36*	9.29
-5	0	7	22.91	23.99
-7	0	7	35.68	34.37
-9	0	7	14.69	16.47
-11	0	7	0.0 *	6.59
-13	0	7	8.95*	5.74
-14	0	8	6.42*	5.44
-12	0	8	5.76*	3.91
-10	0	8	4.67*	0.25
-8	0	8	14.22	13.35
-6	0	8	46.44	47.16
-4	0	8	27.02	27.52
-2	0	8	32.64	32.83
0	0	8	38.87	36.87
2	0	8	0.0 *	2.76
4	0	8	29.85	29.03
6	0	8	6.53*	4.69
8	0	8	0.0 *	1.63
10	0	8	0.0 *	2.67
10	1	8	0.0 *	7.49
9	1	8	9.96*	10.08
8	1	8	8.45*	6.03
7	1	8	2.90*	3.96
6	1	8	5.29*	5.23
5	1	8	8.47*	4.00
4	1	8	0.0 *	0.19
3	1	8	14.72	14.89
2	1	8	0.0 *	2.62
1	1	8	7.39*	4.59
0	1	8	5.01*	4.04
-1	1	8	5.62*	1.08
-2	1	8	0.0 *	2.60
-3	1	8	30.82	28.92
-4	1	8	3.71*	6.52
-5	1	8	17.42	18.03
-6	1	8	12.41	13.00
-7	1	8	13.38	13.26
-8	1	8	8.25*	1.78
-9	1	8	21.19	21.87
-10	1	8	6.99*	10.21
-11	1	8	0.0 *	0.11
-12	1	8	0.0 *	0.73
-13	1	8	0.0 *	1.98
-14	1	8	0.0 *	2.65
-13	2	8	0.0 *	7.87
-12	2	8	0.0 *	9.40
-11	2	8	16.36	17.15
-10	2	8	8.02*	7.14
-9	2	8	0.0 *	6.13
-8	2	8	0.0 *	3.86
-7	2	8	8.52*	1.71
-6	2	8	7.15*	6.72
-5	2	8	0.0 *	8.78
-4	2	8	9.33*	11.72
-3	2	8	29.36	27.44
-2	2	8	5.73*	5.91
-1	2	8	16.21	15.03
0	2	8	17.10	17.60
1	2	8	20.97	19.68
2	2	8	11.67	11.66
3	2	8	0.0 *	0.04
4	2	8	17.16	16.82
5	2	8	9.03*	5.86
6	2	8	2.23*	0.05
7	2	8	0.0 *	0.28
8	2	8	0.0 *	0.65
9	2	8	0.0 *	2.97
8	3	8	8.64*	5.22
7	3	8	4.17*	1.53
6	3	8	9.52*	8.12
5	3	8	0.0 *	0.38
4	3	8	8.96*	8.35
3	3	8	20.72	19.68
2	3	8	5.89*	0.68
1	3	8	15.83	15.47
0	3	8	6.57*	5.79
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-2	3	8	15.81	18.02
-3	3	8	8.79*	11.64
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-5	3	8	7.21*	1.49
-6	3	8	11.76	12.56
-7	3	8	0.0 *	0.20
-8	3	8	17.97	17.50
-9	3	8	29.34	27.95
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-12	3	8	0.0 *	0.80
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-10	4	8	6.26*	7.45
-9	4	8	18.12	17.29
-8	4	8	17.28	15.91
-7	4	8	25.54	25.04
-6	4	8	12.73	12.21
-5	4	8	5.58*	3.08
-4	4	8	4.73*	2.52
-3	4	8	7.31*	9.51
-2	4	8	8.84*	9.90
-1	4	8	6.14*	3.39
0	4	8	7.02*	1.50
1	4	8	10.11*	10.56
2	4	8	20.99	21.42
3	4	8	24.46	24.15
4	4	8	18.83	18.96
5	4	8	14.26	13.28
6	4	8	12.83	14.41
7	4	8	5.91*	6.12
5	5	8	9.25*	8.08
4	5	8	9.83*	8.39
3	5	8	3.18*	8.13
2	5	8	8.04*	6.78
1	5	8	5.70*	2.95
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-2	5	8	8.28*	7.33
-3	5	8	4.31*	6.44
-4	5	8	0.0 *	1.88
-5	5	8	8.78*	8.11
-6	5	8	0.0 *	3.60
-7	5	8	9.28*	9.74
-8	5	8	2.92*	4.32
-9	5	8	3.34*	7.68
-4	6	8	0.0 *	5.63
-3	6	8	0.0 *	2.40
-2	6	8	0.0 *	1.20
-1	6	8	6.45*	2.16
0	6	8	6.31*	2.16
-8	5	9	6.64*	6.01
-7	5	9	2.92*	6.86
-6	5	9	4.31*	2.03
-5	5	9	0.0 *	3.68
-4	5	9	0.0 *	1.94
-3	5	9	5.93*	5.52
-2	5	9	6.70*	10.24
-1	5	9	0.0 *	8.33
0	5	9	8.45*	3.69
1	5	9	10.35*	10.52
2	5	9	0.0 *	2.24
3	5	9	7.60*	2.95
4	5	9	19.64	18.31
6	4	9	9.18*	6.81
5	4	9	21.58	20.77
4	4	9	8.59*	8.71
3	4	9	10.12*	2.75
2	4	9	5.01*	3.98
1	4	9	10.63*	10.73
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-2	4	9	7.19*	8.12
-3	4	9	6.22*	2.14
-4	4	9	0.0 *	4.17
-5	4	9	18.92	20.02
-6	4	9	6.55*	4.18
-7	4	9	28.71	28.39
-8	4	9	8.68*	2.37
-9	4	9	8.06*	4.32
-10	4	9	8.89*	0.24
-12	3	9	9.95*	9.42
-11	3	9	0.0 *	0.70
-10	3	9	19.77	21.22
-9	3	9	19.25	17.67
-8	3	9	8.95*	10.51
-7	3	9	18.69	19.14
-6	3	9	1.77*	5.95
-5	3	9	3.68*	7.99
-4	3	9	3.83*	1.71
-3	3	9	6.79*	6.27
-2	3	9	15.31	14.80
-1	3	9	4.50*	1.94
0	3	9	6.53*	5.00
1	3	9	9.20*	9.57
2	3	9	9.78*	3.09
3	3	9	22.06	21.88
4	3	9	17.90	18.30
5	3	9	8.44*	3.72
6	3	9	6.68*	5.37
7	3	9	5.18*	3.33
8	3	9	0.0 *	3.06
9	2	9	4.70*	9.64
8	2	9	3.51*	2.22
7	2	9	0.0 *	3.93
6	2	9	11.33	11.04
5	2	9	6.83*	9.14
4	2	9	3.95*	3.20
3	2	9	3.64*	2.40
2	2	9	9.01*	10.48
1	2	9	14.92	13.93
0	2	9	0.0 *	3.27
-1	2	9	16.84	16.75
-2	2	9	28.38	27.86
-3	2	9	8.99*	5.33
-4	2	9	0.0 *	1.05
-5	2	9	17.17	17.71
-6	2	9	18.14	18.44
-7	2	9	14.35	14.33
-8	2	9	30.00	27.70
-9	2	9	16.14	15.68
-10	2	9	29.05	28.39
-11	2	9	0.0 *	0.33
-12	2	9	6.21*	5.28
-13	2	9	0.0 *	3.87
-13	1	9	0.0 *	1.88
-12	1	9	8.23*	5.95
-11	1	9	0.0 *	3.17
-10	1	9	0.0 *	2.92
-9	1	9	10.12*	11.36
-8	1	9	31.26	29.81
-7	1	9	8.16*	3.45
-6	1	9	5.88*	2.90
-5	1	9	0.0 *	0.21
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-2	1	9	5.12*	8.37
-1	1	9	5.59*	7.00
0	1	9	17.44	15.42
1	1	9	28.36	27.14
2	1	9	0.0 *	2.16
3	1	9	32.66	32.78
4	1	9	21.38	21.85
5	1	9	6.75*	11.64
6	1	9	6.23*	0.36
7	1	9	3.10*	4.45
8	1	9	0.0 *	3.08
9	1	9	9.10*	9.03
9	0	9	7.22*	8.29
7	0	9	5.51*	1.86
5	0	9	25.41	24.96
3	0	9	26.57	25.72
1	0	9	10.59	10.69
-1	0	9	10.13	10.64
-3	0	9	20.98	19.67
-5	0	9	4.74*	10.34
-7	0	9	10.54	11.55
-9	0	9	7.83*	8.32
-11	0	9	6.24*	7.28
-13	0	9	9.95*	6.78
-12	0	10	7.46*	1.88
-10	0	10	15.53	15.39
-8	0	10	4.26*	3.28
-6	0	10	7.82*	1.00
-4	0	10	13.94	14.52
-2	0	10	0.0 *	2.93
0	0	10	9.54	9.86
2	0	10	19.92	20.81
4	0	10	7.31*	10.54
6	0	10	26.80	27.68
8	0	10	8.56*	9.04
8	1	10	3.24*	6.72
7	1	10	9.39*	13.28
6	1	10	4.21*	0.86
5	1	10	9.16*	8.93
4	1	10	30.45	30.60
3	1	10	37.71	37.26
2	1	10	10.88	11.62
1	1	10	13.08	13.28
0	1	10	6.19*	2.41
-1	1	10	10.93	9.80
-2	1	10	10.52	9.52
-3	1	10	0.0 *	4.01
-4	1	10	4.42*	0.86
-5	1	10	2.31*	0.17
-6	1	10	21.89	21.86
-7	1	10	6.57*	5.46
-8	1	10	27.08	26.82
-9	1	10	13.16	12.96
-10	1	10	0.0 *	4.44
-11	1	10	9.30*	3.36
-12	1	10	6.19*	4.08
-13	1	10	7.89*	9.17
-12	2	10	0.0 *	0.72
-11	2	10	16.49	17.01
-10	2	10	17.81	17.02
-9	2	10	13.07	12.27
-8	2	10	36.08	34.77
-7	2	10	21.60	21.94
-6	2	10	21.22	20.33
-5	2	10	6.90*	0.85
-4	2	10	17.66	17.45
-3	2	10	0.0 *	0.63
-2	2	10	18.99	19.92
-1	2	10	8.10*	7.74
0	2	10	2.39*	1.50
1	2	10	27.25	26.87
2	2	10	15.63	15.63
3	2	10	5.53*	8.58
4	2	10	0.0 *	3.29

h	k	l	Fo	Fc
5	2	10	9.69*	3.91
6	2	10	12.20	11.40
7	2	10	6.56*	2.38
8	2	10	2.57*	7.64
7	3	10	4.20*	1.04
6	3	10	8.15*	1.41
5	3	10	0.0 *	0.68
4	3	10	7.47*	3.99
3	3	10	8.77*	5.95
2	3	10	5.37*	0.29
1	3	10	16.35	15.72
0	3	10	4.06*	2.31
-1	3	10	9.91*	13.59
-2	3	10	0.0 *	1.51
-3	3	10	0.0 *	1.67
-4	3	10	0.0 *	1.58
-5	3	10	0.0 *	0.03
-6	3	10	5.32*	1.84
-7	3	10	16.14	16.50
-8	3	10	22.76	21.29
-9	3	10	19.53	18.78
-10	3	10	7.36*	8.44
-11	3	10	12.23	11.39
-10	4	10	5.77*	6.26
-9	4	10	8.60*	5.36
-8	4	10	10.80*	11.14
-7	4	10	10.00*	13.56
-6	4	10	0.0 *	2.16
-5	4	10	8.99*	13.75
-4	4	10	8.90*	10.64
-3	4	10	11.87*	10.89
-2	4	10	1.33*	1.88
-1	4	10	12.90	14.12
0	4	10	0.0 *	4.95
1	4	10	6.96*	2.96
2	4	10	8.29*	10.65
3	4	10	5.81*	0.42
4	4	10	13.07	13.06
5	4	10	0.0 *	8.60
2	5	10	10.98*	12.64
1	5	10	2.68*	5.88
0	5	10	0.0 *	0.13
-1	5	10	9.32*	1.73
-2	5	10	0.0 *	4.35
-3	5	10	3.52*	8.63
-4	5	10	10.59*	9.48
-5	5	10	18.44	20.46
-6	5	10	0.0 *	3.52
-7	5	10	11.24*	14.16
-5	5	11	0.0 *	0.60
-4	5	11	17.72	19.68
-3	5	11	3.90*	0.70
-2	5	11	0.0 *	3.30
-1	5	11	0.0 *	1.26
0	5	11	0.0 *	3.80
4	4	11	7.13*	3.95
3	4	11	6.99*	5.99
2	4	11	8.48*	10.22
1	4	11	0.0 *	7.77
0	4	11	15.16	13.61
-1	4	11	0.0 *	2.11
-2	4	11	13.27	13.13
-3	4	11	8.39*	3.14
-4	4	11	7.20*	1.96
-5	4	11	0.0 *	9.84
-6	4	11	0.0 *	0.46
-7	4	11	3.51*	2.90
-8	4	11	7.57*	8.47
-9	4	11	0.0 *	1.15
-11	3	11	8.29*	6.04
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-9	3	11	7.07*	5.74
-8	3	11	5.83*	4.33
-7	3	11	0.0 *	8.27
-6	3	11	13.12*	9.88
-5	3	11	5.26*	0.25
-4	3	11	9.39*	9.09
-3	3	11	3.89*	3.37
-2	3	11	0.0 *	3.74
-1	3	11	0.0 *	2.82
0	3	11	2.94*	5.26
1	3	11	17.24	16.35
2	3	11	6.83*	8.35
3	3	11	22.25	21.97
4	3	11	0.0 *	0.29
5	3	11	0.0 *	0.78
6	3	11	0.0 *	3.19
7	2	11	5.93*	4.38
6	2	11	9.21*	3.74
5	2	11	9.61*	9.11
4	2	11	34.97	35.52
3	2	11	14.59	14.37
2	2	11	14.72	15.60
1	2	11	20.14	19.44
0	2	11	5.74*	0.88
-1	2	11	7.23*	6.69
-2	2	11	6.16*	3.98
-3	2	11	0.0 *	0.31
-4	2	11	11.31	10.95
-5	2	11	18.78	19.16
-6	2	11	0.0 *	3.45
-7	2	11	26.38	24.92
-8	2	11	41.46	41.67
-9	2	11	9.41*	7.25
-10	2	11	7.25*	3.03
-11	2	11	25.50	25.81
-12	2	11	0.0 *	7.36
-12	1	11	5.23*	2.56
-11	1	11	7.02*	10.37
-10	1	11	5.09*	5.69
-9	1	11	2.96*	0.48
-8	1	11	8.54*	7.62
-7	1	11	13.35	13.00
-6	1	11	18.91	18.29
-5	1	11	16.48	16.47
-4	1	11	4.37*	4.08
-3	1	11	9.67*	10.89
-2	1	11	0.0 *	0.04
-1	1	11	7.19*	6.37
0	1	11	6.86*	6.95
1	1	11	6.86*	10.25
2	1	11	5.63*	1.09
3	1	11	29.29	28.26
4	1	11	18.50	17.82
5	1	11	22.93	23.30
6	1	11	15.64	16.14
7	1	11	0.0 *	2.07
7	0	11	6.29*	6.00
5	0	11	25.27	25.80
3	0	11	18.96	18.92
1	0	11	3.85*	0.44
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-5	0	11	4.79*	7.84
-7	0	11	5.70*	4.67
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-13	0	11	4.71*	2.36
-12	0	12	7.19*	7.68
-10	0	12	12.15	12.47
-8	0	12	0.0 *	1.20
-6	0	12	0.0 *	9.72
-4	0	12	13.15	12.88
-2	0	12	17.20	16.30
0	0	12	8.60*	5.75
2	0	12	19.57	19.96
4	0	12	7.26*	10.01
6	0	12	9.74*	11.49
6	1	12	15.06	15.29
5	1	12	13.51	15.03
4	1	12	12.90	13.65
3	1	12	9.25*	5.88
2	1	12	6.85*	2.87
1	1	12	7.87*	0.44
0	1	12	3.12*	3.62
-1	1	12	0.0 *	3.66
-2	1	12	7.92*	5.13
-3	1	12	0.0 *	1.95
-4	1	12	5.86*	5.93
-5	1	12	21.05	21.40
-6	1	12	23.34	22.93
-7	1	12	0.0 *	2.13
-8	1	12	24.00	25.24
-9	1	12	5.46*	6.21
-10	1	12	8.75*	7.82
-11	1	12	5.20*	5.70
-12	1	12	14.05	13.18
-11	2	12	19.87	20.16
-10	2	12	12.09	13.23
-9	2	12	11.51	12.47
-8	2	12	22.24	21.55
-7	2	12	0.0 *	0.57
-6	2	12	17.36	18.30
-5	2	12	10.83*	10.42
-4	2	12	9.22*	9.22
-3	2	12	0.0 *	3.33
-2	2	12	8.75*	2.21
-1	2	12	9.66*	7.88
0	2	12	5.56*	6.93
1	2	12	6.24*	3.95
2	2	12	4.69*	5.19
3	2	12	0.0 *	4.18
4	2	12	25.70	26.61
5	2	12	10.06*	6.36
4	3	12	0.0 *	3.37
3	3	12	9.95*	5.56
2	3	12	6.30*	8.19
1	3	12	0.0 *	3.18
0	3	12	7.12*	0.63
-1	3	12	10.52*	5.51
-2	3	12	13.29	12.92
-3	3	12	5.40*	7.62
-4	3	12	19.12	18.08
-5	3	12	4.91*	1.78
-6	3	12	1.38*	4.64
-7	3	12	10.08*	13.02
-8	3	12	17.10	17.27
-9	3	12	6.16*	6.32
-10	3	12	24.91	25.12
-7	4	12	15.86	18.24
-6	4	12	14.38	14.32
-5	4	12	6.44*	14.57
-4	4	12	17.42	17.76
-3	4	12	0.0 *	9.59
-2	4	12	8.69*	0.56
-1	4	12	6.88*	1.23
0	4	12	0.0 *	3.26
1	4	12	0.0 *	4.69
2	4	12	6.99*	8.38
-1	4	13	8.87*	6.88
-2	4	13	2.56*	6.73
-3	4	13	6.60*	0.52
-4	4	13	10.02*	10.33
-5	4	13	9.39*	10.66
-6	3	13	9.76	10.17
-7	3	13	7.10*	6.11
-6	3	13	2.85*	6.37
-5	3	13	0.0 *	1.96
-4	3	13	0.0 *	0.29
-3	3	13	7.68*	4.35
-2	3	13	4.44*	0.82
-1	3	13	0.0 *	0.69
0	3	13	0.0 *	4.08
1	3	13	0.0 *	8.47
2	3	13	14.26	13.83
3	2	13	6.91*	1.37
4	2	13	7.76*	8.06
3	2	13	7.99*	6.00
2	2	13	17.52	17.67
1	2	13	15.39	16.18
0	2	13	14.37	13.86
-1	2	13	5.05*	2.68
-2	2	13	0.0 *	0.06
-3	2	13	7.63*	2.22
-4	2	13	0.0 *	1.76
-5	2	13	18.33	18.05
-6	2	13	7.35*	6.33
-7	2	13	2.00*	3.37
-8	2	13	17.48	16.64
-9	2	13	4.89*	5.02
-10	2	13	7.10*	12.00
-11	1	13	19.28	19.72
-10	1	13	9.24*	9.29
-9	1	13	20.47	21.20
-8	1	13	13.98	14.39
-7	1	13	10.41	8.89
-6	1	13	0.0 *	3.35
-5	1	13	6.69*	8.72
-4	1	13	13.87	13.12
-3	1	13	0.0 *	1.30
-2	1	13	1.87*	2.21
-1	1	13	9.54*	2.25
0	1	13	0.0 *	2.82
1	1	13	2.84*	9.30
2	1	13	5.27*	2.22
3	1	13	7.06*	8.65
4	1	13	7.54*	7.09
5	1	13	0.0 *	5.32
5	0	13	5.41*	2.99
3	0	13	9.87	10.61
1	0	13	8.38*	1.82
-1	0	13	8.41*	12.37
-3	0	13	0.0 *	0.91
-5	0	13	0.0 *	13.26
-7	0	13	12.49	12.37
-9	0	13	22.35	22.89
-11	0	13	6.60*	5.46
-10	0	14	8.27*	7.20
-8	0	14	0.0 *	8.98
-6	0	14	13.27	13.39
-4	0	14	7.33*	2.74
-2	0	14	11.67	12.83
0	0	14	13.42	14.07
2	0	14	0.0 *	2.18
3	1	14	8.28*	10.16
2	1	14	8.73*	8.66
1	1	14	7.39*	1.71
0	1	14	9.58*	2.81
-1	1	14	3.36*	8.31
-2	1	14	9.29*	6.53
-3	1	14	10.76	12.01
-4	1	14	8.61*	10.23
-5	1	14	21.98	23.38
-6	1	14	7.84*	7.04
-7	1	14	15.73	14.42
-8	1	14	6.35*	8.73
-9	1	14	28.82	29.17
-10	1	14	14.32	15.40
-9	2	14	17.63	17.73
-8	2	14	27.85	26.66
-7	2	14		

h	k	l	Fo	Fc
-6	2	14	13.60	13.01
-5	2	14	17.44	19.34
-4	2	14	4.87*	4.76
-3	2	14	9.43*	9.52
-2	2	14	8.78*	2.58
-1	2	14	0.0 *	4.29
0	2	14	0.0 *	1.18
1	2	14	11.25	11.72
2	2	14	7.79*	5.17
0	3	14	10.05*	6.56
-1	3	14	5.29*	8.74
-2	3	14	0.0 *	3.00
-3	3	14	10.90*	8.80
-4	3	14	19.56	19.23
-5	3	14	7.20*	8.90
-6	3	14	3.30*	1.14
-7	3	14	5.38*	2.45
0	2	15	6.99*	9.55
-1	2	15	11.76	11.10
-2	2	15	9.03*	3.00
-3	2	15	10.02*	4.73
-4	2	15	9.90*	10.96
-5	2	15	12.56	12.08
-6	2	15	0.0 *	0.40
-7	2	15	7.15*	3.53
-8	1	15	0.0 *	2.00
-7	1	15	0.0 *	1.08
-6	1	15	6.93*	9.21
-5	1	15	15.07	14.10
-4	1	15	15.70	16.09
-3	1	15	17.06	16.84
-2	1	15	5.65*	3.31
-1	1	15	0.0 *	4.85
0	1	15	0.0 *	4.16
1	1	15	8.99*	15.20
1	0	15	11.93	10.80
-1	0	15	14.24	13.51
-3	0	15	15.85	17.59
-5	0	15	8.43*	7.93
-7	0	15	10.56*	9.53
-4	0	16	0.0 *	2.08
-2	0	16	7.12*	6.65
-3	1	16	25.15	25.16
-4	1	16	0.0 *	0.39

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STRUCTURE FACTOR TABLES FOR THE p-BROMOBENZOATE DERIVATIVE OF THE SECOND ELUTED ISOMER

h	k	l	Fo	Fc
1	0	0	27.13	28.50
2	0	0	44.49	46.06
3	0	0	60.74	59.89
4	0	0	42.57	42.63
5	0	0	17.36	17.68
6	0	0	5.38	5.88
7	0	0	10.65	10.68
8	0	0	5.92	6.46
9	0	0	8.73	8.21
11	1	0	7.56	7.58
6	1	0	3.57	3.29
5	1	0	46.84	46.12
4	1	0	54.02	54.56
3	1	0	128.52	135.60
2	1	0	51.08	53.24
1	1	0	27.22	27.78
0	1	0	22.88	23.43
-1	1	0	6.25	4.33
-2	1	0	27.78	27.78
-3	1	0	31.35	31.87
-4	1	0	42.01	41.80
-5	1	0	11.30	10.94
-6	1	0	13.07	12.54
-8	1	0	11.52	11.39
-9	1	0	10.58	10.40
-8	2	0	5.33	5.38
-7	2	0	3.90	4.34
-6	2	0	6.17	6.23
-5	2	0	25.67	24.91
-4	2	0	24.44	24.48
-3	2	0	50.80	50.69
-2	2	0	30.71	31.68
-1	2	0	28.27	28.43
0	2	0	11.67	11.73
1	2	0	70.85	73.60
2	2	0	107.49	115.00
3	2	0	41.76	43.28
4	2	0	44.86	45.44
5	2	0	3.48	3.90
6	2	0	12.84	13.56
7	2	0	37.88	38.51
8	2	0	24.59	25.05
9	2	0	12.61	12.47
10	2	0	7.81	8.01
11	3	0	5.11	5.86
6	3	0	24.61	24.26
5	3	0	32.60	33.16
3	3	0	11.63	11.33
2	3	0	50.71	50.24
1	3	0	6.46	5.50
0	3	0	98.32	105.65
-1	3	0	62.29	65.58
-2	3	0	18.49	18.75
-3	3	0	37.75	38.43
-4	3	0	16.27	16.07
-6	3	0	7.09	7.02
-7	3	0	8.06	7.77
-8	3	0	11.12	10.70
-10	3	0	5.27	5.01
-11	3	0	6.39	7.16
-10	4	0	4.39	5.40
-6	4	0	15.64	16.19
-5	4	0	15.04	15.33
-4	4	0	8.53	9.98
-3	4	0	33.13	35.40
-2	4	0	19.13	20.16
-1	4	0	60.71	62.48
0	4	0	29.18	29.94
1	4	0	2.91	3.19
2	4	0	32.60	32.15
3	4	0	28.96	28.90
4	4	0	27.30	27.08
5	4	0	25.29	25.73
6	4	0	10.68	10.89
7	4	0	17.23	16.74
9	4	0	6.48	5.91
10	4	0	4.99	5.47
8	5	0	17.64	18.13
7	5	0	9.49	9.68
5	5	0	10.32	9.59
4	5	0	18.30	18.83
3	5	0	22.39	22.92
2	5	0	49.69	49.99
1	5	0	16.05	16.44
0	5	0	29.44	30.20
-1	5	0	26.57	25.44
-2	5	0	5.73	5.63
-3	5	0	16.47	15.67
-4	5	0	17.82	18.15
-5	5	0	14.39	15.25
-6	5	0	4.35	4.74
-7	5	0	11.42	11.31
-9	5	0	8.20	7.32
-9	6	0	8.01	7.78
-8	6	0	12.57	12.00
-7	6	0	6.36	6.10
-6	6	0	12.93	13.15
-5	6	0	10.36	10.12
-4	6	0	5.45	5.75
-3	6	0	8.43	9.03
-2	6	0	5.66	5.95
-1	6	0	4.86	6.31
0	6	0	3.52	3.14
1	6	0	42.51	44.66
2	6	0	42.70	42.26
3	6	0	21.19	20.79
4	6	0	11.93	12.15
5	6	0	28.23	27.33
6	6	0	17.45	16.92
7	6	0	14.82	14.48
9	6	0	8.76	8.82
10	7	0	9.96	10.65
9	7	0	6.76	7.34
7	7	0	4.02	3.86
6	7	0	20.35	20.56
5	7	0	14.87	14.91
4	7	0	9.58	9.77
3	7	0	5.81	5.45
2	7	0	18.85	19.31
1	7	0	4.10	4.33
0	7	0	13.55	12.94
-1	7	0	39.61	40.72
-2	7	0	23.31	23.74
-3	7	0	46.68	47.84
-4	7	0	11.89	11.16
-5	7	0	23.67	24.18
-6	7	0	5.60	5.40
-6	8	0	6.99	6.14
-5	8	0	6.72	6.33
-4	8	0	5.72	5.64
-3	8	0	15.09	14.62
-2	8	0	20.93	21.37
-1	8	0	9.65	10.51
0	8	0	24.55	24.38
1	8	0	17.55	17.32
2	8	0	15.19	14.98
3	8	0	8.43	9.94
4	8	0	5.42	6.15
8	8	0	9.17	9.32
11	8	0	7.30	7.48
9	9	0	5.55	5.69
8	9	0	4.57	5.15
6	9	0	18.16	17.73
5	9	0	5.13	4.38
4	9	0	7.69	6.77
2	9	0	3.26	4.21
1	9	0	11.49	11.54
0	9	0	3.99	3.69
-1	9	0	23.47	23.49
-2	9	0	23.70	23.79
-3	9	0	18.15	18.24
-4	9	0	5.41	4.85
-5	9	0	6.98	6.70
-6	9	0	7.39	7.54
-3	10	0	14.04	13.70
-2	10	0	14.65	14.21
-1	10	0	21.01	21.48
2	10	0	13.03	12.69
3	10	0	6.95	6.87
3	11	0	9.99	10.56
2	11	0	19.05	18.36
1	11	0	12.78	12.39
0	11	0	5.85	7.58
-1	12	0	11.38	10.83
2	12	0	8.44	9.09
4	12	0	4.27	5.98
5	12	0	11.39	10.77
6	12	0	9.40	9.15
4	13	0	9.16	8.27
1	14	0	6.56	5.69
2	13	1	4.68	5.08
7	12	1	8.71	8.64
3	12	1	7.91	7.32
1	12	1	8.50	8.65
0	12	1	7.30	6.89
-2	12	1	9.02	8.69
-5	11	1	4.39	4.57
-4	11	1	6.58	5.32
-2	11	1	5.36	5.35
0	11	1	9.10	8.75
1	11	1	8.13	6.27
2	11	1	5.64	6.12
3	11	1	11.26	11.41
4	11	1	7.05	6.77
5	11	1	6.27	6.15
7	10	1	9.58	9.81
5	10	1	11.62	11.46
4	10	1	14.65	13.65
3	10	1	8.38	7.47
1	10	1	6.11	6.56
-1	10	1	22.95	23.08
-2	10	1	9.83	9.61
-4	10	1	11.81	12.08
-5	10	1	7.15	6.99
-7	9	1	6.32	6.15
-5	9	1	10.59	10.53
-4	9	1	5.39	4.92
-2	9	1	13.49	13.87
0	9	1	24.04	23.94
1	9	1	10.67	10.72
2	9	1	12.42	12.62
4	9	1	12.26	11.97
6	9	1	6.56	6.12
7	9	1	9.84	9.33
8	9	1	7.48	7.51
8	8	1	5.78	5.55
4	8	1	10.35	10.84
3	8	1	15.68	15.06
2	8	1	12.48	12.31
1	8	1	21.82	21.51
0	8	1	11.08	10.31
-1	8	1	6.01	6.12
-2	8	1	8.16	8.62
-3	8	1	38.86	39.48
-5	8	1	12.73	13.17
-8	7	1	4.02	4.08
-7	7	1	7.76	8.16
-4	7	1	20.09	20.53
-3	7	1	11.11	11.64
-2	7	1	24.89	25.32
-1	7	1	20.30	20.57
0	7	1	34.75	35.29
1	7	1	8.11	8.92
2	7	1	22.20	21.57
3	7	1	24.15	23.35
4	7	1	10.04	9.56
5	7	1	6.58	6.71
11	6	1	7.39	7.18
8	6	1	6.34	5.47
7	6	1	16.24	16.04
6	6	1	9.09	9.64
3	6	1	15.65	16.04
2	6	1	5.67	4.73
0	6	1	27.95	27.94
-1	6	1	27.00	26.95
-2	6	1	25.84	26.21
-3	6	1	12.16	12.74
-4	6	1	8.03	7.65
-5	6	1	21.03	20.86
-7	6	1	9.01	9.41
-8	6	1	4.53	3.99
-10	5	1	6.21	5.74
-9	5	1	8.61	9.39
-7	5	1	5.10	5.10
-6	5	1	11.18	11.25
-5	5	1	22.16	22.98
-4	5	1	21.87	22.14
-3	5	1	11.76	12.23
-2	5	1	40.48	40.51
-1	5	1	29.27	30.71
0	5	1	10.32	11.71
1	5	1	36.25	38.07
2	5	1	19.46	19.91
3	5	1	29.59	30.13
4	5	1	22.81	23.63
5	5	1	17.85	17.98
6	5	1	9.54	10.16
8	5	1	18.30	19.16
9	5	1	6.19	5.97
11	5	1	5.01	4.58
9	4	1	11.30	10.81
8	4	1	16.22	15.72
7	4	1	9.85	10.15
6	4	1	10.95	10.95
5	4	1	5.72	5.52
4	4	1	57.79	56.31
3	4	1	29.28	29.12
2	4	1	12.22	11.88
1	4	1	12.98	12.35
0	4	1	44.92	44.64
-1	4	1	27.19	27.85
-2	4	1	39.39	40.59
-3	4	1	29.07	30.40
-4	4	1	9.06	9.97
-5	4	1	3.18	2.47
-8	4	1	4.77	4.51
-9	4	1	8.88	6.28
-10	4	1	20.32	20.58
-11	3	1	10.59	10.44
-9	3	1	7.20	7.68
-7	3	1	21.14	21.14
-6	3	1	23.63	23.96
-5	3	1	24.45	24.83
-4	3	1	31.06	30.63
-3	3	1	29.99	29.33
-2	3	1	13.22	14.00
-1	3	1	65.08	67.86
0	3	1	8.61	8.49
2	3	1	83.99	88.72
			31.36	30.95

h	k	l	Fo	Fc
3	3	1	12.18	11.55
4	3	1	32.36	29.18
6	3	1	14.36	14.62
7	3	1	11.75	11.80
8	3	1	8.21	7.34
10	2	1	4.79	4.75
9	2	1	23.00	24.03
8	2	1	15.84	16.59
7	2	1	10.27	10.83
6	2	1	19.60	19.49
5	2	1	37.66	37.19
4	2	1	23.02	23.37
3	2	1	27.33	27.47
2	2	1	41.89	41.98
1	2	1	21.55	21.34
0	2	1	45.03	45.71
-1	2	1	21.41	21.34
-2	2	1	50.97	52.11
-3	2	1	7.33	7.45
-4	2	1	35.01	36.07
-5	2	1	21.08	21.88
-6	2	1	12.60	12.46
-7	2	1	6.53	6.24
-11	2	1	6.37	6.76
-10	1	1	9.27	8.25
-9	1	1	8.38	8.43
-8	1	1	11.11	10.81
-3	1	1	17.68	17.90
-2	1	1	47.37	46.70
-1	1	1	45.89	46.16
0	1	1	59.33	59.97
1	1	1	95.59	98.63
2	1	1	97.95	100.76
3	1	1	33.68	32.74
4	1	1	48.17	49.06
5	1	1	45.84	45.92
6	1	1	28.32	28.33
7	1	1	6.54	6.38
8	1	1	4.65	6.05
9	1	1	6.82	5.77
10	1	1	6.12	7.10
10	0	1	8.87	9.38
9	0	1	5.16	5.22
7	0	1	14.66	14.58
6	0	1	31.16	30.95
5	0	1	47.77	47.43
4	0	1	34.55	33.58
3	0	1	49.68	50.08
2	0	1	110.42	112.96
1	0	1	109.10	113.93
0	0	1	9.10	9.58
-1	0	1	17.46	17.30
-2	0	1	31.03	30.39
-3	0	1	9.84	9.24
-4	0	1	12.29	12.12
-5	0	1	22.44	22.05
-6	0	1	29.68	28.26
-7	0	1	18.80	17.97
-8	0	1	8.71	8.22
-9	0	1	6.88	7.61
-11	0	1	7.82	7.75
-10	-1	1	8.69	8.59
-9	-1	1	8.60	8.92
-8	-1	1	6.94	6.55
-4	-1	1	10.52	10.74
-3	-1	1	51.37	50.91
-2	-1	1	75.78	80.49
-1	-1	1	58.36	60.31
0	-1	1	65.59	69.79
1	-1	1	80.77	84.72
2	-1	1	125.44	130.45
3	-1	1	44.53	43.39
4	-1	1	18.75	18.42
5	-1	1	14.47	13.95
6	-1	1	4.50	4.20
8	-1	1	10.22	10.23
9	-1	1	4.55	3.91
10	-1	1	8.96	9.66
10	-2	1	4.96	5.17
6	-2	1	33.46	33.51
5	-2	1	26.47	26.20
4	-2	1	4.86	4.85
3	-2	1	10.50	10.39
2	-2	1	64.13	64.32
1	-2	1	17.25	16.70
0	-2	1	53.75	58.42
-1	-2	1	143.68	170.55
-2	-2	1	73.22	76.21
-3	-2	1	52.16	51.55
-4	-2	1	12.31	12.49
-5	-2	1	4.89	4.99
-6	-2	1	21.57	21.50
-7	-2	1	7.04	6.93
-9	-2	1	14.74	15.38
-10	-2	1	12.05	12.10
-9	-3	1	9.75	9.01
-8	-3	1	15.68	15.88
-7	-3	1	21.67	21.56
-6	-3	1	28.24	27.83
-5	-3	1	38.02	37.14
-4	-3	1	9.30	8.84
-3	-3	1	17.15	16.57
-2	-3	1	14.99	16.30
-1	-3	1	7.81	8.12
0	-3	1	26.96	27.05
1	-3	1	54.47	55.20
2	-3	1	47.02	50.91
3	-3	1	52.40	54.55
5	-3	1	32.10	32.01
6	-3	1	17.35	17.37
7	-3	1	8.16	8.30
5	-4	1	6.11	5.33
4	-4	1	20.63	20.67
3	-4	1	38.45	37.93
2	-4	1	53.61	53.04
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-2	-4	1	45.28	47.06
-3	-4	1	47.28	48.19
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-6	-4	1	14.82	14.74
-7	-4	1	23.16	22.77
-8	-4	1	34.34	34.82
-9	-4	1	11.12	10.97
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3	-7	1	20.70	20.34
4	-7	1	3.20	2.34
5	-7	1	9.23	9.78
7	-7	1	5.21	5.42
7	-8	1	8.85	8.67
5	-8	1	10.71	11.27
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-2	-8	1	19.83	20.26
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2	12	3	17.00	17.15
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0	6	4	27.66	28.24
1	6	4	3.69	3.97
2	6	4	30.68	30.94
3	6	4	5.45	5.12
4	6	4	12.30	12.20
6	6	4	9.62	10.29
8	6	4	5.38	5.81
4	7	4	14.78	15.04
3	7	4	11.30	10.68
2	7	4	21.93	21.55
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h	k	l	Fo	Fc
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5	10	4	8.01	7.85
7	10	4	9.26	9.18
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7	4	5	5.22	5.48
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7	1	5	5.58	5.55
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h	k	l	Fo	Fc
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3	2	9	8.11	7.74
2	2	9	7.63	7.42
1	2	9	27.31	27.65
0	2	9	7.28	7.01
-1	2	9	14.99	14.96
-3	2	9	6.30	6.47
-5	2	9	8.67	8.44
-6	2	9	20.47	20.40
-8	2	9	6.98	6.36
-9	2	9	6.32	5.61
-10	2	9	4.67	4.34
-10	1	9	7.72	8.10
-9	1	9	9.94	10.11
-7	1	9	17.05	17.28
-6	1	9	27.20	27.24
-4	1	9	7.77	7.76
-3	1	9	9.13	9.10
-2	1	9	20.93	21.51
-1	1	9	4.78	4.87
0	1	9	5.59	5.42
2	1	9	9.04	9.31
3	1	9	10.92	10.86
4	1	9	7.00	7.10
2	0	9	8.47	7.92
0	0	9	7.29	7.06
-1	0	9	23.98	24.03
-2	0	9	22.65	23.26
-3	0	9	20.74	21.23
-5	0	9	19.47	19.25
-6	0	9	7.48	7.83
-7	0	9	9.81	9.49
-9	0	9	10.62	10.23
-11	0	9	7.47	7.22
-10	-1	9	6.17	5.56
-7	-1	9	5.56	5.79
-5	-1	9	33.97	34.79
-4	-1	9	15.80	15.86
-1	-1	9	20.62	20.69
0	-1	9	6.20	6.48
1	-1	9	11.76	12.05
3	-2	9	6.39	6.76
2	-2	9	10.36	10.43
1	-2	9	12.80	13.06
-4	-2	9	11.01	11.29
-5	-2	9	16.80	17.26
-6	-2	9	27.79	27.70
-7	-2	9	8.25	7.79
-8	-2	9	8.93	8.71
-9	-2	9	6.41	6.99
-9	-3	9	6.51	7.34
-8	-3	9	8.46	8.19
-7	-3	9	14.38	15.21
-3	-3	9	18.72	18.81
-2	-3	9	14.04	13.93
-1	-3	9	27.22	27.26
0	-3	9	10.50	10.02
1	-3	9	12.25	13.45
1	-4	9	5.96	5.60
0	-4	9	8.95	8.08
-1	-4	9	12.03	11.79
-4	-4	9	17.58	17.50
-5	-4	9	12.14	12.42
-6	-4	9	13.18	13.42
-8	-4	9	9.08	10.73
-10	-4	9	4.28	4.00
-10	-5	9	4.58	4.35
-5	-5	9	9.92	10.71
-4	-5	9	9.58	9.70
-3	-5	9	17.42	18.04
-2	-5	9	7.14	7.61
-1	-5	9	9.26	9.55
1	-5	9	5.76	4.61
2	-5	9	7.69	7.93
-1	-6	9	8.72	9.08
-3	-6	9	8.49	7.98
-4	-6	9	5.66	4.44
-5	-6	9	9.73	10.50
-6	-6	9	4.25	4.29
-7	-6	9	11.04	10.51
-8	-6	9	10.50	9.57
-6	-7	9	11.10	11.20
-5	-7	9	4.89	4.15
-3	-8	9	6.05	6.06
-8	-8	9	5.87	5.72
-4	-10	9	9.31	7.74
-8	-8	10	4.99	4.24
-5	-8	10	7.46	7.24
-2	-8	10	4.32	4.52
-1	-7	10	5.62	4.78
-5	-7	10	4.93	6.27
-6	-7	10	7.15	7.70
-7	-7	10	5.84	4.90
-9	-7	10	4.91	5.07
-7	-6	10	7.68	6.95
-6	-6	10	6.13	5.37
-5	-6	10	5.61	5.40
-2	-6	10	7.44	7.63
-2	-5	10	12.52	13.23
-3	-5	10	13.37	13.69
-4	-5	10	7.31	6.41
-8	-4	10	4.17	4.85
-7	-4	10	6.57	6.58
-6	-4	10	7.43	7.70
-5	-4	10	5.27	6.42
0	-4	10	14.87	15.62
0	-3	10	6.77	6.68
-4	-3	10	5.24	6.43
-5	-3	10	5.18	4.73
-6	-3	10	8.44	8.58
-9	-3	10	8.58	8.47
-11	-3	10	5.02	4.86
-8	-2	10	4.97	3.97
-7	-2	10	15.49	15.31
-6	-2	10	7.28	7.26
-4	-2	10	19.62	19.31
-3	-2	10	21.27	20.55
-2	-2	10	6.02	5.64
0	-2	10	6.10	5.48
1	-2	10	4.01	4.50
1	-1	10	8.92	9.21
0	-1	10	11.08	11.58
-3	-1	10	16.13	16.90
-4	-1	10	7.86	7.70
-6	-1	10	6.85	6.48
-7	-1	10	17.52	18.04
-8	-1	10	9.48	9.97
-11	-1	10	6.70	6.16
-11	0	10	8.00	7.06
-9	0	10	9.27	8.83
-8	0	10	6.26	5.57
-4	0	10	19.57	19.64
-2	0	10	8.14	8.37
-1	0	10	11.33	10.83
0	0	10	17.90	17.55
1	0	10	5.99	6.58
3	1	10	4.27	4.53
0	1	10	4.67	4.58
-3	1	10	8.43	8.31
-4	1	10	12.80	12.80
-5	1	10	13.81	13.59
-6	1	10	6.67	6.87
-8	1	10	4.42	5.66
-9	1	10	7.91	8.22
-11	1	10	6.97	7.25
-10	2	10	8.25	8.34
-8	2	10	11.06	10.92
-7	2	10	12.57	12.72
-6	2	10	13.27	13.23
-5	2	10	13.97	14.14
-4	2	10	10.10	9.54
-3	2	10	5.22	5.02
-2	2	10	16.87	17.65
2	2	10	5.19	5.83
2	3	10	8.18	8.29
-1	3	10	15.61	15.83
-2	3	10	12.55	11.89
-3	3	10	11.89	12.27
-4	3	10	8.18	7.98
-5	3	10	10.99	11.10
-6	3	10	10.83	10.02
-7	3	10	9.22	8.69
-8	3	10	13.52	13.36
-10	3	10	6.94	6.44
-6	4	10	5.15	5.89
-5	4	10	8.59	8.57
-4	4	10	5.11	4.86
-3	4	10	21.02	20.84
0	4	10	3.99	4.45
1	4	10	7.92	8.51
2	5	10	9.11	9.28
1	5	10	4.69	5.11
0	5	10	8.39	7.69
-1	5	10	7.42	7.16
-3	5	10	12.12	13.72
-4	5	10	12.00	12.11
-5	5	10	15.55	16.27
-6	5	10	17.75	18.35
-7	5	10	10.01	9.94
-8	6	10	6.73	6.84
-6	6	10	6.10	6.40
-5	6	10	5.62	5.27
-3	6	10	10.90	11.17
-2	6	10	10.41	10.54

h	k	l	Fo	Fc
-1	6	10	13.16	13.03
1	7	10	6.45	6.70
-2	7	10	7.43	7.63
-3	7	10	15.26	15.36
-4	7	10	10.95	11.42
-5	7	10	8.00	7.98
-4	8	10	4.60	5.15
-2	8	10	5.08	4.45
-1	8	10	7.89	8.45
0	8	10	5.67	5.63
-4	7	11	8.04	8.18
-2	7	11	8.10	8.87
-1	6	11	8.70	8.85
-2	6	11	6.88	6.80
-3	6	11	5.83	6.43
-4	6	11	7.46	7.32
-6	5	11	8.20	8.09
-5	5	11	13.50	13.64
-4	5	11	16.40	16.36
-1	5	11	8.03	7.92
0	5	11	6.66	6.30
1	5	11	6.64	6.12
-2	4	11	10.11	9.78
-3	4	11	7.41	7.35
-4	4	11	6.70	5.66
-5	4	11	5.88	6.35
-6	4	11	7.29	6.21
-9	3	11	9.63	8.89
-8	3	11	9.09	8.44
-7	3	11	9.85	10.28
-6	3	11	9.21	9.53
-4	3	11	8.80	9.33
-3	3	11	10.30	10.44
-2	3	11	13.24	13.39
0	3	11	9.30	9.98
1	3	11	8.90	9.39
0	2	11	6.74	6.76
-1	2	11	6.85	7.18
-3	2	11	6.02	5.20
-4	2	11	18.46	18.33
-5	2	11	12.04	12.43
-6	2	11	13.22	13.63
-7	2	11	6.55	6.09
-9	1	11	11.34	11.68
-8	1	11	6.11	6.74
-5	1	11	10.57	10.59
-4	1	11	13.61	14.25
-3	1	11	4.11	4.43
-2	1	11	18.11	18.63
-3	0	11	11.48	11.57
-5	0	11	4.93	4.16
-7	0	11	6.94	6.96
-8	0	11	8.55	8.18
-10	0	11	9.55	8.54
-9	-1	11	5.03	4.24
-6	-1	11	19.21	19.56
-4	-1	11	13.46	13.75
2	-1	11	5.02	5.36
-1	-2	11	8.93	8.87
-2	-2	11	9.55	9.74
-3	-2	11	6.85	7.12
-4	-2	11	9.31	9.07
-5	-2	11	8.58	9.04
-9	-2	11	5.16	5.03
-10	-2	11	5.23	4.27
-8	-3	11	11.09	11.41
-6	-3	11	7.49	6.67
-5	-4	11	7.18	6.46
-6	-4	11	7.55	6.97
-7	-4	11	7.57	7.71
-3	-5	11	7.17	6.53
-6	-6	11	5.57	5.19
-5	-4	12	5.32	5.77
-4	-4	12	7.88	7.49
-6	-3	12	8.32	7.84
-7	-3	12	6.71	7.31
-6	-2	12	4.49	4.85
-3	-2	12	7.57	7.15
-2	-2	12	5.71	6.07
-3	-1	12	7.02	6.86
-6	-1	12	7.72	8.06
-8	0	12	9.24	8.22
-7	0	12	9.97	10.34
-6	0	12	9.00	8.69
-4	0	12	11.61	11.50
-3	0	12	10.73	10.12
-1	0	12	6.99	5.92
-2	1	12	4.69	4.61
-3	1	12	11.63	11.42
-4	1	12	14.66	14.54
-5	1	12	7.30	7.41
-7	1	12	5.87	5.59
-2	2	12	9.19	9.50
-1	2	12	10.65	10.60
-1	3	12	5.57	4.73
-2	3	12	7.17	7.48
-3	3	12	5.58	6.41
-5	3	12	8.06	8.25
-6	3	12	11.46	11.16
-7	3	12	8.23	8.58
-5	4	12	7.27	7.18
-4	4	12	7.27	7.06
-3	4	12	8.90	8.64
-3	5	12	7.45	6.85
-4	5	12	7.37	6.75

2715 reflections

STRUCTURE FACTOR TABLES FOR

CAMPHOR-1,4-HOMOENOL p-BROMOBENZOATE

h	k	l	Fo	Fc
2	0	0	26.21	25.51
4	0	0	60.14	54.59
6	0	0	0.0 *	3.86
7	1	0	0.0 *	2.81
6	1	0	11.94	12.35
5	1	0	7.13*	5.98
4	1	0	0.0 *	4.80
3	1	0	63.02	62.57
2	1	0	25.51	26.26
1	1	0	139.51	144.65
0	2	0	41.63	42.97
1	2	0	87.23	80.99
2	2	0	5.40*	3.88
3	2	0	38.01	37.62
4	2	0	6.89*	2.97
5	2	0	19.53	19.58
6	2	0	5.57*	2.83
7	2	0	4.81*	1.96
7	3	0	0.0 *	3.29
6	3	0	8.86*	1.37
5	3	0	14.08	14.15
4	3	0	0.0 *	6.68
3	3	0	16.46	16.57
2	3	0	63.90	62.71
1	3	0	55.16	48.44
0	4	0	19.12	21.34
1	4	0	40.40	36.21
2	4	0	17.42	16.78
3	4	0	6.69*	1.24
4	4	0	29.42	28.49
5	4	0	0.0 *	1.30
6	4	0	6.73*	1.64
6	5	0	10.55	9.74
5	5	0	13.95	13.77
4	5	0	0.0 *	2.44
3	5	0	10.02	9.74
2	5	0	6.92*	7.92
1	5	0	43.93	40.77
0	6	0	29.26	31.70
1	6	0	32.76	31.74
2	6	0	14.66	14.01
3	6	0	22.15	21.96
4	6	0	7.83*	8.21
5	6	0	18.92	17.67
5	7	0	11.76*	10.62
4	7	0	0.0 *	1.35
3	7	0	11.83	12.30
2	7	0	46.79	44.18
1	7	0	2.00*	1.46
0	8	0	21.92	22.73
1	8	0	14.70	15.19
2	8	0	6.30*	2.59
3	8	0	5.14*	5.05
2	9	0	0.0 *	3.58
1	9	0	12.38	12.21
0	9	1	18.57	19.27
1	9	1	17.64	15.86
2	9	1	0.0 *	5.24
3	8	1	13.96	13.81
2	8	1	15.83	15.74
1	8	1	20.07	19.37
0	8	1	0.0 *	6.46
0	7	1	36.52	37.24
1	7	1	0.0 *	3.65
2	7	1	7.47*	4.54
3	7	1	6.45*	7.48
4	7	1	15.86	14.96
5	7	1	0.0 *	5.21
5	6	1	0.0 *	6.11
4	6	1	7.19*	11.43
3	6	1	18.65	16.87
2	6	1	20.58	19.48
1	6	1	24.34	23.78
0	6	1	9.68	12.34
0	5	1	8.54	8.01
1	5	1	48.40	44.25
2	5	1	9.04*	13.00
3	5	1	19.86	19.25
4	5	1	10.32	10.97
5	5	1	9.96*	13.24
6	5	1	0.0 *	3.28
6	4	1	1.40*	7.55
5	4	1	8.80*	9.29
4	4	1	14.55	14.59
3	4	1	13.64	11.32
2	4	1	69.63	65.82
1	4	1	25.33	22.66
0	4	1	4.83*	2.17
0	3	1	43.14	47.18
1	3	1	39.83	38.74
2	3	1	24.98	24.28
3	3	1	18.81	19.19
4	3	1	27.25	27.71
5	3	1	6.93*	10.00
6	3	1	6.42*	5.19
7	3	1	4.69*	2.98
7	2	1	10.28	10.11
6	2	1	0.0 *	6.38
5	2	1	7.28*	7.23
4	2	1	7.96	8.58
3	2	1	63.89	60.09
2	2	1	30.00	27.60
1	2	1	51.70	49.24
0	2	1	6.84	5.28
0	1	1	26.01	28.60
1	1	1	93.52	95.08
2	1	1	17.41	17.88
3	1	1	41.15	38.41
4	1	1	27.49	26.35
5	1	1	22.48	20.81
6	1	1	11.27	11.55
7	1	1	0.0 *	4.34
7	0	1	10.04	10.03
6	0	1	11.14	11.03
5	0	1	7.15*	8.73
4	0	1	37.55	36.96
3	0	1	41.12	39.01
2	0	1	79.29	82.37
1	0	1	35.42	36.42
0	0	2	89.44	85.61
1	0	2	88.68	95.57
2	0	2	24.13	24.21
3	0	2	23.98	21.66
4	0	2	35.13	35.22
5	0	2	8.29*	10.31
6	0	2	0.0 *	1.36
7	0	2	0.0 *	3.79
7	1	2	7.01*	5.18
6	1	2	13.74	13.95
5	1	2	7.08*	7.71
4	1	2	16.51	15.88
3	1	2	32.41	31.55
2	1	2	59.58	59.73
1	1	2	75.83	77.20
0	1	2	24.04	23.07
0	2	2	61.85	67.94
1	2	2	95.37	93.59
2	2	2	23.32	23.69
3	2	2	37.68	37.43
4	2	2	1.82*	6.41
5	2	2	30.68	29.64
6	2	2	13.54	12.26
7	2	2	9.52*	6.13
7	3	2	0.0 *	2.99
6	3	2	15.34	14.74
5	3	2	20.31	20.31
4	3	2	21.70	22.29
3	3	2	7.01*	6.72
2	3	2	78.02	77.44
1	3	2	21.27	20.47
0	3	2	0.0 *	1.00
0	4	2	95.92	106.61
1	4	2	43.95	38.99
2	4	2	20.53	19.27
3	4	2	15.44	14.55
4	4	2	38.87	37.33
5	4	2	9.52*	10.51
6	4	2	4.65*	5.75
6	5	2	9.61*	3.15
5	5	2	9.95*	6.79
4	5	2	9.66*	7.07
3	5	2	45.12	43.51
2	5	2	14.06	14.65
1	5	2	41.24	40.09
0	5	2	2.71*	0.72
0	6	2	19.32	21.76
1	6	2	22.73	22.12
2	6	2	13.58	13.77
3	6	2	14.52	15.18
4	6	2	8.57*	8.81
5	6	2	13.89	13.35
4	7	2	9.05*	4.79
3	7	2	1.44*	6.80
2	7	2	23.30	21.20
1	7	2	11.05	10.77
0	7	2	0.0 *	5.69
0	8	2	15.90	16.03
1	8	2	6.27*	8.49
2	8	2	4.62*	4.24
3	8	2	0.0 *	4.86
2	9	2	1.74*	3.66
1	9	2	7.29*	10.26
0	9	2	7.05*	5.10
0	9	3	1.57*	2.30
1	9	3	11.33	11.16
3	8	3	9.60*	6.04
2	8	3	0.0 *	9.01
1	8	3	0.0 *	5.60
0	8	3	10.53	11.01
0	7	3	39.95	39.72
1	7	3	5.67*	8.47
2	7	3	7.12*	4.18
3	7	3	6.72*	5.28
4	7	3	12.65	12.25
5	6	3	5.38*	9.22
4	6	3	14.83	14.97
3	6	3	22.31	21.49
2	6	3	16.91	16.35
1	6	3	31.97	31.12
0	6	3	0.0 *	2.49
0	5	3	29.01	29.77
1	5	3	49.59	47.48
2	5	3	13.62	13.58
3	5	3	18.73	18.47
4	5	3	11.08	10.83
5	5	3	20.56	19.88
6	5	3	8.33*	8.46
6	4	3	13.33	12.52
5	4	3	8.13*	3.23
4	4	3	21.01	20.93
3	4	3	44.77	43.23
2	4	3	56.31	55.12
1	4	3	38.55	33.94
0	4	3	7.20*	4.78
0	3	3	85.86	99.77
1	3	3	53.64	50.28
2	3	3	51.23	47.91
3	3	3	16.36	16.86
4	3	3	43.71	41.01
5	3	3	21.78	22.64
6	3	3	12.64	12.18
7	3	3	6.31*	1.76
7	2	3	3.85*	8.90
6	2	3	9.14*	8.89
5	2	3	15.24	15.60
4	2	3	22.79	22.64
3	2	3	52.21	51.19
2	2	3	12.39	12.16
1	2	3	54.63	55.07
0	2	3	39.73	41.17
0	1	3	43.34	40.69
1	1	3	44.83	45.82
2	1	3	8.83	7.95
3	1	3	43.97	42.18
4	1	3	21.42	20.17
5	1	3	28.41	25.81
6	1	3	6.36*	5.46
7	1	3	7.86*	7.56
7	0	3	0.0 *	1.71
6	0	3	18.79	18.48
5	0	3	6.93*	1.47
4	0	3	0.0 *	2.90
3	0	3	4.72*	0.92
2	0	3	121.68	127.26
1	0	3	34.04	36.08
0	0	4	59.64	56.23
1	0	4	89.56	98.68
2	0	4	35.41	36.63
3	0	4	0.0 *	1.19
4	0	4	18.81	17.41
5	0	4	4.50*	4.09
6	0	4	9.62*	12.42
7	0	4	4.06*	2.54
7	1	4	7.34*	4.66
6	1	4	3.91*	5.43
5	1	4	6.08*	8.69
4	1	4	13.09	12.87
3	1	4	29.28	28.28
2	1	4	60.35	63.10
1	1	4	61.06	64.57
0	1	4	66.51	62.34
0	2	4	35.60	35.08
1	2	4	101.62	101.97
2	2	4	5.38*	7.39
3	2	4	22.12	21.85
4	2	4	1.51*	3.04
5	2	4	21.28	20.47
6	2	4	9.08*	9.80
7	2	4	0.0 *	4.76
7	3	4	8.69*	8.14
6	3	4	16.16	15.30
5	3	4	6.65*	5.23
4	3	4	14.55	14.06
3	3	4	21.66	21.91
2	3	4	62.68	61.01
1	3	4	18.20	14.29
0	3	4	5.27*	5.51
0	4	4	51.20	55.06
1	4	4	37.71	34.53
2	4	4	19.70	18.51
3	4	4	6.93*	5.72
4	4	4	33.91	33.89
5	4	4	16.62	16.45
6	4	4	7.71*	6.36
6	5	4	5.30*	5.75
5	5	4	9.80*	10.26
4	5	4	14.68	14.72
3	5	4	38.31	39.05
2	5	4	8.64*	6.82
1	5	4	23.52	23.55

h	k	l	Fo	Fc
0	5	4	12.78	14.03
0	6	4	29.12	33.74
1	6	4	11.76	10.01
2	6	4	8.78*	10.89
3	6	4	20.20	20.46
4	6	4	9.38*	9.12
5	6	4	7.34*	8.74
4	7	4	0.0 *	5.11
3	7	4	2.86*	11.23
2	7	4	20.67	18.37
1	7	4	14.19	13.88
0	7	4	4.65*	1.14
0	8	4	0.0 *	7.19
1	8	4	12.16	12.48
2	8	4	7.71*	9.40
3	8	4	0.0 *	4.44
1	9	4	0.0 *	10.52
0	9	4	9.95*	2.03
0	9	5	0.0 *	3.01
1	9	5	13.21	14.34
3	8	5	16.44	15.62
2	8	5	15.32	15.19
1	8	5	9.14*	8.28
0	8	5	15.30	16.29
0	7	5	36.08	39.17
1	7	5	6.35*	9.48
2	7	5	11.23*	10.25
3	7	5	10.59*	10.67
4	7	5	13.89	12.60
5	6	5	10.80*	11.22
4	6	5	6.50*	6.44
3	6	5	12.10	10.82
2	6	5	31.96	30.30
1	6	5	34.06	32.85
0	6	5	26.76	28.72
0	5	5	7.33	4.91
1	5	5	45.02	44.55
2	5	5	22.41	21.85
3	5	5	16.07	14.96
4	5	5	0.0 *	4.03
5	5	5	20.13	19.90
6	5	5	7.94*	6.21
6	4	5	9.80*	9.83
5	4	5	13.42	14.25
4	4	5	9.42	7.86
3	4	5	18.96	19.99
2	4	5	57.12	58.20
1	4	5	28.82	25.86
0	4	5	14.52	15.53
0	3	5	66.06	67.95
1	3	5	47.84	47.65
2	3	5	22.67	21.34
3	3	5	18.80	19.30
4	3	5	33.84	34.17
5	3	5	6.40*	5.66
6	3	5	0.0 *	3.25
7	3	5	0.0 *	3.32
7	2	5	0.0 *	5.53
6	2	5	5.95*	4.85
5	2	5	18.50	18.18
4	2	5	16.27	16.60
3	2	5	55.99	55.51
2	2	5	24.39	23.70
1	2	5	92.29	98.08
0	2	5	24.17	21.32
0	1	5	176.42	172.69
1	1	5	33.36	37.26
2	1	5	56.75	56.19
3	1	5	30.18	29.32
4	1	5	41.74	41.40
5	1	5	20.99	19.83
6	1	5	6.15*	4.33
7	1	5	3.83*	2.58
7	0	5	0.0 *	6.16
6	0	5	18.78	18.63
5	0	5	20.14	20.16
4	0	5	12.09	11.80
3	0	5	19.78	20.92
2	0	5	67.01	70.14
1	0	5	7.85	7.83
0	0	6	95.11	87.86
1	0	6	45.86	46.72
2	0	6	18.45	20.35
3	0	6	10.94	9.37
4	0	6	38.88	40.25
5	0	6	0.0 *	2.86
6	0	6	14.33	13.32
7	0	6	0.0 *	2.08
7	1	6	6.97*	6.74
6	1	6	3.96*	4.45
5	1	6	11.88	12.78
4	1	6	16.13	15.57
3	1	6	47.21	46.90
2	1	6	36.44	38.73
1	1	6	40.85	43.74
0	1	6	111.65	104.02
0	2	6	108.44	105.45
1	2	6	64.10	70.90
2	2	6	21.96	22.00
3	2	6	48.88	45.81
4	2	6	5.11*	2.60
5	2	6	12.20	11.65
6	2	6	0.0 *	4.54
7	2	6	8.59*	6.05
6	3	6	9.72*	7.82
5	3	6	8.20*	6.95
4	3	6	10.00	10.61
3	3	6	23.20	21.07
2	3	6	60.87	61.03
1	3	6	27.06	25.78
0	3	6	19.44	19.15
0	4	6	38.72	39.59
1	4	6	36.01	31.16
2	4	6	17.71	15.87
3	4	6	5.38*	9.17
4	4	6	16.37	16.56
5	4	6	12.27	11.56
6	4	6	0.0 *	4.43
6	5	6	9.00*	6.15
5	5	6	11.42	11.58
4	5	6	15.25	15.45
3	5	6	20.90	19.97
2	5	6	15.88	15.22
1	5	6	41.20	40.61
0	5	6	16.91	17.47
0	6	6	19.54	20.95
1	6	6	15.60	15.63
2	6	6	7.85*	11.60
3	6	6	12.48	12.87
4	6	6	14.28	14.18
5	6	6	6.98*	7.52
4	7	6	8.45*	8.88
3	7	6	0.0 *	4.88
2	7	6	31.92	28.98
1	7	6	5.32*	7.48
0	7	6	14.07	14.67
0	8	6	0.0 *	1.82
1	8	6	15.71	15.52
2	8	6	8.73*	5.08
3	8	6	10.77*	6.93
1	9	6	5.66*	9.52
0	9	6	5.23*	2.08
0	9	7	10.81*	3.35
1	9	7	10.48*	8.03
3	8	7	15.08	15.21
2	8	7	7.58*	10.44
1	8	7	12.77	13.41
0	8	7	9.19*	15.15
0	7	7	38.72	40.75
1	7	7	10.93*	15.94
2	7	7	11.21*	10.25
3	7	7	6.37*	2.70
4	7	7	10.87*	14.79
5	6	7	9.54*	9.93
4	6	7	0.0 *	1.74
3	6	7	17.21	16.89
2	6	7	26.38	25.66
1	6	7	30.23	31.09
0	6	7	4.96*	10.52
0	5	7	21.99	23.60
1	5	7	39.21	40.08
2	5	7	22.91	22.81
3	5	7	20.22	19.95
4	5	7	10.45	12.28
5	5	7	0.0 *	8.70
6	5	7	0.0 *	2.05
6	4	7	5.35*	3.19
5	4	7	2.94*	4.48
4	4	7	13.32	13.47
3	4	7	13.69	13.02
2	4	7	37.61	38.78
1	4	7	8.57*	8.30
0	4	7	17.90	16.56
0	3	7	55.22	56.65
1	3	7	21.95	21.53
2	3	7	6.88*	7.94
3	3	7	26.11	25.91
4	3	7	26.37	24.76
5	3	7	0.0 *	5.47
6	3	7	6.03*	7.19
7	2	7	0.0 *	1.80
6	2	7	9.07*	8.31
5	2	7	16.89	16.68
4	2	7	17.32	16.40
3	2	7	26.02	26.81
2	2	7	44.14	47.27
1	2	7	71.20	76.43
0	2	7	13.32	13.84
0	1	7	137.07	128.61
1	1	7	69.76	72.27
2	1	7	26.47	27.31
3	1	7	40.75	39.21
4	1	7	21.80	22.19
5	1	7	23.20	23.24
6	1	7	2.66*	10.30
7	1	7	10.77*	3.23
7	0	7	7.01*	2.31
6	0	7	21.28	22.37
5	0	7	0.0 *	1.39
4	0	7	6.34*	1.81
3	0	7	14.89	14.39
2	0	7	78.93	85.40
1	0	7	30.06	30.08
0	0	8	97.18	91.98
1	0	8	24.32	25.41
2	0	8	28.88	29.31
3	0	8	40.40	42.42
4	0	8	28.10	28.20
5	0	8	8.75*	5.36
6	0	8	6.97*	6.48
7	0	8	0.0 *	2.58
7	1	8	5.72*	5.19
6	1	8	8.18*	5.72
5	1	8	5.45*	3.96
4	1	8	18.41	17.20
3	1	8	51.64	51.83
2	1	8	38.56	41.96
1	1	8	52.95	52.17
0	1	8	107.60	98.07
0	2	8	15.07	15.16
1	2	8	47.70	48.77
2	2	8	37.78	38.64
3	2	8	24.94	26.41
4	2	8	10.57	9.96
5	2	8	15.78	15.56
6	2	8	12.78	13.36
7	2	8	2.59*	2.77
6	3	8	10.63*	12.02
5	3	8	17.56	17.66
4	3	8	22.96	23.27
3	3	8	14.36	14.51
2	3	8	57.74	57.24
1	3	8	60.28	58.62
0	3	8	43.67	42.54
0	4	8	42.80	44.68
1	4	8	38.01	36.23
2	4	8	18.49	17.64
3	4	8	15.61	15.84
4	4	8	25.65	25.05
5	4	8	17.83	17.62
6	4	8	5.76*	3.24
6	5	8	0.0 *	4.61
5	5	8	9.20*	5.98
4	5	8	13.25	13.11
3	5	8	26.13	25.69
2	5	8	14.38	13.95
1	5	8	19.59	19.83
0	5	8	6.71*	4.38
0	6	8	22.88	25.67
1	6	8	20.45	21.05
2	6	8	16.54	16.10
3	6	8	19.67	19.98
4	6	8	7.68*	4.37
5	6	8	3.58*	8.08
4	7	8	3.60*	10.28
3	7	8	0.0 *	0.86
2	7	8	20.89	19.36
1	7	8	0.0 *	1.40
0	7	8	0.0 *	3.06
0	8	8	5.43*	8.85
1	8	8	15.14	14.77
2	8	8	0.0 *	8.58
3	8	8	7.53*	3.70
0	9	8	0.0 *	6.82
3	8	9	15.64	15.18
2	8	9	11.91*	7.88
1	8	9	12.36	13.98
0	8	9	0.0 *	2.75
0	7	9	18.06	18.96
1	7	9	8.52*	2.54
2	7	9	9.89*	7.23
3	7	9	9.78*	9.15
4	7	9	8.56*	10.42
5	6	9	3.89*	2.74
4	6	9	11.15	11.35
3	6	9	12.68	11.51
2	6	9	13.11	13.23
1	6	9	15.10	17.39
1	6	9	18.73	16.92
0	5	9	8.45*	9.59
1	5	9	32.36	35.06
2	5	9	19.26	19.38
3	5	9	20.91	20.57
4	5	9	13.75	14.71
5	5	9	14.02	13.86
6	4	9	6.94*	9.30
5	4	9	10.71	8.87
4	4	9	14.01	13.97
3	4	9	21.15	21.58
2	4	9	29.52	31.68

h	k	l	Fo	Fc
1	4	9	20.50	19.51
0	4	9	12.64	12.77
0	3	9	46.68	47.55
1	3	9	17.31	17.13
2	3	9	27.00	28.05
3	3	9	19.20	20.10
4	3	9	21.97	21.82
5	3	9	6.45*	6.48
6	3	9	11.00	9.81
7	2	9	6.90*	4.89
6	2	9	8.93*	10.27
5	2	9	8.05*	6.49
4	2	9	18.90	17.92
3	2	9	26.15	26.88
2	2	9	39.12	40.80
1	2	9	53.39	52.35
0	2	9	16.86	15.93
0	1	9	38.36	35.45
1	1	9	44.81	45.97
2	1	9	27.46	28.80
3	1	9	27.24	27.83
4	1	9	16.27	15.98
5	1	9	22.64	21.38
6	1	9	8.64*	8.14
7	1	9	1.49*	8.66
7	0	9	6.98*	8.14
6	0	9	7.31*	2.69
5	0	9	9.73	9.40
4	0	9	8.25*	14.08
3	0	9	0.0 *	2.27
2	0	9	38.13	43.77
1	0	9	22.25	21.79
0	0	10	72.95	69.10
1	0	10	43.31	40.36
2	0	10	7.76	7.20
3	0	10	9.02	8.57
4	0	10	41.46	41.31
5	0	10	5.15*	1.06
6	0	10	9.38	10.14
7	0	10	3.68*	1.59
7	1	10	0.0 *	2.42
6	1	10	10.69	10.97
5	1	10	18.98	19.52
4	1	10	8.14*	8.03
3	1	10	44.80	45.54
2	1	10	47.00	51.17
1	1	10	53.13	51.02
0	1	10	23.19	22.11
0	2	10	13.12	13.09
1	2	10	43.53	39.44
2	2	10	55.56	58.35
3	2	10	12.54	12.65
4	2	10	8.13*	8.61
5	2	10	21.51	22.29
6	2	10	3.69*	4.94
6	3	10	11.08	11.20
5	3	10	10.91	11.26
4	3	10	28.21	28.08
3	3	10	13.25	14.07
2	3	10	34.28	37.41
1	3	10	55.30	51.00
0	3	10	0.0 *	5.42
0	4	10	45.42	47.76
1	4	10	27.34	24.53
2	4	10	4.13*	7.47
3	4	10	19.00	18.91
4	4	10	21.49	19.94
5	4	10	18.13	17.41
6	4	10	0.0 *	1.82
5	5	10	0.0 *	3.13
4	5	10	15.55	15.63
3	5	10	27.69	27.75
2	5	10	8.41*	9.82
1	5	10	26.40	26.52
0	5	10	42.78	43.19
0	6	10	22.46	22.60
1	6	10	16.29	16.25
2	6	10	15.13	14.98
3	6	10	17.90	18.48
4	6	10	12.70	12.51
5	6	10	0.0 *	5.51
4	7	10	0.0 *	4.88
3	7	10	11.45	11.84
2	7	10	19.45	19.50
1	7	10	8.70*	11.59
0	7	10	0.0 *	0.86
0	8	10	12.27	13.59
1	8	10	10.92*	13.38
2	8	10	6.92*	7.32
2	8	11	7.56*	5.20
1	8	11	5.49*	10.16
0	8	11	8.61*	8.20
0	7	11	0.0 *	5.54
1	7	11	14.99	15.18
2	7	11	0.0 *	7.11
3	7	11	8.35*	6.43
4	7	11	4.95*	8.65
5	6	11	0.0 *	3.31
4	6	11	13.20	13.18
3	6	11	9.89*	8.88
2	6	11	22.35	21.87
1	6	11	14.20	15.34
0	6	11	37.46	39.90
0	5	11	4.82*	7.89
1	5	11	25.85	25.59
2	5	11	11.36	12.99
3	5	11	0.0 *	7.44
4	5	11	2.96*	2.98
5	5	11	11.51	11.85
6	4	11	7.84*	5.41
5	4	11	3.07*	7.29
4	4	11	5.25*	4.62
3	4	11	22.59	23.71
2	4	11	25.68	28.03
1	4	11	39.03	37.38
0	4	11	3.96*	6.48
0	3	11	70.95	71.58
1	3	11	47.13	41.67
2	3	11	32.09	32.43
3	3	11	23.71	24.19
4	3	11	23.31	22.16
5	3	11	11.08	11.41
6	3	11	9.22*	3.31
6	2	11	9.37*	3.39
5	2	11	0.0 *	1.78
4	2	11	15.19	15.36
3	2	11	23.94	25.90
2	2	11	13.87	13.15
1	2	11	28.62	29.62
0	2	11	53.56	50.41
0	1	11	13.89	14.05
1	1	11	49.42	46.27
2	1	11	37.98	39.23
3	1	11	17.86	18.39
4	1	11	10.31	10.80
5	1	11	7.96*	5.19
6	1	11	13.05	13.92
7	0	11	7.75*	1.55
6	0	11	0.0 *	2.93
5	0	11	18.44	17.93
4	0	11	12.73	12.79
3	0	11	19.75	20.77
2	0	11	28.07	28.04
1	0	11	52.46	51.99
0	0	12	81.26	77.67
1	0	12	7.29*	7.74
2	0	12	3.13*	1.20
3	0	12	32.13	33.42
4	0	12	7.97*	10.72
5	0	12	5.49*	1.02
6	0	12	0.0 *	0.93
6	1	12	12.11	13.05
5	1	12	5.65*	5.70
4	1	12	5.83*	7.09
3	1	12	23.47	24.81
2	1	12	50.53	53.18
1	1	12	37.76	35.55
0	1	12	48.75	45.68
0	2	12	21.96	20.82
1	2	12	51.26	47.91
2	2	12	38.25	39.45
3	2	12	23.89	25.18
4	2	12	4.19*	8.98
5	2	12	11.37	11.73
6	2	12	0.0 *	1.81
6	3	12	9.34*	2.70
5	3	12	4.47*	3.95
4	3	12	13.54	13.63
3	3	12	18.81	20.28
2	3	12	29.44	31.48
1	3	12	47.82	43.17
0	3	12	0.0 *	4.57
0	4	12	31.66	31.29
1	4	12	24.60	22.87
2	4	12	12.86	12.42
3	4	12	11.09	12.92
4	4	12	16.40	16.96
5	4	12	10.14*	10.89
6	4	12	0.0 *	0.45
5	5	12	1.45*	7.82
4	5	12	0.0 *	8.93
3	5	12	16.83	18.09
2	5	12	7.23*	3.82
1	5	12	37.88	35.50
0	5	12	19.39	18.84
0	6	12	31.34	32.28
1	6	12	13.55	12.92
2	6	12	20.40	21.46
3	6	12	9.56*	4.52
4	6	12	0.0 *	9.22
3	7	12	0.0 *	9.18
2	7	12	17.63	17.48
1	7	12	10.03*	10.14
0	7	12	3.57*	2.94
0	8	12	13.17	14.02
1	8	12	7.25*	12.85
2	8	12	0.0 *	5.35
2	8	13	0.0 *	4.80
1	8	13	10.53*	10.85
0	8	13	5.70*	7.80
0	7	13	11.11	11.13
1	7	13	12.67	12.33
2	7	13	0.0 *	6.98
3	7	13	11.41	11.36
4	6	13	7.22*	13.12
3	6	13	8.70*	5.77
2	6	13	16.58	16.75
1	6	13	7.38*	9.34
0	6	13	26.41	26.60
0	5	13	7.78*	5.83
1	5	13	30.74	29.79
2	5	13	25.24	28.05
3	5	13	10.10*	11.23
4	5	13	7.91*	9.65
5	5	13	10.00*	12.21
5	4	13	11.80	10.89
4	4	13	9.03*	3.15
3	4	13	14.60	14.94
2	4	13	19.65	21.06
1	4	13	37.88	36.47
0	4	13	10.01	10.65
0	3	13	58.86	56.07
1	3	13	38.43	36.93
2	3	13	0.0 *	5.27
3	3	13	12.53	14.83
4	3	13	0.0 *	7.07
5	3	13	9.67*	6.32
6	3	13	0.0 *	4.07
6	2	13	0.0 *	5.88
5	2	13	12.10	12.24
4	2	13	23.57	25.14
3	2	13	23.04	22.55
2	2	13	23.05	24.16
1	2	13	39.58	35.91
0	2	13	72.94	70.46
0	1	13	29.88	28.53
1	1	13	47.82	46.11
2	1	13	24.54	26.02
3	1	13	21.94	22.71
4	1	13	19.51	21.36
5	1	13	10.92	10.99
6	1	13	8.85*	10.05
6	0	13	0.0 *	7.83
5	0	13	15.66	15.17
4	0	13	6.88*	4.00
3	0	13	19.64	16.35
2	0	13	43.29	45.91
1	0	13	24.73	24.20
0	0	14	23.29	22.65
1	0	14	40.91	39.04
2	0	14	21.83	20.38
3	0	14	32.95	35.61
4	0	14	14.21	14.27
5	0	14	8.48*	9.53
6	0	14	10.29*	7.37
6	1	14	0.0 *	3.65
5	1	14	0.0 *	4.04
4	1	14	17.67	17.86
3	1	14	22.74	24.96
2	1	14	13.49	14.44
1	1	14	31.55	28.21
0	1	14	48.16	46.51
0	2	14	12.05	12.25
1	2	14	8.62*	9.27
2	2	14	19.60	21.39
3	2	14	19.32	19.48
4	2	14	14.69	14.57
5	2	14	7.94*	7.75
6	2	14	9.37*	9.77
6	3	14	0.0 *	4.52
5	3	14	9.22*	8.95
4	3	14	12.01	12.54
3	3	14	3.01*	3.49
2	3	14	36.89	39.29
1	3	14	24.85	24.14
0	3	14	0.0 *	3.61
0	4	14	27.69	25.71
1	4	14	22.43	19.76
2	4	14	8.11*	10.22
3	4	14	17.79	17.84
4	4	14	6.44*	7.91
5	4	14	6.41*	7.43
5	5	14	8.80*	3.17
4	5	14	9.28*	8.67
3	5	14	8.85*	14.53
2	5	14	15.06	16.15
1	5	14	4.99*	8.13
0	5	14	12.05	11.11
0	6	14	19.02	19.17
1	6	14	19.91	19.82

h	k	l	Fo	Fc
2	6	14	21.55	23.71
3	6	14	10.51	11.16
4	6	14	0.0 *	6.41
3	7	14	9.17*	9.00
2	7	14	10.40*	11.26
1	7	14	11.05*	13.29
0	7	14	5.36*	1.41
0	8	14	0.0 *	2.29
1	8	14	11.91	12.12
0	8	15	10.50	10.20
0	7	15	10.94*	13.14
1	7	15	4.52*	6.73
2	7	15	7.73*	2.73
3	7	15	14.24	14.37
4	6	15	9.30*	11.30
3	6	15	11.18	11.60
2	6	15	4.45*	8.65
1	6	15	6.99*	4.65
0	6	15	16.23	16.49
0	5	15	0.0 *	1.36
1	5	15	19.82	18.83
2	5	15	16.16	16.98
3	5	15	8.42*	8.50
4	5	15	7.12*	9.09
5	5	15	0.0 *	8.57
5	4	15	0.0 *	3.58
4	4	15	0.0 *	4.92
3	4	15	16.38	16.97
2	4	15	11.17	12.57
1	4	15	33.60	32.55
0	4	15	7.12*	3.77
0	3	15	39.43	35.75
1	3	15	13.76	12.42
2	3	15	19.61	21.04
3	3	15	17.00	17.14
4	3	15	2.30*	9.29
5	3	15	11.77	11.92
6	3	15	6.80*	3.31
6	2	15	8.18*	2.56
5	2	15	0.0 *	10.76
4	2	15	26.58	28.65
3	2	15	13.26	14.36
2	2	15	19.49	20.69
1	2	15	32.14	28.08
0	2	15	42.51	40.22
0	1	15	28.59	29.45
1	1	15	40.74	38.19
2	1	15	27.89	29.30
3	1	15	16.45	16.67
4	1	15	15.27	16.29
5	1	15	12.90	12.79
6	1	15	0.0 *	7.25
6	0	15	9.48*	3.15
5	0	15	22.21	23.62
4	0	15	7.77*	2.29
3	0	15	18.86	18.75
2	0	15	37.02	38.84
1	0	15	28.71	28.15
0	0	16	13.92	13.66
1	0	16	42.21	39.77
2	0	16	10.45	11.24
3	0	16	25.55	26.79
4	0	16	12.31	12.51
5	0	16	5.42*	6.22
6	0	16	7.90*	6.54
6	1	16	3.01*	6.36
5	1	16	8.40*	10.75
4	1	16	16.45	16.94
3	1	16	21.78	22.40
2	1	16	9.49*	12.24
1	1	16	32.02	30.54
0	1	16	23.19	22.40
0	2	16	11.22	12.05
1	2	16	14.20	15.42
2	2	16	43.02	42.71
3	2	16	3.59*	3.34
4	2	16	16.11	16.49
5	2	16	11.36	12.52
6	2	16	8.59*	8.73
5	3	16	9.78*	8.34
4	3	16	5.26*	9.86
3	3	16	10.55	11.42
2	3	16	3.95*	10.31
1	3	16	17.11	16.80
0	3	16	31.95	30.23
0	4	16	8.14*	8.68
1	4	16	27.61	26.69
2	4	16	9.09*	8.45
3	4	16	1.35*	13.01
4	4	16	0.0 *	6.63
5	4	16	7.26*	3.89
4	5	16	11.73	11.57
3	5	16	11.49	12.41
2	5	16	4.60*	5.21
1	5	16	9.39*	10.39
0	5	16	28.78	29.45
0	6	16	0.0 *	7.47
1	6	16	5.11*	5.95
2	6	16	9.85*	8.94
3	6	16	3.27*	10.03
4	6	16	8.01*	6.04
2	7	16	7.33*	13.76
1	7	16	12.76	13.20
0	7	16	9.33*	4.24
0	7	17	6.94*	7.26
1	7	17	10.46*	11.27
2	7	17	9.02*	4.82
3	6	17	3.00*	4.39
2	6	17	14.33	13.89
1	6	17	3.59*	10.58
0	6	17	12.57	12.65
0	5	17	10.57*	10.08
1	5	17	8.32*	8.08
2	5	17	24.89	25.26
3	5	17	1.44*	3.32
4	5	17	6.46*	5.55
5	4	17	7.69*	7.23
4	4	17	0.0 *	3.02
3	4	17	11.83	12.24
2	4	17	10.44	10.03
1	4	17	32.10	30.86
0	4	17	17.31	16.55
0	3	17	16.30	15.99
1	3	17	19.37	19.07
2	3	17	13.80	13.22
3	3	17	8.06*	10.30
4	3	17	7.22*	6.02
5	3	17	3.60*	3.89
5	2	17	4.08*	3.18
4	2	17	10.72*	15.17
3	2	17	15.89	16.69
2	2	17	9.13*	9.92
1	2	17	13.20	13.20
0	2	17	33.17	31.92
0	1	17	38.23	37.64
1	1	17	31.63	27.82
2	1	17	24.38	24.90
3	1	17	15.23	16.37
4	1	17	13.97	14.75
5	1	17	0.0 *	3.71
6	1	17	8.53*	3.89
6	0	17	3.75*	5.60
5	0	17	4.52*	1.51
4	0	17	10.07*	13.15
3	0	17	29.50	31.98
2	0	17	32.71	34.37
1	0	17	22.91	21.73
0	0	18	8.25*	8.77
1	0	18	27.54	26.61
2	0	18	0.0 *	3.01
3	0	18	8.93*	8.62
4	0	18	9.74	10.67
5	0	18	0.0 *	7.95
5	1	18	0.0 *	0.81
4	1	18	11.25	13.23
3	1	18	9.95*	8.77
2	1	18	9.84*	7.41
1	1	18	8.32*	10.82
0	1	18	34.50	33.00
0	2	18	0.0 *	0.66
1	2	18	17.33	15.76
2	2	18	30.04	31.35
3	2	18	12.70	13.31
4	2	18	0.0 *	6.55
5	2	18	8.59*	5.76
5	3	18	11.63	12.16
4	3	18	0.0 *	4.73
3	3	18	10.02	10.60
2	3	18	20.09	19.61
1	3	18	29.79	28.38
0	3	18	23.20	22.48
0	4	18	23.04	23.39
1	4	18	38.58	35.91
2	4	18	0.0 *	9.03
3	4	18	19.53	21.13
4	4	18	0.0 *	7.11
5	4	18	8.13*	10.13
4	5	18	11.74	11.58
3	5	18	9.86*	9.78
2	5	18	7.90*	8.61
1	5	18	12.42	12.88
0	5	18	34.18	30.84
0	6	18	8.69*	6.13
1	6	18	0.0 *	3.45
2	6	18	17.11	16.25
3	6	18	10.37*	1.80
1	7	18	7.62*	8.34
0	7	18	0.0 *	6.05
0	7	19	0.0 *	5.57
1	7	19	5.86*	10.86
3	6	19	5.03*	0.76
2	6	19	12.85*	10.05
1	6	19	5.55*	3.83
0	6	19	10.89*	12.49
0	5	19	14.61	14.58
1	5	19	18.96	17.53
2	5	19	17.36	16.72
3	5	19	10.03*	8.60
4	5	19	3.79*	0.50
4	4	19	8.99*	8.37
3	4	19	6.75*	11.24
2	4	19	17.61	16.05
1	4	19	17.61	17.25
0	4	19	18.36	18.75
0	3	19	21.06	20.70
1	3	19	19.20	19.24
2	3	19	14.27	13.66
3	3	19	15.40	15.01
4	3	19	4.84*	6.19
5	3	19	0.0 *	0.69
5	2	19	6.21*	3.11
4	2	19	9.81*	14.45
3	2	19	6.68*	9.12
2	2	19	8.70*	10.95
1	2	19	14.68	14.54
0	2	19	31.23	31.75
0	1	19	10.00	10.13
1	1	19	7.86*	8.42
2	1	19	17.46	16.89
3	1	19	0.0 *	5.73
4	1	19	0.0 *	4.13
5	1	19	8.18*	5.13
5	0	19	0.0 *	3.59
4	0	19	8.61*	13.80
3	0	19	24.77	26.10
2	0	19	10.08*	7.93
1	0	19	39.09	37.39
0	0	20	0.0 *	2.20
1	0	20	16.24	16.45
2	0	20	0.0 *	3.70
3	0	20	30.72	31.12
4	0	20	12.22	11.35
5	0	20	9.77*	2.07
5	1	20	4.82*	8.89
4	1	20	17.39	17.58
3	1	20	5.10*	3.52
2	1	20	13.68	13.82
1	1	20	20.77	20.20
0	1	20	28.06	27.79
0	2	20	8.77*	10.20
1	2	20	11.89	11.44
2	2	20	26.56	26.07
3	2	20	6.74*	9.23
4	2	20	5.87*	7.64
5	2	20	10.21*	5.74
4	3	20	9.69*	7.64
3	3	20	5.30*	6.65
2	3	20	7.08*	7.51
1	3	20	13.97	13.96
0	3	20	9.61*	10.72
0	4	20	22.53	21.53
1	4	20	18.45	18.85
2	4	20	0.0 *	4.85
3	4	20	4.55*	6.47
4	4	20	0.0 *	5.93
3	5	20	9.43*	10.68
2	5	20	11.04*	3.62
1	5	20	6.42*	10.22
0	5	20	15.90	15.71
0	6	20	6.77*	11.34
1	6	20	0.0 *	5.96
2	6	20	10.48*	12.87
2	6	21	2.43*	2.09
1	6	21	4.53*	7.66
0	6	21	7.11*	13.51
0	5	21	7.39*	4.98
1	5	21	3.45*	4.54
2	5	21	10.90*	13.86
3	5	21	11.32*	2.17
4	4	21	0.0 *	2.02
3	4	21	6.97*	8.81
2	4	21	5.16*	3.70
1	4	21	11.55*	6.76
0	4	21	9.84*	1.13
0	3	21	14.39	13.91
1	3	21	6.31*	7.38
2	3	21	7.74*	2.19
3	3	21	8.43*	7.69
4	3	21	0.0 *	4.07
5	2	21	6.29*	3.57
4	2	21	12.17	13.17
3	2	21	7.54*	9.05
2	2	21	5.84*	5.36
1	2	21	10.18*	8.79
0	2	21	28.57	28.32
0	1	21	10.04*	6.54
1	1	21	21.88	21.83
2	1	21	26.10	25.08
3	1	21	11.67	12.67
4	1	21	3.66*	7.04

h	k	l	Fo	Fc
5	1	21	0.0 *	6.22
5	0	21	13.52	13.86
4	0	21	7.51*	8.20
3	0	21	0.0 *	3.29
2	0	21	4.99*	13.15
1	0	21	29.95	29.41
0	0	22	20.81	20.68
1	0	22	21.87	21.32
2	0	22	0.0 *	4.17
3	0	22	20.19	20.12
4	0	22	0.0 *	11.62
4	1	22	7.81*	10.14
3	1	22	7.80*	9.78
2	1	22	11.61	12.14
1	1	22	2.97*	1.31
0	1	22	9.93*	10.00
0	2	22	0.0 *	4.27
1	2	22	12.37	11.67
2	2	22	18.03	19.18
3	2	22	8.19*	7.87
4	2	22	6.19*	0.34
4	3	22	0.0 *	5.30
3	3	22	4.11*	6.74
2	3	22	0.0 *	1.68
1	3	22	22.50	22.81
0	3	22	6.04*	10.80
0	4	22	9.41*	4.51
1	4	22	0.0 *	1.13
2	4	22	12.13*	10.14
3	4	22	7.09*	8.22
2	5	22	8.59*	5.79
1	5	22	11.46*	7.12
0	5	22	8.15*	12.82
0	6	22	0.0 *	5.97
1	6	22	5.88*	5.64
0	5	23	2.68*	1.81
1	5	23	0.0 *	6.46
2	5	23	14.88	14.46
3	4	23	5.94*	8.24
2	4	23	0.0 *	3.92
1	4	23	14.38	13.93
0	4	23	0.0 *	9.43
0	3	23	4.83*	2.95
1	3	23	22.79	22.35
2	3	23	11.10*	12.46
3	3	23	10.24*	12.29
4	3	23	0.0 *	1.24
4	2	23	8.25*	9.00
3	2	23	6.26*	7.98
2	2	23	8.91*	10.50
1	2	23	0.0 *	7.95
0	2	23	33.77	33.18
0	1	23	5.83*	5.22
1	1	23	16.40	17.24
2	1	23	16.38	17.26
3	1	23	5.36*	4.54
4	1	23	2.72*	1.17
4	0	23	16.83	16.08
3	0	23	9.24*	4.37
2	0	23	6.05*	0.58
1	0	23	15.13	13.90
0	0	24	9.43*	8.70
1	0	24	0.0 *	2.67
2	0	24	9.30*	2.61
3	0	24	9.21*	10.58
4	0	24	3.60*	2.70
4	1	24	0.0 *	7.30
3	1	24	5.97*	11.91
2	1	24	5.88*	4.50
1	1	24	13.95	14.50
0	1	24	14.68	14.34
0	2	24	6.04*	0.93
1	2	24	5.77*	6.66
2	2	24	16.58	16.34
3	2	24	8.23*	4.28
4	2	24	0.0 *	2.32
3	3	24	10.02*	3.39
2	3	24	5.91*	7.49
1	3	24	0.0 *	11.73
0	3	24	8.83*	8.20
0	4	24	5.08*	2.63
1	4	24	9.89*	11.49
2	4	24	10.39*	4.97
1	5	24	0.0 *	3.39
0	5	24	16.68	16.15
2	4	25	0.0 *	4.34
1	4	25	10.22*	8.09
0	4	25	8.37*	8.83
0	3	25	13.15	12.83
1	3	25	18.01	17.26
2	3	25	0.0 *	10.94
3	3	25	11.90*	9.76
3	2	25	0.0 *	3.31
2	2	25	6.89*	2.61
1	2	25	8.18*	6.06
0	2	25	23.41	23.25
0	1	25	0.0 *	5.30
1	1	25	0.0 *	4.77
2	1	25	8.48*	11.54
3	1	25	8.07*	7.97
3	0	25	6.12*	6.30
2	0	25	3.88*	4.03
1	0	25	16.43	16.92
0	0	26	0.0 *	2.00
1	0	26	10.63	10.04
2	0	26	0.0 *	0.55
3	0	26	0.0 *	6.69
3	1	26	0.0 *	8.20
2	1	26	0.0 *	2.38
1	1	26	10.13*	11.58
0	1	26	8.19*	9.38
0	2	26	0.0 *	3.61
1	2	26	0.0 *	1.86
2	2	26	12.03*	12.49
3	2	26	0.0 *	2.21
2	3	26	0.0 *	3.54
1	3	26	7.54*	6.77
0	3	26	8.84*	9.46
0	4	26	0.0 *	2.24
1	4	26	4.36*	5.07
0	3	27	0.0 *	2.35
1	3	27	10.63*	2.96
2	2	27	0.0 *	5.93
1	2	27	10.19*	8.62
0	2	27	0.0 *	5.82
0	1	27	2.69*	0.45
1	1	27	0.0 *	6.60
2	1	27	6.43*	13.88
2	0	27	10.10*	3.31
1	0	27	8.73*	9.09
0	0	28	5.27*	0.29
1	0	28	10.42*	8.31
2	0	28	9.73*	1.64
2	1	28	10.69*	5.02
1	1	28	6.31*	7.11
0	1	28	12.16	10.83
0	2	28	0.0 *	0.50
1	2	28	0.0 *	4.69
0	1	29	0.0 *	3.53
1	0	29	10.64*	8.84

1300 reflections

STRUCTURE FACTOR TABLES FOR

RAUCUBAINE

h	k	l	Fo	Fc
0	0	1	16.78	17.90
0	0	2	0.0 *	0.52
0	0	3	3.78	3.88
0	0	4	33.51	31.56
0	0	5	2.22	2.27
0	0	6	13.60	13.18
0	0	7	8.92	9.14
0	0	8	10.06	9.55
0	0	9	2.54	2.26
0	0	10	5.48	5.32
0	0	11	0.97	1.01
0	1	11	4.23	3.91
0	1	10	3.42	3.05
0	1	9	8.81	9.16
0	1	8	10.09	10.07
0	1	7	4.49	4.41
0	1	6	5.84	5.74
0	1	5	11.41	11.35
0	1	4	18.65	18.54
0	1	3	4.33	4.19
0	1	2	39.16	39.33
0	1	1	45.85	50.24
0	2	0	64.42	67.69
0	2	1	58.68	61.00
0	2	2	14.83	14.58
0	2	3	15.91	15.51
0	2	4	22.46	21.89
0	2	5	11.27	10.49
0	2	6	7.50	7.50
0	2	7	2.02	2.04
0	2	8	8.07	7.27
0	2	9	5.11	4.70
0	2	10	1.96	1.91
0	2	11	3.10	3.11
0	3	11	0.95*	1.19
0	3	10	2.68	2.52
0	3	9	7.29	7.00
0	3	8	13.13	12.78
0	3	7	13.36	13.31
0	3	6	8.52	8.29
0	3	5	21.50	20.37
0	3	4	17.19	17.49
0	3	3	3.26	3.37
0	3	2	43.73	43.46
0	3	1	5.61	5.95
0	4	0	59.14	57.04
0	4	1	24.34	23.36
0	4	2	27.09	26.11
0	4	3	29.19	27.71
0	4	4	10.78	10.52
0	4	5	3.85	4.00
0	4	6	6.98	7.30
0	4	7	9.22	9.38
0	4	8	5.03	5.10
0	4	9	0.31*	0.99
0	4	10	0.70*	1.13
0	4	11	1.71	1.63
0	5	10	2.19	2.30
0	5	9	2.10	2.21
0	5	8	8.91	8.88
0	5	7	7.92	7.56
0	5	6	7.20	7.05
0	5	5	11.70	11.32
0	5	4	2.09	2.04
0	5	3	9.97	10.58
0	5	2	19.01	17.89
0	5	1	15.94	14.51
0	6	0	27.66	26.51
0	6	1	30.24	27.94
0	6	2	2.44	2.81
0	6	3	18.97	18.79
0	6	4	22.44	21.20
0	6	5	18.28	17.56
0	6	6	9.50	9.18
0	6	7	7.45	7.67
0	6	8	3.39	3.33
0	6	9	2.24	2.02
0	6	10	2.31	2.22
0	7	10	3.53	3.61
0	7	9	4.12	3.96
0	7	8	2.11	1.94
0	7	7	12.20	12.03
0	7	6	18.15	17.76
0	7	5	10.20	9.83
0	7	4	14.32	13.73
0	7	3	10.54	10.21
0	7	2	13.26	12.52
0	7	1	2.28	2.06
0	8	0	14.77	15.03
0	8	1	8.88	8.59
0	8	2	13.25	12.24
0	8	3	10.58	10.57
0	8	4	9.81	9.27
0	8	5	10.34	10.63
0	8	6	13.36	12.52
0	8	7	5.01	4.86
0	8	8	3.47	3.03
0	8	9	4.96	4.81
0	9	9	1.88	1.49
0	9	8	3.00	2.52
0	9	7	7.89	7.81
0	9	6	2.23	2.20
0	9	5	10.28	10.06
0	9	4	4.68	4.82
0	9	3	13.57	12.75
0	9	2	5.57	5.14
0	9	1	4.58	3.91
0	10	0	3.86	3.46
0	10	1	4.62	5.26
0	10	2	14.04	13.63
0	10	3	5.63	5.91
0	10	4	4.10	4.01
0	10	5	5.37	4.58
0	10	6	8.32	8.45
0	10	7	3.05	3.06
0	10	8	3.87	3.69
0	10	9	5.72	5.36
0	11	8	3.74	3.56
0	11	7	2.57	2.28
0	11	6	3.18	3.07
0	11	5	6.68	6.49
0	11	4	5.94	5.65
0	11	3	6.31	6.43
0	11	2	2.15	2.11
0	11	1	9.40	9.07
0	12	0	4.64	4.87
0	12	1	2.79	2.37
0	12	2	4.83	4.76
0	12	3	3.60	3.25
0	12	4	6.48	5.89
0	12	5	3.93	3.90
0	12	6	0.88*	0.67
0	12	7	0.0 *	0.35
0	13	6	1.03	1.04
0	13	5	4.88	4.82
0	13	4	3.00	3.18
0	13	3	0.36*	0.82
0	13	2	5.64	5.77
0	13	1	3.53	3.41
0-14		0	3.15	3.34
0	14	1	5.97	6.02
0	14	2	1.72	1.28
0	14	3	5.86	5.91
0	14	4	1.59	1.94
0	14	5	4.85	4.64
0	15	4	1.77	1.69
0	15	3	4.28	4.23
0	15	2	4.73	4.62
0	15	1	0.29*	4.54
0-16		0	2.73	2.31
-1-15		3	2.77	2.05
-1-15		2	3.31	2.76
-1-15		1	5.55	4.97
-1-15		0	1.80	1.94
-1-15		-1	2.79	2.78
1	15	2	0.0 *	1.42
1	15	3	2.51	3.02
1	14	5	3.90	3.34
1	14	4	1.98	1.88
1	14	3	6.28	6.19
1	14	2	2.98	3.21
1	14	1	2.63	3.10
-1-14		0	6.35	6.03
-1-14		1	2.04	1.85
-1-14		2	5.88	5.32
-1-14		3	3.66	3.81
-1-14		4	3.46	2.92
-1-14		5	4.79	4.03
-1-13		6	1.62	1.31
-1-13		5	1.30	1.66
-1-13		4	3.02	2.37
-1-13		3	1.42	1.09
-1-13		2	4.81	4.42
-1-13		1	9.45	8.38
1	13	0	3.24	3.23
1	13	1	4.60	4.25
1	13	2	4.49	4.19
1	13	3	4.06	4.08
1	13	4	5.09	4.90
1	13	5	6.70	6.47
1	13	6	0.86*	0.67
1	12	7	2.98	2.47
1	12	6	1.47	1.31
1	12	5	2.74	2.77
1	12	4	2.42	2.45
1	12	3	3.06	3.13
1	12	2	4.47	4.39
1	12	1	3.18	3.80
1	12	0	4.66	4.81
1	12	-1	11.71	11.15
1	12	-2	3.27	3.42
-1-12		3	8.76	8.24
-1-12		4	5.62	5.26
-1-12		5	5.86	4.46
-1-12		6	1.75	1.48
-1-12		7	1.03	1.08
1	11	-8	1.58	1.75
-1-11		7	1.63	1.80
-1-11		6	4.07	3.65
1	11	-5	4.98	5.72
1	11	-4	1.58	1.31
1	11	-3	8.31	7.99
1	11	-2	8.39	8.22
1	11	-1	0.91*	1.19
1	11	0	5.69	5.28
1	11	1	6.45	6.14
1	11	2	8.57	8.20
1	11	3	6.25	6.49
1	11	4	2.27	2.50
1	11	5	3.21	2.88
1	11	6	2.19	2.49
1	11	7	3.31	3.35
1	11	8	4.32	4.08
1	10	8	0.83*	1.16
1	10	7	3.43	3.36
1	10	6	4.86	4.48
1	10	5	6.57	6.21
1	10	4	2.37	2.48
1	10	3	13.08	13.30
1	10	2	5.98	5.26
1	10	1	7.02	6.93
1	10	0	9.09	9.28
1	10	-1	12.19	11.99
1	10	-2	16.09	15.27
1	10	-3	7.70	7.44
1	10	-4	14.48	15.77
1	10	-5	2.37	2.42
1	10	-6	2.63	2.97
1	10	-7	1.98	2.13
1	10	-8	2.69	2.78
1	9	-9	2.42	2.57
1	9	-8	4.17	3.94
1	9	-7	3.53	3.61
1	9	-6	13.04	13.57
1	9	-5	9.87	10.53
1	9	-4	3.44	3.69
1	9	-3	6.91	7.03
1	9	-2	12.28	11.71
1	9	-1	2.16	2.25
1	9	0	11.83	11.48
1	9	1	2.98	2.80
1	9	2	14.11	14.25
1	9	3	10.35	9.87
1	9	4	5.75	6.33
1	9	5	3.77	3.30
1	9	6	5.51	5.47
1	9	7	0.73*	0.84
1	9	8	3.15	2.99
1	9	9	7.19	6.60
1	8	9	4.93	4.63
1	8	8	2.70	2.83
1	8	7	4.29	4.32
1	8	6	9.56	9.26
1	8	5	11.01	10.71
1	8	4	6.33	6.43
1	8	3	5.25	5.26
1	8	2	5.86	5.11
1	8	1	9.72	10.09
1	8	0	7.60	7.11
1	8	-1	18.93	18.01
1	8	-2	5.86	5.84
1	8	-3	9.91	10.19
1	8	-4	5.95	5.82
1	8	-5	7.52	8.03
1	8	-6	5.46	5.67
1	8	-7	7.46	7.67
1	8	-8	6.71	7.04
1	8	-9	1.47	1.48
1	7	-10	3.74	3.69
1	7	-9	6.92	7.10
1	7	-8	1.95	1.82
1	7	-7	5.44	5.81
1	7	-6	13.48	14.48
1	7	-5	10.79	11.44
1	7	-4	3.73	3.80
1	7	-3	14.12	14.53
1	7	-2	7.81	7.81
1	7	-1	20.66	20.12
1	7	0	29.32	29.12
1	7	1	22.81	23.46
1	7	2	4.84	4.64
1	7	3	17.13	15.90
1	7	4	10.48	10.51
1	7	5	17.41	17.81
1	7	6	1.89	2.11
1	7	7	5.69	5.79
1	7	8	3.08	3.30
1	7	9	6.28	5.75
1	7	10	4.14	4.11
1	6	10	3.02	3.06

h	k	l	Fo	Fc
1	6	9	3.39	3.29
1	6	8	6.39	6.54
1	6	7	7.95	7.81
1	6	6	10.46	10.60
1	6	5	2.53	2.53
1	6	4	10.36	10.24
1	6	3	8.12	8.01
1	6	2	6.61	5.69
1	6	1	17.45	17.75
1	6	0	7.62	7.38
1	6	-1	12.75	13.02
1	6	-2	17.42	17.88
1	6	-3	14.17	14.28
1	6	-4	7.13	7.85
1	6	-5	7.22	8.01
1	6	-6	6.16	6.56
1	6	-7	7.98	8.11
1	6	-8	10.75	11.25
1	6	-9	4.57	4.46
1	6	-10	5.11	4.95
1	5	-10	3.02	2.71
1	5	-9	2.69	2.91
1	5	-8	7.34	8.22
1	5	-7	11.05	11.80
1	5	-6	9.70	9.91
1	5	-5	10.70	11.47
1	5	-4	4.67	4.98
1	5	-3	12.67	13.74
1	5	-2	22.28	22.32
1	5	-1	37.22	35.67
1	5	0	26.64	27.60
1	5	1	8.19	8.69
1	5	2	10.92	11.05
1	5	3	21.62	20.33
1	5	4	10.65	10.97
1	5	5	5.04	4.99
1	5	6	12.69	12.45
1	5	7	5.73	5.62
1	5	8	8.03	8.01
1	5	9	6.36	6.14
1	5	10	4.77	4.74
1	4	11	5.42	5.21
1	4	10	2.85	2.75
1	4	9	7.05	7.08
1	4	8	7.14	6.87
1	4	7	12.02	11.78
1	4	6	5.73	5.97
1	4	5	6.63	5.88
1	4	4	7.59	6.53
1	4	3	6.85	6.95
1	4	2	17.18	16.97
1	4	1	14.63	14.70
1	4	0	20.34	22.48
1	4	-1	24.03	23.75
1	4	-2	38.78	39.04
1	4	-3	20.32	20.76
1	4	-4	5.97	6.37
1	4	-5	12.53	12.69
1	4	-6	5.10	5.24
1	4	-7	13.82	14.49
1	4	-8	10.30	10.30
1	4	-9	3.98	4.37
1	4	-10	2.71	2.71
1	4	-11	2.16	2.21
1	3	-11	4.09	4.08
1	3	-10	1.41	1.38
1	3	-9	4.32	4.52
1	3	-8	7.55	8.13
1	3	-7	8.96	9.71
1	3	-6	9.84	10.91
1	3	-5	10.98	10.60
1	3	-4	13.43	13.94
1	3	-3	19.08	19.66
1	3	-2	25.26	25.60
1	3	-1	33.37	32.30
1	3	0	36.40	38.54
1	3	1	34.87	37.33
1	3	2	32.46	32.70
1	3	3	14.98	14.61
1	3	4	17.87	16.92
1	3	5	7.50	7.30
1	3	6	17.96	17.30
1	3	7	9.81	10.02
1	3	8	8.96	8.95
1	3	9	2.39	2.08
1	3	10	1.64	1.47
1	3	11	2.64	2.72
1	2	11	0.57*	0.69
1	2	10	2.37	2.26
1	2	9	0.79*	0.91
1	2	8	10.87	10.69
1	2	7	8.79	8.48
1	2	6	1.48	1.27
1	2	5	12.53	12.10
1	2	4	18.45	18.01
1	2	3	9.73	9.52
1	2	2	56.10	56.96
1	2	1	56.68	61.05
1	2	0	25.52	26.26
1	2	-1	14.15	13.97
1	2	-2	9.80	9.20
1	2	-3	12.60	13.69
1	2	-4	16.92	17.71
1	2	-5	9.44	9.28
1	2	-6	15.89	16.57
1	2	-7	20.12	20.68
1	2	-8	12.38	13.08
1	2	-9	6.42	6.72
1	2	-10	0.83*	0.63
1	2	-11	0.94*	0.80
1	1	-11	2.41	2.39
1	1	-10	0.73*	0.60
1	1	-9	4.23	4.45
1	1	-8	7.91	8.19
1	1	-7	4.73	4.60
1	1	-6	1.70	1.64
1	1	-5	14.76	15.11
1	1	-4	14.95	14.50
1	1	-3	19.81	19.67
1	1	-2	26.34	25.52
1	1	-1	32.80	31.18
1	1	0	73.82	76.46
1	1	1	55.64	58.41
1	1	2	35.99	37.25
1	1	3	40.59	40.43
1	1	4	38.35	36.06
1	1	5	9.13	8.76
1	1	6	12.79	12.83
1	1	7	7.85	7.71
1	1	8	3.63	3.38
1	1	9	4.50	4.38
1	1	10	4.25	3.89
1	1	11	0.82*	1.15
1	0	11	5.49	5.43
1	0	10	1.03*	0.92
1	0	9	3.43	3.56
1	0	8	1.67	1.58
1	0	7	0.65*	0.47
1	0	6	23.48	22.75
1	0	5	3.87	3.81
1	0	4	2.67	2.76
1	0	3	13.78	14.23
1	0	2	5.37	5.13
1	0	1	68.41	70.20
1	0	0	19.87	19.92
1	0	-1	6.74	6.88
1	0	-2	14.87	14.51
1	0	-3	19.68	20.04
1	0	-4	0.65*	0.32
1	0	-5	19.67	19.53
1	0	-6	17.25	17.38
1	0	-7	10.62	10.86
1	0	-8	2.73	2.64
1	0	-9	10.27	11.02
1	0	-10	4.89	4.94
1	0	-11	3.25	3.15
2	0	-11	3.14	3.26
2	0	-10	0.62*	0.75
2	0	-9	6.23	6.58
2	0	-8	5.94	6.83
2	0	-7	4.41	4.44
2	0	-6	1.55	1.57
2	0	-5	12.01	11.90
2	0	-4	29.22	28.71
2	0	-3	12.71	11.80
2	0	-2	29.88	28.04
2	0	-1	25.88	25.70
2	0	0	13.45	13.72
2	0	1	26.02	27.44
2	0	2	0.84*	2.17
2	0	3	2.87	3.00
2	0	4	13.66	14.51
2	0	5	20.10	19.34
2	0	6	9.54	9.65
2	0	7	0.95	0.93
2	0	8	0.0 *	0.19
2	0	9	0.92*	1.08
2	0	10	2.42	2.66
2	0	11	2.30	2.02
2	1	11	2.59	2.66
2	1	10	4.10	3.86
2	1	9	8.35	7.91
2	1	8	10.31	9.90
2	1	7	10.54	10.55
2	1	6	10.43	10.57
2	1	5	5.43	5.34
2	1	4	6.69	6.52
2	1	3	22.78	23.88
2	1	2	7.79	8.16
2	1	1	9.01	9.12
2	1	0	41.42	39.82
2	1	-1	39.29	36.14
2	1	-2	5.94	5.80
2	1	-3	30.16	28.26
2	1	-4	12.30	11.82
2	1	-5	8.48	8.39
2	1	-6	2.96	3.30
2	1	-7	6.64	6.77
2	1	-8	6.12	6.82
2	1	-9	1.55	1.58
2	1	-10	3.09	3.02
2	1	-11	2.06	1.73
2	2	-11	0.97*	1.03
2	2	-10	2.58	2.56
2	2	-9	2.98	3.27
2	2	-8	4.88	5.09
2	2	-7	4.18	4.75
2	2	-6	9.72	9.85
2	2	-5	14.87	15.14
2	2	-4	14.31	13.66
2	2	-3	42.35	39.22
2	2	-2	22.45	21.49
2	2	-1	14.49	14.06
2	2	0	10.74	10.47
2	2	1	16.72	15.98
2	2	2	34.98	36.00
2	2	3	24.25	24.03
2	2	4	27.18	26.73
2	2	5	13.41	13.36
2	2	6	11.60	11.88
2	2	7	6.59	6.91
2	2	8	10.56	10.54
2	2	9	7.75	7.60
2	2	10	1.17	1.27
2	3	10	3.66	3.43
2	3	9	5.02	5.07
2	3	8	13.28	13.03
2	3	7	9.62	10.14
2	3	6	10.63	10.57
2	3	5	9.70	9.16
2	3	4	13.32	13.28
2	3	3	34.32	34.49
2	3	2	19.25	19.11
2	3	1	37.77	37.24
2	3	0	17.57	17.72
2	3	-1	36.23	33.48
2	3	-2	10.69	10.38
2	3	-3	23.61	22.53
2	3	-4	14.20	14.22
2	3	-5	14.46	14.40
2	3	-6	2.04	1.93
2	3	-7	12.30	12.56
2	3	-8	9.62	10.38
2	3	-9	3.56	3.81
2	3	-10	1.83	1.64
2	3	-11	2.89	2.58
2	4	-10	2.40	2.39
2	4	-9	5.27	5.31
2	4	-8	4.64	4.94
2	4	-7	6.49	7.48
2	4	-6	8.71	8.91
2	4	-5	7.74	7.75
2	4	-4	21.64	21.12
2	4	-3	18.05	17.37
2	4	-2	8.03	8.22
2	4	-1	13.94	12.85
2	4	0	14.82	14.74
2	4	1	30.28	30.84
2	4	2	12.92	12.43
2	4	3	26.89	26.22
2	4	4	13.97	13.64
2	4	5	5.99	6.28
2	4	6	3.95	3.86
2	4	7	10.66	10.88
2	4	8	7.18	6.94
2	4	9	4.58	4.60
2	4	10	2.86	2.85
2	5	10	1.64	1.71
2	5	9	5.84	5.38
2	5	8	3.43	3.47
2	5	7	2.82	2.38
2	5	6	5.07	5.12
2	5	5	4.51	4.66
2	5	4	4.16	3.45
2	5	3	5.87	5.41
2	5	2	31.68	32.59
2	5	1	8.67	5.24
2	5	0	13.45	13.22
2	5	-1	14.91	14.79
2	5	-2	12.23	11.05
2	5	-3	15.26	15.17
2	5	-4	12.88	12.47
2	5	-5	8.63	9.28
2	5	-6	9.27	8.96
2	5	-7	13.20	13.65
2	5	-8	3.68	3.64
2	5	-9	3.88	3.88
2	5	-10	2.55	2.38
2	6	-10	1.78	1.82
2	6	-9	4.42	4.54

h	k	l	Fo	Fc
2	6	-8	4.56	4.55
2	6	-7	2.01	1.97
2	6	-6	4.62	4.78
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2	6	-4	5.17	5.35
2	6	-3	12.70	12.52
2	6	-2	18.81	17.43
2	6	-1	13.70	13.36
2	6	0	0.67*	1.17
2	6	1	3.65	3.86
2	6	2	16.22	17.34
2	6	3	12.94	13.17
2	6	4	9.59	10.31
2	6	5	12.58	12.28
2	6	6	8.27	8.52
2	6	7	3.26	2.75
2	6	8	3.89	3.43
2	6	9	2.53	2.51
2	6	10	4.59	4.16
2	7	9	1.47	1.42
2	7	8	5.59	5.31
2	7	7	6.97	6.82
2	7	6	3.65	3.69
2	7	5	12.54	13.27
2	7	4	5.33	5.36
2	7	3	9.30	9.14
2	7	2	6.03	3.84
2	7	1	11.63	12.19
2	7	0	5.44	5.64
2	7	-1	13.25	13.37
2	7	-2	5.80	5.71
2	7	-3	10.23	10.14
2	7	-4	9.39	9.95
2	7	-5	7.00	7.56
2	7	-6	10.36	11.54
2	7	-7	0.97	1.04
2	7	-8	5.62	5.92
2	7	-9	4.43	4.13
2	7	-10	2.28	2.36
2	8	-9	5.79	5.55
2	8	-8	2.63	2.88
2	8	-7	3.63	3.67
2	8	-6	11.08	11.72
2	8	-5	4.01	4.23
2	8	-4	9.14	8.95
2	8	-3	6.81	6.54
2	8	-2	8.84	8.65
2	8	-1	6.90	7.23
2	8	0	14.31	15.17
2	8	1	4.87	4.58
2	8	2	4.85	5.15
2	8	3	3.88	3.78
2	8	4	15.47	15.75
2	8	5	2.79	2.53
2	8	6	10.63	10.59
2	8	7	0.68*	1.17
2	8	8	6.90	6.66
2	8	9	4.47	4.54
2	9	9	5.55	5.18
2	9	8	2.11	2.11
2	9	7	3.52	3.51
2	9	6	3.52	3.22
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2	9	2	6.74	7.35
2	9	1	9.28	9.43
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2	9	-1	10.76	12.19
2	9	-2	6.96	7.01
2	9	-3	2.87	3.32
2	9	-4	9.44	10.53
2	9	-5	1.68	1.77
2	9	-6	1.06*	1.20
2	9	-7	1.47	1.51
2	9	-8	2.05	1.84
2	9	-9	2.94	3.06
2	10	-8	4.42	4.51
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2	10	-6	4.07	4.06
2	10	-5	1.68	2.00
2	10	-4	2.93	3.22
2	10	-3	5.53	6.27
2	10	-2	6.73	6.95
2	10	-1	10.53	11.42
2	10	0	6.89	7.76
2	10	1	4.42	4.84
2	10	2	13.57	14.59
2	10	3	3.04	3.04
2	10	4	5.62	5.87
2	10	5	4.28	4.31
2	10	6	1.90	1.99
2	10	7	2.43	2.60
2	10	8	2.19	2.17
2	11	7	4.79	4.56
2	11	6	2.66	2.68
2	11	5	0.93*	0.86
2	11	4	3.96	4.33
2	11	3	6.59	7.14
2	11	2	8.81	9.24
2	11	1	14.40	15.76
2	11	0	11.42	12.83
2	11	-1	8.17	8.83
2	11	-2	6.64	7.76
2	11	-3	1.55	1.42
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2	12	-1	5.08	5.32
2	12	0	5.01	5.10
2	12	1	5.48	6.29
2	12	2	6.73	6.49
2	12	3	4.26	4.23
2	12	4	1.98	2.02
2	12	5	3.48	3.59
2	12	6	4.15	4.38
2	12	7	3.71	3.76
2	13	6	2.03	2.45
2	13	5	3.30	3.23
2	13	4	4.90	4.78
2	13	3	3.95	3.74
2	13	2	2.23	1.92
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-2	13	3	1.95	1.71
-2	13	4	2.07	1.78
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-2	13	6	4.16	3.68
-2	14	5	5.97	5.52
-2	14	4	3.34	2.78
-2	14	3	1.00*	0.90
-2	14	2	4.10	3.82
-2	14	1	3.95	3.53
-2	14	0	4.19	3.69
-2	14	-1	5.03	4.81
2	14	2	2.57	3.84
2	14	3	2.25	2.24
2	14	4	2.50	2.44
2	15	3	0.41*	1.26
-2	15	-2	1.55	1.29
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-2	15	0	6.59	5.39
-2	15	1	2.17	2.35
-2	15	2	2.92	2.72
-2	15	3	0.96*	0.91
-3	15	1	2.60	2.31
-3	15	0	2.98	2.95
-3	15	-1	1.30*	1.47
3	14	3	1.08	1.91
3	14	2	0.0 *	3.29
-3	14	-1	5.11	4.22
-3	14	0	1.32	1.09
-3	14	1	5.97	5.29
-3	14	2	3.33	2.56
-3	14	3	2.64	2.39
-3	14	4	5.05	4.30
-3	13	5	11.84	10.04
-3	13	4	2.55	2.37
-3	13	3	5.84	5.03
-3	13	2	3.59	3.37
-3	13	1	4.53	4.39
-3	13	0	4.41	3.87
-3	13	-1	5.40	4.93
3	13	2	6.48	6.10
3	13	3	3.29	3.49
3	13	4	2.39	2.20
3	13	5	5.05	4.91
3	12	6	4.12	4.45
3	12	5	1.31	1.49
3	12	4	5.31	5.29
3	12	3	5.09	5.03
3	12	2	6.92	7.13
3	12	1	3.45	3.86
3	12	0	4.84	5.64
-3	12	1	1.43	1.41
-3	12	2	4.16	3.81
-3	12	3	4.17	3.65
-3	12	4	7.36*	6.47
-3	12	5	4.17	3.77
-3	12	6	4.25	3.49
-3	11	7	1.19	1.17
-3	11	6	0.0 *	0.86
-3	11	5	6.59	5.43
-3	11	4	9.22	8.63
-3	11	3	4.45	4.22
3	11	-2	0.85*	0.63
3	11	-1	5.90	6.11
3	11	0	7.71	9.03
3	11	1	7.21	7.86
3	11	2	7.20	7.70
3	11	3	4.42	4.67
3	11	4	2.11	2.04
3	11	5	4.02	3.80
3	11	6	6.43	5.97
3	11	7	3.68	3.67
3	10	7	3.79	3.38
3	10	6	2.27	2.13
3	10	5	2.09	2.19
3	10	4	6.58	6.71
3	10	3	13.80	13.86
3	10	2	11.47	12.04
3	10	1	8.78	9.52
3	10	0	4.00	4.55
3	10	-1	3.87	3.64
3	10	-2	0.79*	1.47
3	10	-3	11.78	11.71
3	10	-4	1.09*	1.24
3	10	-5	3.97	4.10
-3	10	6	5.36	5.04
-3	10	7	3.41	2.64
3	10	-8	1.47	1.60
3	9	-8	1.45	1.43
3	9	-7	0.95*	0.75
3	9	-6	8.94	9.28
3	9	-5	6.25	6.19
3	9	-4	7.36	7.81
3	9	-3	8.72	8.93
3	9	-2	12.31	11.88
3	9	-1	3.08	3.29
3	9	0	15.13	16.49
3	9	1	1.77	2.06
3	9	2	13.87	14.63
3	9	3	4.35	4.81
3	9	4	7.41	7.36
3	9	5	8.54	8.64
3	9	6	5.72	6.12
3	9	7	2.89	2.93
3	9	8	2.21	2.05
3	8	9	1.10	0.95
3	8	8	1.83	1.71
3	8	7	3.99	4.10
3	8	6	2.18	2.26
3	8	5	4.15	4.81
3	8	4	5.84	6.22
3	8	3	13.19	13.34
3	8	2	2.95	2.49
3	8	1	5.65	6.06
3	8	0	6.36	6.75
3	8	-1	4.67	5.15
3	8	-2	7.80	7.23
3	8	-3	7.74	8.01
3	8	-4	2.82	3.06
3	8	-5	10.68	11.34
3	8	-6	2.36	2.72
3	8	-7	1.55	1.60
3	8	-8	1.76	1.89
3	8	-9	0.96	0.92
3	7	-9	2.62	2.44
3	7	-8	1.31	1.32
3	7	-7	1.93	1.84
3	7	-6	7.15	7.42
3	7	-5	7.87	8.27
3	7	-4	6.93	6.70
3	7	-3	7.95	7.46
3	7	-2	9.76	9.32
3	7	-1	15.36	15.07
3	7	0	10.60	10.56
3	7	1	7.02	7.57
3	7	2	4.25	4.74
3	7	3	5.82	6.02
3	7	4	6.45	6.55
3	7	5	1.38	1.46
3	7	6	7.72	7.73
3	7	7	4.65	4.12
3	7	8	2.05	1.97
3	7	9	2.92	2.95
3	6	9	2.38	2.24
3	6	8	3.22	3.54
3	6	7	8.64	8.59
3	6	6	5.91	6.39
3	6	5	8.61	8.69
3	6	4	7.24	7.19
3	6	3	4.18	4.09
3	6	2	12.37	13.11
3	6	1	8.79	9.32
3	6	0	16.17	15.91
3	6	-1	21.39	20.70
3	6	-2	4.74	4.63
3	6	-3	8.71	8.92
3	6	-4	12.78	12.70

h	k	l	Fo	Fc
3	6	-5	9.19	9.43
3	6	-6	3.58	3.97
3	6	-7	5.35	5.35
3	6	-8	2.78	2.62
3	6	-9	3.14	3.19
3	6	-10	2.73	2.48
3	5	-10	3.56	3.67
3	5	-9	3.38	3.37
3	5	-8	2.52	2.71
3	5	-7	3.96	4.32
3	5	-6	0.79*	1.87
3	5	-5	14.25	14.89
3	5	-4	13.49	13.01
3	5	-3	13.66	13.04
3	5	-2	16.91	16.06
3	5	-1	14.75	13.53
3	5	0	11.61	11.18
3	5	1	19.73	20.54
3	5	2	10.67	11.13
3	5	3	6.41	6.31
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3	5	6	5.06	5.17
3	5	7	5.61	5.79
3	5	8	4.40	4.53
3	5	9	0.31*	0.52
3	5	10	3.27	2.93
3	4	10	2.63	2.46
3	4	9	4.57	4.45
3	4	8	7.72	8.27
3	4	7	2.82	2.70
3	4	6	7.78	7.86
3	4	5	3.79	3.45
3	4	4	8.97	9.11
3	4	3	10.85	11.70
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3	4	1	10.30	9.64
3	4	0	14.60	14.62
3	4	-1	8.33	8.65
3	4	-2	13.70	12.86
3	4	-3	14.89	14.35
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3	4	-5	8.51	8.80
3	4	-6	8.17	8.60
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3	3	-10	2.69	2.91
3	3	-9	2.47	2.58
3	3	-8	1.03*	1.17
3	3	-7	9.46	10.39
3	3	-6	8.23	8.85
3	3	-5	8.96	8.83
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3	3	-1	25.69	24.18
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3	3	1	7.56	7.94
3	3	2	5.27	5.21
3	3	3	8.17	8.69
3	3	4	2.59	2.96
3	3	5	3.82	3.82
3	3	6	7.36	7.69
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3	2	8	3.10	3.54
3	2	7	9.29	9.54
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3	2	4	11.85	12.43
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3	2	-1	17.75	16.67
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3	2	-3	9.81	9.81
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3	2	-6	2.39	2.25
3	2	-7	8.60	9.32
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3	1	-10	2.77	2.63
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3	1	-8	2.20	1.91
3	1	-7	8.11	8.65
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3	1	-5	11.44	10.94
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3	1	-1	23.12	22.36
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3	1	4	10.72	11.24
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3	1	6	4.35	4.56
3	1	7	5.68	5.59
3	1	8	6.00	5.66
3	1	9	9.33	8.96
3	1	10	2.90	2.80
3	0	10	4.58	4.83
3	0	9	0.0 *	0.86
3	0	8	1.45	1.35
3	0	7	1.81	1.63
3	0	6	1.06*	1.60
3	0	5	11.14	12.19
3	0	4	4.28	3.94
3	0	3	13.74	14.45
3	0	2	13.45	13.08
3	0	1	17.44	16.79
3	0	0	12.29	11.82
3	0	-1	12.85	12.08
3	0	-2	25.10	23.12
3	0	-3	23.26	22.00
3	0	-4	3.27	3.24
3	0	-5	8.83	9.11
3	0	-6	0.63*	1.89
3	0	-7	2.99	2.96
3	0	-8	2.06	1.99
3	0	-9	4.53	4.59
3	0	-10	4.65	4.26
3	0	-11	1.23	1.06
4	0	-10	0.93*	1.93
4	0	-9	0.88*	0.33
4	0	-8	0.82*	0.44
4	0	-7	6.50	6.33
4	0	-6	1.38	1.39
4	0	-5	2.78	2.93
4	0	-4	1.12	1.16
4	0	-3	9.56	9.52
4	0	-2	2.88	2.60
4	0	-1	27.85	25.26
4	0	0	40.09	36.57
4	0	1	15.73	14.68
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4	0	3	18.27	18.66
4	0	4	7.87	7.92
4	0	5	9.30	9.29
4	0	6	10.11	10.29
4	0	7	8.12	8.25
4	0	8	1.41	1.25
4	0	9	1.93	2.05
4	0	10	1.67	1.56
4	1	10	3.21	3.07
4	1	9	8.30	7.82
4	1	8	2.64	2.82
4	1	7	5.46	5.03
4	1	6	3.46	3.50
4	1	5	23.79	23.77
4	1	4	15.14	15.55
4	1	3	1.19	1.24
4	1	2	5.30	5.23
4	1	1	2.83	2.83
4	1	0	20.31	18.68
4	1	-1	9.11	8.19
4	1	-2	17.23	15.52
4	1	-3	9.34	9.34
4	1	-4	12.16	11.71
4	1	-5	6.56	6.99
4	1	-6	8.16	8.46
4	1	-7	3.89	4.04
4	1	-8	6.51	6.72
4	1	-9	2.38	2.36
4	1	-10	4.95	4.86
4	2	-10	3.43	3.07
4	2	-9	1.10*	1.19
4	2	-8	3.32	3.42
4	2	-7	12.06	12.30
4	2	-6	9.68	9.89
4	2	-5	5.26	5.17
4	2	-4	8.54	8.18
4	2	-3	8.65	8.18
4	2	-2	6.54	6.67
4	2	-1	10.54	9.84
4	2	0	18.15	17.74
4	2	1	4.68	4.40
4	2	2	9.52	9.07
4	2	3	8.53	8.21
4	2	4	11.27	11.51
4	2	5	0.0 *	1.06
4	2	6	10.07	10.41
4	2	7	2.07	1.98
4	2	8	2.15	2.24
4	2	9	3.80	3.87
4	3	9	2.04	2.23
4	3	8	3.40	3.57
4	3	7	3.02	2.86
4	3	6	2.10	1.97
4	3	5	5.76	6.56
4	3	4	6.32	6.38
4	3	3	6.68	7.09
4	3	2	12.61	13.08
4	3	1	8.09	7.22
4	3	0	10.54	10.55
4	3	-1	15.98	14.64
4	3	-2	6.35	6.34
4	3	-3	6.91	6.90
4	3	-4	4.15	3.96
4	3	-5	9.64	9.79
4	3	-6	8.41	8.59
4	3	-7	5.06	5.60
4	3	-8	4.56	4.90
4	3	-9	4.89	4.80
4	3	-10	3.24	3.43
4	4	-10	2.47	2.69
4	4	-9	1.76	1.73
4	4	-8	3.04	3.24
4	4	-7	5.42	5.68
4	4	-6	6.71	7.08
4	4	-5	10.78	10.42
4	4	-4	6.17	6.30
4	4	-3	19.84	19.59
4	4	-2	9.32	9.20
4	4	-1	9.62	8.78
4	4	0	4.94	4.78
4	4	1	8.95	8.79
4	4	2	6.26	6.72
4	4	3	3.92	4.14
4	4	4	2.99	3.14
4	4	5	9.69	10.16
4	4	6	0.94*	1.39
4	4	7	4.76	5.13
4	4	8	2.12	2.19
4	4	9	1.78	1.69
4	5	9	4.75	4.53
4	5	8	4.35	4.86
4	5	7	2.18	1.93
4	5	6	2.88	3.05
4	5	5	4.81	4.84
4	5	4	4.17	4.41
4	5	3	11.80	12.27
4	5	2	4.45	5.06
4	5	1	7.18	7.75
4	5	0	8.75	8.57
4	5	-1	10.37	9.37
4	5	-2	8.52	8.20
4	5	-3	3.03	2.87
4	5	-4	6.05	5.88
4	5	-5	9.31	9.84
4	5	-6	4.93	5.12
4	5	-7	6.92	7.34
4	5	-8	3.05	2.68
4	5	-9	3.19	3.38
4	6	-9	2.93	2.94
4	6	-8	0.39*	0.63
4	6	-7	3.22	3.36
4	6	-6	3.98	4.75
4	6	-5	12.43	12.83
4	6	-4	14.24	14.00
4	6	-3	3.18	3.02
4	6	-2	7.42	7.70
4	6	-1	4.69	4.11
4	8	0	13.88	13.45
4	6	1	2.53	2.60
4	6	2	15.01	16.11
4	6	3	14.56	15.18
4	6	4	11.38	11.86
4	6	5	6.09	6.71
4	6	6	6.14	6.50
4	6	7	3.52	3.75
4	6	8	2.81	2.76
4	6	9	0.22*	0.20
4	7	9	3.80	3.66
4	7	8	2.35	2.42
4	7	7	0.30*	0.65
4	7	6	1.00	1.03
4	7	5	8.81	8.69
4	7	4	5.23	6.26
4	7	3	10.15	10.72
4	7	2	10.47	11.10
4	7	1	0.0 *	1.26
4	7	0	4.29	4.80
4	7	-1	9.12	8.57
4	7	-2	5.87	6.04
4	7	-3	2.80	3.08
4	7	-4	9.88	10.71
4	7	-5	5.24	5.87
4	7	-6	2.63	2.77
4	7	-7		

h	k	l	Fo	Fc
4	7	-8	3.21	3.35
4	7	-9	1.70	1.59
4	8	-8	1.54	1.47
4	8	-7	3.36	3.44
4	8	-6	4.06	4.21
4	8	-5	9.10	9.10
4	8	-4	6.02	5.76
4	8	-3	5.33	5.38
4	8	-2	9.43	9.82
4	8	-1	12.70	12.44
4	8	0	1.76	1.96
4	8	1	9.20	9.81
4	8	2	10.51	10.90
4	8	3	12.01	12.82
4	8	4	7.22	6.93
4	8	5	7.51	7.77
4	8	6	6.01	6.33
4	8	7	3.76	3.75
4	8	8	2.00	1.78
4	9	7	1.58	1.48
4	9	6	2.20	2.30
4	9	5	4.68	4.63
4	9	4	5.81	5.66
4	9	3	7.44	7.26
4	9	2	5.55	5.62
4	9	1	4.72	5.45
4	9	0	5.80	5.87
4	9	-1	11.93	12.17
4	9	-2	7.39	7.19
4	9	-3	10.74	10.76
4	9	-4	4.56	4.57
4	9	-5	3.42	3.35
4	9	-6	5.03	5.82
4	9	-7	2.88	3.18
4	9	-8	0.51*	0.67
4	10	-7	0.0 *	1.33
-4	-10	6	1.71	1.49
-4	-10	5	5.42	4.58
-4	-10	4	3.60	3.19
4	10	-3	1.10*	0.23
4	10	-2	5.73	5.43
4	10	-1	5.69	5.62
4	10	0	2.43	2.99
4	10	1	5.37	6.06
4	10	2	3.78	3.90
4	10	3	0.72*	0.69
4	10	4	0.95*	0.67
4	10	5	3.25	3.14
4	10	6	0.98	0.97
4	10	7	3.58	3.51
4	11	6	2.96	2.95
4	11	5	1.82	1.84
4	11	4	2.12	1.82
4	11	3	3.30	3.11
4	11	2	3.69	3.72
4	11	1	2.07	2.45
4	11	0	1.91	2.20
4	11	-1	5.99	6.11
-4	-11	2	8.30	6.92
-4	-11	3	5.23	4.71
-4	-11	4	5.14	4.60
-4	-11	5	7.13	6.29
-4	-11	6	3.85	3.13
-4	-12	5	7.62	6.40
-4	-12	4	7.04	5.62
-4	-12	3	4.07	3.59
-4	-12	2	1.57	1.58
-4	-12	1	8.45	8.02
-4	-12	0	4.92	4.29
-4	-12	-1	1.81	1.82
4	12	2	4.96	5.19
4	12	3	3.21	3.06
4	12	4	0.92	0.92
4	12	5	1.90	1.89
4	13	4	1.82	1.92
4	13	3	2.60	2.30
4	13	2	1.97	3.24
-4	-13	-1	3.79	3.28
-4	-13	0	5.14	4.36
-4	-13	1	2.73	2.80
-4	-13	2	3.49	3.43
-4	-13	3	3.34	3.04
-4	-13	4	6.86	6.24
-4	-14	2	4.12	3.81
-4	-14	1	2.93	2.68
-4	-14	0	5.99	5.40
-4	-14	-1	3.17	3.00
4	14	2	0.0 *	4.15
-5	-13	2	2.73	2.31
-5	-13	1	1.06*	1.04
-5	-13	0	6.49	5.32
5	13	1	0.0 *	3.10
5	12	3	3.91	4.12
5	12	2	2.20	2.18
5	12	1	1.89	1.99
-5	-12	0	0.76*	1.18
-5	-12	1	3.70	3.28
-5	-12	2	3.45	2.80
-5	-12	3	3.77	2.68
-5	-12	4	4.92	4.11
-5	-11	5	2.97	2.28
-5	-11	4	5.03	4.53
-5	-11	3	3.06	2.42
-5	-11	2	2.04	1.81
5	11	-1	3.54	3.91
5	11	0	2.85	2.89
5	11	1	5.13	5.70
5	11	2	6.04	6.54
5	11	3	2.20	2.41
5	11	4	0.89*	0.74
5	11	5	4.71	5.01
5	10	6	4.08	4.05
5	10	5	2.23	2.37
5	10	4	3.64	3.67
5	10	3	3.36	3.43
5	10	2	2.79	3.36
5	10	1	3.01	3.16
5	10	0	3.05	3.28
5	10	-1	8.84	9.11
5	10	-2	8.03	7.97
5	10	-3	5.40	5.55
-5	-10	4	1.29	1.49
-5	-10	5	1.05	0.87
5	10	-6	0.45*	1.40
5	9	-7	2.35	2.45
5	9	-6	1.27	1.40
5	9	-5	0.65*	1.06
5	9	-4	2.27	2.50
5	9	-3	2.81	2.43
5	9	-2	4.43	4.43
5	9	-1	6.23	6.14
5	9	0	4.60	4.93
5	9	1	3.97	3.99
5	9	2	6.02	6.16
5	9	3	1.94	1.77
5	9	4	4.26	4.44
5	9	5	3.49	3.81
5	9	6	2.10	2.13
5	8	7	2.67	2.72
5	8	6	1.35	1.24
5	8	5	5.04	4.67
5	8	4	3.50	3.65
5	8	3	11.73	12.13
5	8	2	1.60	1.56
5	8	1	1.83	2.11
5	8	0	5.73	5.89
5	8	-1	9.80	9.94
5	8	-2	8.45	8.27
5	8	-3	7.34	7.56
5	8	-4	1.01*	0.72
5	8	-5	1.71	1.88
5	8	-6	3.11	3.28
5	8	-7	1.29	1.21
5	8	-8	2.84	2.88
5	7	-8	2.57	2.39
5	7	-7	2.72	3.16
5	7	-6	0.71*	0.54
5	7	-5	2.11	2.03
5	7	-4	4.88	5.30
5	7	-3	5.37	5.50
5	7	-2	9.55	9.86
5	7	-1	8.11	8.58
5	7	0	7.43	8.03
5	7	1	6.16	6.25
5	7	2	6.77	7.33
5	7	3	10.23	11.31
5	7	4	4.30	4.36
5	7	5	8.66	9.24
5	7	6	7.25	7.39
5	7	7	1.10	1.23
5	6	8	2.02	1.81
5	6	7	1.57	1.12
5	6	6	2.81	2.67
5	6	5	1.34	1.29
5	6	4	5.76	6.12
5	6	3	7.78	8.52
5	6	2	10.34	10.76
5	6	1	6.57	6.39
5	6	0	4.90	5.20
5	6	-1	6.00	5.81
5	6	-2	13.94	14.37
5	6	-3	8.52	8.34
5	6	-4	3.93	3.61
5	6	-5	4.32	4.80
5	6	-6	6.35	6.56
5	6	-7	2.33	2.51
5	6	-8	1.06	1.15
5	5	-9	4.29	4.13
5	5	-8	0.89	0.90
5	5	-7	3.58	3.76
5	5	-6	6.48	6.61
5	5	-5	6.71	6.75
5	5	-4	5.54	5.35
5	5	-3	6.31	6.02
5	5	-2	2.06	2.22
5	5	-1	12.09	12.02
5	5	0	5.75	5.31
5	5	1	9.85	10.08
5	5	2	9.26	9.26
5	5	3	9.59	10.17
5	5	4	4.30	4.12
5	5	5	3.21	3.50
5	5	6	3.04	3.13
5	5	7	5.09	4.83
5	5	8	2.01	2.01
5	4	8	1.70	1.86
5	4	7	4.03	4.04
5	4	6	3.00	2.79
5	4	5	4.53	4.41
5	4	4	9.66	9.72
5	4	3	8.88	9.30
5	4	2	7.63	7.32
5	4	1	4.38	4.16
5	4	0	2.52	2.72
5	4	-1	13.84	13.20
5	4	-2	17.51	17.09
5	4	-3	6.66	6.45
5	4	-4	8.28	8.54
5	4	-5	5.70	6.03
5	4	-6	4.35	4.37
5	4	-7	5.69	5.67
5	4	-8	3.56	3.49
5	4	-9	2.83	2.94
5	3	-9	2.02	2.03
5	3	-8	1.08	1.16
5	3	-7	3.89	3.89
5	3	-6	5.00	5.08
5	3	-5	3.84	3.86
5	3	-4	11.02	10.60
5	3	-3	9.81	9.40
5	3	-2	15.07	14.69
5	3	-1	5.45	5.67
5	3	0	6.38	6.01
5	3	1	5.09	5.13
5	3	2	7.63	7.78
5	3	3	3.69	3.79
5	3	4	11.78	11.70
5	3	5	7.28	7.54
5	3	6	6.70	7.06
5	-3	7	2.21	2.28
5	3	8	0.87	0.86
5	3	9	2.31	2.33
5	2	9	2.89	2.46
5	2	8	4.75	4.75
5	2	7	0.91*	0.57
5	2	6	4.77	4.90
5	2	5	7.37	7.28
5	2	4	5.14	5.27
5	2	3	6.66	6.62
5	2	2	12.81	12.84
5	2	1	11.12	10.75
5	2	0	12.28	11.64
5	2	-1	9.26	8.57
5	2	-2	5.27	5.03
5	2	-3	10.09	9.92
5	2	-4	10.73	10.48
5	2	-5	13.39	13.44
5	2	-6	12.31	12.27
5	2	-7	5.24	5.31
5	2	-8	1.56	1.45
5	2	-9	0.79*	0.41
5	1	-9	4.02	3.84
5	1	-8	2.98	3.24
5	1	-7	7.71	7.35
5	1	-6	7.15	7.28
5	1	-5	11.95	11.89
5	1	-4	14.24	14.00
5	1	-3	1.70	1.39
5	1	-2	9.89	9.28
5	1	-1	10.21	9.03
5	1	0	6.47	6.13
5	1	1	11.77	10.88
5	1	2	7.92	8.27
5	1	3	14.52	14.87
5	1	4	15.60	15.65
5	1	5	2.12	2.10
5	1	6	8.31	8.09
5	1	7	2.70	2.84
5	1	8	3.34	3.66
5	1	9	2.44	2.57
5	0	9	3.06	2.44
5	0	8	3.04	2.70
5	0	7	2.22	2.41
5	0	6	0.78*	0.98
5	0	5	11.12	10.93
5	0	4	0.92	0.93
5	0	3	3.03	3.26
5	0	2	11.28	11.31
5	0	1	4.26	3.98
5	0	0	5.93	5.61

h	k	l	Fo	Fc
5	0	-1	8.39	8.17
5	0	-2	4.86	4.91
5	0	-3	1.86	1.55
5	0	-4	12.88	11.99
5	0	-5	2.63	2.48
5	0	-6	2.82	2.98
5	0	-7	5.17	4.73
5	0	-8	5.73	5.54
5	0	-9	0.78*	1.56
6	0	-9	2.67	2.26
6	0	-8	0.0 *	0.26
6	0	-7	0.93*	1.02
6	0	-6	2.86	2.78
6	0	-5	3.82	3.19
6	0	-4	14.38	13.84
6	0	-3	1.89	1.77
6	0	-2	1.05*	0.18
6	0	-1	7.80	7.61
6	0	0	7.63	7.65
6	0	1	1.08	0.97
6	0	2	17.64	17.96
6	0	3	7.54	7.56
6	0	4	9.08	9.32
6	0	5	7.61	7.57
6	0	6	3.97	3.99
6	0	7	1.66	1.50
6	0	8	5.34	5.41
6	1	8	2.10	2.08
6	1	7	1.62	1.55
6	1	6	2.07	1.79
6	1	5	1.41	1.27
6	1	4	0.82*	1.24
6	1	3	11.90	11.63
6	1	2	5.09	5.19
6	1	1	4.29	4.45
6	1	0	9.15	8.60
6	1	-1	6.38	6.46
6	1	-2	6.74	6.67
6	1	-3	17.02	16.90
6	1	-4	7.59	7.52
6	1	-5	7.90	7.56
6	1	-6	1.06*	0.47
6	1	-7	1.00*	0.99
6	1	-8	1.03*	1.03
6	2	-8	3.23	3.16
6	2	-7	3.25	3.37
6	2	-6	6.84	7.06
6	2	-5	7.60	7.31
6	2	-4	5.07	5.24
6	2	-3	7.25	6.35
6	2	-2	3.92	3.51
6	2	-1	0.98*	0.87
6	2	0	7.01	6.89
6	2	1	14.82	15.17
6	2	2	3.02	3.06
6	2	3	3.42	3.52
6	2	4	9.39	9.72
6	2	5	4.65	4.82
6	2	6	0.94	1.00
6	2	7	1.35	1.13
6	2	8	5.02	4.80
6	3	7	1.54	1.26
6	3	6	4.29	4.03
6	3	5	3.75	3.60
6	3	4	7.79	7.64
6	3	3	11.25	11.25
6	3	2	5.70	5.91
6	3	1	7.85	8.07
6	3	0	7.02	6.85
6	3	-1	9.47	9.61
6	3	-2	10.50	10.75
6	3	-3	6.21	5.98
6	3	-4	14.05	13.81
6	3	-5	5.83	6.16
6	3	-6	3.04	3.22
6	3	-7	2.18	2.13
6	3	-8	2.87	2.74
6	4	-8	2.50	2.59
6	4	-7	3.98	4.30
6	4	-6	3.59	3.29
6	4	-5	4.57	4.63
6	4	-4	5.80	5.21
6	4	-3	6.08	6.16
6	4	-2	8.61	8.57
6	4	-1	9.86	9.86
6	4	0	3.96	3.90
6	4	1	4.06	3.59
6	4	2	11.90	12.04
6	4	3	5.12	5.15
6	4	4	2.61	2.67
6	4	5	5.14	4.87
6	4	6	1.82	1.60
6	4	7	5.16	4.82
6	5	7	4.58	4.48
6	5	6	3.99	3.66
6	5	5	3.05	2.79
6	5	4	5.59	5.71
6	5	3	0.94*	0.83
6	5	2	8.73	8.46
6	5	1	4.37	4.37
6	5	0	0.79*	0.68
6	5	-1	6.22	5.88
6	5	-2	11.22	11.53
6	5	-3	7.69	7.59
6	5	-4	6.55	6.88
6	5	-5	1.93	1.83
6	5	-6	2.23	1.90
6	5	-7	2.16	2.34
6	5	-8	3.11	2.83
6	6	-7	3.59	3.55
6	6	-6	2.34	2.42
6	6	-5	3.38	3.13
6	6	-4	3.09	3.09
6	6	-3	3.02	2.59
6	6	-2	5.49	5.87
6	6	-1	5.22	5.36
6	6	0	1.94	2.02
6	6	1	3.47	3.51
6	6	2	2.26	2.10
6	6	3	3.20	3.01
6	6	4	5.52	5.71
6	6	5	5.18	5.13
6	6	6	3.72	3.58
6	6	7	3.90	3.79
6	7	6	0.72*	0.59
6	7	5	3.60	3.54
6	7	4	1.02*	0.92
6	7	3	3.23	3.34
6	7	2	5.81	6.00
6	7	1	2.76	2.84
6	7	0	4.69	4.95
6	7	-1	5.58	5.54
6	7	-2	2.37	2.57
6	7	-3	1.18*	1.51
6	7	-4	2.57	2.84
6	7	-5	4.08	4.05
6	7	-6	1.97	2.15
6	7	-7	3.21	3.12
6	8	-6	3.52	3.78
6	8	-5	1.68	1.68
6	8	-4	3.97	3.94
6	8	-3	1.00*	1.18
6	8	-2	2.94	2.54
6	8	-1	1.98	1.94
6	8	0	3.87	4.15
6	8	1	5.61	5.97
6	8	2	3.71	3.75
6	8	3	3.58	3.41
6	8	4	3.93	4.14
6	8	5	3.88	4.01
6	8	6	2.32	2.35
6	9	5	3.29	3.76
6	9	4	2.95	3.02
6	9	3	3.89	3.74
6	9	2	1.68	1.75
6	9	1	3.98	4.38
6	9	0	4.68	4.76
6	9	-1	5.47	5.50
6	9	-2	3.60	3.87
6	9	-3	2.77	3.05
6	9	-4	1.08*	1.53
6	9	-5	0.75*	0.69
6	9	-6	0.58*	1.11
6	10	-5	0.0 *	1.26
6	10	-4	0.0 *	1.18
6	10	-3	1.79	3.16
6	10	-2	0.81*	1.33
6	10	-1	0.89*	1.00
6	10	0	0.89*	0.78
6	10	1	4.18	4.47
6	10	2	4.44	4.86
6	10	3	3.24	3.28
6	10	4	2.80	2.94
6	11	3	3.87	4.08
6	11	2	3.91	4.33
6	11	1	1.30	1.50
6	11	0	2.60	2.90
6	11	-1	0.22*	3.14
6	11	-2	0.26*	3.21
6	11	-3	2.27	2.05
7	10	0	0.47*	0.33
7	10	-1	2.13	2.63
7	9	-3	2.03	2.11
7	9	-2	1.66	2.02
7	9	-1	1.27	1.37
7	9	0	2.71	3.04
7	9	1	2.30	2.10
7	9	2	4.96	5.10
7	8	4	1.76	1.64
7	8	3	2.68	2.50
7	8	2	2.77	2.61
7	8	1	0.70*	0.93
7	8	0	1.97	1.67
7	8	-1	3.79	3.90
7	8	-2	3.17	3.22
7	8	-3	2.54	2.82
7	8	-4	1.17	1.26
7	7	-5	3.03	3.15
7	7	-4	3.83	3.96
7	7	-3	3.07	2.92
7	7	-2	3.07	2.74
7	7	-1	3.34	3.24
7	7	0	2.67	2.63
7	7	1	1.84	1.97
7	7	2	2.38	2.37
7	7	3	3.86	3.87
7	7	4	3.80	3.81
7	6	5	4.19	4.17
7	6	4	1.20	1.27
7	6	3	5.13	5.38
7	6	2	2.59	2.76
7	6	1	4.32	4.08
7	6	0	2.52	2.56
7	6	-1	2.07	2.15
7	6	-2	3.48	3.59
7	6	-3	0.96	0.94
7	6	-4	4.76	4.61
7	6	-5	4.60	4.76
7	6	-6	4.12	4.29
7	5	-6	4.05	4.14
7	5	-5	6.18	6.02
7	5	-4	2.82	2.92
7	5	-3	3.39	3.39
7	5	-2	6.42	6.23
7	5	-1	3.52	3.78
7	5	0	6.90	6.76
7	5	1	2.55	2.47
7	5	2	9.52	9.38
7	5	3	2.81	2.79
7	5	4	0.86*	0.76
7	5	5	3.28	3.22
7	4	6	2.17	1.99
7	4	5	3.23	3.21
7	4	4	4.17	4.00
7	4	3	3.97	3.77
7	4	2	5.65	5.92
7	4	1	5.12	5.26
7	4	0	2.26	2.32
7	4	-1	7.18	7.23
7	4	-2	8.46	8.54
7	4	-3	2.96	3.21
7	4	-4	3.33	3.06
7	4	-5	0.68*	0.75
7	4	-6	4.02	3.96
7	4	-7	2.59	2.36
7	3	-7	3.40	3.37
7	3	-6	2.67	2.85
7	3	-5	2.06	1.96
7	3	-4	3.46	3.42
7	3	-3	4.55	4.39
7	3	-2	10.94	10.60
7	3	-1	4.75	4.76
7	3	0	2.49	2.24
7	3	1	4.08	4.48
7	3	2	8.93	8.86
7	3	3	2.78	3.01
7	3	4	6.47	6.42
7	3	5	5.77	5.50
7	3	6	1.59	1.61
7	2	6	3.63	3.39
7	2	5	3.22	3.16
7	2	4	6.11	6.02
7	2	3	1.80	1.85
7	2	2	4.03	4.50
7	2	1	2.32	2.60
7	2	0	2.26	2.31
7	2	-1	3.76	3.74
7	2	-2	9.67	9.06
7	2	-3	4.20	3.96
7	2	-4	3.97	3.73
7	2	-5	3.59	3.75
7	2	-6	1.94	1.70
7	2	-7	1.62	1.61
7	1	-7	1.72	1.41
7	1	-6	1.01	1.04
7	1	-5	2.66	2.55
7	1	-4	2.65	2.30
7	1	-3	7.54	7.31
7	1	-2	1.59	1.57
7	1	-1	3.65	3.78
7	1	0	6.08	5.84
7	1	1	4.22	3.92
7	1	2	6.30	6.28
7	1	3	3.96	3.86
7	1	4	2.55	2.28
7	1	5	2.23	2.06
7	1	6	0.43*	0.53
7	0	5	1.62	1.72
7	0	4	1.98	1.56
7	0		1.98	1.63

h	k	l	Fo	Fc
7	0	3	2.15	2.02
7	0	2	4.54	4.45
7	0	1	9.90	9.30
7	0	0	0.71*	0.18
7	0	-1	4.00	4.00
7	0	-2	2.00	2.19
7	0	-3	10.13	9.99
7	0	-4	2 33	2 22
7	0	-5	4.88	4.86
7	0	-6	0.98*	0.93
7	0	-7	0.38*	0.60
8	0	-5	0.15*	0.40
8	0	-4	1.76	1.77
8	0	-3	5.17	4.78
8	0	-2	2.54	2.66
8	0	-1	4.10	3.95
8	0	0	3.30	3.22
8	0	1	3.01	3.03
8	0	2	2.76	2.82
8	0	3	0.16*	0.27
8	0	4	4.14	4.13
8	1	4	3.47	3.03
8	1	3	3.33	3.39
8	1	2	1.90	1.76
8	1	1	3.72	3.82
8	1	0	3.35	3.38
8	1	-1	2.48	2.42
8	1	-2	3.32	2.89
8	1	-3	3.40	3.34
8	1	-4	1.03*	1.17
8	1	-5	1.01	1.02
8	2	-5	0.0 *	0.33
8	2	-4	0.89*	0.96
8	2	-3	4.00	3.96
8	2	-2	2.99	2.84
8	2	-1	3.49	3.49
8	2	0	1.63	1.44
8	2	1	2.37	2.24
8	2	2	1.46	1.52
8	2	3	0.0 *	0.35
8	2	4	3.10	2.87
8	3	4	5.29	4.72
8	3	3	2.98	2.93
8	3	2	2.27	2.21
8	3	1	3.45	3.47
8	3	0	2.30	2.71
8	3	-1	5.21	5.08
8	3	-2	3 46	3.39
8	3	-3	0.86*	0.89
8	3	-4	1.25	1.30
8	3	-5	2.26	2.36
8	4	-5	1 09	1.30
8	4	-4	5.02	5.49
8	4	-3	3.87	3.90
8	4	-2	2.75	2.63
8	4	-1	2 55	2 43
8	4	0	0 67*	0.78
8	4	1	3.54	3.44
8	4	2	0.94*	1 46
8	4	3	1.34	1.28
8	5	3	2.65	2.73
8	5	2	3 29	3.07
8	5	1	2.13	2 01
8	5	0	3.28	3.21
8	5	-1	3.96	4.17
8	5	-2	5.02	5.10
8	5	-3	2.39	2.28
8	5	-4	3.78	3.71
8	6	-3	2.25	2.27
8	6	-2	0.0 *	0.74
8	6	-1	2.37	2.42
8	6	0	2.66	2.69
8	6	1	3 27	3.49
8	6	2	1.77	1.83
8	7	1	4.78	4.91
8	7	0	2.70	3.06
8	7	-1	2.79	3.10
8	7	-2	2.53	2.54
9	1	-1	0.77*	1.17
9	1	0	1.95	1.66
9	0	0	3.05	3.31
9	0	-1	0 0 *	0.75

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STRUCTURE FACTOR TABLES FOR METHYL 3-C-(CARBOMETHOXYMETHYL)-4,6-DI-O-p-CHLOROBENZOYL-2,3-DIDEOXY-α-D-RIBO-HEXOPYRANOSIDE

h	k	l	Fo	Fc
0	0	1	14.53	15.45
0	0	2	19.95	21.08
0	0	3	1.79*	1.92
0	0	4	6.67	7.00
0	0	5	7.37	7.11
0	0	6	25.76	24.30
0	0	7	29.24	29.40
0	0	8	19.62	19.61
0	0	9	12.18	12.25
0	0	10	3.96	2.67
0	0	11	1.97*	1.05
0	0	12	1.24*	2.30
0	2	0	23.66	23.79
0	2	1	40.74	41.63
0	2	2	13.82	12.85
0	2	3	127.08	131.32
0	2	4	39.06	39.11
0	2	5	6.68	6.90
0	2	6	11.40	10.97
0	2	7	16.18	15.64
0	2	8	5.90	5.96
0	2	9	1.28*	0.13
0	2	10	15.46	15.58
0	2	11	8.39	8.75
0	2	12	5.54	4.62
0	4	0	80.66	80.60
0	4	1	43.61	42.13
0	4	2	38.37	38.53
0	4	3	28.68	28.35
0	4	4	23.35	23.76
0	4	5	10.54	10.31
0	4	6	17.44	18.03
0	4	7	37.28	38.47
0	4	8	11.39	10.49
0	4	9	18.73	18.54
0	4	10	9.60	9.83
0	4	11	3.80	4.11
0	4	12	4.28	2.18
0	6	0	29.37	28.31
0	6	1	29.63	29.50
0	6	2	15.35	14.06
0	6	3	36.45	36.87
0	6	4	35.13	35.86
0	6	5	9.69	10.70
0	6	6	5.45	5.59
0	6	7	8.31	8.30
0	6	8	3.31*	4.94
0	6	9	15.58	15.65
0	6	10	16.22	17.88
0	6	11	8.70	8.83
0	8	0	31.54	31.33
0	8	1	13.52	12.59
0	8	2	5.23	4.76
0	8	3	8.93	8.70
0	8	4	11.14	11.58
0	8	5	13.45	12.86
0	8	6	5.48	5.53
0	8	7	11.49	11.04
0	8	8	4.48	4.71
0	8	9	15.11	14.35
0	8	10	5.25	5.26
0	10	0	12.01	10.53
0	10	1	6.69	6.42
0	10	2	2.30*	2.64
0	10	3	19.55	18.86
0	10	4	3.85	3.51
0	10	5	19.21	19.38
0	10	6	17.16	17.35
0	10	7	0.60*	1.49
0	10	8	5.53	5.02
0	12	0	21.48	21.11
0	12	1	5.88	5.40
0	12	2	15.82	16.25
0	12	3	15.65	15.89
0	12	4	4.21	4.39
0	12	5	6.07	5.56
0	12	6	5.79	4.31
0	14	0	16.95	17.27
0	14	1	11.87	12.31
0	1	1	16.98	17.96
0	1	2	6.25	6.34
0	1	3	46.95	45.54
0	1	4	5.99	6.51
0	1	5	8.19	7.61
0	1	6	42.57	42.33
0	1	7	6.07	6.81
0	1	8	13.66	12.92
0	1	9	0.0*	1.06
0	1	10	3.42	1.59
0	1	11	13.38	13.35
0	1	12	3.63	3.54
0	3	1	38.05	39.42
0	3	2	46.13	46.38
0	3	3	82.25	86.18
0	3	4	17.47	18.19
0	3	5	21.15	21.31
0	3	6	6.02	6.01
0	3	7	0.0*	4.53
0	3	8	18.48	18.23
0	3	9	5.48	5.31
0	3	10	13.91	13.58
0	3	11	4.46	3.65
0	3	12	5.55	6.49
0	5	1	12.58	11.58
0	5	2	41.09	40.72
0	5	3	14.92	15.13
0	5	4	15.07	14.79
0	5	5	14.33	13.51
0	5	6	12.90	12.83
0	5	7	22.00	22.58
0	5	8	15.74	15.80
0	5	9	10.66	10.39
0	5	10	6.44	6.00
0	5	11	9.01	8.33
0	7	1	16.09	15.18
0	7	2	19.13	19.71
0	7	3	36.13	36.41
0	7	4	14.05	12.97
0	7	5	26.45	26.21
0	7	6	9.23	10.15
0	7	7	15.77	15.69
0	7	8	3.50	4.15
0	7	9	8.19	7.58
0	7	10	5.34	5.06
0	9	1	1.09*	2.77
0	9	2	13.00	13.49
0	9	3	12.43	12.89
0	9	4	4.06	5.12
0	9	5	16.58	16.95
0	9	6	9.24	8.78
0	9	7	4.51	5.34
0	9	8	7.70	7.64
0	9	9	2.69*	0.94
0	11	1	3.70	2.87
0	11	2	13.36	13.60
0	11	3	7.41	6.66
0	11	4	6.20	6.49
0	11	5	11.06	11.09
0	11	6	9.41	8.98
0	11	7	0.0*	1.30
0	13	1	6.01	4.64
0	13	2	14.72	15.47
0	13	3	6.34	6.41
0	13	4	2.00*	2.95
1	0	0	64.23	64.56
1	0	1	38.57	36.76
1	0	-1	93.02	95.46
1	0	2	98.73	101.44
1	0	-2	63.90	64.99
1	0	3	156.23	165.99
1	0	-3	59.68	58.56
1	0	4	33.41	32.09
1	0	-4	31.56	31.39
1	0	5	53.55	53.19
1	0	-5	9.13	8.50
1	0	6	56.48	55.07
1	0	-6	3.71	2.59
1	0	7	24.67	22.66
1	0	-7	13.32	13.76
1	0	8	3.52	3.34
1	0	-8	6.88	5.94
1	0	9	6.87	6.65
1	0	-9	5.16	4.95
1	0	10	7.81	6.98
1	0	-10	16.18	15.64
1	0	11	2.32*	1.59
1	0	-11	6.29	5.82
1	0	12	5.25	4.52
1	0	-12	3.62*	5.70
1	2	0	89.50	92.23
1	2	1	19.31	19.12
1	2	-1	26.81	27.84
1	2	2	34.39	33.90
1	2	-2	25.45	25.59
1	2	3	67.14	67.45
1	2	-3	15.11	14.71
1	2	4	33.60	33.84
1	2	-4	13.91	14.25
1	2	5	27.79	28.76
1	2	-5	18.19	18.16
1	2	6	43.87	44.45
1	2	-6	3.78	5.08
1	2	7	12.77	12.35
1	2	-7	19.16	18.68
1	2	8	11.64	11.64
1	2	-8	19.48	20.05
1	2	9	16.08	15.71
1	2	-9	2.42*	4.21
1	2	10	5.94	5.19
1	2	-10	2.03	1.67
1	2	11	6.78	6.19
1	2	-11	2.84*	2.14
1	2	12	2.53*	2.87
1	2	-12	3.19*	3.86
1	4	0	21.99	21.82
1	4	1	42.24	42.70
1	4	-1	76.24	76.98
1	4	2	17.37	17.32
1	4	-2	17.56	17.35
1	4	3	21.69	21.17
1	4	-3	25.69	25.05
1	4	4	14.44	14.66
1	4	-4	38.53	38.40
1	4	5	12.22	11.98
1	4	-5	29.92	27.43
1	4	6	39.06	38.44
1	4	-6	13.67	14.40
1	4	7	9.08	9.68
1	4	-7	10.84	10.60
1	4	8	11.16	11.82
1	4	-8	9.64	10.00
1	4	9	2.43*	3.67
1	4	-9	2.83*	3.59
1	4	10	11.15	10.82
1	4	-10	16.94	17.79
1	4	11	4.37	4.57
1	4	-11	0.0*	1.49
1	6	0	28.55	27.52
1	6	1	11.64	10.66
1	6	-1	35.81	34.15
1	6	2	29.27	29.62
1	6	-2	15.93	15.36
1	6	3	11.60	11.04
1	6	-3	23.48	23.05
1	6	4	3.59	3.54
1	6	-4	69.70	68.32
1	6	5	12.62	12.24
1	6	-5	8.21	7.74
1	6	6	6.67	6.81
1	6	-6	14.18	13.99
1	6	7	10.34	10.31
1	6	-7	15.88	16.31
1	6	8	5.38	5.15
1	6	-8	6.81	6.08
1	6	9	22.72	23.58
1	6	-9	6.19	5.74
1	6	10	4.07	3.53
1	6	-10	3.30*	2.80
1	6	-11	6.99	6.65
1	8	0	23.07	22.95
1	8	1	4.43	3.65
1	8	-1	33.13	32.95
1	8	2	26.15	26.24
1	8	-2	10.66	10.14
1	8	3	8.84	8.43
1	8	-3	19.03	18.24
1	8	4	10.13	9.79
1	8	-4	24.17	25.04
1	8	5	10.84	10.09
1	8	-5	4.02	2.85
1	8	6	15.71	15.61
1	8	-6	8.55	7.65
1	8	7	2.58*	0.84
1	8	-7	7.41	7.06
1	8	8	5.33	6.05
1	8	-8	13.41	14.08
1	8	9	11.82	11.84
1	8	-9	3.74	2.50
1	8	-10	9.06	9.15
1	10	0	16.81	17.06
1	10	1	6.83	7.47
1	10	-1	10.84	10.38
1	10	2	12.90	12.81
1	10	-2	14.48	14.05
1	10	3	11.27	11.41
1	10	-3	16.61	17.23
1	10	4	2.30*	2.20
1	10	-4	6.13	5.74
1	10	5	3.09*	2.45
1	10	-5	11.24	11.32
1	10	6	3.81	4.61
1	10	-6	3.01*	2.49
1	10	7	2.83*	1.43
1	10	-7	3.52*	2.75
1	10	8	11.86	12.66
1	10	-8	6.36	6.68
1	12	0	9.17	9.23
1	12	1	9.17	9.07
1	12	-1	13.56	13.60
1	12	2	9.37	9.86
1	12	-2	6.85	7.43
1	12	3	6.74	6.17
1	12	-3	7.49	8.94
1	12	4	4.16	4.91
1	12	-4	2.80*	0.79
1	12	5	12.70	13.56
1	12	-5	3.18*	3.27
1	12	6	11.60	11.69
1	12	-6	5.21	5.89
1	1	0	49.37	49.27

h	k	l	Fo	Fc
1	1	1	45.11	43.27
1	1	-1	41.65	41.97
1	1	2	22.81	23.57
1	1	-2	20.17	20.24
1	1	3	68.04	68.67
1	1	-3	37.24	38.15
1	1	4	44.14	44.08
1	1	-4	31.47	32.25
1	1	5	36.38	36.28
1	1	-5	16.80	16.48
1	1	6	6.37	5.79
1	1	-6	5.10	5.53
1	1	7	9.95	10.68
1	1	-7	6.95	6.79
1	1	8	5.12	5.13
1	1	-8	5.28	4.89
1	1	9	4.90	5.23
1	1	-9	12.37	11.95
1	1	10	5.52	5.25
1	1	-10	2.14*	1.81
1	1	11	3.70	3.83
1	1	-11	7.56	8.54
1	1	12	0.0 *	2.56
1	1	-12	10.30	9.68
1	3	0	57.76	59.95
1	3	1	38.08	37.20
1	3	-1	21.02	21.25
1	3	2	12.28	12.63
1	3	-2	3.83	4.07
1	3	3	29.57	30.95
1	3	-3	12.32	12.41
1	3	4	9.06	7.91
1	3	-4	19.62	19.82
1	3	5	10.33	10.82
1	3	-5	24.34	23.98
1	3	6	50.68	51.38
1	3	-6	9.29	9.03
1	3	7	29.47	29.42
1	3	-7	34.59	35.01
1	3	8	6.23	5.49
1	3	-8	15.46	15.60
1	3	9	4.61	3.87
1	3	-9	5.21	5.08
1	3	10	2.61*	1.76
1	3	-10	0.0 *	3.23
1	3	11	11.20	10.66
1	3	-11	9.91	9.77
1	3	-12	12.57	11.79
1	5	0	4.93	3.75
1	5	1	24.46	25.39
1	5	-1	8.29	8.71
1	5	2	10.86	11.90
1	5	-2	8.93	8.74
1	5	3	16.80	16.70
1	5	-3	9.01	9.57
1	5	4	19.58	19.94
1	5	-4	34.34	34.54
1	5	5	19.73	20.38
1	5	-5	30.12	30.17
1	5	6	7.38	6.07
1	5	-6	2.77	2.10
1	5	7	17.47	17.46
1	5	-7	2.32*	1.16
1	5	8	4.16	4.59
1	5	-8	9.55	10.12
1	5	9	10.35	11.20
1	5	-9	15.20	14.26
1	5	10	0.0 *	2.09
1	5	-10	17.83	17.62
1	5	11	4.99	5.12
1	5	-11	4.38	2.47
1	7	0	21.79	20.54
1	7	1	16.41	16.76
1	7	-1	17.41	18.00
1	7	2	13.00	13.01
1	7	-2	45.74	45.50
1	7	3	11.26	10.31
1	7	-3	11.50	12.20
1	7	4	16.00	16.47
1	7	-4	14.64	14.76
1	7	5	8.75	8.55
1	7	-5	16.84	16.86
1	7	6	8.60	8.82
1	7	-6	8.85	7.41
1	7	7	5.93	5.93
1	7	-7	5.99	4.77
1	7	8	5.76	6.07
1	7	-8	4.18	4.57
1	7	9	1.98*	4.42
1	7	-9	14.28	14.54
1	7	10	6.08	6.63
1	7	-10	11.91	12.98
1	9	0	6.73	6.03
1	9	1	4.61	4.15
1	9	-1	7.02	7.06
1	9	2	2.33*	2.86
1	9	-2	4.92	4.77
1	9	3	8.42	8.78
1	9	-3	12.83	12.09
1	9	4	9.56	9.57
1	9	-4	10.19	9.58
1	9	5	7.19	7.58
1	9	-5	8.87	8.33
1	9	6	5.59	6.11
1	9	-6	2.98*	4.47
1	9	7	8.96	8.15
1	9	-7	7.66	7.38
1	9	8	6.34	5.92
1	9	-8	10.33	10.36
1	9	9	10.40	10.56
1	9	-9	0.0 *	1.51
1	11	0	10.90	11.21
1	11	1	4.46	5.84
1	11	-1	9.85	9.91
1	11	2	9.25	9.31
1	11	-2	15.50	15.65
1	11	3	5.37	7.21
1	11	-3	8.69	9.05
1	11	4	5.01	5.51
1	11	-4	7.71	6.92
1	11	5	8.05	8.14
1	11	-5	6.67	6.43
1	11	6	12.40	12.96
1	11	-6	2.85*	2.77
1	11	7	3.58*	5.07
1	11	-7	4.65	4.12
1	13	0	14.34	14.47
1	13	1	5.82	6.13
1	13	-1	6.00	5.40
1	13	2	9.86	10.11
1	13	-2	4.67	4.65
1	13	3	4.96	4.57
1	13	-3	3.12*	1.65
1	13	4	3.08*	4.34
1	13	-4	6.23	6.19
2	0	0	11.24	10.54
2	0	1	41.56	42.00
2	0	-1	72.96	72.28
2	0	2	14.61	14.15
2	0	-2	17.52	17.61
2	0	3	0.21*	1.58
2	0	-3	5.23	5.37
2	0	4	0.98*	1.44
2	0	-4	0.67*	1.82
2	0	5	7.63	8.00
2	0	-5	18.68	20.19
2	0	6	39.94	40.16
2	0	-6	13.95	15.04
2	0	7	14.10	14.95
2	0	-7	8.08	8.57
2	0	8	2.83*	2.72
2	0	-8	1.94*	0.22
2	0	9	2.55*	2.22
2	0	-9	3.70	1.35
2	0	10	0.60*	2.35
2	0	-10	2.69*	1.35
2	0	11	0.0 *	0.54
2	0	-11	3.77	4.99
2	2	0	41.45	41.81
2	2	1	16.49	16.54
2	2	-1	14.14	14.00
2	2	2	10.39	10.34
2	2	-2	16.76	17.25
2	2	3	21.46	21.57
2	2	-3	10.09	10.13
2	2	4	15.04	13.72
2	2	-4	22.01	22.15
2	2	5	19.53	18.51
2	2	-5	21.75	21.70
2	2	6	30.31	30.12
2	2	-6	5.10	4.34
2	2	7	13.82	13.82
2	2	-7	9.92	9.82
2	2	8	11.54	10.90
2	2	-8	7.06	7.42
2	2	9	5.92	6.03
2	2	-9	8.05	7.14
2	2	10	1.59*	2.49
2	2	-10	4.69	2.88
2	2	11	2.59*	2.72
2	2	-11	6.59	6.30
2	4	0	10.66	10.79
2	4	1	19.62	20.01
2	4	-1	17.61	17.81
2	4	2	14.82	14.70
2	4	-2	11.54	12.06
2	4	3	21.63	21.90
2	4	-3	6.93	7.28
2	4	4	20.60	20.62
2	4	-4	31.97	33.53
2	4	5	13.60	13.91
2	4	-5	13.07	11.52
2	4	6	11.64	12.45
2	4	-6	8.88	9.10
2	4	7	10.91	10.98
2	4	-7	7.09	7.07
2	4	8	10.01	10.04
2	4	-8	18.36	18.30
2	4	9	16.00	15.72
2	4	-9	4.26	4.02
2	4	10	9.19	7.69
2	4	-10	3.84	3.79
2	4	-11	5.84	4.74
2	6	0	23.88	22.87
2	6	1	23.09	22.97
2	6	2	7.02	6.89
2	6	-2	32.36	31.02
2	6	3	16.11	15.80
2	6	-3	7.99	8.27
2	6	-4	21.88	22.09
2	6	5	3.58	3.49
2	6	-5	2.72*	1.49
2	6	6	2.71*	2.16
2	6	-6	6.04	6.19
2	6	7	3.41	2.16
2	6	-7	3.90	3.41
2	6	8	4.15	4.37
2	6	-8	3.44	2.56
2	6	9	2.27*	2.05
2	6	-9	8.09	7.49
2	6	10	9.02	9.53
2	6	-10	6.57	7.05
2	8	0	6.61	7.74
2	8	1	24.38	24.87
2	8	-1	3.32	3.31
2	8	2	14.71	14.69
2	8	-2	17.36	17.34
2	8	3	6.02	6.80
2	8	-3	9.88	9.31
2	8	-4	4.86	4.30
2	8	4	8.84	8.67
2	8	5	0.95*	3.55
2	8	-5	12.72	13.21
2	8	6	0.0 *	0.73
2	8	-6	8.71	8.99
2	8	7	0.60*	0.64
2	8	-7	3.41*	1.20
2	8	8	5.85	5.47
2	8	-8	7.00	7.32
2	8	-9	7.44	7.98
2	10	0	2.57*	0.41
2	10	1	5.41	5.05
2	10	-1	8.16	7.75
2	10	2	0.71*	1.80
2	10	-2	5.12	4.58
2	10	3	5.78	5.35
2	10	-3	7.41	7.33
2	10	4	8.34	8.54
2	10	-4	6.79	6.93
2	10	5	4.13	3.32
2	10	-5	7.97	8.41
2	10	6	0.0 *	1.90
2	10	-6	6.96	6.71
2	10	7	0.65*	0.79
2	10	-7	13.81	13.04
2	12	0	4.73	3.96
2	12	1	13.82	14.07
2	12	-1	7.11	6.64
2	12	2	1.45*	2.95
2	12	-2	2.79*	3.84
2	12	3	2.64*	1.63
2	12	-3	4.65	4.16
2	12	4	5.23	6.44
2	12	-4	2.67*	3.64
2	1	0	21.43	20.76
2	1	1	27.75	27.39
2	1	-1	29.60	28.88
2	1	2	18.50	18.30
2	1	-2	19.96	21.34
2	1	3	33.81	33.95
2	1	-3	33.37	33.75
2	1	4	4.71	4.66
2	1	-4	26.24	26.22
2	1	5	23.00	23.64
2	1	-5	19.38	19.54
2	1	6	28.15	29.13
2	1	-6	14.13	13.95
2	1	7	22.11	22.34
2	1	-7	13.84	13.82
2	1	8	6.79	6.00
2	1	-8	6.61	5.64
2	1	9	3.43	3.06
2	1	-9	7.32	7.15
2	1	10	3.38*	1.64
2	1	-10	9.17	8.85
2	1	11	3.56*	2.70
2	1	-11	3.29*	1.00
2	3	0	23.05	23.38
2	3	1	12.21	12.09
2	3	-1	13.14	12.73

h	k	l	Fo	Fc
2	3	2	34 17	34 83
2	3	-2	18.99	18.19
2	3	3	6.60	6.58
2	3	-3	24.71	25.70
2	3	4	11.17	11.51
2	3	-4	18.89	19.70
2	3	5	8.06	8.45
2	3	-5	22.10	22.25
2	3	6	21.30	21.32
2	3	-6	21.66	21.89
2	3	7	10.32	10.59
2	3	-7	12.05	11.94
2	3	8	7.62	8.18
2	3	-8	10.53	11.48
2	3	9	17.73	18 37
2	3	-9	8.29	7.87
2	3	10	4.16	5.68
2	3	-10	7.61	7.98
2	3	-11	6.79	5.63
2	5	0	14.38	14.96
2	5	1	10.26	10.96
2	5	-1	8.97	9.26
2	5	2	29.68	28.48
2	5	-2	23.78	23.56
2	5	3	16.93	15.83
2	5	-3	35.73	34.08
2	5	4	9.96	9.22
2	5	-4	20.83	20.64
2	5	5	9.60	9.68
2	5	-5	16.66	17.30
2	5	6	7.99	7.39
2	5	-6	6.76	7.12
2	5	7	7.23	7.41
2	5	-7	1.89*	0.67
2	5	8	4.20	4.58
2	5	-8	14.23	14.64
2	5	9	10.41	9.94
2	5	-9	9.43	8.88
2	5	10	6.30	5.11
2	5	-10	6.35	6.49
2	5	-11	7.19	7 26
2	7	0	11.18	10.54
2	7	1	16.91	16.70
2	7	-1	20.63	20.37
2	7	2	6.01	5.49
2	7	-2	12.45	11.03
2	7	3	11.47	11.84
2	7	-3	19.56	19.14
2	7	4	2.96	2.89
2	7	-4	16.32	16.96
2	7	5	15.16	14.79
2	7	-5	13.68	14.16
2	7	6	5.37	5.71
2	7	-6	12.16	12.12
2	7	7	5.46	4 87
2	7	-7	3.73	1 88
2	7	8	6.70	6 81
2	7	-8	10.35	10 76

h	k	l	Fo	Fc
2	7	9	8 17	8.44
2	7	-9	7.23	6.89
2	7	-10	8 53	8.03
2	9	0	12.04	12.52
2	9	1	2.71*	2.15
2	9	-1	4.51	4.44
2	9	2	19 03	18.43
2	9	-2	17.05	17.52
2	9	3	6.25	7.35
2	9	-3	3 37	1.60
2	9	4	4 15	3.98
2	9	-4	9 62	9.02
2	9	5	4.38	3.99
2	9	-5	14.99	13.95
2	9	6	4.43	3.30
2	9	-6	6.21	5.31
2	9	7	4.41	3.60
2	9	-7	7.79	7.90
2	9	8	6.71	6.69
2	9	-8	4.39	2.87
2	11	0	11.60	11.29
2	11	1	9.01	9.02
2	11	-1	2.53*	3.57
2	11	2	3.38*	3.34
2	11	-2	9.35	9.85
2	11	3	8.58	9.03
2	11	-3	3.81	3 74
2	11	4	6.97	6.81
2	11	-4	5.56	6.00
2	11	5	9.95	9.43
2	11	-5	5.74	4.47
2	11	-6	0.0 *	1.64
2	13	0	7.68	6.65
2	13	1	9.40	9.15
2	13	-1	3.90	5 67
3	0	0	35.16	34.13
3	0	1	4.46	4.35
3	0	-1	23.77	23.32
3	0	2	37.92	38.42
3	0	-2	19.80	19.43
3	0	3	5.36	5.07
3	0	-3	26 59	25.35
3	0	4	9.17	8.34
3	0	-4	8.17	8.23
3	0	5	3.23	0.68
3	0	-5	10 51	10.30
3	0	6	11.12	10.96
3	0	-6	4.50	4.07
3	0	7	18.98	18.33
3	0	-7	3.74*	3.95
3	0	8	9.77	9.98
3	0	-8	5.16	3.55
3	0	9	7.19	6.46
3	0	-9	6 75	7 16
3	0	-10	3.28*	1.21
3	2	0	11.68	10.68
3	2	1	10.88	11 92
3	2	-1	29 72	28.53

h	k	l	Fo	Fc
3	2	2	6.62	6 20
3	2	-2	7.95	7 09
3	2	3	10.34	10 23
3	2	-3	9 99	8.55
3	2	4	3 37	3 10
3	2	-4	10 52	10 07
3	2	5	3.31	3.44
3	2	-5	3 49	3.34
3	2	6	11 24	12 23
3	2	-6	17.03	16 52
3	2	7	11.49	11.46
3	2	-7	8.46	8 52
3	2	8	0.60*	3.36
3	2	-8	6 65	6.50
3	2	9	10.95	10.61
3	2	-9	8.75	8.35
3	2	-10	11.02	10.76
3	4	0	13 44	14 18
3	4	1	4.88	4.62
3	4	-1	13 32	13.53
3	4	2	8.11	8.56
3	4	-2	13 65	13 85
3	4	3	5.41	5.00
3	4	-3	11 78	12.01
3	4	4	5.19	5.42
3	4	-4	3.25	4.13
3	4	5	6.25	6 62
3	4	-5	3.72	4 34
3	4	6	4 28	4 76
3	4	-6	13.38	13.74
3	4	7	5.34	5 88
3	4	-7	7 00	7 74
3	4	8	7 94	8 05
3	4	-8	9.49	9.58
3	4	9	3.12*	1 00
3	4	-9	12 68	12.99
3	4	-10	2.53*	1.03
3	6	0	1.56*	2 33
3	6	1	14.68	14 15
3	6	-1	10.62	10.73
3	6	2	20.42	19 66
3	6	-2	13.91	14.07
3	6	3	7.86	8.65
3	6	-3	21 31	21.72
3	6	4	5.82	5 19
3	6	-4	8.24	7.23
3	6	5	8.12	8 19
3	6	-5	9.33	9 56
3	6	6	4 47	5 17
3	6	-6	11.87	10 92
3	6	7	7.00	8.32
3	6	-7	5 28	3.53
3	6	8	7.78	7 74
3	6	-9	3.58*	3 32
3	6	-8	5 35	5.69
3	8	0	5 81	5.53
3	8	1	4.09	4 61
3	8	-1	2 67*	1 58

h	k	l	Fo	Fc
3	8	2	3.79	3.31
3	8	-2	9.37	8.60
3	8	3	4.42	3.55
3	8	-3	12 85	13.29
3	8	4	9.83	9 06
3	8	-4	14 58	14.26
3	8	5	8 57	8.52
3	8	-5	6.06	6.16
3	8	6	4.01	2.90
3	8	-6	8.15	8.17
3	8	7	2.52*	0.75
3	8	-7	7.62	7.16
3	10	0	4.10	4.37
3	10	1	4.71	5.48
3	10	-1	8.04	8 47
3	10	2	7 56	'6 34
3	10	-2	3.40*	4.75
3	10	3	4.36	2.43
3	10	-3	6.72	7.00
3	10	4	5.54	4.99
3	10	-4	6.25	5.54
3	10	-5	11.37	10.63
3	1	0	41.18	39.65
3	1	1	38.52	36.98
3	1	-1	19 51	19.56
3	1	2	4.79	4.95
3	1	-2	17.43	16.49
3	1	3	2.78*	3.48
3	1	-3	16 29	17.27
3	1	4	16.47	16.74
3	1	-4	8.66	7.70
3	1	5	11.59	10.84
3	1	-5	12 81	12.39
3	1	6	7.58	7.43
3	1	-6	11.07	11.29
3	1	7	8.88	8.89
3	1	-7	2.80*	5.17
3	1	8	7.15	7.94
3	1	-8	2.64*	2.74
3	1	9	7.63	8.63
3	1	-9	8.98	9 01
3	1	-10	4.05	3.62
3	3	0	17.19	15.73
3	3	1	10.32	9.27
3	3	-1	14.17	13.19
3	3	2	13.96	14.12
3	3	-2	7.41	6.98
3	3	3	6.39	6 42
3	3	4	15.49	15.40
3	3	-3	23.80	24.48
3	3	-4	15.29	16.52
3	3	5	11.55	10.73
3	3	-5	5.52	5.30
3	3	6	3.49	4.69
3	3	-6	3.80	3.20
3	3	7	15.48	14.55
3	3	-7	7.01	5.24
3	3	8	3 83	4.47

h	k	l	Fo	Fc
3	3	-8	4.85	5.78
3	3	9	5.82	6.55
3	3	-9	12.16	12.05
3	3	-10	7.43	6.52
3	5	0	13.81	13.85
3	5	1	16.07	15.73
3	5	-1	12.48	12 37
3	5	2	12.81	13.81
3	5	-2	9.15	8.42
3	5	3	2.93*	4.59
3	5	-3	11.11	10.78
3	5	4	13.24	13.48
3	5	-4	12.68	12.72
3	5	5	7 03	7.21
3	5	-5	4.69	4.03
3	5	6	8.37	9.43
3	5	-6	12.90	14.26
3	5	7	9.69	10.19
3	5	-7	5.01	4.10
3	5	8	4.43	4.71
3	5	-8	5.38	4.57
3	5	-9	8.54	9.33
3	7	0	10.79	10.89
3	7	1	11.30	10.78
3	7	-1	18.49	18.87
3	7	-2	7.56	6.45
3	7	3	0.0 *	0.78
3	7	-3	15.87	16.60
3	7	4	6.74	7.46
3	7	-4	21.79	22.64
3	7	5	5.32	5.51
3	7	-5	6.20	6.22
3	7	6	5.58	6.55
3	7	-6	4.69	2.55
3	7	7	7.18	7.05
3	7	-7	7.23	7.12
3	7	-8	6.56	5.94
3	9	0	4.92	3.67
3	9	1	8.39	9.20
3	9	-1	6.66	5.99
3	9	2	2.56*	1.70
3	9	-2	2.52*	3.84
3	9	3	4.15	4 29
3	9	-3	10.17	9.24
3	9	4	12.23	12.59
3	9	-4	11.16	10.70
3	9	5	5.28	4.99
3	9	-5	6.48	6.36
3	9	6	3.52*	3.14
3	9	-6	7.34	7.50
3	9	-7	9.69	10.93
3	11	0	2.59*	1.67
3	11	1	5.98	5.83
3	11	-1	2.61*	1.63
3	11	2	4.64	4.09
3	11	-2	4.95	5.74
3	11	-3	5 18	4 52
4	0	0	4.79	5.06

h	k	l	Fo	Fc
4	0	1	3.05*	2.42
4	0	-1	3.62	2.53
4	0	2	6.69	6.24
4	0	-2	7.40	7.04
4	0	3	14.20	14.44
4	0	-3	8.21	7.74
4	0	4	0.0 *	0.87
4	0	-4	0.0 *	0.45
4	0	5	16.36	16.73
4	0	-5	8.04	8.08
4	0	6	13.29	13.63
4	0	-6	10.57	11.06
4	0	7	11.65	12.19
4	0	-7	6.61	7.52
4	0	-8	4.40	3.76
4	2	0	9.88	9.81
4	2	1	9.41	9.56
4	2	-1	6.61	7.23
4	2	2	5.77	5.70
4	2	-2	0.0 *	0.59
4	2	3	0.01*	1.13
4	2	-3	11.58	11.29
4	2	4	8.91	9.08
4	2	-4	7.30	8.59
4	2	5	11.91	11.36
4	2	-5	9.64	10.37
4	2	6	10.13	11.77
4	2	-6	3.79	2.65
4	2	7	0.0 *	1.36
4	2	-7	6.39	7.00
4	2	-8	8.20	8.35
4	4	0	8.18	8.08
4	4	1	6.09	5.57
4	4	-1	12.81	11.74
4	4	2	7.15	6.67
4	4	-2	6.94	7.36
4	4	3	8.38	8.91
4	4	-3	5.94	6.16
4	4	4	4.18	4.38
4	4	-4	11.23	10.91
4	4	5	4.43	4.98
4	4	-5	3.09*	5.37
4	4	6	2.67*	2.24
4	4	-6	12.68	13.79
4	4	-7	5.04	4.50
4	6	0	9.99	9.73
4	6	1	3.36*	3.25
4	6	-1	12.09	12.89
4	6	2	10.07	9.28
4	6	-2	5.34	5.45
4	6	3	5.32	5.33
4	6	-3	13.73	14.13
4	6	4	12.19	11.99
4	6	-4	18.37	19.30
4	6	5	3.12*	2.69
4	6	-5	17.51	18.02
4	6	-6	8.72	8.03
4	8	0	2.80*	3.32
4	8	1	3.59	2.09
4	8	-1	6.74	7.37
4	8	2	4.00	3.77
4	8	-2	12.68	11.76
4	8	3	6.96	7.45
4	8	-3	13.91	15.28
4	8	-4	7.90	8.09
4	1	0	11.77	12.05
4	1	1	12.16	11.93
4	1	-1	14.65	13.96
4	1	2	12.13	12.09
4	1	-2	12.83	13.60
4	1	3	4.78	5.43
4	1	-3	14.66	14.69
4	1	4	3.77	2.73
4	1	-4	4.75	4.07
4	1	5	2.51*	2.88
4	1	-5	6.43	7.59
4	1	6	14.26	15.53
4	1	-6	5.29	4.13
4	1	7	9.37	9.08
4	1	-7	7.23	6.21
4	1	-8	2.18*	3.25
4	3	0	8.08	7.63
4	3	1	2.29*	2.30
4	3	-1	6.40	6.53
4	3	2	7.24	7.19
4	3	-2	0.0 *	1.57
4	3	3	8.35	8.65
4	3	-3	11.47	11.75
4	3	4	0.0 *	1.73
4	3	-4	4.12	3.60
4	3	5	9.84	9.81
4	3	-5	9.28	9.29
4	3	6	7.97	7.18
4	3	-6	8.29	8.40
4	3	7	3.51	1.39
4	3	-7	9.96	10.55
4	3	-8	9.40	10.16
4	5	0	13.99	13.34
4	5	1	5.44	4.27
4	5	-1	9.46	9.11
4	5	2	7.42	6.13
4	5	-2	12.49	12.58
4	5	3	7.47	7.05
4	5	-3	10.36	10.43
4	5	4	3.42*	5.23
4	5	-4	8.58	8.98
4	5	5	4.99	4.24
4	5	-5	8.53	8.71
4	5	6	8.12	7.63
4	5	-6	10.27	11.02
4	5	-7	4.65	3.73
4	7	0	6.25	5.46
4	7	1	7.52	7.49
4	7	-1	11.22	11.33
4	7	2	7.74	7.06
4	7	-2	4.74	4.90
4	7	3	6.76	5.58
4	7	-3	15.90	16.60
4	7	4	2.60*	1.53
4	7	-4	9.72	9.49
4	7	-5	6.34	5.49
4	9	0	7.68	7.78
4	9	1	3.89	3.25
4	9	-1	5.17	5.74
4	9	-2	5.19	5.63
5	0	0	2.92*	4.67
5	0	1	5.59	6.06
5	0	-1	3.32*	0.94
5	0	2	3.60	1.35
5	0	-2	6.78	6.60
5	0	3	4.62	4.06
5	0	-3	13.09	12.87
5	0	-4	4.35	3.60
5	2	0	3.23*	2.64
5	2	1	4.00	3.40
5	2	-1	4.37	3.79
5	2	2	1.95*	1.81
5	2	-2	6.83	7.95
5	2	-3	9.25	9.28
5	2	-4	7.19	6.87
5	4	0	0.0 *	1.61
5	4	1	5.23	5.25
5	4	-1	6.44	6.46
5	4	-2	5.99	7.15
5	1	0	2.77*	3.48
5	1	1	2.73*	0.58
5	1	-1	3.69*	3.53
5	1	2	3.63*	4.48
5	1	-2	4.08	3.14
5	1	3	2.53*	1.26
5	1	-3	6.34	5.97
5	1	-4	4.70	4.72
5	3	0	4.96	4.45
5	3	1	0.0 *	2.08
5	3	-1	4.66	3.15
5	3	2	4.28	3.67
5	3	-2	5.25	4.21
5	3	-3	3.13*	1.24

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STRUCTURE FACTOR TABLES FOR NAPHTHIDINE

h	k	l	Fo	Fc
2	0	0	8.68	8.25
4	0	0	26.23	26.85
6	0	0	2.02	1.59
8	0	0	10.76	10.93
9	1	0	0.44*	0.65
8	1	0	1.26*	2.16
7	1	0	1.84	1.82
6	1	0	9.49	9.58
5	1	0	9.86	10.19
4	1	0	1.76	0.99
3	1	0	9.85	10.27
2	1	0	32.81	32.79
1	1	0	39.93	43.65
2	2	0	2.20	1.58
3	2	0	12.84	12.08
4	2	0	17.71	18.35
5	2	0	4.17	3.53
6	2	0	4.84	5.20
7	2	0	1.39*	1.49
8	2	0	1.67	2.85
9	2	0	0.0 *	0.98
8	3	0	1.97	1.96
7	3	0	5.78	5.57
6	3	0	3.32	2.61
5	3	0	8.39	8.85
4	3	0	7.31	7.08
3	3	0	19.63	19.22
4	4	0	6.42	6.29
5	4	0	1.40*	0.99
6	4	0	3.94	3.79
7	4	0	1.18*	1.52
8	4	0	0.62*	1.47
7	5	0	0.0 *	0.08
6	5	0	1.80	0.76
5	5	0	0.89*	0.99
6	6	0	3.02	3.13
7	6	0	0.99*	2.15
7	6	1	0.74*	1.25
6	6	1	2.27	2.69
5	5	1	3.66	3.68
6	5	1	2.17	2.15
7	5	1	1.27	1.17
8	4	1	0.59*	0.61
7	4	1	0.82*	1.63
6	4	1	4.35	4.15
5	4	1	3.02	3.38
4	4	1	1.16*	1.59
3	3	1	8.28	7.45
4	3	1	8.82	8.40
5	3	1	5.98	6.01
6	3	1	10.44	9.97
7	3	1	3.28	3.20
8	3	1	1.93	1.88
9	2	1	0.91*	0.61
8	2	1	0.87*	0.87
7	2	1	5.36	5.37
6	2	1	11.84	13.22
5	2	1	12.17	11.26
4	2	1	17.28	16.63
3	2	1	12.49	12.04
2	2	1	21.40	21.60
1	1	1	12.27	13.51
2	1	1	17.08	16.59
3	1	1	22.04	21.44
4	1	1	16.85	15.97
5	1	1	10.58	9.25
6	1	1	4.43	4.25
7	1	1	5.43	5.61
8	1	1	2.62	2.36
9	1	1	2.82	3.25
9	0	1	1.96	2.25
8	0	1	3.50	3.37
7	0	1	0.45*	1.02
6	0	1	0.95*	0.17
5	0	1	1.79	1.03
4	0	1	1.39	1.35
3	0	1	1.62	1.40
2	0	1	2.75	0.50
1	0	1	11.34	10.01
1	0	2	53.28	53.88
2	0	2	67.70	65.89
3	0	2	0.21*	0.94
4	0	2	1.76	1.16
5	0	2	5.16	4.97
6	0	2	4.09	3.96
7	0	2	3.02	3.45
8	0	2	6.49	6.89
9	0	2	1.18*	1.58
9	1	2	1.65	1.55
8	1	2	4.92	4.42
7	1	2	4.42	4.65
6	1	2	6.63	6.53
5	1	2	5.42	5.22
4	1	2	9.78	9.19
3	1	2	8.61	7.30
2	1	2	10.34	9.56
1	1	2	71.81	78.14
2	2	2	12.03	12.95
3	2	2	20.09	20.80
4	2	2	23.74	23.54
5	2	2	11.59	11.05
6	2	2	10.69	10.92
7	2	2	5.80	5.10
8	2	2	1.72	2.10
9	2	2	0.65*	1.34
8	3	2	1.05*	0.25
7	3	2	2.50	2.49
6	3	2	6.90	7.15
5	3	2	6.26	6.88
4	3	2	7.80	7.60
3	3	2	16.25	15.90
4	4	2	5.27	5.13
5	4	2	3.99	3.66
6	4	2	3.30	3.12
7	4	2	1.53	1.56
8	4	2	0.35*	1.38
7	5	2	2.32	2.13
6	5	2	4.60	4.26
5	5	2	1.77	2.81
6	6	2	3.00	4.30
7	6	2	0.0 *	1.72
7	6	3	0.0 *	1.95
6	6	3	1.13*	2.22
5	5	3	5.34	5.61
6	5	3	1.97	1.43
7	5	3	1.01*	0.42
8	4	3	0.86*	1.25
7	4	3	1.46	1.42
6	4	3	6.22	6.31
5	4	3	4.54	4.55
4	4	3	0.91*	1.90
3	3	3	4.54	4.88
4	3	3	10.22	9.50
5	3	3	3.76	4.15
6	3	3	7.67	8.01
7	3	3	1.13*	1.64
8	3	3	0.0 *	0.43
9	2	3	1.01*	2.46
8	2	3	0.0 *	0.73
7	2	3	5.79	5.72
6	2	3	6.46	6.67
5	2	3	13.37	13.52
4	2	3	5.41	4.97
3	2	3	29.81	29.99
2	2	3	2.75	2.92
1	1	3	77.77	86.30
2	1	3	25.12	25.56
3	1	3	9.01	8.52
4	1	3	20.55	19.59
5	1	3	6.10	6.66
6	1	3	2.50	1.74
7	1	3	8.60	8.61
8	1	3	3.10	2.65
9	1	3	1.22*	1.11
9	0	3	3.09	3.43
8	0	3	0.0 *	1.00
7	0	3	4.22	4.25
6	0	3	4.54	4.05
5	0	3	2.98	3.38
4	0	3	0.94*	0.33
3	0	3	1.34	0.35
2	0	3	52.38	48.93
1	0	3	64.78	67.31
0	0	4	11.06	9.89
1	0	4	6.80	5.75
2	0	4	23.57	23.73
3	0	4	10.99	11.30
4	0	4	12.41	12.62
5	0	4	2.28	2.46
6	0	4	3.25	3.80
7	0	4	11.43	11.22
8	0	4	1.18*	1.54
9	0	4	1.20*	1.03
9	1	4	0.90*	1.09
8	1	4	3.34	3.10
7	1	4	9.58	9.62
6	1	4	6.46	6.17
5	1	4	6.84	6.37
4	1	4	10.21	9.11
3	1	4	14.85	14.32
2	1	4	25.81	26.19
1	1	4	37.57	42.33
2	2	4	14.65	15.18
3	2	4	13.17	12.81
4	2	4	4.42	4.73
5	2	4	4.53	4.70
6	2	4	6.21	6.28
7	2	4	3.54	2.63
8	2	4	1.09*	1.57
8	3	4	0.67*	2.20
7	3	4	1.43	0.72
6	3	4	5.79	5.84
5	3	4	11.45	11.70
4	3	4	1.26*	0.62
3	3	4	11.04	9.80
4	4	4	2.04	0.41
5	4	4	6.60	6.89
6	4	4	3.08	3.10
7	4	4	2.67	3.06
8	4	4	1.12*	1.31
7	5	4	0.58*	1.28
6	5	4	3.52	3.75
5	5	4	5.47	5.93
6	6	4	0.0 *	1.46
6	6	5	0.0 *	0.61
5	5	5	2.70	2.18
6	5	5	4.74	4.56
7	5	5	0.91*	1.46
8	4	5	0.0 *	1.43
7	4	5	1.16*	0.63
6	4	5	4.60	4.90
5	4	5	8.15	8.14
4	4	5	1.91	1.48
3	3	5	2.67	3.42
4	3	5	1.99	1.16
5	3	5	2.38	2.29
6	3	5	4.98	5.45
7	3	5	1.16*	0.66
8	3	5	1.11*	1.22
8	2	5	0.70*	0.42
7	2	5	0.52*	1.57
6	2	5	4.78	4.41
5	2	5	4.99	4.62
4	2	5	10.34	10.03
3	2	5	19.13	18.61
2	2	5	0.0 *	2.19
1	1	5	101.87	111.25
2	1	5	57.99	59.69
3	1	5	13.22	13.12
4	1	5	9.41	9.47
5	1	5	1.11*	1.71
6	1	5	9.17	9.06
7	1	5	8.99	9.06
8	1	5	1.37*	2.39
9	1	5	0.0 *	2.14
9	0	5	2.28	2.28
8	0	5	3.46	3.07
7	0	5	10.87	9.89
6	0	5	0.64*	0.23
5	0	5	4.59	4.26
4	0	5	10.05	10.09
3	0	5	18.59	18.68
2	0	5	40.47	40.63
1	0	5	86.57	94.55
1	0	6	21.08	22.23
2	0	6	23.66	23.60
3	0	6	34.59	33.67
4	0	6	4.89	5.00
5	0	6	7.71	7.13
6	0	6	1.40	0.99
7	0	6	0.63*	0.69
8	0	6	3.74	3.90
9	0	6	0.90*	1.17
9	1	6	0.0 *	0.72
8	1	6	1.33*	2.15
7	1	6	7.39	7.58
6	1	6	7.13	6.87
5	1	6	5.58	6.27
4	1	6	8.38	6.81
3	1	6	30.06	29.26
2	1	6	12.15	12.79
1	1	6	2.46	3.66
2	2	6	3.98	4.77
3	2	6	16.68	16.49
4	2	6	0.45*	0.80
5	2	6	9.41	8.72
6	2	6	4.57	4.40
7	2	6	1.64	1.69
8	2	6	0.0 *	0.49
8	3	6	0.95*	1.91
7	3	6	1.55	1.41
6	3	6	6.69	6.84
5	3	6	2.38	1.23
4	3	6	4.63	4.60
3	3	6	4.84	5.18
4	4	6	0.58*	1.34
5	4	6	11.40	11.24
6	4	6	3.71	4.08
7	4	6	0.0 *	0.98
8	4	6	0.0 *	1.09
7	5	6	0.0 *	0.56
6	5	6	1.40	1.72
5	5	6	6.42	6.50
6	6	6	1.16*	1.44
6	6	7	1.01*	0.80
5	5	7	5.06	6.22
6	5	7	1.09*	1.84
7	5	7	0.0 *	1.01
8	4	7	0.0 *	2.01
7	4	7	2.11	2.33

h	k	l	Fo	Fc
6	4	7	4.29	3.28
5	4	7	12.40	12.17
4	4	7	0.0 *	0.35
3	3	7	15.95	15.44
4	3	7	2.69	3.70
5	3	7	5.59	5.93
6	3	7	2.41	1.59
7	3	7	3.40	2.90
8	3	7	0.0 *	0.45
8	2	7	0.60*	0.83
7	2	7	1.66	2.05
6	2	7	3.02	2.80
5	2	7	2.55	2.49
4	2	7	6.78	6.38
3	2	7	13.92	13.79
2	2	7	31.76	33.67
1	1	7	15.44	15.53
2	1	7	29.70	30.47
3	1	7	22.86	22.37
4	1	7	12.64	11.51
5	1	7	4.09	4.76
6	1	7	10.79	10.99
7	1	7	4.01	3.89
8	1	7	3.53	2.60
9	0	7	0.26*	1.05
8	0	7	0.0 *	0.11
7	0	7	13.57	12.71
6	0	7	11.61	10.84
5	0	7	1.17*	2.23
4	0	7	9.05	8.92
3	0	7	10.56	10.52
2	0	7	14.05	13.71
1	0	7	3.22	2.35
0	0	8	22.62	21.90
1	0	8	0.86*	0.78
2	0	8	11.22	11.31
3	0	8	17.84	17.27
4	0	8	2.78	2.85
5	0	8	2.12	1.07
6	0	8	1.39*	0.49
7	0	8	4.69	4.14
8	0	8	4.88	5.73
8	1	8	0.65*	0.28
7	1	8	4.36	3.69
6	1	8	4.72	4.89
5	1	8	7.06	6.78
4	1	8	8.56	8.65
3	1	8	28.65	27.83
2	1	8	8.97	7.80
1	1	8	51.57	53.39
2	2	8	17.68	17.63
3	2	8	24.76	24.79
4	2	8	9.49	8.93
5	2	8	2.55	3.03
6	2	8	5.79	5.44
7	2	8	1.32*	1.24
8	2	8	0.74*	1.19
8	3	8	0.83*	1.06
7	3	8	0.0 *	0.55
6	3	8	2.81	2.32
5	3	8	6.20	6.92
4	3	8	3.74	1.05
3	4	8	3.01	2.92
4	4	8	1.25	0.96
5	4	8	7.28	7.95
6	4	8	5.83	6.15
7	4	8	1.43	1.11
7	5	8	0.57*	1.33
6	5	8	0.0 *	0.51
5	5	8	6.07	6.71
6	6	8	0.0 *	1.02
6	6	9	0.73*	0.40
5	5	9	6.45	7.62
6	5	9	1.08*	1.33
7	5	9	0.0 *	0.75
7	4	9	0.67*	0.08
6	4	9	3.11	3.05
5	4	9	1.53	2.36
4	4	9	6.06	6.97
3	3	9	3.68	3.57
4	3	9	4.16	4.91
5	3	9	5.68	5.56
6	3	9	4.23	3.85
7	3	9	1.30	1.71
8	3	9	0.0 *	0.81
8	2	9	0.94*	0.80
7	2	9	2.29	1.30
6	2	9	3.61	3.25
5	2	9	7.20	7.50
4	2	9	6.91	6.81
3	2	9	13.31	12.65
2	2	9	3.96	4.53
1	1	9	8.16	7.64
2	1	9	27.93	27.23
3	1	9	13.50	13.58
4	1	9	4.95	5.05
5	1	9	4.90	4.03
6	1	9	9.06	8.89
7	1	9	5.08	4.46
8	1	9	2.20	2.15
8	0	9	4.99	5.35
7	0	9	9.13	8.95
6	0	9	11.20	11.15
5	0	9	6.81	6.70
4	0	9	11.11	10.41
3	0	9	6.14	5.57
2	0	9	3.52	3.97
1	0	9	10.53	10.74
1	0	10	17.29	18.08
2	0	10	13.79	13.06
3	0	10	3.10	3.93
4	0	10	13.11	12.63
5	0	10	5.34	5.60
6	0	10	1.22*	1.78
7	0	10	3.71	4.10
8	0	10	3.28	2.50
8	1	10	2.51	3.06
7	1	10	2.82	2.44
6	1	10	5.17	4.71
5	1	10	4.49	4.93
4	1	10	14.46	14.37
3	1	10	20.52	20.99
2	1	10	14.02	10.93
1	1	10	8.33	8.03
2	2	10	30.87	30.31
3	2	10	9.00	9.03
4	2	10	5.49	5.32
5	2	10	10.00	10.43
6	2	10	4.46	4.72
7	2	10	1.16*	1.76
8	2	10	1.45	1.91
8	3	10	0.93*	2.52
7	3	10	0.0 *	1.04
6	3	10	5.14	4.12
5	3	10	3.58	2.74
4	3	10	3.49	4.27
3	3	10	3.18	2.56
4	4	10	0.0 *	1.36
5	4	10	7.47	8.30
6	4	10	3.32	3.01
7	4	10	0.76*	1.86
7	5	10	0.27*	0.64
6	5	10	1.85	2.27
5	5	10	6.45	7.90
6	6	10	0.28*	0.86
6	6	11	0.0 *	0.11
5	5	11	4.01	5.27
6	5	11	0.0 *	2.32
7	4	11	1.02*	1.86
6	4	11	4.17	4.13
5	4	11	4.28	3.94
4	4	11	2.41	2.88
3	3	11	4.97	4.76
4	3	11	6.10	6.57
5	3	11	5.00	4.90
6	3	11	2.79	3.03
7	3	11	1.20*	1.13
8	3	11	0.0 *	1.28
8	2	11	0.73*	1.81
7	2	11	2.31	2.67
6	2	11	1.40	1.52
5	2	11	4.23	4.52
4	2	11	6.88	6.13
3	2	11	5.77	5.71
2	2	11	12.81	12.76
1	1	11	11.41	10.95
2	1	11	12.71	12.71
3	1	11	14.10	13.40
4	1	11	17.58	17.81
5	1	11	5.78	5.57
6	1	11	5.04	5.13
7	1	11	3.49	3.13
8	1	11	1.82	3.28
8	0	11	0.40*	1.33
7	0	11	1.00*	0.82
6	0	11	2.11	2.51
5	0	11	0.54*	2.42
4	0	11	2.67	3.33
3	0	11	14.98	14.81
2	0	11	11.72	12.43
1	0	11	22.90	22.50
0	0	12	1.42	1.44
1	0	12	2.97	2.80
2	0	12	11.52	11.35
3	0	12	4.82	3.80
4	0	12	1.29*	2.30
5	0	12	4.57	3.58
6	0	12	0.96*	1.67
7	0	12	2.97	3.04
8	0	12	0.0 *	0.45
8	1	12	2.12	2.49
7	1	12	4.92	4.25
6	1	12	4.87	4.15
5	1	12	5.89	4.98
4	1	12	11.72	10.93
3	1	12	4.66	4.03
2	1	12	0.68*	1.07
1	1	12	7.97	7.95
2	2	12	3.45	3.00
3	2	12	18.45	18.71
4	2	12	4.76	4.03
5	2	12	7.39	7.78
6	2	12	5.68	5.32
7	2	12	2.59	2.56
8	2	12	0.0 *	1.93
7	3	12	0.0 *	1.84
6	3	12	3.47	3.58
5	3	12	5.70	5.67
4	3	12	6.56	8.12
3	3	12	5.40	5.04
4	4	12	4.98	5.95
5	4	12	4.88	4.86
6	4	12	1.22*	2.12
7	4	12	0.0 *	2.33
6	5	12	1.33*	1.07
5	5	12	3.40	3.43
5	5	13	3.62	5.12
6	5	13	0.0 *	1.36
7	4	13	0.0 *	2.27
6	4	13	1.28*	2.94
5	4	13	0.94*	2.33
4	4	13	1.17*	0.69
3	3	13	2.32	2.15
4	3	13	8.31	8.80
5	3	13	2.76	2.28
6	3	13	3.23	3.78
7	3	13	1.48	1.72
8	2	13	0.0 *	2.59
7	2	13	1.11*	0.86
6	2	13	2.52	3.05
5	2	13	8.23	7.42
4	2	13	13.14	12.10
3	2	13	11.38	11.58
2	2	13	12.78	12.63
1	1	13	15.62	15.55
2	1	13	4.38	3.97
3	1	13	5.25	5.14
4	1	13	1.99	1.70
5	1	13	5.58	5.68
6	1	13	0.72*	0.20
7	1	13	1.87	2.17
8	1	13	0.0 *	0.90
8	0	13	0.56*	0.15
7	0	13	0.0 *	3.23
6	0	13	1.08*	0.04
5	0	13	4.82	3.61
4	0	13	6.00	5.44
3	0	13	3.63	2.28
2	0	13	13.90	12.41
1	0	13	14.82	14.43
1	0	14	16.38	15.64
2	0	14	5.26	5.05
3	0	14	1.32	1.10
4	0	14	7.48	8.23
5	0	14	2.76	2.12
6	0	14	2.71	1.64
7	0	14	2.15	2.39
8	0	14	1.34	2.39
8	1	14	0.52*	2.17
7	1	14	1.18*	1.43
6	1	14	2.91	2.43
5	1	14	4.24	4.20
4	1	14	3.95	3.81
3	1	14	10.10	9.79
2	1	14	3.89	2.90
1	1	14	4.33	4.16
2	2	14	8.32	8.96
3	2	14	5.57	5.40
4	2	14	13.13	12.81
5	2	14	11.43	10.35
6	2	14	3.32	3.15
7	2	14	0.0 *	0.42
7	3	14	0.0 *	1.07
6	3	14	1.96	1.82
5	3	14	2.94	3.22
4	3	14	3.13	3.55
3	3	14	0.59*	0.13
4	4	14	0.0 *	0.54
5	4	14	1.34	1.65
6	4	14	0.82*	1.60
7	4	14	0.0 *	1.28
6	5	14	0.0 *	2.36
5	5	14	0.46*	0.54
5	5	15	1.14*	0.81
6	5	15	0.62*	0.33
6	4	15	0.0 *	1.99
5	4	15	1.17*	0.71
4	4	15	1.96	1.30
3	3	15	4.70	4.33
4	3	15	4.93	3.98

h	k	l	Fo	Fc
5	3	15	2 88	3 37
6	3	15	3 40	4 84
7	3	15	0 20*	1 03
7	2	15	1 44	2 21
6	2	15	1 38*	0 73
5	2	15	2 28	1 41
4	2	15	14 98	16 03
3	2	15	2 74	3 51
2	2	15	1 83	1 16
1	1	15	5 33	4 84
2	1	15	0 0 *	0 41
3	1	15	5 14	5 26
4	1	15	1 94	1 55
5	1	15	2 74	2 71
6	1	15	0 0 *	1 26
7	1	15	0 45*	1 13
7	0	15	1 20*	1 73
6	0	15	1 91	1 10
5	0	15	7 33	6 57
4	0	15	8 59	8 04
3	0	15	1 26*	2 64
2	0	15	3 09	3 44
1	0	15	6 19	5 37
0	0	16	39 77	38 82
1	0	16	3 71	3 67
2	0	16	0 0 *	0 36
3	0	16	2 37	1 26
4	0	16	3 19	2 63
5	0	16	0 85*	1 91
6	0	16	1 27*	0 40
7	0	16	0 0 *	0 28
7	1	16	0 0 *	0 65
6	1	16	1 18*	1 32
5	1	16	4 88	4 53
4	1	16	10 50	9 49
3	1	16	2 33	2 33
2	1	16	1 01*	0 73
1	1	16	4 55	3 79
2	2	16	5 08	5 07
3	2	16	4 80	4 89
4	2	16	8 51	8 78
5	2	16	3 35	3 80
6	2	16	0 69*	1 05
7	2	16	0 82*	0 42
7	3	16	0 0 *	2 20
6	3	16	1 21*	0 98
5	3	16	5 12	5 57
4	3	16	4 75	4 61
3	3	16	5 56	5 36
4	4	16	0 0 *	1 55
5	4	16	0 67*	1 23
6	4	16	0 0 *	3 56
5	5	16	0 0 *	2 47
5	5	17	0 0 *	0 55
6	4	17	0 0 *	0 72
5	4	17	0 0 *	1 79
4	4	17	0 0 *	1 20
3	3	17	12 36	13 02
4	3	17	1 41	1 40
5	3	17	1 73	1 66
6	3	17	1 53*	2 34
7	2	17	0 0 *	1 24
6	2	17	2 80	3 40
5	2	17	1 86	1 93
4	2	17	6 43	4 94
3	2	17	6 25	6 94
2	2	17	2 74	2 43
1	1	17	0 0 *	0 31
2	1	17	1 26	1 31
3	1	17	4 28	4 22
4	1	17	2 99	2 14
5	1	17	1 28*	1 11
6	1	17	1 10*	1 14
7	1	17	0 35*	1 11
7	0	17	1 08*	1 06
6	0	17	0 90*	0 62
5	0	17	1 38*	1 05
4	0	17	9 05	7 36
3	0	17	4 33	4 01
2	0	17	0 57*	0 31
1	0	17	3 24	3 47
1	0	18	3 64	3 29
2	0	18	5 52	5 47
3	0	18	0 0 *	0 76
4	0	18	6 76	5 80
5	0	18	0 0 *	0 78
7	0	18	0 0 *	0 23
7	1	18	0 76*	0 79
6	1	18	0 0 *	1 02
5	1	18	2 09	2 32
4	1	18	4 07	4 11
3	1	18	6 84	6 92
2	1	18	3 50	3 73
1	1	18	8 80	8 03
2	2	18	2 24	2 32
3	2	18	3 83	3 24
4	2	18	1 90	2 51
5	2	18	4 95	5 23
6	2	18	1 32*	2 33
6	3	18	0 0 *	3 30
5	3	18	2 10	3 22
4	3	18	2 28	2 47
3	3	18	9 68	9 51
4	4	18	0 76*	0 15
5	4	18	0 88*	2 52
5	5	18	0 0 *	0 16
5	4	19	0 0 *	0 56
4	4	19	0 81*	1 26
3	3	19	5 30	5 28
4	3	19	0 0 *	0 85
5	3	19	1 46*	2 54
6	3	19	0 0 *	1 32
6	2	19	0 0 *	2 07
5	2	19	1 31	1 69
4	2	19	1 27*	1 09
3	2	19	9 78	9 82
2	2	19	3 58	3 82
1	1	19	5 98	6 00
2	1	19	0 83*	1 15
3	1	19	7 26	7 35
4	1	19	1 45*	1 46
5	1	19	1 65	1 31
6	1	19	0 0 *	0 77
6	0	19	1 72	1 81
5	0	19	0 78*	0 41
4	0	19	0 51*	0 08
3	0	19	1 17*	2 60
2	0	19	2 22	2 34
1	0	19	0 65*	0 68
0	0	20	2 98	3 50
1	0	20	3 22	2 85
2	0	20	3 00	3 26
3	0	20	3 22	4 18
4	0	20	0 95*	1 72
5	0	20	1 40*	1 68
6	0	20	1 01*	0 39
6	1	20	1 08*	0 53
5	1	20	1 58	1 93
4	1	20	5 54	5 51
3	1	20	1 33	1 80
2	1	20	4 30	4 89
1	1	20	1 76	1 66
2	2	20	0 0 *	0 51
3	2	20	7 95	8 17
4	2	20	2 14	1 87
5	2	20	1 50	1 86
6	2	20	0 0 *	0 90
5	3	20	0 37*	2 88
4	3	20	1 51*	1 91
3	3	20	1 42*	2 70
4	4	20	0 97*	0 91
5	4	20	0 0 *	1 36
4	4	21	0 83*	0 85
3	3	21	0 85*	0 07
4	3	21	1 23*	2 64
5	3	21	0 0 *	1 80
5	2	21	1 45*	2 16
4	2	21	2 11	2 45
3	2	21	3 72	3 20
2	2	21	2 78	3 21
1	1	21	4 79	4 59
2	1	21	5 16	4 95
3	1	21	4 35	4 08
4	1	21	2 95	2 72
5	1	21	1 32	1 24
6	1	21	0 57*	0 81
6	0	21	0 20*	0 05
5	0	21	0 0 *	0 14
4	0	21	2 06	2 23
3	0	21	4 21	4 65
2	0	21	3 49	3 20
1	0	21	4 09	4 09
1	0	22	1 02*	0 89
2	0	22	1 33	1 66
3	0	22	3 28	2 85
4	0	22	0 0 *	0 31
5	0	22	1 03*	0 88
5	1	22	0 0 *	0 69
4	1	22	3 62	4 25
3	1	22	2 79	3 26
2	1	22	3 06	2 95
1	1	22	2 30	2 81
2	2	22	2 84	2 90
3	2	22	3 20	3 50
4	2	22	2 79	3 85
5	2	22	0 0 *	0 28
5	3	22	0 89*	0 54
4	3	22	0 0 *	0 87
3	3	22	0 0 *	0 10
4	4	22	0 43*	0 25
3	3	23	0 99*	1 35
4	3	23	0 0 *	1 00
5	2	23	0 0 *	0 85
4	2	23	0 0 *	1 52
3	2	23	2 05	2 64
2	2	23	3 65	3 85
1	1	23	1 89	1 52
2	1	23	2 84	2 83
3	1	23	4 09	4 76
4	1	23	1 31*	1 47
5	1	23	0 0 *	0 31
5	0	23	1 10*	0 97
4	0	23	3 16	3 39
3	0	23	2 48	2 76
2	0	23	0 0 *	0 25
1	0	23	1 16*	1 39
0	0	24	4 15	4 41
1	0	24	0 43*	0 31
2	0	24	0 0 *	0 30
3	0	24	2 78	2 69
4	0	24	2 94	3 99
4	1	24	1 13*	2 48
3	1	24	1 47*	2 53
2	1	24	1 05*	1 97
1	1	24	0 35*	1 15
2	2	24	0 53*	1 48
3	2	24	1 01*	3 51
4	2	24	0 0 *	1 35
3	3	24	0 0 *	0 10
3	3	25	0 0 *	1 73
3	2	25	0 36*	1 93
2	2	25	1 39*	1 94
1	1	25	1 00*	1 13
2	1	25	1 02*	1 65
3	1	25	1 85	3 02
4	1	25	0 27*	1 08
4	0	25	0 0 *	1 78
3	0	25	3 70	4 55
2	0	25	3 80	4 57
1	0	25	1 09*	0 87
1	0	26	0 0 *	0 48
2	0	26	0 65*	1 58
3	0	26	0 57*	1 90
3	1	26	0 0 *	0 96
2	1	26	0 43*	1 24
1	1	26	0 0 *	0 34
2	2	26	0 0 *	3 30
3	2	26	0 97*	0 52
2	2	27	0 0 *	3 34
1	1	27	0 0 *	0 07
2	1	27	0 0 *	1 81
2	0	27	0 49*	0 57
1	0	27	1 72	2 44
0	0	28	0 0 *	0 46
1	0	28	0 68*	0 09
1	1	28	0 61*	1 10

826 reflections

STRUCTURE FACTOR TABLES FOR

1,1'-BINAPHTHYL

h	k	l	Fo	Fc
2	0	0	18.73	19.11
4	0	0	15.37	15.40
6	0	0	11.51	11.94
8	0	0	8.24	8.50
8	1	0	6.41	6.50
6	1	0	10.33	10.09
5	1	0	19.19	19.03
4	1	0	5.58	5.76
3	1	0	2.43	2.41
2	1	0	36.09	37.58
1	1	0	22.11	21.73
2	2	0	19.20	19.59
3	2	0	19.21	18.50
4	2	0	16.91	16.71
5	2	0	5.55	5.29
6	2	0	11.08	11.18
7	2	0	2.43	2.33
6	3	0	10.76	10.68
4	3	0	4.24	4.26
3	3	0	5.17	5.34
4	4	0	4.85	4.49
5	4	0	2.15	2.26
6	6	0	4.44	4.24
6	6	1	9.82	9.01
3	3	1	8.76	8.59
4	3	1	4.84	4.60
5	3	1	8.53	8.53
6	3	1	6.63	6.45
7	3	1	3.55	3.78
8	2	1	4.01	4.15
6	2	1	13.55	13.60
5	2	1	7.53	7.79
4	2	1	17.97	17.93
3	2	1	15.52	14.91
1	1	1	13.87	14.12
2	1	1	14.60	14.70
3	1	1	23.56	23.78
4	1	1	8.35	8.30
5	1	1	6.82	6.51
6	1	1	11.49	11.36
7	1	1	6.53	6.37
8	1	1	4.66	4.36
6	0	1	3.97	4.11
5	0	1	2.69	2.90
4	0	1	8.97	8.92
3	0	1	4.86	4.81
2	0	1	20.23	21.32
1	0	1	16.49	17.06
1	0	2	65.82	70.24
2	0	2	45.01	46.64
3	0	2	2.72	2.43
4	0	2	4.08	4.18
5	0	2	4.47	4.34
6	0	2	3.90	3.90
7	0	2	9.16	9.21
8	0	2	6.39	6.47
8	1	2	3.13	2.98
6	1	2	10.41	10.14
5	1	2	13.84	13.92
4	1	2	19.74	19.52
3	1	2	7.92	8.00
2	1	2	27.48	27.78
1	1	2	73.77	76.69
2	2	2	4.57	5.14
3	2	2	23.54	23.08
4	2	2	9.00	9.17
5	2	2	17.43	17.71
6	2	2	3.68	3.96
7	2	2	5.98	5.76
7	3	2	3.63	3.57
6	3	2	5.11	5.18
5	3	2	15.63	15.68
4	4	2	6.27	5.79
7	4	2	3.38	3.26
6	6	2	4.82	4.61
6	5	3	4.48	4.01
5	4	3	5.68	5.90
4	3	3	6.89	6.88
5	3	3	9.45	9.60
6	3	3	3.81	3.93
8	2	3	4.02	3.68
6	2	3	8.05	8.25
5	2	3	12.96	13.20
4	2	3	20.03	19.85
3	2	3	21.77	20.49
2	2	3	22.73	22.09
1	1	3	92.71	97.31
2	1	3	13.96	14.20
3	1	3	12.80	12.40
4	1	3	21.29	20.68
5	1	3	7.16	7.40
6	1	3	12.87	12.42
7	1	3	3.49	3.50
8	0	3	4.39	4.27
7	0	3	4.01	4.11
6	0	3	11.54	11.58
5	0	3	19.19	18.85
4	0	3	39.71	40.95
3	0	3	64.68	65.46
2	0	3	19.32	20.35
1	0	3	15.56	15.95
0	0	4	26.18	25.44
1	0	4	12.68	12.77
2	0	4	4.59	4.76
3	0	4	9.32	8.87
4	0	4	12.94	13.30
5	0	4	4.38	4.43
7	0	4	9.36	9.30
8	1	4	7.49	7.58
7	1	4	3.32	3.63
6	1	4	3.84	3.99
5	1	4	8.07	8.21
4	1	4	26.63	27.07
3	1	4	25.06	26.12
2	1	4	22.07	21.54
1	1	4	11.19	10.65
2	2	4	9.19	9.21
3	2	4	10.56	10.66
4	2	4	4.46	4.35
5	2	4	2.57	2.84
6	2	4	6.51	6.36
6	3	4	11.39	11.37
5	3	4	7.17	6.79
5	4	4	4.41	4.30
7	4	4	6.03	6.17
6	5	4	4.32	4.43
5	5	4	9.41	9.59
5	5	5	7.24	7.09
6	4	5	4.37	5.08
5	4	5	10.67	10.64
4	4	5	3.32	3.63
4	3	5	12.16	12.26
5	3	5	4.52	4.73
6	3	5	5.59	5.61
7	2	5	7.22	7.25
5	2	5	7.03	6.96
4	2	5	5.66	4.76
3	2	5	9.89	9.65
2	2	5	110.66	116.68
1	1	5	60.43	61.67
2	1	5	10.47	10.39
3	1	5	12.21	12.13
4	1	5	9.30	9.44
6	1	5	20.97	20.54
7	0	5	2.42	2.44
5	0	5	9.31	9.05
4	0	5	28.11	27.73
3	0	5	39.78	41.12
2	0	5	73.12	77.93
1	0	5	6.15	6.45
0	0	6	14.16	14.11
2	0	6	21.53	21.68
3	0	6	15.62	15.46
4	0	6	9.52	9.16
5	0	6	5.00	4.95
6	0	6	13.66	13.45
7	0	6	4.09	4.09
8	1	6	7.76	7.58
7	1	6	6.36	6.31
6	1	6	5.28	5.65
5	1	6	8.99	8.93
4	1	6	34.71	34.94
3	1	6	13.01	13.21
2	1	6	2.40	2.44
1	1	6	11.13	10.87
2	2	6	10.63	10.19
3	2	6	5.25	5.28
5	2	6	3.24	3.24
7	3	6	3.23	3.20
6	3	6	4.05	3.78
5	3	6	8.19	7.87
4	3	6	4.96	4.80
3	3	6	11.62	11.64
4	4	6	9.90	10.09
6	4	6	6.19	6.03
6	5	6	5.80	5.48
5	5	6	8.61	8.00
5	5	7	12.69	13.39
6	4	7	4.01	3.79
4	4	7	13.89	13.73
3	3	7	5.83	5.65
4	3	7	7.62	7.44
5	3	7	12.59	12.36
6	3	7	4.44	3.92
7	2	7	4.80	4.88
6	2	7	2.18	2.19
5	2	7	6.39	5.96
4	2	7	2.85	2.78
3	2	7	26.77	26.92
2	2	7	36.51	36.79
1	1	7	24.53	25.19
2	1	7	23.57	24.22
3	1	7	26.48	25.82
4	1	7	5.26	5.13
5	1	7	5.45	5.33
6	1	7	10.35	10.61
7	1	7	4.89	4.48
6	0	7	23.67	23.09
4	0	7	11.28	11.40
3	0	7	4.58	4.09
2	0	7	24.20	24.24
1	0	7	6.87	7.01
0	0	8	63.17	66.40
2	0	8	19.34	19.57
3	0	8	5.52	5.49
5	0	8	9.61	9.03
6	0	8	15.37	15.67
7	0	8	4.12	3.97
8	1	8	4.35	4.37
7	1	8	4.07	3.89
6	1	8	5.43	5.61
5	1	8	5.08	5.36
4	1	8	4.49	4.56
3	1	8	37.59	37.65
2	1	8	19.83	20.38
1	1	8	45.37	46.44
2	2	8	36.71	36.40
3	2	8	23.76	23.24
4	2	8	8.64	7.99
5	2	8	4.66	4.46
7	3	8	2.81	2.86
5	3	8	10.52	10.02
3	3	8	5.88	5.43
4	4	8	22.03	22.73
5	4	8	5.50	5.64
6	4	8	4.23	4.63
7	4	8	2.54	2.44
5	5	8	6.34	6.51
5	5	9	9.07	9.52
6	4	9	4.56	4.53
5	4	9	5.80	5.70
4	4	9	16.53	15.52
7	2	9	3.93	3.93
6	2	9	4.45	4.34
5	2	9	2.70	3.00
4	2	9	7.49	7.04
3	2	9	10.76	10.98
2	2	9	7.98	8.40
1	1	9	8.20	7.85
2	1	9	36.10	36.04
3	1	9	8.38	9.05
4	1	9	10.10	10.03
5	1	9	6.08	6.32
6	1	9	7.06	7.15
7	1	9	8.62	8.66
8	0	9	4.65	4.66
6	0	9	14.94	15.61
4	0	9	6.00	5.75
3	0	9	11.00	10.84
2	0	9	6.28	6.27
1	0	9	5.59	5.73
1	0	10	6.13	6.25
2	0	10	29.95	30.76
3	0	10	6.87	7.46
4	0	10	11.82	11.70
5	0	10	12.37	12.43
7	0	10	4.12	4.53
8	0	10	3.83	3.60
7	1	10	9.29	9.45
6	1	10	4.90	4.95
5	1	10	7.19	7.19
4	1	10	10.06	10.25
3	1	10	20.85	20.96
2	1	10	7.11	7.22
1	1	10	14.86	14.64
2	2	10	39.67	39.76
3	2	10	15.31	16.06
4	2	10	4.81	5.16
5	2	10	7.32	6.91
6	3	10	4.11	4.03
5	3	10	6.11	6.71
4	3	10	4.69	4.82
3	3	10	7.50	7.29
4	4	10	6.76	6.76
5	4	10	16.58	16.56
6	4	10	6.05	6.53
6	4	11	3.72	3.59
5	4	11	9.84	9.45
4	4	11	7.69	7.79
4	3	11	8.52	8.31
5	3	11	9.62	9.46
7	2	11	4.32	4.00
6	2	11	4.06	4.07
5	2	11	8.84	8.86
4	2	11	6.09	5.86
3	2	11	10.42	10.55
2	2	11	13.79	13.03
1	1	11	10.85	10.86
2	1	11	1.91	2.00
3	1	11	14.71	14.64

h	k	l	Fo	Fc
4	1	11	16.57	16.74
5	1	11	9.38	9.79
6	1	11	8.68	8.61
4	0	11	12.55	12.29
3	0	11	20.80	20.29
2	0	11	22.65	22.92
1	0	11	14.23	13.47
0	0	12	14.19	14.08
1	0	12	15.37	15.21
2	0	12	16.09	16.35
3	0	12	17.19	16.65
5	0	12	5.64	5.18
7	0	12	6.23	6.52
7	1	12	6.45	5.86
5	1	12	5.31	5.01
4	1	12	12.86	12.82
3	1	12	11.28	11.07
2	1	12	5.28	5.27
1	1	12	5.19	5.17
2	2	12	3.68	3.42
3	2	12	17.98	17.93
4	2	12	9.20	9.11
5	2	12	10.09	10.09
6	2	12	3.55	3.37
7	2	12	5.19	5.30
6	3	12	6.37	6.03
5	3	12	11.03	10.48
4	3	12	14.75	14.81
3	3	12	12.06	12.15
5	4	12	6.48	6.48
6	4	12	3.62	3.26
6	4	13	2.94	3.36
5	4	13	6.41	7.08
4	4	13	5.32	5.45
4	3	13	17.79	18.28
5	3	13	4.32	4.50
6	3	13	7.70	7.61
6	2	13	3.86	4.10
5	2	13	8.65	9.15
4	2	13	9.88	9.90
3	2	13	17.12	17.05
2	2	13	26.11	26.60
1	1	13	30.88	30.86
2	1	13	4.01	3.65
3	1	13	5.31	5.37
4	1	13	12.53	12.59
5	1	13	8.52	8.48
6	1	13	6.03	6.42
6	0	13	4.75	5.14
5	0	13	6.74	6.94
4	0	13	4.84	4.91
3	0	13	7.19	6.75
2	0	13	9.51	9.50
1	0	13	14.78	14.34
1	0	14	27.15	26.36
2	0	14	10.88	10.38
4	0	14	6.19	6.07
7	0	14	3.81	3.85
7	1	14	2.64	2.51
6	1	14	5.66	5.66
5	1	14	9.11	9.36
3	1	14	14.12	14.07
2	1	14	3.53	3.82
2	2	14	18.47	18.27
3	2	14	2.94	2.90
4	2	14	15.14	15.34
5	2	14	9.02	9.05
7	3	14	5.21	4.87
5	3	14	4.25	4.49
4	3	14	10.83	10.67
4	4	14	4.37	4.51
5	4	14	4.04	4.43
6	4	14	4.62	4.09
6	4	15	2.75	2.48
3	3	15	2.09	2.02
4	3	15	6.82	6.85
5	3	15	10.66	11.20
6	3	15	6.33	6.20
7	2	15	3.12	2.86
6	2	15	3.46	3.51
5	2	15	7.57	7.46
4	2	15	22.17	22.49
3	2	15	7.58	7.80
1	1	15	8.47	8.34
2	1	15	2.64	2.69
3	1	15	3.12	2.88
4	1	15	8.76	8.81
5	1	15	6.14	6.68
6	0	15	5.08	5.27
5	0	15	7.36	7.54
4	0	15	6.62	6.83
3	0	15	2.56	2.71
2	0	15	5.33	5.34
1	0	15	18.03	17.23
0	0	16	52.42	51.16
1	0	16	4.65	4.85
5	0	16	3.07	2.81
5	1	16	4.86	4.81
4	1	16	7.31	7.45
3	1	16	6.93	6.42
2	1	16	3.77	3.66
1	1	16	2.55	2.45
2	2	16	5.67	5.63
3	2	16	12.15	12.84
4	2	16	20.39	20.65
5	2	16	2.77	2.87
6	2	16	6.74	6.99
5	3	16	6.28	6.63
5	4	16	4.11	3.79
6	4	16	3.05	3.01
6	4	17	4.70	4.27
4	4	17	2.32	2.66
3	3	17	16.03	16.03
4	3	17	4.25	4.41
5	3	17	4.97	4.85
6	3	17	3.73	3.60
6	2	17	6.05	5.91
5	2	17	5.28	5.85
4	2	17	5.97	6.13
3	2	17	15.26	15.58
2	2	17	4.48	4.49
1	1	17	7.64	7.65
2	1	17	2.71	2.56
3	1	17	8.30	8.75
4	1	17	9.92	10.20
6	1	17	3.22	3.23
7	1	17	2.31	2.46
4	0	17	3.56	3.58
2	0	17	2.71	2.78
1	0	17	16.74	16.34
1	0	18	8.24	7.91
2	0	18	6.50	6.35
3	0	18	14.08	14.02
4	0	18	2.50	2.26
5	0	18	4.06	4.20
6	1	18	2.71	2.67
3	1	18	14.21	14.17
2	1	18	6.48	6.22
1	1	18	12.90	12.76
3	2	18	4.13	4.07
4	2	18	8.23	8.87
5	2	18	8.74	9.17
6	3	18	6.28	6.38
5	3	18	5.31	6.05
3	3	18	17.34	17.36
5	4	18	5.13	5.32
5	4	19	4.10	4.19
3	3	19	3.05	2.95
5	3	19	8.44	8.83
6	3	19	3.62	3.81
6	2	19	2.68	2.56
4	2	19	2.38	2.66
3	2	19	11.42	12.10
2	2	19	2.95	2.87
1	1	19	11.09	10.74
2	1	19	6.58	6.26
3	1	19	9.34	9.06
4	1	19	7.88	7.67
6	1	19	3.29	3.38
4	0	19	4.48	4.37
3	0	19	14.54	14.85
2	0	19	6.61	6.66
1	0	19	3.94	4.06
0	0	20	7.65	7.24
1	0	20	10.20	10.17
3	0	20	12.35	12.82
4	0	20	6.49	6.55
5	0	20	5.66	5.65
6	0	20	2.57	2.41
5	1	20	2.55	2.55
4	1	20	9.80	10.09
3	1	20	8.54	8.33
2	1	20	9.65	9.76
1	1	20	2.40	2.83
3	2	20	17.55	17.82
5	2	20	2.77	2.76
6	3	20	2.81	3.27
5	3	20	3.65	3.68
3	3	20	6.70	6.26
4	3	21	8.18	7.97
5	2	21	2.90	2.82
3	2	21	8.21	8.00
2	2	21	8.08	7.93
1	1	21	8.71	8.64
2	1	21	13.80	13.46
5	0	21	5.79	5.78
4	0	21	8.05	8.10
3	0	21	6.13	6.37
1	0	21	8.62	8.45
2	0	22	7.06	6.80
5	1	22	2.37	2.15
4	1	22	5.34	5.45
3	1	22	14.53	14.25
2	1	22	9.97	9.92
1	1	22	3.66	3.87
2	2	22	10.67	10.47
3	2	22	5.79	5.57
4	2	22	8.15	7.90
5	3	22	3.07	2.94
4	4	23	3.77	3.72
4	3	23	4.94	4.93
4	2	23	4.47	4.39
3	2	23	6.61	6.55
2	2	23	7.34	7.19
1	1	23	7.30	7.15
2	1	23	4.48	4.80
3	1	23	9.43	9.65
4	1	23	5.77	6.01
5	0	23	4.06	4.12
4	0	23	4.11	4.62
2	0	23	7.88	7.63
0	0	24	6.33	5.88
1	0	24	3.52	3.51
2	0	24	10.26	10.75
3	0	24	3.37	3.73
4	0	24	9.43	9.65
4	1	24	3.79	3.90
3	1	24	5.66	5.68
2	1	24	2.73	3.36
1	1	24	3.33	3.66
2	2	24	4.43	4.48
3	2	24	7.93	8.20
3	3	24	3.33	3.36
4	4	24	4.90	4.68
3	3	25	3.52	3.54
4	2	25	2.77	2.54
2	2	25	3.60	3.39
1	1	25	4.89	4.77
2	1	25	5.13	5.24
3	1	25	6.87	6.80
4	1	25	4.17	3.97
4	0	25	4.57	4.62
3	0	25	13.68	13.36
2	0	25	15.69	15.57
1	0	25	6.83	6.68
3	0	26	7.29	7.26
5	1	26	4.34	3.71
4	1	26	4.25	4.32
3	1	26	5.21	5.42
2	1	26	6.48	6.22
1	1	26	5.16	4.83
2	2	26	5.10	5.30
3	3	26	3.50	3.13
3	3	27	4.40	3.95
3	2	27	2.62	3.06
2	2	27	5.59	5.84
1	1	27	2.91	3.25
2	1	27	11.95	11.73
3	1	27	4.27	3.92
4	1	27	5.77	5.92
4	0	27	4.18	4.04
3	0	27	4.10	4.25
0	0	28	5.60	5.48
2	0	28	3.17	3.73
4	0	28	2.45	2.41
4	1	28	6.04	5.86
2	1	28	6.86	7.07
3	3	28	3.03	3.42
3	2	29	3.91	3.78
2	2	29	7.64	7.50
1	1	29	3.02	3.14
2	1	29	6.22	6.23
4	0	29	3.17	3.07
3	0	29	4.17	4.31
1	0	30	7.51	7.07
3	1	30	3.64	3.43
1	1	30	5.35	5.24
2	2	30	5.12	4.85
2	2	31	4.43	4.36
2	1	31	3.22	2.89
0	0	32	6.57	6.34
1	1	32	4.66	4.50

562 reflections

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