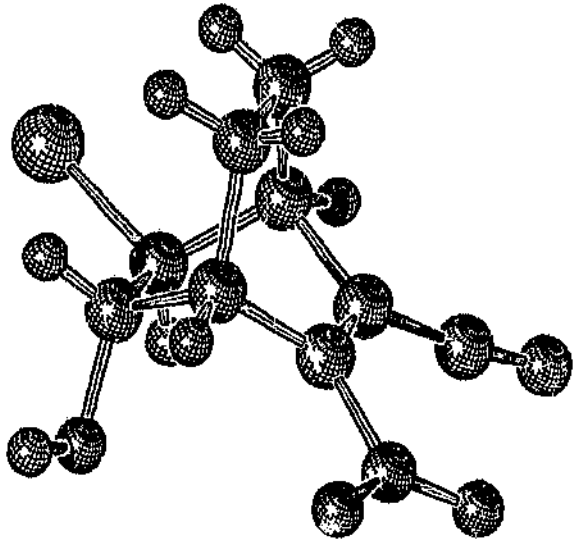
Applications of low-temperature crystallography

by

David Gordon Billing



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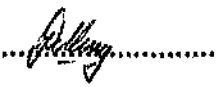
Abstract

Two cryostats, a liquid nitrogen gas flow cryostat and a Be walled flow cryostat, suitable for use on Enraf Nonius CAD4 four circle diffractometers have been commissioned and tested. The necessary techniques relating to crystal mounting and centring for low-temperature data collection were also developed. The complete experimental procedure was tested by determining the crystal structures of the following three compounds at low-temperatures:N-arylpyrrolidin-2-one,4-p-hydroxyphenyl-9,10,11-trihydroxy-8-ethyl-1,7-dioxaspiro[4,4]-nona-2,6-dione and 5,6-trans,-6-Bromo-5-hydroxy-3-nitrobicyclo[2.2.2]oct-2-ene. In performing these structure determinations, some of the applications of low-temperature crystallography were considered.

Declaration

I declare that this dissertation is my own, unaided work. It is being submitted for the degree of Master of Science in the University of the Witwatersrand, Johannesburg. It has not been submitted for any degree or examination in any other University.



D G Billing

January, 1991

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Prof. J.P.Michael for supplying me with crystals and his interest in the work.

Prof. G.W.Perold for the excellent crystals of leucodrin and conocarpin.

List of publications

The following publications have arisen from the work presented in this thesis or from related work.

1. The structure and conformation of 1,1'-Bipyrrolidene-2,2'dithione.
   J.P.Michael, D.G.Billing, J.C.A.Boeyens, L.Carlton, L.Denner, I.Hindmarch.
   J.Mol.Struc.,238,(1990),391-401.
2. Conformational analysis of rotational barriers in N-Arylpyrrolidin-2-ones.
   D.G.Billing, J.C.A.Boeyens, L.Denner, K.du Plooy, G.C.Long, J.P.Michael.
   Acta Cryst.(B) (In Press)
3. Comparison of the NMR coupling paths in leucodrin and conocarpin by low-temperature x-ray structure analysis.
   D.G.Billing, J.C.A.Boeyens, L.Carlton, D.C.Levendis, G.W.Perold.
   J. Chem. Soc. Perkin Transactions 2 (In Press)
4. Crystallographic study of N-arylpyrrolidin-2-ones
   D.G.Billing, J.C.A.Boeyens, D.C.Levendis and J.P.Michael
   S.A.J. of Chem. (Submitted for Publication)

Work from this thesis presented as papers at conferences attended.

1. Conformational preferences in N-Arylpyrrolidin-2-ones.
   Frank Warren Conference, South Africa, April 1988.
   D.G.Billing,J.C.A.Boeyens,L.Denner and J.P.Michael.

2. Low Temperature Crystallography using a Kappa geometry diffractometer.
   SACI Convention, Johannesburg, South Africa, January 1889.
   D.G.Billing, D.C.Levendis.

3. Low Temperature crystallographic study of 1-Phenylpyrrolidin-2-one.
   4th International Chemistry Conference in Africa, Zomba, Malawi, July 1989.
   D.G.Billing, D.C.Levendis.

4. Crystallographic study of N-Phenylpyrrolidin-2-one at selected low temperatures.
   15th Congress and General Assembly of the International Union of Crystallography, Bordeaux, France, July 1990.
   D.G.Billing, D.C.Levendis.

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Chapter One

Introduction

When an ideally imperfect crystal is exposed to a homogeneous beam of monochromatic x-rays, a three dimensional diffraction pattern results. The spatial positions of the diffracted rays are determined by the geometric size and shape of the unit cell. The relative intensities of each of these rays on the other hand may be related to the electron density distribution contained within the unit cell. One of the first applications of x-ray diffraction was the investigation of the structure of crystals. This application was and still is of paramount importance in the fields of chemistry, physics and mineralogy, and is made possible by the Fourier relationship which exists between the distribution of the electron density within the crystal and the intensity of the x-rays diffracted by the crystal. The positions of the atoms are inferred to be at the centres of ovoids of charge. This assumption, valid for the accuracy involved in routine investigations, unfortunately leads to results which are biased.

Technical breakthroughs in recent years, such as automated diffractometry, improved cryogenics and high speed computing have increased the overall resolution attainable in the diffraction experiment, and hence have led to more precise positional parameters for the atoms in the crystals. Along with this improved resolution, the following reasons also exist as to why it would be desirable to perform the diffraction experiment at low temperatures:

1.In general the stability of crystals are improved by cooling, thus it may be possible to study crystals at low temperatures which are unstable at room temperature.

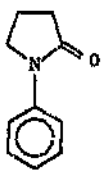
2.Compounds which are liquids at room temperature may be cooled to below their melting point, crystallised and studied.

3.Interesting phase transitions occurring below room temperatures can be studied in detail.

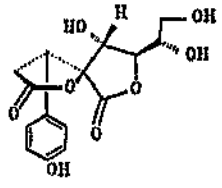
4.Disorder, frequently present in some crystals, may be resolved.

5.Total and deformation densities may be studied in detail, allowing for comparison with theoretically derived values, and investigations into the nature of chemical bonding.

The initial aim of this project was thus to assemble and commission the necessary equipment for performing low temperature diffraction studies. During this stage of the projects, tests are also to be carried out to determine the temperature stability of the cryostats, as well as the maximum time that a crystal could be kept at a temperature of interest. Following this the techniques and methods for collecting low-temperature x-ray data have to be developed. These include methods for mounting crystals, as the normal methods used in routine x-ray diffraction studies tend to fail. Also the problem of centring a crystal in the x-ray beam when it cannot be seen had to be overcome. The complete experimental configuration was then tested by determining the low temperature structures of some compounds, in the process of doing so some of the applications of low-temperature crystallography were considered. The compounds chosen for these tests are shown below:



I



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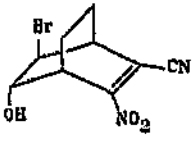


III

The first compound studied was N-phenyl-pyrrolidin-2-one (I). In this compound and derivatives of it, containing mono-substituted aromatic rings, the nature of the bond linking the two rings has been uncertain. A low-temperature study of this compound, conducted at a number of temperatures, allows for the effect of temperature on this bond to be evaluated. If data of a sufficiently high accuracy can be obtained, then the distribution of electronic charge in the bond can be investigated. The structures of II and III are both concerned with the determination of the positions of hydrogen atoms in molecular crystals. Hydrogen atoms have only one valence electron and no core electrons, making it difficult to locate using routine x-ray diffraction techniques. The presence of the heavy element, bromine, in III which tends to dominate the electron density distribution in such crystals, makes it even more difficult to locate hydrogen positions in such cases.

Chapter Two

The effect of temperature on the x-ray diffraction pattern of crystals[1].

Upon cooling, the diffraction pattern obtained from crystalline matter would change. The extent of the changes that would take place depends on the crystal as well as the temperature range through which the cooling takes place. A small change in temperature could lead to hardly any discernable change in the diffraction pattern, unless the crystal undergoes a phase change during the temperature change. In this case the diffraction pattern could change dramatically, depending on the nature of the phase change that takes place. Thus for example a disorder-order phase transition could result in diffuse layer lines in the diffraction pattern resolving into distinct reflections. This and other types of phase transitions could also be associated with changes in the unit cell, which on its own has a dramatic effect on the spatial distribution of reflections.

On the other hand if a crystal does not undergo a phase change on cooling the changes in the diffraction pattern are more subtle, even for large changes in temperature. Firstly the unit cell of the crystal will undergo a slight reduction in size due to thermal contraction. This would cause a slight spreading out of the reflections in the diffraction pattern.The intensity,I, of a diffracted reflection is related to the structure factor by the following

[1]The material for this chapter was summarised from similar work contained in the books listed in the bibliography.

expression:

$$F=\sqrt{\frac{KI}{Lp}}$$

where p, the polarisation factor, describing the polarisation which takes place in the primary beam because of the monochromator, is given by

$$p=(1+\cos^2 2\theta)/2$$

where θ is the angle of diffraction.

The Lorentz factor L, is dependant on the precise measurement technique used. While K represents a scaling factor dependent on effects such as crystal size and primary beam intensity. We thus have that

$$F\propto\sqrt{I}$$

The effect of cooling on the intensities of individual reflections is now most readily seen by considering the structure factor expression:

$$F(h)=\sum_{j=1}^{N} f_j e^{2\pi i \mathbf{h}\mathbf{r}_j}$$

where F(h) is the structure factor associated with the reciprocal space vector h, f denotes the atomic scattering factor for atom j, having position vector r. This expression is only valid for molecules at rest, as the scattering factors used, f_j, correspond to a stationary atom model. The scattering factors cause the inherent intensity decrease with respect to $\sin\theta/\lambda$ depicted in figure 2.1.

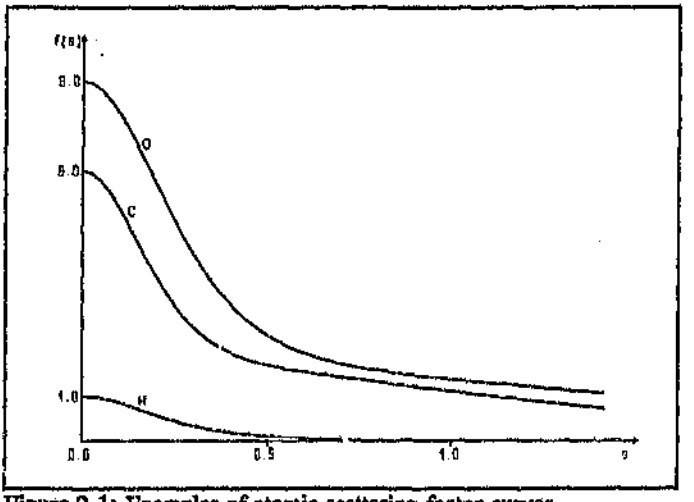


Figure 2.1: Examples of atomic scattering factor curves.

The thermal motion of an atom can be taken into account if the scattering factor for the stationary atom is replaced by the scattering factor for a vibrating atom, f_t which has the form:

$$f_t = f_0 e^{(-B\sin^2\theta)/\lambda^2}$$

The quantity B, known as the Debye-Waller factor is related to the atomic vibration by:

$$B = 8\pi^2 u^2 = 8\pi^2 U$$

where u is the root-mean-square amplitude of atomic vibration, U is the temperature factor generally refined. The effect of the additional term in the structure factor expression, is to cause an increased fall-of of intensity with respect to $\sin\theta/\lambda$, as is shown in figure 2.2 for various values of B.

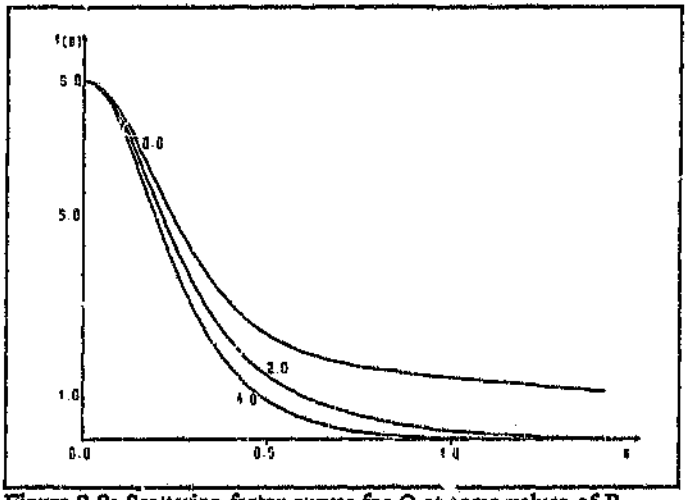


Figure 2.2: Scattering factor curves for O at some values of B

Thus on cooling, the high order reflections show the most obvious change in that they become more intense. More importantly, they become measurable. Thus the number of measured reflections included in a data set increases. This results in better, more precise structure refinements, including lower R-factors. Lower temperatures also improve the precision of the diffraction experiment. This is because intrinsic curvature of the electron density is increased and the magnitude of the thermal diffuse scattering is reduced, allowing for more accurate measurement of reflection intensities.

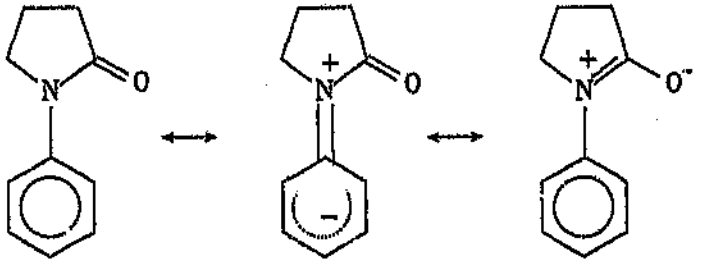
Thus for a crystal that does not undergo a phase transition, the most striking change in its x-ray diffraction pattern on cooling is the large increase in the number of observable reflections. On closer inspection each of the reflections will also be found to have become sharper, due to the reduction of the thermal motion within the crystal. This results in more precise structure determinations with significantly smaller standard deviations associated with the various structural parameters.

Chapter Three

The crystal structure of N-phenylpyrrolidin-2-one at selected low temperatures.

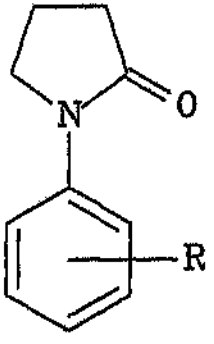
3.1 Introduction

In molecules such as mono-substituted N-arylpyrrolidin-2-ones (eg. I to VIII), restricted rotation around the inter-ring bond could arise from resonance involving the carbonyl group, the nitrogen lone pair and the aromatic system:



Under this scheme the occurrence of the barrier should be accompanied by shorter N-C(Ar) and N-C(O) bonds, a longer C-O distance and a planar system.

The group of related structures I to VIII formed the basis for investigating the barrier to rotation, and the effect of different substituents on such a barrier, both electronically and sterically in mono-substituted N-phenylpyrroldin-2-ones.



I : R=H

II : R=*o*-CH_3

III : R=*m*-CH_3

IV : R=*p*-CH_3

V : R=*m*-OCH_3

VI : R=*p*-OCH_3

VII : R=*m*-NO_2

VIII : R=*o*-OH

By performing the study at various low temperatures in the range of 90K to room temperature, the possible effects of temperature on the solid state conformation of the molecule could possibly be assessed. For the purposes of such a study, compound I was chosen due to its crystal quality, relative high symmetry and availability. It was also hoped that data of a sufficiently high quality could be obtained to allow for a study of the electron density distribution around the central bond. This would hopefully lead to a better understanding of the conformational preferences exhibited by this series of molecules.

3.2 Experimental

Good quality near cubic crystals, grown from CCl_4 across a hexane interface, with edges approximately 0.5mm in size were selected for study. Data were collected on Enraf Nonius CAD4 diffractometers, equipped with graphite single-crystal monochromators, using the cryostats described in chapter 6. Cell constants were obtained from least-squares refinement of 25 high angle θ values. Data reduction

consisted of correction for background, decay (linear), Lorentz and polarisation effects. Table 3.1 contains the crystallographic data at the various temperatures. The structures were solved by direct methods and refined using full matrix least-squares techniques, with the SHELX set of programs.[Sheldrick, 1985]. Table 3.2 contains the fractional coordinates of the 160K structure, which at present is the most precise determination of this structure. Figure 3.1 shows an Ortep diagram of the structure as well as the numbering scheme used throughout this series of structures. The coordinates of the other structures, as well as tables containing bond lengths and bond angles are included in appendix 1. Tables of thermal parameters and structure factors are included as supplementary material on microfiche.

Table 3.1: Crystallographic data for N-phenylpyrroldin-2-one at various temperatures

Temperature/K	R.T.	160	100	90
a/Å	14.114(3)	13.871(2)	13.787(3)	13.758(3)
b/Å	6.879(4)	6.8581(7)	6.8431(8)	6.821(3)
c/Å	17.836(5)	17.335(2)	17.315(3)	17.291(3)
V/Å^3	1687.93	1649.00	1633.55	1622.73
Space group	Pbca	Pbca	Pbca	Pbca
Range	$2<\theta<30$	$3<\theta<40$	$3<\theta<30$	$3<\theta<30$
no. of refs	1110	2260	1470	1552
no. parms	110	143	145	143
R	0.059	0.059	0.065	0.096
Rw	0.054	0.057	0.067	0.080
Cryostat used	-	gas flow	gas flow	flow

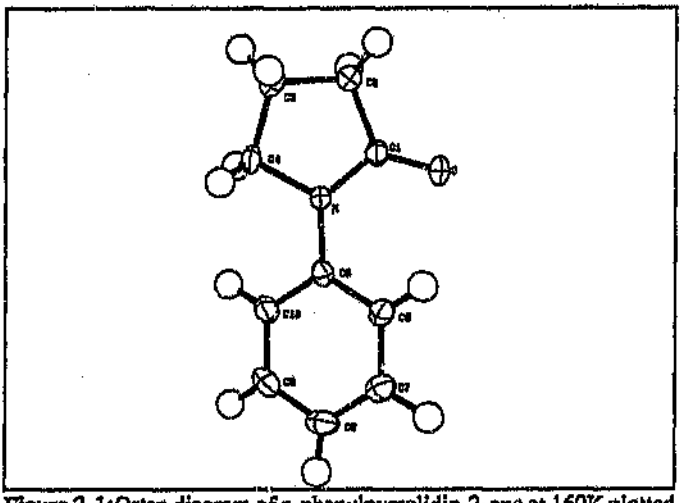


Figure 3.1:Ortep diagram of n-phenylpyrrolidin-2-one at 160K plotted at 50% probability.

Table 3.2: Fractional coordinates ($\times 10^4$) and equivalent isotropic temperature factors ($Å^2, \times 10^3$) for 160K structure of N-phenylpyrrolidin-2-one.

	x/a	y/b	z/c	U_{eq}
N	1253(1)	729(2)	4073(1)	20(0)
O	1422(1)	-1906(2)	4884(1)	28(0)
C1	1351(1)	-151(2)	4775(1)	21(0)
C2	1388(1)	1421(2)	5383(1)	26(0)
C3	1021(1)	3233(2)	4975(1)	28(0)
C4	1271(1)	2877(2)	4136(1)	25(0)
C5	1265(1)	-211(2)	3346(1)	20(0)
C6	874(1)	-2070(2)	3249(1)	25(0)
C7	852(1)	-2910(3)	2523(1)	32(0)
C8	1219(1)	-1932(3)	1891(1)	34(0)
C9	1618(1)	-105(3)	1993(1)	30(0)

C10	1648(1)	756(2)	2714(1)	26(0)
H21	1023(13)	1130(25)	5838(9)	30(1)
H22	2044(14)	1572(25)	5533(9)	30(1)
H31	1324(13)	4445(27)	5168(9)	30(1)
H32	336(14)	3368(25)	5037(9)	30(1)
H41	1870(14)	3266(25)	3997(9)	30(1)
H42	834(13)	3346(25)	377i(9)	30(1)
H6	631(12)	-2718(27)	3684(9)	30(1)
H7	582(13)	-4310(28)	2473(8)	30(1)
H8	1202(12)	-2352(28)	1387(10)	30(1)
H9	1913(13)	559(26)	1573(9)	30(1)
H10	1913(13)	2037(27)	2770(9)	30(1)

3.3 Discussion

Crystallographic studies of the structures, of compounds of the type presently under consideration, have resulted in the bond length and bond angle information given in Tables 3.3 and 3.4, for different substituents (R) on the phenyl rings. From this data it would appear that the central bond between the two rings (N-C5) remains effectively constant in length, (average = 1.419 Å), independent of the torsion angle between the two rings. Further, from molecular mechanics studies a rotational barrier of 7kJmol^{-1} [Billing, Boeyens, Denner, du Plooy, Long and Michael, 1990] has been inferred to exist around the bond.

Table 3.3:Selected bond lengths for related substituted compounds.

	R	Bond Length/Å		
		N-C1	O-C1	N-C5
I	H	1.357(3)	1.218(3)	1.404(4)
II	*o*-CH_3	1.356(3)	1.225(3)	1.432(3)
III	*m*-CH_3	1.373(3)	1.217(3)	1.415(3)
IV	*p*-CH_3	1.368(3)	1.228(2)	1.421(2)
V	*m*-OCH_3	1.366(2)	1.211(2)	1.420(2)
VI	*p*-OCH_3	1.363(3)	1.223(3)	1.419(3)
VII	*m*-NO_3	1.370(2)	1.211(2)	1.416(2)
VIII	*o*-OH	1.345(2)	1.233(2)	1.424(2)

Table 3.4:Selected angles for related substituted compounds.

	R	Torsion Angles/°		Angles between mean planes/°
		C1-N-C5-C6	C1-N-C5-C10	
I	H	34.9(2)	-147.4(4)	33.63(13)
II	*o*-CH_3	63.3(2)	-119.6(3)	58.66(12)
III	*m*-CH_3	155.2(3)	-26.6(1)	25.04(11)
IV	*p*-CH_3	-151.0(2)	31.0(1)	27.90(8)
V	*m*-OCH_3	177.9(2)	-2.4(1)	5.10(8)
VI	*p*-OCH_3	-13.4(1)	166.8(3)	11.63(10)
VII	*m*-NO_3	-25.7(1)	156.2(2)	24.29(8)
VIII	*o*-OH	123.7(2)	-57.7(2)	59.29(9)

Table 3.5: Selected bond lengths and angles for n-phenylpyrrolidin-2-one at various low temperatures.

	Bond length	Torsion angles/°		Angle between
T/K	N-C5	C1-N-C5-C6	C1-N-C5-C10	mean planes/°
R.T	1.404(4)	34.9(2)	-147.4(4)	33.6(1)
160	1.415(2)	35.6(1)	-146.4(2)	34.23(6)
100	1.406(3)	36.1(2)	-145.6(3)	35.2(1)
90	1.421(4)	35.8(2)	-145.7(4)	24.5(2)

It would thus appear as if this barrier to rotation can be ascribed to either packing interactions or to nuclear screening [Boeyens, 1982]. Table 3.5 lists the values obtained for the N-C5 bond length at the different temperatures as well as some of the torsion angles for n-phenylpyrrolidin-2-one. From this table it is clear that the conformation of the molecule does not change with temperature. This can also be seen from figures 3.2 and 3.3, which show stereo projections of the structure at 90K and room temperature respectively. This would seem to suggest that the barrier to rotation does not exist due to packing interactions. Further evidence of this is the success of the molecular mechanics calculations in reproducing the crystal structures. Attributing the barrier to nuclear screening, compels one to consider the distribution of electronic charge throughout the molecule. Now although low-temperature crystallography as a technique is eminently suited to such a task, it is not feasible in the present case. This is because of the deterioration of the crystal quality on cooling which makes it impossible to collect a sufficiently large and accurate data set for performing electron density studies.

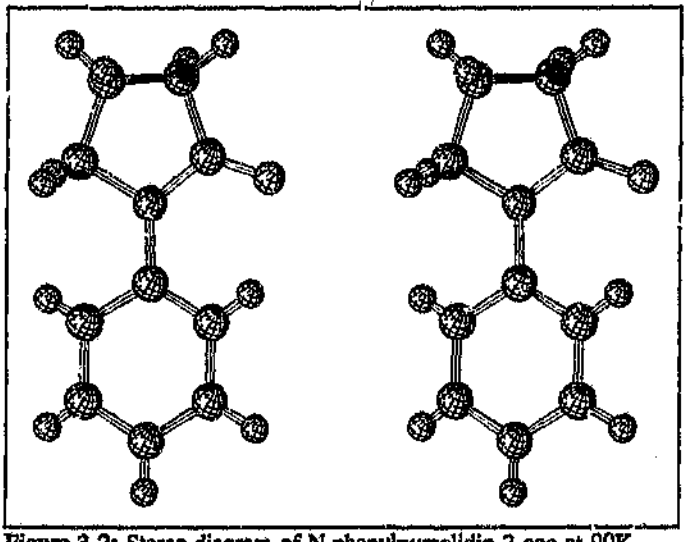
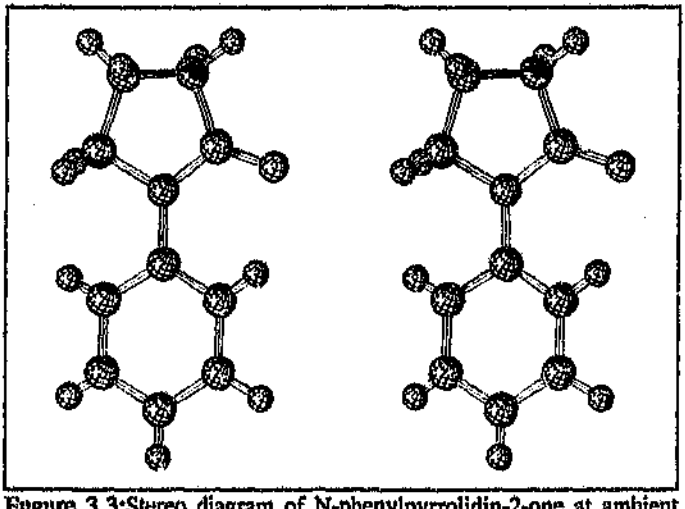


Figure 3.2: Stereo diagram of N-phenylpyrrolidin-2-one at 90K.



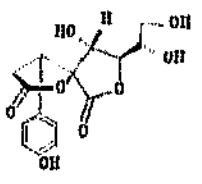
Fugure 3.3:Stereo diagram of N-phenylpyrrolidin-2-one at ambient temperature

Chapter Four.

The low temperature structure of Leucodrin

4.1 Introduction

Leucodrin (I) is obtained from *leucacendron salignum* (ex *adscendens*) [Perold and Pachler, 1966] a species of the Protacean family. Its stereochemistry was demonstrated by X-ray analysis of the dibromo derivative [Diamand and Rogers, 1964], for which no coordinates were reported. The diastereomer of leucodrin, conocarpin (II) comes from *leucospermum conocarpodendron* (L) Buek. [Kruger and Perold, 1970] also of the Protaceae family.



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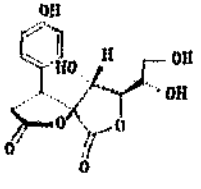
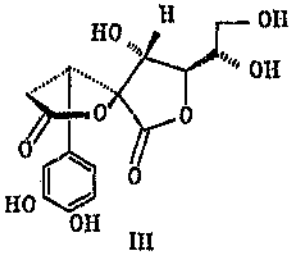


II

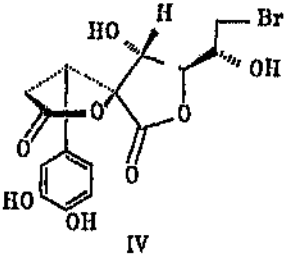
An NMR study of leucodrin[1] and conocarpin revealed five-bond proton-proton couplings of the order of 1 Hz for leucodrin, whilst the corresponding value for conocarpin is reduced to 0.4 Hz. It is of interest to note that three congeners of leucodrin, viz. leudrin (III), its 11-bromo-11-deoxy derivative (IV) and its *ar*-dimethyl ether (V) also show five-bond proton-proton couplings of the order of 1 Hz, over the

[1] IUPAC: 4-p-hydroxyphenyl-9,10,11-trihydroxy-8-ethyl-1,7-dioxaspiro[4,4]-nona-2,6-dione. The detailed numbering is shown in VI

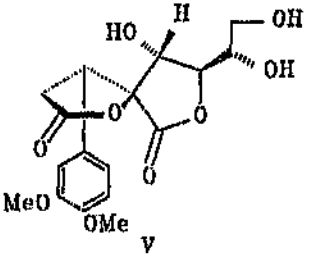
same six atom chain: 3a-H,3-C,4-C,5-C,9-C,9-H [Perold, Carlton, Howard and Michael, 1988]. An interesting aspect of this coupling is that no coupling is evident for the similar chain starting with the 3b-H atom.



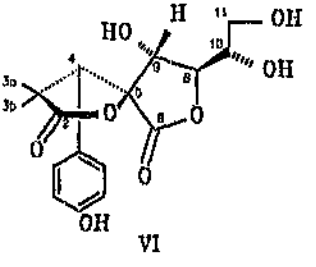
III



IV



V



VI

In order to rationalise this clearly observable $^{5}J_{3\text{-}H,9\text{-}H}$ coupling in the NMR spectra of leucodrin and conocarpin, it is necessary to consider the relative co-planarity of the six atoms involved in terms of the detailed geometry of the molecules. In this respect the positional parameters derived for hydrogen atoms from routine x-ray structure determinations are possibly not sufficiently precise. Thus the structure of leucodrin was determined by x-ray diffraction methods at ambient temperature and at 100K. From this study it is hoped that observed coupling may be rationalised. Furthermore by comparing the two structures the reliability of the hydrogen positions in the room temperature structure of conocarpin can be inferred. This became necessary because conocarpin crystals shattered on cooling.

4.2 Experimental

Crystals of good quality were obtained from Prof. G.W. Perold, and used for the data collection using an Enraf-Nonius CAD4 diffractometer, equipped with an incident beam graphite crystal monochromator. For the low temperature data the gas flow croystat described in chapter 6 was also used. Cell constants were determined by least-squares refinement based on the optimised setting angles of 25 high θ reflections, each measured at four positions. Data reduction consisted of correction for background, Lorentz and polarisation effects. In each case three standard reflections were also monitored hourly, but these only showed random fluctuations. The structures were solved by direct methods and refined by full-matrix least-squares using the SHELX set of programs. [Sheldrick, 1985]. In the room temperature structure all the hydrogen atoms were placed in geometrically calculated positions with a refined common U_{iso}(=0.060(4)). In the low temperature structure the hydrogen atom positions were located from difference Fourier maps and refined with a common refined U_{iso}(=0.016(1)). Although the absolute structure could not be refined with confidence, the known stereochemistry [Diamand and Rogers, 1964] gave a slightly better numerical fit with the numerical data in each case. Crystal and diffraction data are contained in tables 4.1 and 4.2, whilst refined atomic coordinates are collated in tables 4.3 to 4.5. Labelled Ortep drawings of the molecules are shown in figures 4.1 to 4.3. Tables of bond lengths and bond angles are included in appendix 2. Structure factors and anisotropic thermal parameters are included as supplementary material on microfiche.

Table 4.1:Crystal and Diffraction data for Leucodrin.		
Temperature/K	R.T.	100
Chemical formula	$C_{15}H_{16}O_8$	$C_{15}H_{16}O_8$
Crystal Colour	Clear	Clear
Crystal Shape	near Cubic	near Cubic
Crystal Dimensions/mm^3	0.6×0.6×0.6	0.4×0.4×0.4
Mol. weight/$g.mol^{-1}$	324.28	324.28
Crystal system	Orthorhombic	Orthorhombic
Space group	$P2_12_12_1$	$P2_12_12_1$
Cell Dimensions: a/Å b/Å c/Å V/$Å^3$	 8.079(1) 9.047(1) 25.785(3) 1418.3	 6.0196(8) 8.9837(9) 24.645(1) 1386.8
z	4	4
$D_c/g.cm^{-3}$	1.519	1.553
F(000)	680	680
Radiation used	Cu-Kα	Mo-Kα
λ/Å	1.5418	0.71069
μ/cm^{-1}	9.6	0.81
Scan type	ω-2θ	ω-2θ
θ range	3≤θ≤65	3≤θ≤46
hkl range h: k: l:	 0→7 0→10 0→30	 -2→11 -3→17 -4→48
Vertival aperture/mm	4	4
Horizontal aperture/mm	1.5	1.3
Variable scan speed: max:/°min^{-1} min:/s.$refl^{-1}$	 5.49 60	 5.49 90
Measured reflections	1458	8961
Unique reflections	1424	7268

cut-off criteria	F>2σ(F)	F>4σ(F)
ls. parameters	221	257
Max. shift, Δz	0.090	0.002
R	0.051	0.040
R_w, $w=1/\sigma^2(F)$	0.050	0.039
Residual density /eÅ^{-3}: max: min:	 2.33 -0.30	 0.94 -0.52

Table 4.2:Crystal and Diffraction data for Conocarpin	
Temperature/K	R.T.
Chemical formula	$C_{15}H_{16}O_8$
Crystal Colour	Clear
Crystal Shape	near Cubic
Crystal Dimensions/mm^3	
Mol. weight/g.n	324.28
Crystal system	Orthorhombic
Space group	$P2_12_12_1$
Cell Dimensions: a/Å b/Å c/Å V/Å^3	 6.0657(5) 12.021(1) 18.772(1) 1482.6
z	4
D_c/g.cm^{-3}	1.454
F(000)	680
Radiation used	Mo-Kα
λ/Å	0.71069
μ/cm^{-1}	0.76
Scan type	ω-2θ

θ range	3≤θ≤28
hkl range h: k: l:	 -8 → 8 0 → 17 0 → 24
Vertival aperture/mm.	4
Horizont aperture/mm	1.5
Variable scan speed: max:/°min^{-1} min:/s.refl^{-1}	 5.4° 60
Measured reflections	3494
Unique reflections	3270
cut-off criteria	F>4σ(F)
ls. parameters	221
Max. shift,Δz	0.89
R	0.049
R_w,w∝1/σ^2(F)	0.046
Residual density /eÅ^{-3}: max: min:	 0.50 -0.51

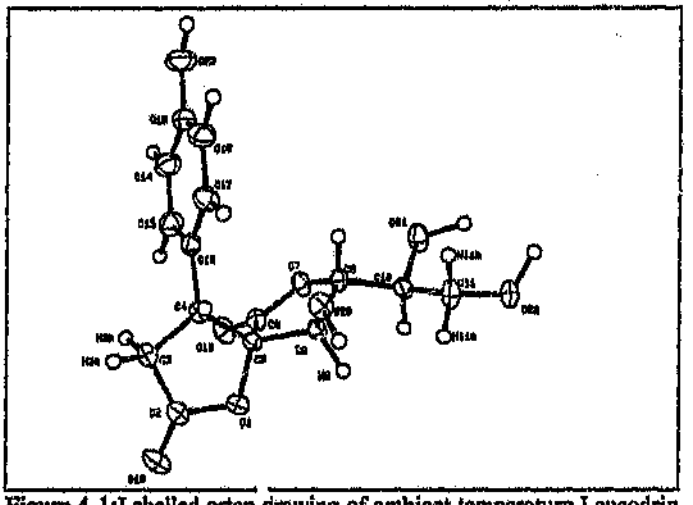


Figure 4.1:Labelled ortep drawing of ambient temperature Leucodrin structure.

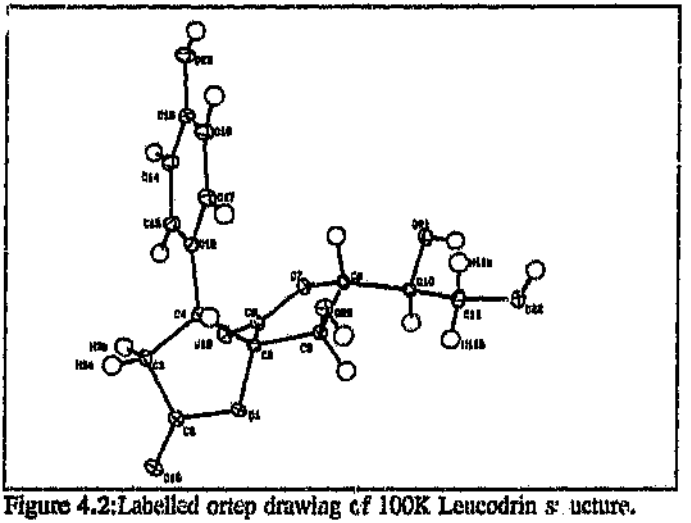


Figure 4.2:Labelled ortep drawing of 100K Leucodrin structure.

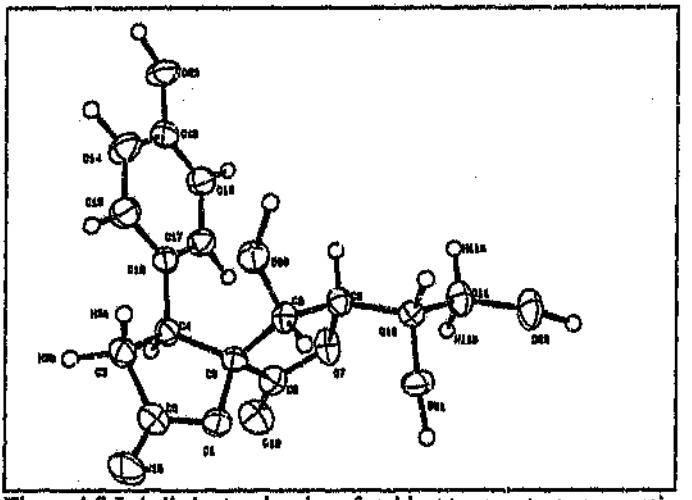


Figure 4.3:Labelled ortep drawing of ambient temperature conocarpin structure.

Table 4.3:Fractional atomic coordinates ($\times 10^4$) and Equivalent isotropic temperature factors ($Å^2, \times 10^3$) for leucodrin.

	x/a	y/b	z/c	U_{eq}
O1	-3536(5)	5893(3)	8021(1)	24(1)
O7	1265(5)	4155(3)	8406(1)	26(1)
O18	-5029(5)	8141(3)	7933(1)	31(1)
O19	922(5)	6569(3)	8276(1)	31(1)
O20	-4007(5)	2682(3)	8667(1)	27(1)
O21	2747(5)	1161(3)	8289(1)	37(1)
O22	178(6)	-735(3)	7745(1)	32(1)
O23	2295(6)	4492(4)	10650(1)	47(1)
C2	-4254(7)	7217(4)	8213(2)	24(1)
C3	-3884(9)	7275(5)	8789(2)	28(1)
C4	-3312(7)	5664(4)	8935(1)	19(1)

C5	-2365(7)	5075(4)	8412(1)	18(1)
C6	80(8)	5386(4)	8346(2)	23(1)
C8	-168(7)	2869(4)	8453(2)	21(1)
C9	2510(7)	3408(4)	8327(2)	20(1)
C10	686(7)	1681(4)	8086(2)	21(1)
C11	-879(8)	382(4)	8046(2)	32(1)
C12	-1853(7)	5426(4)	9403(2)	23(1)
C13	112(9)	6202(5)	9485(2)	35(1)
C14	1479(9)	5893(5)	9903(2)	38(1)
C15	882(8)	4778(5)	10249(2)	33(1)
C16	-1127(9)	4044(5)	10193(2)	34(1)
C17	-2437(8)	4346(5)	9767(2)	30(1)
H20	-5495(5)	2325(3)	8475(1)	60(4)
H21	3044(5)	-7(3)	8237(1)	60(4)
H22	710(6)	-1644(3)	8986(1)	60(4)
H23	1627(6)	3764(4)	10342(1)	60(4)
H3b	-2543(9)	8012(5)	8881(2)	60(4)
H3a	-5354(9)	7635(5)	8987(2)	60(4)
H4	-4732(7)	5077(4)	9079(1)	60(4)
H8	-192(7)	2383(4)	8836(2)	60(4)
H9	-3079(7)	3167(4)	7939(2)	60(4)
H10	850(7)	2152(4)	7703(2)	60(4)
H11a	-2385(8)	720(4)	7859(2)	60(4)
H11b	-1245(8)	-34(4)	8429(2)	60(4)
H13	575(9)	7053(5)	9212(2)	60(4)
H14	2977(9)	6514(5)	9960(2)	60(4)
H16	-1646(9)	3239(5)	10477(2)	60(4)
H17	-3950(8)	3739(5)	9713(2)	60(4)

Table 4.4:Fractional atomic coordinates($\times 10^5$, $\times 10^4$ for H) and equivalent isotropic temperature factors ($Å^2 \times 10^4$, $\times 10^3$ for H) for Leucodrin at 100K.

	x/a	y/b	z/c	U_{eq}
O1	-35359(13)	59653(8)	80199(3)	94(1)
O7	13261(12)	42097(8)	84063(3)	107(1)
O18	-50139(14)	82363(9)	79364(3)	119(1)
O19	9770(13)	66435(8)	82682(3)	125(1)
O20	-39747(13)	27174(9)	86768(3)	118(1)
O21	27777(13)	11411(8)	83056(3)	143(1)
O22	1848(15)	-6967(9)	77243(3)	128(1)
O23	24307(16)	44398(10)	106475(3)	168(1)
C2	-42413(17)	72957(11)	82168(4)	98(1)
C3	-38550(20)	73387(11)	87979(4)	111(1)
C4	-32838(17)	57241(11)	89399(4)	94(1)
C5	-23550(17)	51366(10)	84128(4)	84(1)
C6	1292(17)	54468(11)	83474(4)	94(1)
C8	-11.4(17)	29084(10)	84514(4)	94(1)
C9	-24963(17)	34473(11)	83326(4)	88(1)
C10	7532(17)	17078(11)	80878(4)	93(1)
C11	-8740(18)	4209(11)	80260(4)	126(1)
C12	-18096(18)	54605(11)	94090(4)	103(1)
C13	1968(19)	62260(12)	94883(4)	127(1)
C14	15857(19)	58755(13)	99022(4)	145(2)
C15	9870(19)	47567(12)	102535(4)	127(2)
C16	-10463(20)	40402(12)	101960(4)	132(2)
C17	-24094(19)	43811(12)	97745(4)	124(2)
H3a	-5342(26)	7684(16)	8987(5)	16(1)
H3b	-2721(26)	8080(16)	8852(5)	16(1)
H4	-4681(24)	5110(16)	9014(5)	16(1)

H8	19(28)	2568(16)	8855(5)	16(1)
H9	-2821(24)	3249(15)	7954(5)	16(1)
H10	1062(24)	2102(16)	7760(5)	16(1)
H11a	-1334(23)	-33(15)	8379(5)	16(1)
H11b	-2032(24)	802(16)	7830(5)	16(1)
H13	556(25)	6998(15)	9254(5)	16(1)
H14	2775(25)	6457(15)	9963(5)	16(1)
H16	-1502(24)	3313(15)	10460(5)	16(1)
H17	-4007(23)	3780(15)	9716(5)	16(1)
H20	-5121(26)	2351(15)	8512(5)	16(1)
H21	3040(27)	551(15)	8148(6)	16(1)
H22	394(26)	-1334(16)	7904(6)	16(1)
H23	2037(27)	3828(15)	10814(5)	16(1)

Table 4.5:Fractional atomic coordinates ($\times 10^4$) and equivalent isotropic temperature factors ($Å^2 \times 10^3$) for Conocarpin.

	x/a	y/b	z/c	U_{eq}
O1	2503(3)	182(1)	7298(1)	35(0)
O7	5795(3)	2028(2)	7594(1)	42(0)
O18	521(4)	-944(1)	6697(1)	54(1)
O19	7079(3)	678(2)	7391(1)	48(1)
O20	527(3)	2584(1)	7264(1)	39(0)
O21	3085(3)	1648(1)	9157(1)	29(0)
O22	4636(3)	3342(2)	9995(1)	54(1)
O23	3839(4)	5453(1)	5334(1)	47(1)
C2	1341(5)	-104(2)	6724(2)	35(1)
C3	1316(5)	728(2)	6181(1)	39(1)
C4	3332(5)	1362(2)	6389(1)	29(1)
C5	3296(4)	1235(2)	7208(1)	27(1)

C6	5614(5)	1244(2)	7530(1)	32(1)
C8	3705(4)	2584(2)	8079(1)	30(1)
C9	1954(4)	1963(2)	7673(1)	30(1)
C10	3265(5)	2657(2)	8874(1)	28(1)
C11	5059(5)	3264(2)	9246(1)	45(1)
C12	3479(5)	2456(2)	6096(1)	30(1)
C13	1846(5)	2852(2)	5655(2)	46(1)
C14	1996(5)	3853(2)	5400(2)	48(1)
C15	3772(5)	4465(2)	5592(1)	36(1)
C16	5423(5)	4074(2)	6018(2)	40(1)
C17	5274(5)	3072(2)	6266(1)	37(1)
H20	38(3)	3381(1)	7292(1)	82(3)
H21	3439(3)	921(1)	9409(1)	82(3)
H22	5633(3)	3415(2)	10469(1)	82(3)
H23	2494(4)	581(1)	5052(1)	82(3)
H3b	1481(5)	420(2)	5650(1)	82(3)
H3a	-173(5)	1181(2)	6214(1)	82(3)
H4	4834(5)	1095(2)	6142(1)	82(3)
H8	3728(4)	3354(2)	7866(1)	82(3)
H9	845(4)	1569(2)	8031(1)	82(3)
H10	1734(5)	3060(2)	8964(1)	82(3)
H11a	5132(5)	4026(2)	9020(1)	82(3)
H11b	6619(5)	2881(2)	9166(1)	82(3)
H13	449(5)	2382(2)	5510(2)	82(3)
H14	747(5)	4152(2)	5046(2)	82(3)
H16	6816(5)	4549(2)	6160(2)	82(3)
H17	6583(5)	2765(2)	6593(1)	82(3)

4.3 Discussion

In the current study it is of interest to compare the two structures obtained or Leucodrin. This is most readily achieved by overlaying transparencies of the structural diagrams of figures 4.1 and 4.2. The overlay is almost exact, with the obvious differences being in the hydrogen positions of the hydroxyl groups. The experimental average O-H bond length of 0.73(1)Å is significantly shorter than the 1.08Å assumed in the geometrical calculations involved when positioning the hydrogens in the ambient temperature structure. Similarly the observed C-O-H angles, an average 107(1)°, are less than the 114° assumed for the geometrical calculation. The causes for these differences are many, but the most significant must be the use of spherical scattering factors for H, even though the electron density cloud of the hydrogen atom would be heavily distorted. Also important is the use of 1.08Å by the refinement program for the geometrical calculation. This is the bond length of C-H obtained by neutron diffraction, the equivalent O-H distance being 0.967Å [Allen, Kennard, Watson, Brummer and Orpen, 1987]. The calculated hydrogen positions seem far more reliable however when looking at C-H bonds and angles. The observed average of 0.997Å compares quite favourably with the 1.08Å assumed, The calculated hydrogen atom positions with respect to the five-bond couplings of interest, from C3 to C9 are therefore not in doubt.

Because of the presence of several hydroxyl and carbonyl groups in these molecules a variety of intermolecular hydrogen bonds is expected in the solid state. Several such possibilities are observed, but only in the case of the low-temperature leucodrin structure has any attempt been made to identify hydrogen bonds in terms of non-bonded

intermolecular O···H contacts. The only contact of less than 2Å, between O21···H20 (d=1.75Å) can be so interpreted with confidence. Other possibilities are presented by the contacts d(O20···H23)=2.00Å, d(O19···H22)=2.07Å.

Cases of 5-bond proton-proton coupling over single bonds are rare. It is therefore of interest to note that three congeners of leucodrin, showed five-bond coupling, of the order of 1 Hz over the six atom chain; 3a-H,3-C,4-C,5-C,9-C,9-H in solution, while no such effect is observable for the similar chain starting with 3b-H. [Perold, Carlton, Howard and Michael, 1988]. This effect has now also been established for leucodrin itself, as well as for Conocarpin. Long range couplings of this order are generally rationalised in terms of a planar disposition of the atoms, the so-called W-coupling mechanism or possibly using a through space effect. In the case of leucodrin it was found that the relative planarity of the chain does not distinguish between the two cases, table 4.6. Similarly the linear separation, table 4.6, between 3-H and 9-H does not offer any specific rationale for the coupling effects observed. The differences in the linear separation between leucodrin and conocarpin merely indicate conformational differences between the two molecules, as can be seen from the stereo diagrams of figures 4.4 and 4.5. At this stage one now considers coupling interactions over single bonds to occur via the bonding electrons, and that vicinal orbital transmissions are at a maximum when these orbitals are trans to each other. Then, for a six atom system three torsion angles of 180° along a planar array would offer the most favourable coupling condition. In the case of leucodrin and conocarpin the three torsion angles in the chain 3-H to 9-H are given in table 4.7. In the case of the chain 3b-H to 9-H for leucodrin the first torsion angle (at -94°) indicates that the first σ-orbital is orthoganally aligned to the plane of

the other two orbitals, hence minimal coupling is expected, as is found table 4.8. Similar reasoning can also be applied in the case of conocarpin, however here one should note that the second torsion angle in the series is also close to orthogonal. From this one can only conclude that whereas an orthogonal torsion angle at the terminus of a six atom chain minimises coupling, its location midway along the chain has a far lesser effect.

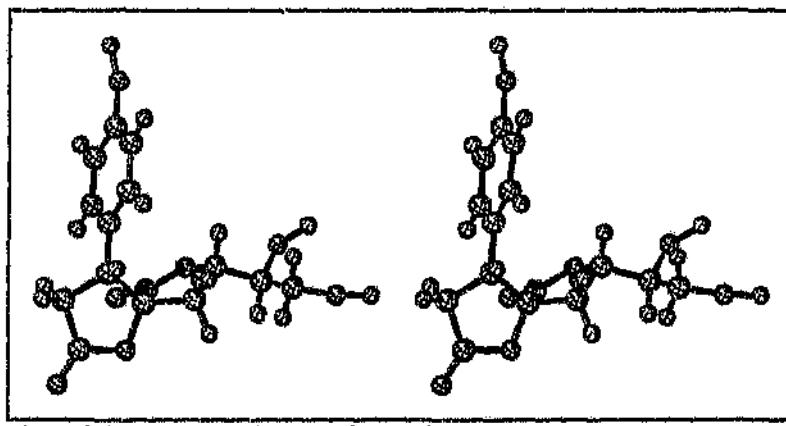


Figure 4.4:Stereoscopic diagram of Leucodrin.

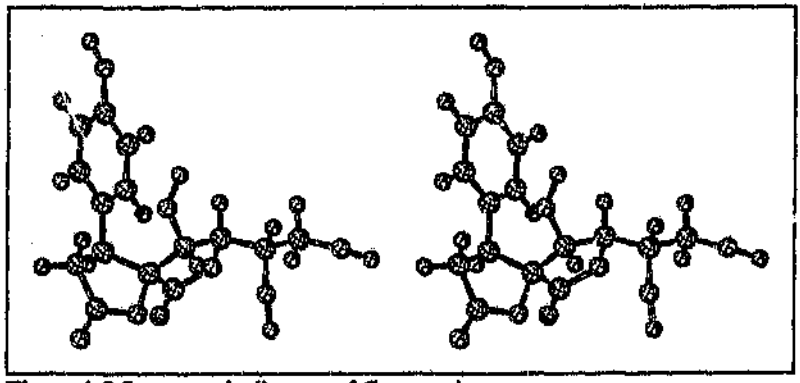


Figure 4.5:Stereoscopic diagram of Conocarpin

Table 4.6: Mean plane deviations and linear seperations.							
	Deviation(Å) from mean plane:3-H,3-C,4-C,5-C,9-C,9-H						Linear seperation(Å)
	3-H	3-C	4-C	5-C	9-C	9-H	between 3-H and 9-H
Leucodrin 3a-H2	.385(7)	-.362(6)	.229(6)	-.526(5)	.173(5)	.120(6)	5.055(1)
Leucodrin 3b-H	.331(7)	-.509(6)	-.140(6)	.307(5)	.186(5)	-.251(5)	5.022(1)
Conocarpin 3a-H	.234(4)	-.361(4)	.265(4)	-.308(3)	.408(3)	-.232(3)	3.502(1)
Conocarpin 3b-H	.022(4)	-.021(4)	-.225(4)	.442(3)	-.355(3)	.114(3)	4.729(1)

Table 4.7: Torsion angles.			
	3-H,3-C,4-C,5-C/°	3-C,4-C,5-C,9-C/°	4-C,5-C,9-C,9-H/°
Leucodrin, 3a-H	144.8(6)	-155.0(5)	135.1(5)
Leucodrin, 3b-H	-93.3(5)	-155.0(5)	135.1(5)
Conocarpin, 3a-H	85.2(3)	-87.3(2)	127.1(3)
Conocarpin, 3b-H	-152.6(4)	-87.3(2)	127.1(3)

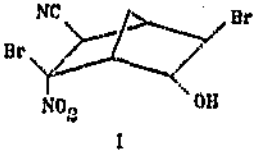
Table 4.8: Proton-proton coupling constants J_{H-H}(Hz) for Leucodrin and Conocarpin.										
	3a-3b	3a-4	3a-9	3b-9	3b-4	8-9	8-10	10-11a	10-11b	11a-11b
Leucodrin	-17.4	8.8	0.8	-	12.6	8.2	-	6.1	6.6	-11.0
Conocarpin	-16.7	13.2	-	0.4	7.8	8.8	3.0	-	-	-

Chapter Five

Hydrogen positions in the presence of Bromine.

5.1 Introduction.

Previous studies of norbornyl type systems, containing nitro and hydroxy side chains, have shown that the possibility of forming intramolecular hydrogen bonds in such systems is of interest.The presence of bromine, or other heavy elements, in a crystal tends to make it difficult to locate hydrogen atom positions. This was the case in the determination of the structure of I [Blom, Edwards, Field and Michael, 1980]. A method around this problem was put forward by Boeyens, Denner and Michael (1984), when they determined the structure of II. Their method involves the replacement of the bromine atoms, by chlorine which is far lighter. The method then hinges on the assumption that the two structures are identical.



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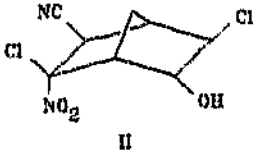
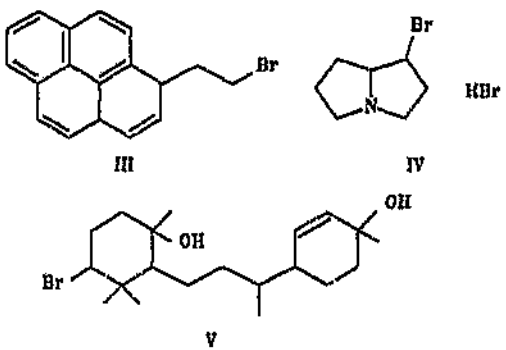
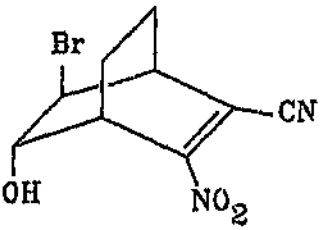


II

The method of choice for determining hydrogen positions in crystals is without doubt neutron diffraction. Facilities for using this method are however not readily available, hence the search for alternatives. Locating hydrogen positions using low temperature x-ray data seems possible considering that the hydrogen atoms were located and refined isotropically for III [Foss and Stevens, 1985], IV [Wilson and Sawicki, 1979] and V [Schmitz, Hollenbeak, Carter, Hossain and van der Helm, 1979].



While testing the cryostats, we decided to see if, by lowering the temperature at which the experiment is done, hydrogen atom positions could be determined, and possibly be refined in the presence of bromine atoms. For the purposes of our investigation suitable crystals of VI (IUPAC: 5,6-trans,-6-Bromo-5-hydroxy-3-nitrobicyclo[2.2.2]oct-2-ene-carbonitrile) were obtained from Prof. J.P Michael. This compound is sufficiently similar to those studied previously to allow for a consideration of the method, even though no intramolecular hydrogen bonding is expected in this case.



VI

5.2 Experimental

Using the crystals obtained from J.P.Michael, data were collected on a Nonius CAD4 single crystal diffractometer using graphite monochromated Mo-Kα radiation. For the 100K data the gas flow cryostat described in chapter seven was again used. Cell parameters were determined using four equivalents each of 25 high θ reflections. Crystal decay was monitored using three standard reflections, remeasured after each hour of exposure, but showed only random fluctuations. Data reduction consisted of corrections for background, Lorentz, polarisation and absorption, the latter empirically (North, Phillips and Mathews, 1968). The structures were solved by direct methods and refined with full-matrix least-squares techniques using the SHELX set of programs. For the ambient temperature structure no hydrogen atoms could reliably be located from difference Fourier maps, and hence all were placed in geometrically calculated positions (C-H 1.08Å), with a common refined isotropic temperature factor. For the 100K structure all hydrogen atoms were located, and their positions refined, again with a

common refined isotropic temperature factor. Crystal and diffraction data for the structures are contained in table 5.1. Fractional atomic coordinates are given in tables 5.2 and 5.3. The numbering scheme used is shown in the Ortep drawings in figures 5.1 and 5.2. Tables containing bond lengths and angles are included in the appendix 3. Anisotropic thermal parameters and tables of observed and calculated structures factors are included as suplementary material on microfiche.

Table 5.1:Crystal and diffraction data for VI		
Temperature/K:	RT	100
Chemical formula:	$C_9H_9N_2O_3Br$	$C_9H_9N_2O_3Br$
Crystal colour:	yellow	yellow
Crystal shape:	rectangular	rectangular
Crystal dimension/mm^3:	1.0×0.8×0.6	1.0×0.8×0.6
Mol. weight/g.mol^{-1}:	273.09	273.09
Crystal system:	Monoclinic	Monoclinic
Space group:	P 2_1/n	P 2_1/n
Space group no.:	14	14
Cell dimensions: a/Å: b/Å: c/Å: β/°: V/Å^3:	 8.29(1) 10.252(2) 12.12(2) 95.37(7) 1026(2)	 8.279(6) 10.044(2) 12.083(8) 95.03(3) 1001.9(9)
z	4	4
D_c/g.cm^{-3}:	1.768	1.810
F(000):	540	540
Radiation used:	Mo-Kα	Mo-Kα
λ/Å:	0.71069	0.71069
μ/cm^{-1}:	38.83	39.78
Scan type:	ω-2θ	ω-2θ
θ range/°:	$2 \leq \theta \leq 30$	$2 \leq \theta \leq 35$

hkl range: h: k: l:	 -11 → 11 -1 → 14 -1 → 17	 -13 → 13 -1 → 16 -1 → 19
Vertical aperture/mm:	4	4
Horizontal aperture/mm:	1.3	1.3
Variable scan speed range: Max: Min:	 5.49 ° min^{-1} 60 s $refl^{-1}$	 5.49 ° min^{-1} 90 s $refl^{-1}$
no. unique reflections:	1662	3027
cut off criteria:	$F > 4\sigma(F)$	$F > 3\sigma(F)$
no. L.S. parameters:	137	164
Max. shift, ΔZ:	0.001	-1.24
Residual electron density/e.$Å^{-3}$ $(\Delta\rho)_{max}$: $(\Delta\rho)_{min}$:	 1.52 -1.39	 3.01 -3.11
R:	0.072	0.079
R_w: $w=1/\sigma^2(F)-F^2$	0.077	0.085

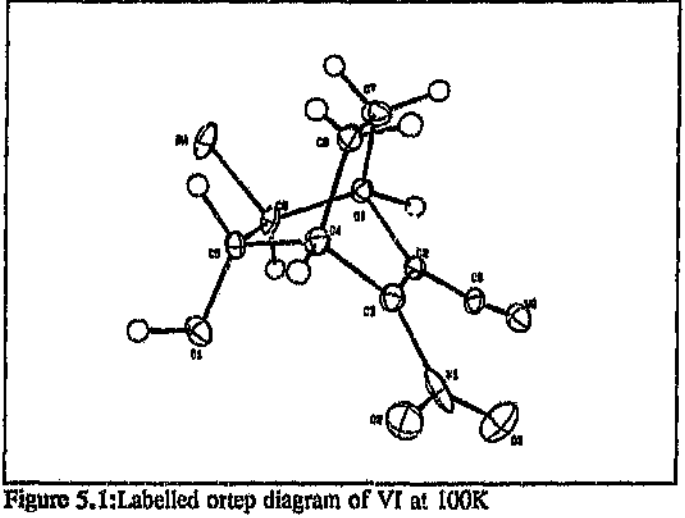


Figure 5.1:Labelled ortep diagram of VI at 100K

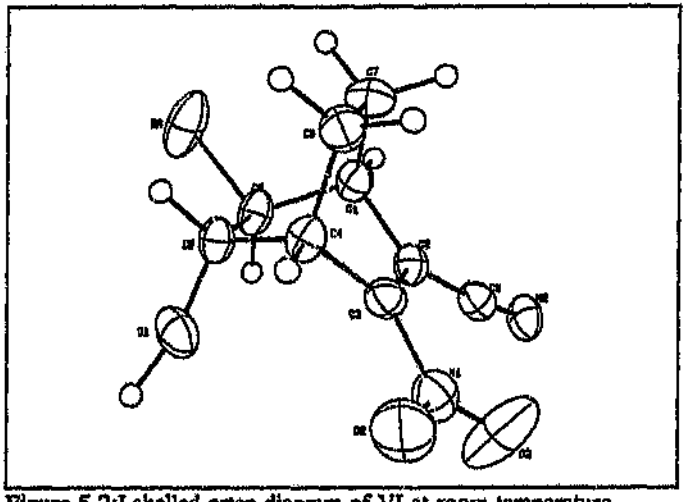


Figure 5.2:Labelled ortep diagram of VI at room temperature.

Table 5.2: Fractional coordinates ($\times 10^4$) and equivalent isotropic temperature factors ($Å^2$, $\times 10^3$) for VI at room temperature.

	x/a	y/b	z/c	U_{eq}
BR	240(1)	598(1)	6986(1)	61(0)
O1	1995(5)	-793(4)	9660(3)	49(1)
O2	672(6)	-3993(6)	10819(4)	54(1)
O3	-1763(8)	-3403(7)	10970(5)	77(2)
N1	-590(6)	-3418(5)	10523(4)	38(1)
N2	-4670(6)	-1670(6)	9711(5)	57(1)
C1	-1600(6)	-1385(6)	7948(4)	31(1)
C2	-1876(6)	-2052(5)	9036(4)	32(1)
C3	-602(6)	-2719(5)	9441(4)	30(1)
C4	810(6)	-2755(5)	8768(4)	29(1)
C5	1362(6)	-1315(5)	8652(4)	33(1)
C6	-127(6)	-515(6)	8253(5)	39(1)

C7	-1210(7)	-2432(6)	7107(4)	38(1)
C8	241(7)	-3261(6)	7620(5)	42(1)
C9	-3399(6)	-1870(5)	9483(4)	38(1)
HO1	2742(5)	-364(4)	10342(3)	73(8)
H1	-2629(6)	-839(6)	7584(4)	73(8)
H4	1764(6)	-3371(5)	9144(4)	73(8)
H5	2293(6)	-1286(5)	8086(4)	73(8)
H6	-367(6)	101(6)	8941(5)	73(8)
H71	-2253(7)	-3051(6)	6918(4)	73(8)
H71	-892(7)	-1968(6)	6357(4)	73(8)
H81	1223(7)	-3198(6)	7098(5)	73(8)
H82	-136(7)	-4265(6)	7678(5)	73(8)

Table 5.3: Fractional coordinates ($\times 10^4$) and equivalent isotropic temperature factors ($Å^2$, $\times 10^3$) for VI at 100K.

	x/a	y/b	z/c	U_{eq}
BR	259(1)	699(0)	7027(0)	22(0)
O1	2013(3)	-792(3)	9700(3)	19(0)
O2	713(4)	-4028(3)	10838(3)	20(1)
O3	-1789(4)	-3490(4)	10984(3)	32(1)
N1	-589(4)	-3496(3)	10561(3)	16(1)
N2	-4678(4)	-1673(3)	9740(3)	22(1)
C1	-1603(4)	-1355(3)	7962(3)	13(1)
C2	-1889(4)	-2062(3)	9038(3)	14(1)
C3	-594(4)	-2773(3)	9450(3)	15(1)
C4	820(4)	-2773(3)	8760(3)	15(1)
C5	1370(4)	-1308(3)	8668(3)	13(1)
C6	-118(4)	-462(3)	8273(3)	15(1)
C7	-1198(4)	-2415(4)	7111(3)	17(1)
C8	249(4)	-3262(4)	7583(3)	17(1)

C9	-3393(4)	-1875(3)	9492(3)	15(1)
H1	-3066(38)	-1125(34)	8133(33)	21(3)
H4	1778(37)	-3366(35)	9151(32)	21(3)
H5	2149(37)	-1518(34)	7950(32)	21(3)
H6	-544(36)	124(36)	8885(32)	21(3)
H71	-775(38)	-1997(34)	6426(31)	21(3)
H72	-2233(37)	-2896(34)	6852(32)	21(3)
H81	-86(38)	-4256(34)	7566(35)	21(3)
H82	610(38)	-2948(36)	7195(32)	21(3)
H0	3104(39)	-543(33)	9657(34)	21(3)

5.3 Discussion

By determining the structure of VI at ambient temperature and at 100K, and keeping all other experimental conditions as nearly identical as possible it was possible to show that by reducing the thermal motion of the atoms in the crystal, the determination of hydrogen positions in the presence of heavy elements becomes feasible. The results are somewhat detracted from by the large residual electron density and the elongated thermal ellipsoids. These effect are almost certainly due to absorption, or other effects relating to the large crystal used. In this case the optimum crystal thickness, given by $t_{opt}=2/\mu$,where μ is the linear absorption coefficient of the crystal, would be about 0.5mm. The crystal is thus almost twice the optimum size based on absorption effects alone. More importantly though, for a crystal of this size it is quite possible that the whole of it is not exposed to a homogeneous beam. This could introduce systematic errors into the data that cannot be corrected for. It can only be hoped that since the

structural parameters seem reasonable the systematic errors introduced in this manner, were not too severe.

When comparing the two structures of VI, shown in stereographic projection in figures 5.3 and 5.4, it is apparent that the hydroxyl hydrogen position is different for the two structures. On closer inspection it is evident that there are differences in the positions of the other hydrogens as well. This is most likely due to the manner in which the hydrogen positions are determined for the ambient temperature structure. That is, they were placed in geometrically calculated positions. This procedure tends to work well for hydrogen atoms attached to carbon, but generally fails when dealing with hydrogens attached to other elements. The reason for this is that only the geometries associated with carbon are provided for by the refinement program.

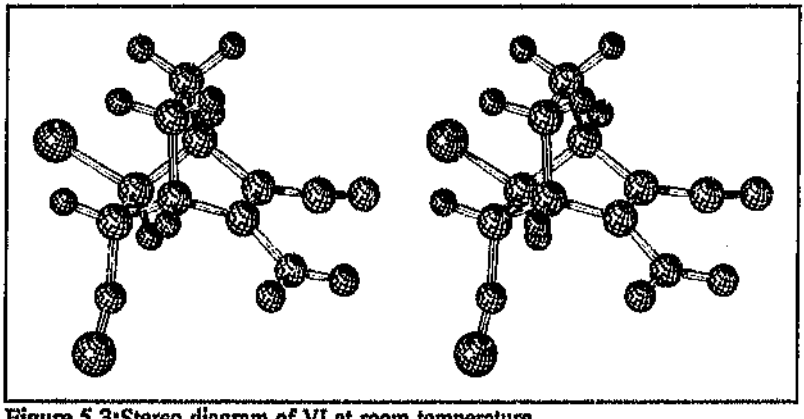


Figure 5.3:Stereo diagram of VI at room temperature

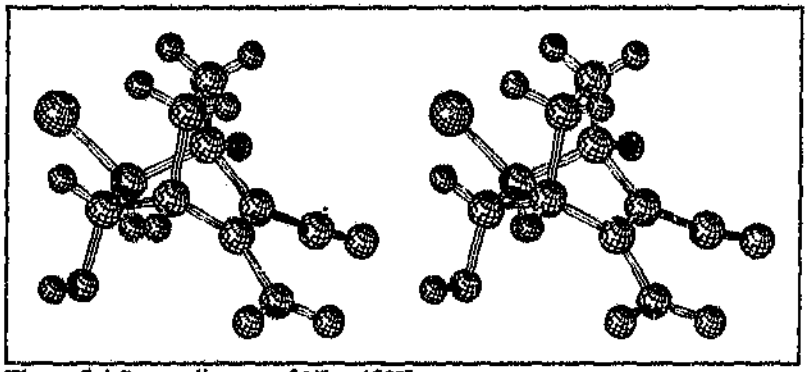


Figure 5.4:Stereo diagram of VI at 100K

Chapter Six

Experimental, equipment and techniques.

6.1 Introduction

Essentially the cryostats used in low temperature x-ray diffraction experiments may be divided into two categories, depending on the cryogen used. For liquid helium the cryostat can be based on a liquid flow cryostat or it can incorporate a closed cycle helium refrigerator. The latter having the advantage of allowing for long term data collections. Liquid helium cryostats require that the crystal under study be enclosed in the cryostat, generally using Be windows, to reduce thermal losses. Liquid nitrogen cryostats on the other hand are generally of the continuous gas flow type. In this case a stream of cold nitrogen gas is used to cool the crystal to the desired temperature, typically in the range 100K to 273K.

In this project two cryostats, a liquid helium flow cryostat and a nitrogen gas flow cryostat, designed and manufactured by Enraf Nonius, were assembled, commissioned and tested. The Helium cryostat was further modified to allow it to operate using liquid nitrogen as cryogen as well.

Although an operational cryostat would be the first step to performing a low temperature diffraction experiment, a number of other factors also need to be taken into

consideration to fully exploit the capabilities of the technique. The most important among these would be the alignment of the diffractometer as well as the centring of the crystal within the x-ray beam.

6.2 CAD4 alignment.

After aligning the diffractometer a few times according to the method set out in the operators manual, it was found that the method could be improved by slight modification.

For the alignment, the CAD4 routine ALIGN and a suitable crystal, ie. a good, stable diffractor free of absorption problems is required. For the routine ALIGN at least one reflection with $80 \leq \chi \leq 100$ (eulerian diffractometer angles are defined in figure 6.1) is required in the reflection list. (two or more reflections give more consistent results). These reflections have to be found and centred before invoking ALIGN, which would then accurately measure the positions of the eight equivalents of the selected reflection. From this the errors in the alignment are calculated and corrected for as set out in table 6.1 using the adjusters indicated in figures 6.2 and 6.3. Note that the vertical may be adjusted in one of two places, indicated as a course and a fine adjustment. Generally it should only be necessary to use the course adjustment, the fine adjustment being more intimately linked with the monochromator. During the final stages of alignment or when a very accurate alignment is desired, the centring of the crystal in the x-ray beam will become crucial, and should also be corrected for according to the coordinate axis set out in figure 6.4.

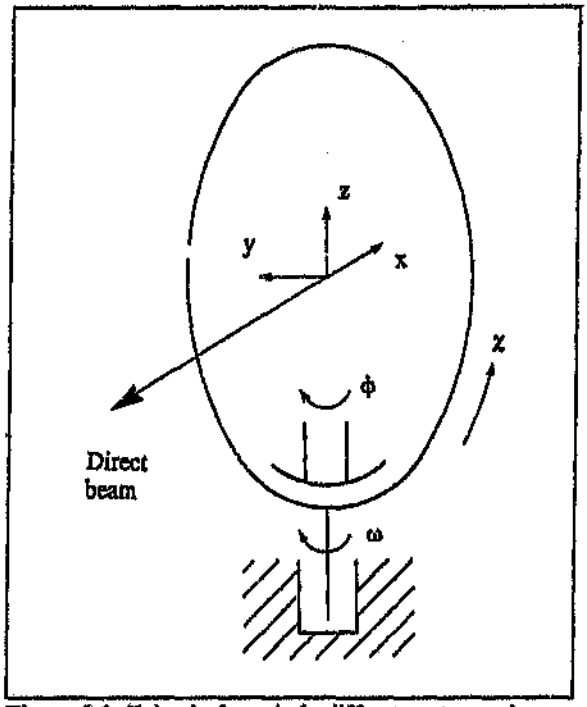


Figure 6.1: Eulerain four circle diffractometer angles.

Table 6.1: Alignment correction to perform if reported error is greater than zero.

Deter(mm)	Move detector towards negative θ.
TubeHo(mm)	Move tubeshield towards HV cable, ie turn anti-clockwise.
CrystX(mm)	Use VIEW 0, move crystal towards left when looking through the telescope.
CrystY(mm)	Use VIEW 0, set ϕ_k to 101°, move crystal left when looking through the telescope.
CrystX(mm)	Move crystal down.
Mondeg(°)	Increase monochromator angle.
TubeVe(mm)	Coarse adjustment: Move tubeshield down, ie turn anticlockwise. Fine adjustment: Move tubeshield down.

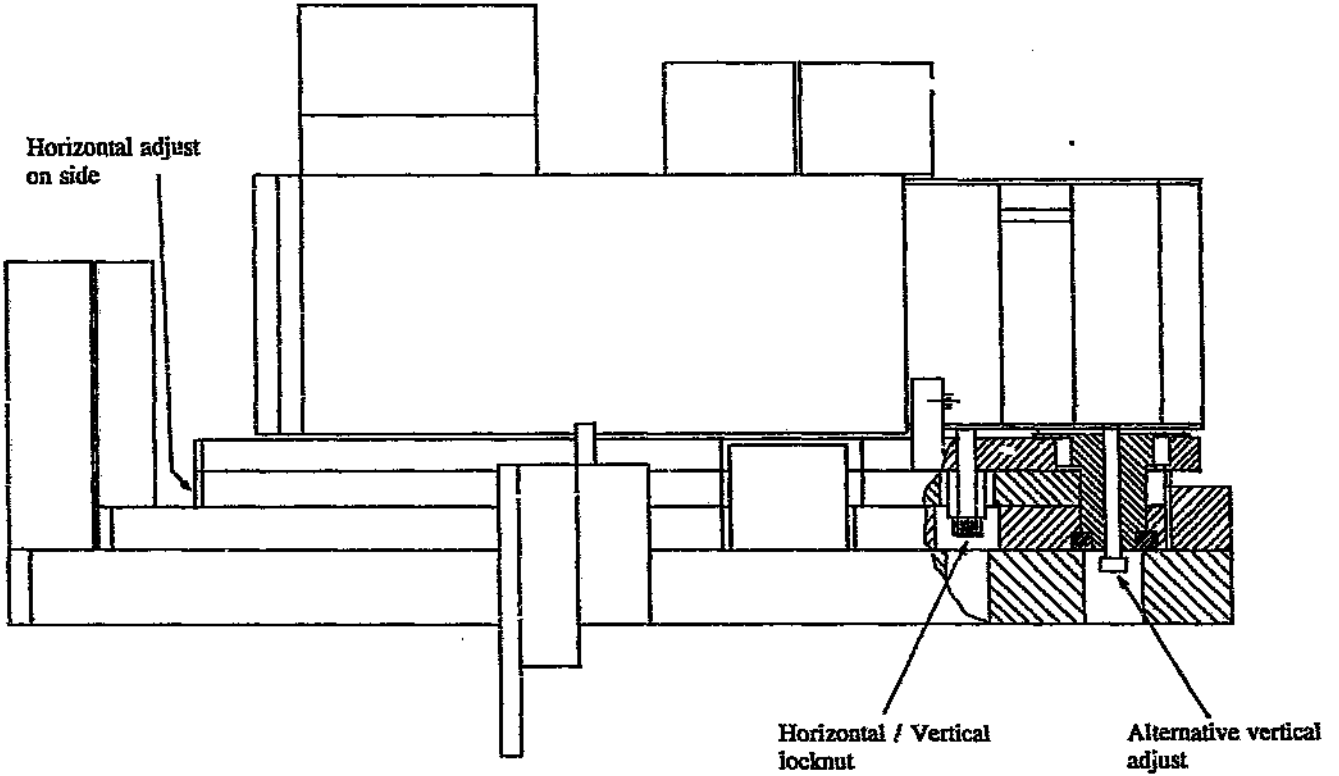


Figure 6.2: Rear view of tubeshield

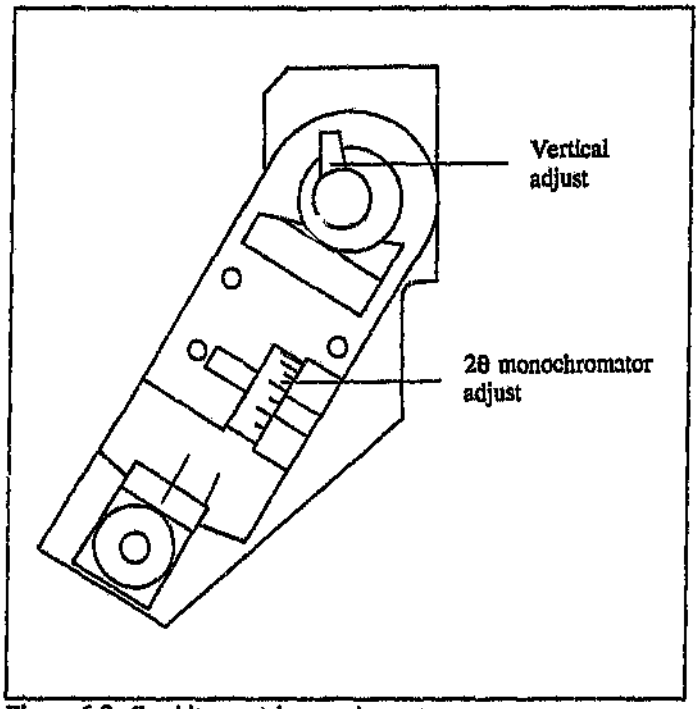


Figure 6.3: Graphite crystal monochromator.

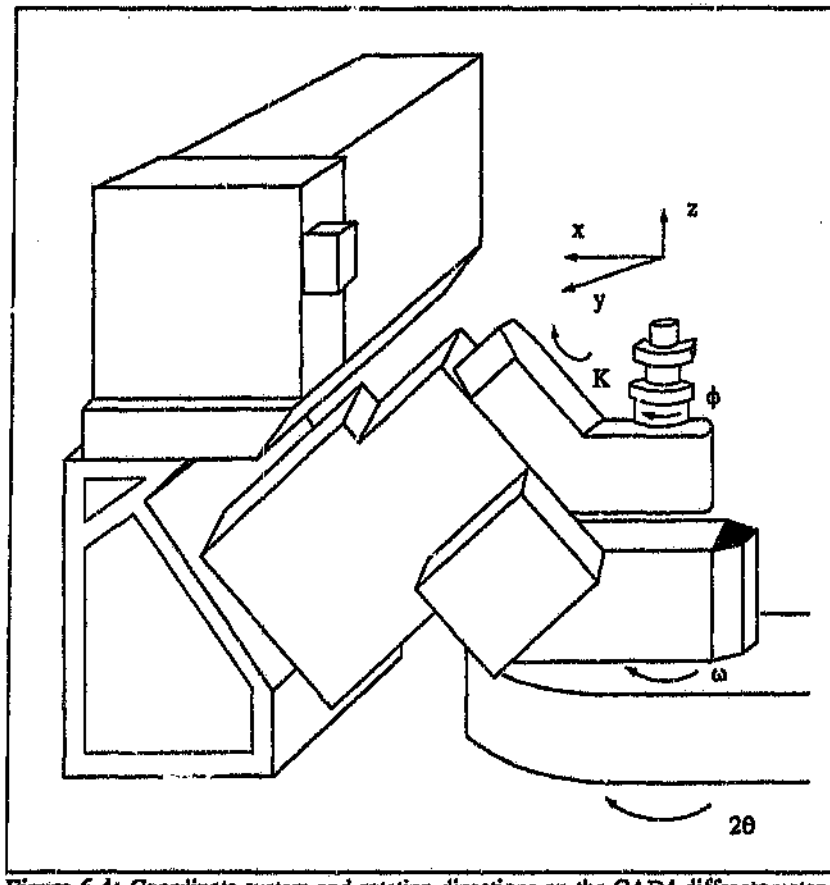


Figure 6.4: Coordinate system and rotation directions on the CAD4 diffractometer.

6.3 Operation of the LN_2 gas flow cryostat

The cryostat when assembled as shown in figure 6.5, cools the crystal by boiling of nitrogen and then blowing it over the crystal. In this, condensation and icing can be a problem. Icing on the goniometer head is minimised by surrounding the cold stream with a concentric warm one. To get around all the other possible condensation problems it is necessary to seal the CAD4 enclosure and to then dry the inside atmosphere to below 30% relative humidity. This is most readily accomplished by the use of silica gel.For the best temperature stability it is important that the level of liquid nitrogen in the transfer dewar be kept as constant as possible. This is done by regular refills from a large (100l) supply dewar. It is vitally important that the level sensors in the transfer dewar are in proper working order. Finally by stepping through reciprocal space in a different pattern ice formation could also by limited and the quality of the data improved.

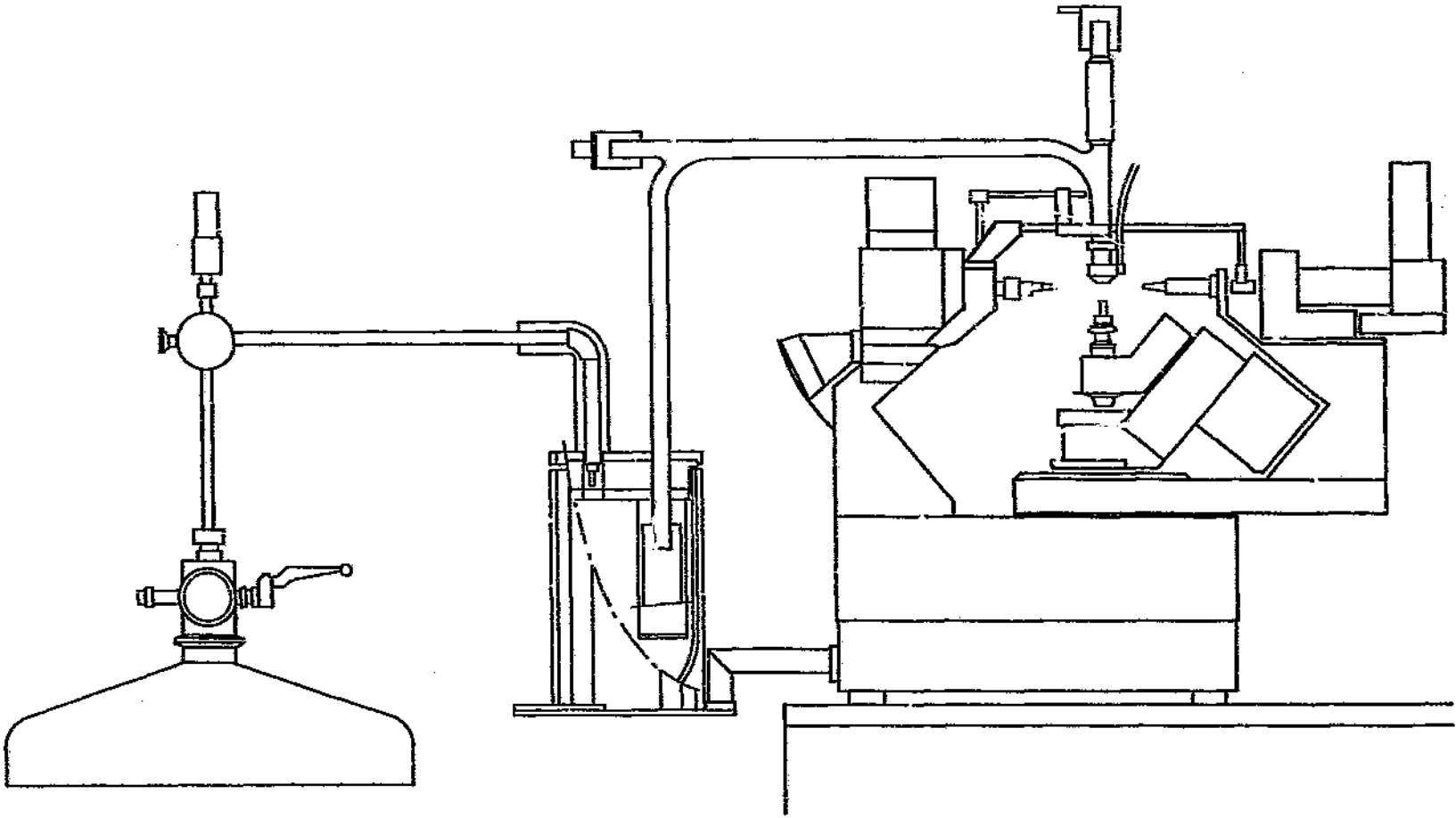


Figure 6.5: Assembled gas flow cryostat.

6.4 Operation of the Be walled flow cryostat.

The flow cryostat cools the crystal by keeping it in a cold chamber, to which a heat exchanger is attached. Coolant is then allowed to pass through the heat exchanger. The beryllium walled cryostat, when assembled as in figure 6.6, may be used with either liquid helium or with liquid nitrogen acting as cryogen. It is important to note that when using liquid nitrogen as cryogen the diffusion pump should be on for the duration of the experiment, so as to provide sufficiently good insulating vacuum. This is not necessary when using liquid helium as it operates as a cryo-pump, thus producing its own insulating vacuum.

The crystal is mounted on an insulating cup, as shown in figure 6.7, the one supplied with the cryostat, is double walled, with a vacuum in between the walls. This provides excellent insulation between the cold chamber and the goniometer. When a cup with inferior insulating properties is used then condensation is likely to occur on the goniometer head and possibly on the outside of the cryostat. At 15K the cryostat consumes approximately 30 litres of liquid helium per day. Because of the higher heat capacity of liquid nitrogen the cryostat is far more efficient when it is used. In the latter case the minimum temperature attainable is approximately 90K.

Compared to the gas flow cryostat, where a lot of scope for variations exists, the flow cryostat requires a far more detailed and rigid routine to successfully mount and operate it. In this respect the following procedure was found to work and is thus recommended.

1.Set software and hardware limits

2.Mount crystal on CAD4

3.Centre the crystal optically.

4.Get an orientations matrix (by searching for reflections and then indexing those found).

5.Mount the cryostat.

6.Check the orientation matrix. This is in case the crystal got knocked off the mount whilst mounting the cryostat. The quality of the cell would have deteriorated somewhat due to the absorption of the x-rays by the beryllium windows of the cryostat.

7.Purge the cold chamber with high purity helium.

8.Again check the orientation matrix.

9.Start cooling down by switching on the pump and opening the necessary valves. While cooling down follow the orientation matrix by reentering the reflections used for its determination. This is useful as the crystal will most definitely move during this stage of the experiment.

10.When the temperature has stabilised recentre the crystal using the four equivalents as set out in the next section.

11.When the crystal has been centered, data collection may proceed as normal.

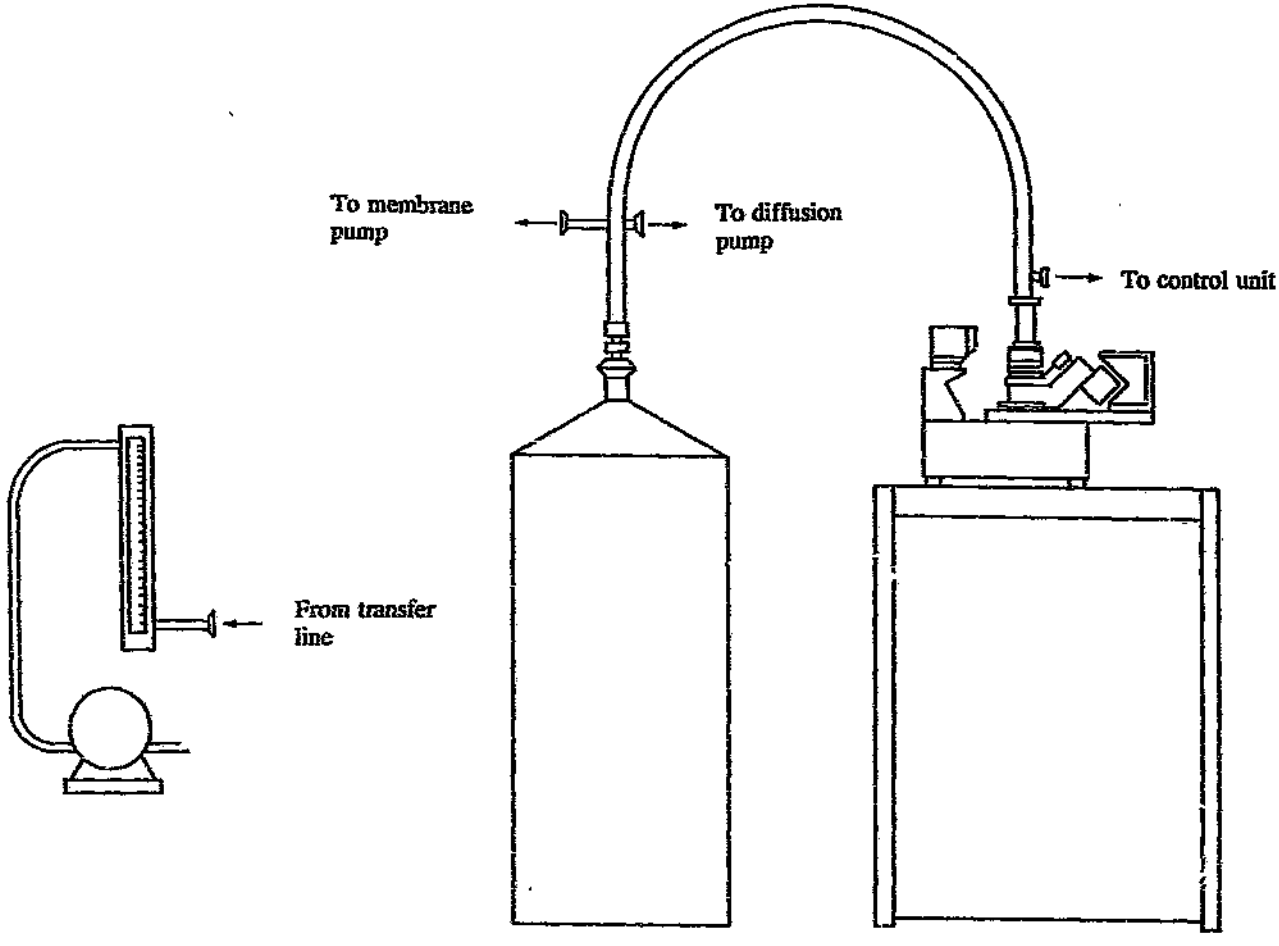


Figure 6.6: Assembled Be-walled flow cryostat.

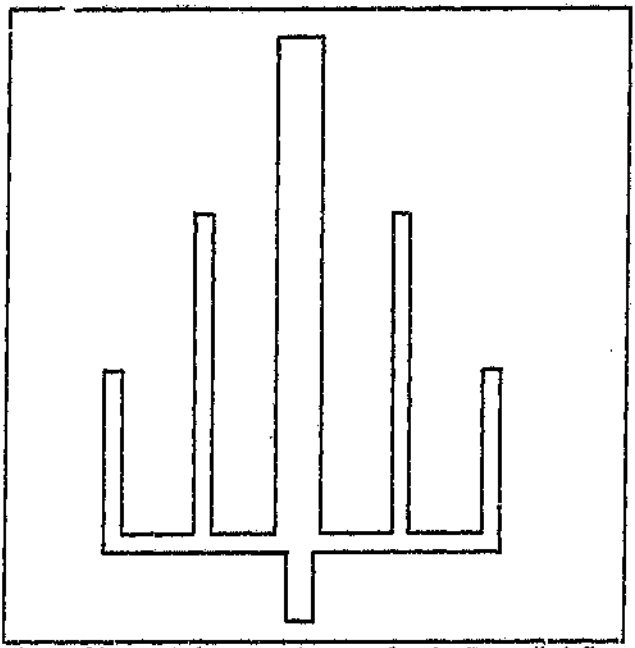


Figure 6.7: Insulating mounting cup for the Be walled flow cryostat.

6.5 Centring

As has been mentioned previously, accurately centring the crystal is very important when intending to collect an accurate data set. The desired centring precision is often greater than can be obtained optically when using a telescope. Furthermore with some experimental configurations it is not possible to see the crystal. Hence it is necessary to centre the crystal by using the equivalent reflections, of which there are eight on a four-circle diffractometer. When the motion of the goniometer is not restricted, then all eight of these equivalents are accessible and the crystal may be centred using the routine 'ALIGN' in the same manner as when aligning the diffractometer. However when the Beryllium-walled flow cryostat is mounted on the goniometer, the motion of the Kappa

arm necessarily becomes restricted to $-60° \leq K \leq 60°$. Thus making it impossible to reach high χ values. The resultant of this is that only four of the eight settings listed in table 6.2 [Ibers and James, 1974] are accessible.

Table 6.2:Eulerian angles for the eight equivalent settings accessible on a four circle diffractometer.				
Indices				
hkl	ϕ	χ	2θ	ω
-h-k-l	$\pi+\phi$	$-\chi$	2θ	ω
hkl	ϕ	χ	-2θ	$-\omega$
-h-k-l	$\pi+\phi$	$-\chi$	-2θ	$-\omega$
hkl	$\pi+\phi$	$-\chi$	2θ	$-\omega$
-h-k-l	ϕ	χ	2θ	$-\omega$
hkl	$\pi+\phi$	$-\chi$	-2θ	ω
-h-k-l	ϕ	χ	-2θ	ω

Table 6.3:Observed χ and 2θ values for the accessible settings.			
Setting	Indices	Observed χ	Observed 2θ
1	hkl	$\chi+\chi_o+\Delta\chi_{zl}+\Delta\chi_c$	$2\theta+2\theta_o+S_x-S_y+C_x+C_y$
2	-h-k-l	$-\chi+\chi_o+\Delta\chi_{zl}+\Delta\chi_c$	$2\theta+2\theta_o-S_x+S_y-C_x-C_y$
3	hkl	$-\chi+\chi_o-\Delta\chi_{zl}-\Delta\chi_c$	$-2\theta+2\theta_o+S_x+S_y+C_x-C_y$
4	-h-k-l	$\chi+\chi_o-\Delta\chi_{zl}-\Delta\chi_c$	$-2\theta+2\theta_o-S_x-S_y-C_x+C_y$

The various terms added to and subtracted from χ and 2θ to get the observed values listed in table 6.3, come from the different errors that are caused by a badly centred

crystal. From the observed χ values the following expression was derived, during this project, for Δz, the error in the vertical height of the crystal.

$$\Delta z = \left[4\chi_m - \frac{\Delta h}{2R_c \sin\theta}\right]\left(\frac{2\sin\theta}{\left[\frac{1}{R_s}+\frac{1}{R_c}\right]}\right)$$

where Δh = Error in detector height

R_c = Crystal to counter distance

R_s = Crystal to source distance

$\chi_m = \chi_1+\chi_2-\chi_3-\chi_4$, χ_i is the observed χ value for setting 1.

Similarly the errors Δx and Δy in the diffraction plane may be derived from the observed values of 2θ, resulting in the following relationships.

$$\Delta x = \frac{\theta_x}{4\sin\theta\left[\frac{1}{R_s}+\frac{1}{R_c}\right]}$$

$$\Delta y = \frac{\theta_y}{4\cos\theta\left[\frac{1}{R_s}-\frac{1}{R_c}\right]}$$

where $\theta_x = \theta_1-\theta_2+\theta_3-\theta_4$

$\theta_y = \theta_2+\theta_3-\theta_1-\theta_4$, θ_i is the observed 2θ value for setting 1.

Thus for a well aligned diffractometer, the crystal may be centred using information from only four settings of the equivalents. These ideas have been incorporated into a

FORTRAN program CENTRE, a listing of which is included in the appendix.

6.6 Crystal mounting

The seemingly simple operation for routine work is often the cause of great frustration when it comes to low-temperature studies. This is because a contraction of the mount as well as changes in the properties of the adhesive used can take place. Contraction generally leads to the crystal moving away from the centre of the x-ray beam. This is readily corrected for by reentering the crystal when the temperature has stabilised. The adhesive used can cause more severe problems as some crack on cooling to a very low temperature. This could lead to the crystal falling of the mount or to it being damaged as well. High vacuum grease was found to be the most effective for attaching a crystal to a glass fibre. Mounting crystals in capillaries is not recommended, as the capillary still introduces absorption effects noticeable in high resolution studies, it may require elaborate correction of the data, as is done by the program of De Titta (). Also the crystal is often damaged when it is inserted into the capillary.

6.7 Conclusion

The Be-walled flow cryostat has a temperature range of 10K to ambient temperature. For work below 90K liquid helium needs to be used instead of liquid nitrogen, making data collection at such low temperatures very costly. Also the time spent collecting data is limited as it is not possible to refill the supply dewar while the cryostat is operational. That is after about 3 days when using liquid helium or about a week for liquid nitrogen,

the cryostat must necessarily be allowed to warm up to room temperature. If data collection is to be continued after refilling the supply dewar, then the crystal will have to be re-centered after cooling down and reaching a stable temperature. At this stage it might not be possible to stabilize the cryostat at exactly the same temperature as before. Thus if data is to be collected in this manner then it might be neccessary to scale each set of data independently. This and the limited area of reciprocal space accessible with the cryostat makes it very difficult to collect extended accurate data sets with it. It is probably more appropriate to use this cryostat for the study of phase transitions in crystals with relatively small unit cells.

In the case of the liquid nitrogen gas-flow cryostat, data collection could in principle be continued indefinitely, as it is possible to refill the supply dewar without affecting the stability of the cryostat. This makes this cryostat extremely suitable for the collection of accurate data requiring long counting times as well as the collection of many equivalents. Such data sets would be suitable for charge density and other studies that require very accurate data.

It was possible to show that the temperature stability of both cryostats is better than 1K with the use of a Chromel-alumel thermocouple. The absolute temperatures attainable with the cryostats were also verified with this thermocouple.

Chapter Seven.

Future trends and developments.

For routine purposes only data contained within the copper sphere of reflection need generally be collected, this corresponds to a resolution of about 0.8Å. This is sufficient to resolve atomic positions and to produce the conformational data that is generally required from such studies. When a low-temperature diffraction experiment is combined with an attempt to remove all systematic errors during data capture, it becomes possible to obtain more information about the molecules in a crystal than just their xyz coordinates. To distinguish this technique from routine work Angermund, Claus, Goddard and Kruger (1985) in their review article referred to it as high resolution crystallography. In a high resolution study, typically the full Ewald sphere of reflections should be collected. The resolution attainable using this technique is of the order of 0.4 to 0.5Å.

With the aid of accurate data obtained from a high resolution x-ray diffraction experiment, it becomes feasible to look at the electron density distribution in detail. This should lead to a better understanding of chemical bonding [Coppens, 1984]. The total electron density obtained, however is rather difficult to interpret as it is dominated by the core electrons, even though this is a complete representation of the nature of the bonding in the molecule. A more useful representation is contained in the concept of an electron deformation density, [Coppens, 1989; Schwartz, Valtazanos and Ruedenberg,

1985]. In an analysis involving this concept, the spherically symmetric electron density of a free atom is subtracte ' from that found experimentally for the atom in a crystal. Thus information is at hand as to how the electron density associated with the atom changed upon bond formation or interaction in a crystal. This exercise is a very definite attempt to answer the question posed by C.A Coulson in 1971 when he said: "Clearly a molecule is much more complicated than an atom. So the question arises: what happens to an atom when it becomes part of a molecule?" [Coppens, 1977]

Another field of activity to gain from the availability of more accurate Bragg reflection data, is the interpretation of anisotropic displacement parameters (ADP's) in terms of molecular motion. What were originally termed anisotropic "vibrational parameters" or "thermal parameters" are more precisely described as anisotropic Gaussian displacement parameters. This indicates that they are not only influenced by thermal motion but are subject to disorder effects as well. The need for accurate data, stems from the recognitic that in routine structures the ADP's are frequently adversely affected by absorption, truncation or some other systematic error in the data. This has led to some authors referring to ADP's as the rubbish bin of crystallography, since most of the errors seem to collect in them even without affecting other structural parameters severely. The usefulness of ADP's were first realised by Cruickshank (1956) when he showed that such quantities for anthracene and naphthalene could be interpreted in terms of molecular rigid-body motion and hence related to spectroscopic and thermodynamic properties [Cruickshank, 1956b]. A more recent treatment by Dunitz, Schomaker and Trueblood (1988) shows how force constants, barriers to rotation and frequencies can be derived from ADP's, by treating the molecule a rigid or semi- rigid body. A more

graphical approach to this is presented by Hummel, Roselli and Burgi (1990)

Lastly the advances in high resolution crystallography is probably best exemplified by the work of Paturle, Graafsma Boviatis, Legrand, Coppens, Kvick and Wing (1989) in which they studied the influence of an external electric field on the x-ray scattering of the non-linear optical crystal 2-methyl-4-nitroaniline. The most astounding feature of this work was their ability to detect variations in integrated intensity caused by the applied field of the of the order of 0.1%.

High resolution crystallography is the logical next step to determining crystal structures at low-temperatures. This is because it requires the same experimental equipment. It only demands greater care during data capture and better data reduction methods. This greatly increases the number of applications of modern single crystal x-ray diffraction, especially within a field such as chemistry.

Chapter Eight

Concluding Discussion

During the course of this project, the main objectives set out at the start were achieved. That is the cryostats have been assembled and tested on the diffractometers also the experimental methods and techniques necessary for collecting data at low-temperatures have been developed. Initial tests relating to temperature stability and the minimum temperature reachable at the crystal position were conducted successfully. The cryostats were found to have a temperature stability of better than 1K, making them suitable for collecting accurate data. The use of the Be-walled flow cryostat in this respect is somewhat restricted due to the limited movement around the kappa axis of the diffractometer. Also its supply dewar cannot be refilled whilst the cryostat is operational.

A final evaluation of the cryostats as well as a consideration of some of the applications of low-temperature crystallography involved the determination of some crystal structures at low-temperatures. The compounds chosen for this were :N-arylpyrrolidin-2-one,4-p-hydroxyphenyl-9,10,11-trihydroxy-8-ethyl-1,7-dioxaspiro[4,4]-nona-2,6-dioneand5,6-trans,-6-Bromo-5-hydroxy-3-nitrobicyclo[2.2.2]oct-2-ene. The structure of the first was determined at three different low-temperatures.

Experience gained from these studies indicate that even with an operational cooling device, the collection of low-temperature diffraction data is not straight forward.

Unexpected events such as the crystal shattering or moving on cooling can always take place.

The consideration of hydrogen atom positions, which motivated the study of 4-p-hydroxyphenyl-9,10,11-trihydroxy-8-ethyl-1,7-dioxaspiro[4,4]-nona-2,6-dioneand5,6-trans,-6-Bromo-5-hydroxy-3-nitrobicyclo[2.2.2]oct-2-ene, as well as that of leucodrin, indicated that the technique has considerable application in this field, as the positions given to hydrogen atoms from ambient temperature data is often suspect, especially if they are associated with atoms other than carbon. In this respect, low-temperature structure determination offers significantly better and more reliable structural parameters for the hydrogen atoms, in particular and for all atoms in general.

Appendix 1

All distance measurements are quoted in Å and angles in °

Selected Bond lengths for N-phenylpyrrolidin-2-one at 160K			
C1 -N	1.365(2)	C4 -N	1.478(2)
C5 -N	1.415(2)	C1 -O	1.222(2)
C2 -C1	1.509(2)	C3 -C2	1.518(2)
C4 -C3	1.516(2)	C6 -C5	1.396(2)
C10 -C5	1.387(2)	C7 -C6	1.384(2)
C8 -C7	1.382(3)	C9 -C8	1.381(3)
C10 -C9	1.383(2)		
H21 -C2	.959(16)	H22 -C2	.932(19)
H31 -C3	.990(18)	H32 -C3	.961(19)
H41 -C4	.905(18)	H42 -C4	.933(17)
H6 -C6	.937(17)	H7 -C7	1.035(19)
H8 -C8	.921(17)	H9 -C9	.951(17)
H10 -C10	.958(18)		

Selected Bond angles for N-phenylpyrrolidin-2-one at 160K			
C4 -N -C1	111.9(1)	C5 -N -C1	126.2(1)
C5 -N -C4	121.4(1)	O -C1 -N	125.5(1)
C2 -C1 -N	108.1(1)	C2 -C1 -O	126.4(1)
C3 -C2 -C1	104.4(1)	C4 -C3 -C2	103.8(1)
C3 -C4 -N	103.2(1)	C6 -C5 -N	121.3(1)
C10 -C5 -N	119.3(1)	C10 -C5 -C6	119.4(1)
C7 -C6 -C5	119.9(2)	C8 -C7 -C6	120.7(2)
C9 -C8 -C7	119.1(2)	C10 -C9 -C8	121.0(2)
C9 -C10 -C5	119.9(2)		
H21 -C2 -C1	114.1(10)	H21 -C2 -C3	112.1(10)
H22 -C2 -C1	107.6(10)	H22 -C2 -C3	111.1(11)

H22 -C2 -H21	107.5(14)	H31 -C3 -C2	112.8(10)
H31 -C3 -C4	111.2(9)	H32 -C3 -C2	111.1(10)
H32 -C3 -C4	1[illegible].4(9)	H32 -C3 -H31	107.6(14)
H41 -C4 -N	106.8(11)	H41 -C4 -C3	114.8(10)
H42 -C4 -N	106.4(11)	H42 -C4 -C3	116.5(11)
H42 -C4 -H41	108.3(14)	H6 -C6 -C5	118.4(11)
H6 -C6 -C7	121.8(11)	H7 -C7 -C6	118.0(8)
H7 -C7 -C8	121.2(8)	H8 -C8 -C7	126.3(12)
H8 -C8 -C9	114.5(12)	H9 -C9 -C8	120.6(11)
H9 -C9 -C10	118.3(11)	H10 -C10 -C5	120.3(10)
H10 -C10 -C9	119.7(10)		

Fractional coordinates ($\times 10^4$) and equivalent isotropic temperature factors ($Å^2, \times 10^3$) for N-phenylpyrr lidin-2-one at 90K

	x/a	y/b	z/c	U_{eq}
N	1250(2)	740(5)	4092(1)	15(1)
O	1429(1)	-1921(5)	4903(1)	17(1)
C1	1355(2)	-144(6)	4800(2)	13(1)
C2	1394(2)	1404(7)	5407(2)	17(1)
C3	1014(2)	3245(7)	5007(2)	19(1)
C4	1269(2)	2883(7)	4154(2)	16(1)
C5	1261(2)	-200(6)	3359(2)	12(1)
C6	870(2)	-2066(7)	3264(2)	16(1)
C7	844(2)	-2922(7)	2535(2)	19(1)
C8	1218(2)	-1919(7)	1893(2)	19(1)
C9	1624(2)	-64(7)	1999(2)	15(1)
C10	1650(2)	792(7)	2730(2)	18(1)
H7	564(22)	-4242(66)	2478(16)	17(3)
H10	1973(22)	1853(71)	2806(17)	17(3)
H8	1191(21)	-2469(57)	1417(16)	17(3)

H41	778(20)	3566(61)	3752(15)	17(3)
H9	1953(21)	403(63)	1610(16)	17(3)
H6	632(20)	-2623(56)	3704(16)	17(3)
H42	1923(21)	3224(63)	3978(15)	17(3)
H21	2080(21)	1378(64)	5620(15)	17(3)
H22	1630(20)	819(58)	5076(15)	17(3)
H31	270(21)	3484(57)	5031(15)	17(3)
H32	1342(20)	4450(63)	5181(16)	17(3)

Selected Bond lengths for N-phenylpyrrolidin-2-one at 90K			
C1 -N	1.372(4)	C4 -N	1.466(5)
C5 -N	1.421(4)	C1 -O	1.229(5)
C2 -C1	1.490(5)	C3 -C2	1.527(6)
C4 -C3	1.536(4)	C6 -C5	1.392(5)
C10 -C5	1.388(4)	C7 -C6	1.390(4)
C8 -C7	1.401(5)	C9 -C8	1.395(6)
C10 -C9	1.392(4)		
H21 -C2	1.014(28)	H22 C2	1.061(28)
H31 -C3	1.037(28)	H32 -C3	.985(38)
H41 -C4	1.075(30)	H42 -C4	.979(29)
H6 -C6	.912(29)	H7 -C7	.985(42)
H8 -C8	.906(29)	H9 -C9	.872(30)
H10 -C10	.860(42)		

Selected Bond angles for N-phenylpyrrolidin-2-one at 90K			
C4 -N -C1	111.8(3)	C5 -N -C1	126.6(3)
C5 -N -C4	121.0(3)	O -C1 -N	124.8(3)
C2 -C1 -N	108.7(4)	C2 -C1 -O	126.4(3)
C3 -C2 -C1	104.5(3)	C4 -C3 -C2	103.0(3)
C3 -C4 -N	103.1(3)	C6 -C5 -N	120.9(3)
C10 -C5 -N	118.9(4)	C10 -C5 -C6	120.2(3)
C7 -C6 -C5	120.1(3)	C8 -C7 -C6	120.2(4)
C9 -C8 -C7	119.1(3)	C10 -C9 -C8	120.7(3)
C9 -C10 -C5	119.8(4)		
H21 -C2 -C1	106.1(20)	H21 -C2 -C3	120.0(23)
H22 -C2 -C1	106.1(20)	H22 -C2 -C3	120.6(19)
H22 -C2 -H21	98.3(22)	H3. -C3 -C2	116.7(20)
H31 -C3 -C4	106.8(15)	H32 -C3 -C2	113.0( )
H32 -C3 -C4	108.8(17)	H32 -C3 -H31	108.0(2
H41 -C4 -N	111.9(22)	H41 -C4 -C3	114.0(16)
H42 -C4 -N	103.3(25)	H42 -C4 -C3	118.1(17)
H42 -C4 -H41	106.0(26)	H6 -C6 -C5	115.0(22)
H6 -C6 -C7	124.9(23)	H7 -C7 -C6	119.0(17)
H7 -C7 -C8	120.8(17)	H8 -C8 -C7	120.2(24)
H8 -C8 -C9	120.8(23)	H9 -C9 -C8	115.9(26)
H9 -C9 -C10	122.3(25)	H10 -C10 -C5	119.3(21)
H10 -C10 -C9	120.4(21)		

Fractional coordinates ($\times 10^4$) and equivalent isotropic temperature factors ($Å^2, \times 10^3$) for N-phenylpyrrolidin-2-one at 100K				
	x/a	y/b	z/c	U_{eq}
N	1252(1)	717(3)	4091(1)	33(0)
O	1432(1)	-1924(3)	4902(1)	29(0)

C1	1354(2)	-142(4)	4791(2)	26(1)
C2	1394(2)	1407(4)	5406(2)	26(1)
C3	1012(2)	3239(4)	4997(2)	29(1)
C4	1264(2)	2894(4)	4157(2)	28(1)
C5	1266(2)	-185(3)	3360(2)	24(0)
C6	866(2)	-2086(4)	3259(1)	25(1)
C7	850(2)	-2898(4)	2534(2)	30(1)
C8	1215(2)	-1928(4)	1895(2)	29(1)
C9	1627(2)	-67(4)	2004(2)	26(1)
C10	1645(2)	769(4)	2724(2)	26(1)
H21	2115(17)	1564(33)	5578(13)	31(2)
H22	1067(16)	1075(35)	5888(14)	31(2)
H31	248(17)	3306(35)	5074(13)	31(2)
H32	1288(16)	4380(37)	5214(14)	31(2)
H41	810(16)	3419(36)	3787(13)	31(2)
H42	1933(17)	3397(34)	4021(13)	31(2)
H6	652(15)	-2714(37)	3727(13)	31(2)
H7	564(17)	-4223(37)	2456(13)	31(2)
H8	1208(14)	-2428(39)	1342(14)	31(2)
H9	1872(16)	496(36)	1531(14)	31(2)
H10	1940(16)	2036(38)	2782(13)	31(2)

Selected Bond lengths for N-phenylpyrrolidin-2-one at 100K			
C1 -N	1.352(3)	C4 -N	1.489(3)
C5 -N	1.406(3)	C1 -O	1.235(3)
C2 -C1	1.500(3)	C3 -C2	1.529(3)
C4 -C3	1.513(3)	C6 -C5	1.419(3)
C10 -C5	1.381(3)	C7 -C6	1.370(3)
C8 -C7	1.383(4)	C9 -C8	1.402(4)

C10 -C9	1.370(4)		
H21 -C2	1.041(22)	H22 -C2	.973(24)
H31 -C3	1.061(22)	H32 -C3	.943(25)
H41 -C4	.963(23)	H42 -C4	1.011(23)
H6 -C6	.962(23)	H7 -C7	.996(24)
H8 -C8	1.013(24)	H9 -C9	.965(23)
H10 -C10	.960(24)		

Selected Bond angles for N-phenylpyrrolidin-2-one at 100K			
C4 -N -C1	111.3(2)	C5 -N -C1	127.8(2)
C5 -N -C4	120.3(2)	O -C1 -N	125.0(2)
C2 -C1 -N	109.5(2)	C2 -C1 -O	125.4(2)
C3 -C2 -C1	103.6(2)	C4 -C3 -C2	103.8(2)
C3 -C4 -N	103.0(2)	C6 -C5 -N	120.3(2)
C10 -C5 -N	121.0(2)	C10 -C5 -C6	118.6(2)
C7 -C6 -C5	119.2(2)	C8 -C7 -C6	122.2(2)
C9 -C8 -C7	118.2(3)	C10 -C9 -C8	120.4(2)
C9 -C10 -C5	121.4(2)		
H21 -C2 -C1	108.0(13)	H21 -C2 -C3	112.0(13)
H22 -C2 -C1	115.3(14)	H22 -C2 -C3	115.2(14)
H22 -C2 -H21	102.8(18)	H31 -C3 -C2	108.6(13)
H31 -C3 -C4	110.7(12)	H32 -C3 -C2	110.6(15)
H32 -C3 -C4	114.7(15)	H32 -C3 -H31	108.3(19)
H41 -C4 -N	108.2(14)	H41 -C4 -C3	115.5(14)
H42 -C4 -N	109.3(13)	H42 -C4 -C3	112.3(13)
H42 -C4 -H41	108.2(19)	H6 -C6 -C5	115.0(15)
H6 -C6 -C7	125.8(15)	H7 -C7 -C6	119.8(14)
H7 -C7 -C8	118.0(14)	H8 -C8 -C7	126.1(15)
H8 -C8 -C9	115.7(15)	H9 -C9 -C8	112.8(15)
H9 -C9 -C10	126.7(15)	H10 -C10 -C5	120.1(15)

H10 -C10 -C9	118.5(15)		

Fractional coordinates ($\times 10^4$) and equivalent isotropic temperature factors ($Å^2, \times 10^3$) for N-phenylpyrrolidin-2-one at Ambient temperature

	x/a	y/b	z/c	U_{eq}
N	1257(2)	711(3)	4044(1)	44(1)
O	1406(2)	-1882(3)	4861(1)	62(1)
C1	1342(2)	-142(5)	4745(2)	44(1)
C2	1377(2)	1421(5)	5358(2)	55(1)
C3	1035(3)	3214(5)	4938(2)	63(1)
C4	1282(3)	2860(4)	4098(2)	57(1)
C5	1279(2)	-210(4)	3323(2)	42(1)
C6	882(2)	-2069(4)	3230(2)	52(1)
C7	864(2)	-2903(5)	2511(2)	68(1)
C8	1222(2)	-1945(6)	1877(2)	71(1)
C9	1617(3)	-122(5)	1982(2)	58(1)
C10	1643(2)	728(5)	2696(2)	54(1)
H21	2091(2)	1621(5)	5567(2)	84(3)
H22	916(2)	1071(5)	5835(2)	84(3)
H31	280(3)	3385(5)	5011(2)	84(3)
H32	1393(3)	4497(5)	5148(2)	84(3)
H41	1975(3)	3423(4)	3958(2)	84(3)
H42	760(3)	3510(4)	3723(2)	84(3)
H6	597(2)	-2852(4)	3716(2)	84(3)
H7	556(2)	-4329(5)	2439(2)	84(3)
H8	1204(2)	-2618(6)	1316(2)	84(3)
H9	1905(3)	646(5)	1493(2)	84(3)
H10	1955(2)	2152(5)	2763(2)	84(3)

Selected Bond lengths for N-phenylpyrrolidin-2-one at ambient temperature			
C1 -N	1.357(3)	C4 -N	1.482(3)
C5 -N	1.404(4)	C1 -O	1.218(3)
C2 -C1	1.515(4)	C3 -C2	1.512(4)
C4 -C3	1.522(4)	C6 -C5	1.403(4)
C10 -C5	1.371(4)	C7 -C6	1.375(4)
C8 -C7	1.380(5)	C9 -C8	1.384(5)
C10 -C9	1.372(4)		
H21 -C2	1.080(0)	H22 -C2	1.080(0)
H31 -C3	1.080(0)	H32 -C3	1.080(0)
H41 -C4	1.080(0)	H42 -C4	1.080(0)
H6 -C6	1.080(0)	H7 -C7	1.080(0)
H8 -C8	1.080(0)	H9 -C9	1.080(0)
H10 -C10	1.080(0)		

Selected Bond angles for N-phenylpyrrolidin-2-one at ambient temperature			
C4 -N -C1	111.9(3)	C5 -N -C1	127.2(2)
C5 -N -C4	120.4(3)	O -C1 -N	125.4(3)
C2 -C1 -N	109.2(3)	C2 -C1 -O	125.4(3)
C3 -C2 -C1	103.2(2)	C4 -C3 -C2	105.1(3)
C3 -C4 -N	102.4(2)	C6 -C5 -N	120.6(3)
C10 -C5 -N	120.3(3)	C10 -C5 -C6	119.1(3)
C7 -C6 -C5	119.5(3)	C8 -C7 -C6	121.3(3)
C9 -C8 -C7	118.3(3)	C10 -C9 -C8	121.1(3)
C9 -C10 -C5	120.6(3)		
H21 -C2 -C1	110.9(2)	H22 -C2 -C1	111.2(2)
H22 -C2 -H21	109.5(0)	C3 -C2 -H21	110.8(2)
C3 -C2 -H22	111.1(2)	H31 -C3 -C2	110.3(2)

H32 -C3 -C2	110.8(2)	H32 -C3 -H31	109.5(0)
C4 -C3 -H31	110.8(2)	C4 -C3 -H32	110.3(2)
H41 -C4 -N	111.4(2)	H41 -C4 -C3	111.5(2)
H42 -C4 -N	111.1(2)	H42 -C4 -C3	110.9(2)
H42 -C4 -H41	109.5(0)	H6 -C6 -C5	120.7(2)
C7 -C6 -H6	119.8(2)	H7 -C7 -C6	119.4(2)
C8 -C7 -H7	119.3(2)	H8 -C8 -C7	120.5(2)
C9 -C8 -H8	121.2(2)	H9 -C9 C8	119.3(2)
C10 -C9 -H9	119.5(2)	H10 -C10 -C5	119.8(2)
H10 -C10 -C9	119.6(2)		

Appendix 2

Selected bond lengths for Leucodrin at 100K			
C2 -O1	1.365(1)	C5 -O1	1.440(1)
C6 -O7	1.333(1)	C8 -O7	1.459(1)
C2 -O18	1.203(1)	C6 -O19	1.207(1)
C9 -O20	1.415(1)	C10 -O21	1.434(1)
C11 -O22	1.419(1)	C15 -O23	1.363(1)
C3 -C2	1.509(1)	C4 -C3	1.535(1)
C5 -C4	1.555(1)	C12 -C4	1.514(1)
C6 -C5	1.530(1)	C9 -C5	1.534(1)
C9 -C8	1.545(1)	C10 -C8	1.518(1)
C11 -C10	1.524(1)	C13 -C12	1.405(1)
C17 -C12	1.396(1)	C14 -C13	1.387(1)
C15 -C14	1.397(1)	C16 -C15	1.391(2)
C17 -C16	1.391(1)		
H20 -O20	.874(15)	H21 -O21	.686(14)
H22 -O22	.745(14)	H23 -O23	.735(14)
H3a -C3	1.064(15)	H3b -C3	.964(15)
H4 -C4	1.024(14)	H8 -C8	1.083(12)
H9 -C9	1.005(13)	H10 -C10	.931(14)
H11a-C11	1.031(14)	H11b-C11	.926(14)
H13 -C13	.942(13)	H14 -C14	.900(14)
H16 -C16	.980(13)	H17 -C17	1.113(14)

Selected bond angles for Leucodrin at 100K			
C5 -O1 -C2	110.3(1)	C8 -O7 -C6	110.9(1)
O18 -C2 -O1	120.9(1)	C3 -C2 -O1	109.9(1)

C3 -C2 -O18	129.2(1)	C4 -C3 -C2	104.2(1)
C5 -C4 -C3	101.2(1)	C12 -C4 -C3	117.9(1)
C12 -C4 -C5	115.3(1)	C4 -C5 -O1	104.8(1)
C6 -C5 -O1	108.1(1)	C6 -C5 -C4	112.6(1)
C9 -C5 -O1	113.0(1)	C9 -C5 -C4	115.6(1)
C9 -C5 -C6	102.7(1)	O19 -C6 -O7	122.2(1)
C5 -C6 -O7	111.3(1)	C5 -C6 -O19	126.4(1)
C9 -C8 -O7	106.5(1)	C10 -C8 -O7	108.5(1)
C10 -C8 -C9	114.9(1)	C5 -C9 -O20	114.2(1)
C8 -C9 -O20	108.4(1)	C8 -C9 -C5	103.4(1)
C8 -C10 -O21	107.7(1)	C11 -C10 -O21	108.5(1)
C11 -C10 -C8	112.5(1)	C10 -C11 -O22	107.8(1)
C13 -C12 -C4	122.9(1)	C17 -C12 -C4	119.4(1)
C17 -C12 -C13	117.7(1)	C14 -C13 -C12	121.2(1)
C15 -C14 -C13	120.1(1)	C14 -C15 -O23	117.6(1)
C16 -C15 -O23	122.9(1)	C16 -C15 -C14	119.5(1)
C17 -C16 -C15	120.0(1)	C16 -C17 -C12	121.5(1)
H20 -O20 -C9	111.6(9)	H21 -O21 -C10	103.9(13)
H22 -O22 -C11	106.4(11)	H23 -O23 -C15	112.4(12)
H3a -C3 -C2	109.1(8)	H3a -C3 -C4	110.9(8)
H3b -C3 -C2	105.6(8)	H3b -C3 -C4	117.4(9)
H3b -C3 -H3a	107.2(11)	H4 -C4 -C3	111.7(8)
H4 -C4 -C5	105.8(8)	H4 -C4 -C12	104.5(8)
H8 -C8 -O7	105.0(8)	H8 -C8 -C9	110.2(9)
H8 -C8 -C10	111.2(8)	H9 -C9 -O20	113.4(8)
H9 -C9 -C5	108.4(8)	H9 -C9 -C8	108.4(8)
H10 -C10 -O21	108.5(9)	H10 -C10 -C8	110.7(9)
H10 -C10 -C11	108.8(9)	H11a-C11 -O22	108.7(7)
H11a-C11 -C10	112.4(8)	H11b-C11 -O22	107.7(9)
H11b-C11 -C10	105.1(9)	H11b-C11 -H11a	115.0(11)

H13 -C13 -C12	117.7(9)	H13 -C13 -C14	121.1(9)
H14 -C14 -C13	118.7(9)	H14 -C14 -C15	120.8(9)
H16 -C16 -C15	118.8(8)	H16 -C16 -C17	121.2(9)
H17 -C17 -C12	118.0(7)	H17 -C17 -C16	120.5(7)

Selected bond lengths for Leucodrin at room temperature.			
C2 -O1	1.368(5)	C5 -O1	1.439(5)
C6 -O7	1.335(5)	C8 -O7	1.458(4)
C2 -O18	1.201(5)	C6 -O19	1.200(5)
C9 -O20	1.424(5)	C10 -O21	1.4[illegible]5)
C11 -O22	1.427(5)	C15 -O23	1.370(5)
C3 -C2	1.503(5)	C4 -C3	1.545(5)
C5 -C4	1.559(5)	C12 -C4	1.515(5)
C6 -C5	1.523(6)	C9 -C5	1.526(5)
C9 -C8	1.540(6)	C10 -C8	1.525(5)
C11 -C10	1.515(6)	C13 -C12	1.401(6)
C17 -C12	1.399(5)	C14 -C13	1.390(6)
C15 -C14	1.395(6)	C16 -C15	1.398(7)
C17 -C16	1.384(6)		
H20 -O20	1.080(0)	H21 -O21	1.080(0)
H22 -O22	1.080(0)	H23 -O23	1.080(0)
H3b -C3	1.080(0)	H3a -C3	1.080(0)
H4 -C4	1.080(0)	H8 -C8	1.080(0)
H9 -C9	1.080(0)	H10 -C10	1.080(0)
H11a-C11	1.080(0)	H11b-C11	1.080(0)
H13 -C13	1.080(0)	H14 -C14	1.080(0)
H16 -C16	1.080(0)	H17 -C17	1.080(0)

Selected bond angles for Leucodrin at room temperature			
C5 -O1 -C2	110.8(3)	C8 -O7 -C6	110.7(3)
O18 -C2 -O1	121.2(4)	C3 -C2 -O1	109.9(3)
C3 -C2 -O18	129.0(4)	C4 -C3 -C2	104.0(3)
C5 -C4 -C3	101.3(3)	C12 -C4 -C3	117.4(3)
C12 -C4 -C5	115.2(3)	C4 -C5 -O1	104.3(3)
C6 -C5 -O1	108.0(3)	C6 -C5 -C4	113.2(3)
C9 -C5 -O1	112.2(3)	C9 -C5 -C4	116.2(3)
C9 -C5 -C6	102.9(4)	O19 -C6 -O7	122.1(4)
C5 -C6 -O7	111.0(3)	C5 -C6 -O19	126.8(4)
C9 -C8 -O7	106.4(3)	C10 -C8 -O7	107.9(3)
C10 -C8 -C9	114.0(3)	C5 -C9 -O20	113.8(3)
C8 -C9 -O20	108.3(3)	C8 -C9 -C5	105.2(3)
C8 -C10 -O21	107.5(3)	C11 -C10 -O21	108.6(3)
C11 -C10 -C8	112.0(4)	C10 -C11 -O22	107.6(4)
C13 -C12 -C4	123.2(4)	C17 -C12 -C4	119.0(4)
C17 -C12 -C13	117.8(4)	C14 -C13 -C12	121.7(4)
C15 -C14 -C13	119.1(5)	C14 -C15 -O23	117.2(4)
C16 -C15 -O23	122.4(4)	C16 -C15 -C14	120.3(4)
C17 -C16 -C15	119.4(4)	C16 -C17 -C12	121.5(4)
C9 -O20 -H20	113.0(2)	C10 -O21 -H21	114.9(2)
C11 -O22 -H22	111.2(2)	C15 -O23 -H23	114.0(2)
H3b -C3 -C2	110.6(2)	H3a -C3 -C2	110.9(3)
H3a -C3 -H3b	109.5(0)	C4 -C3 -H3b	111.0(2)
C4 -C3 -H3a	110.8(2)	H4 -C4 -C3	111.6(3)
C5 -C4 -H4	115.2(2)	C12 -C4 -H4	97.0(2)
H8 -C8 -O7	114.2(2)	C9 -C8 -H8	108.0(2)
C10 -C8 -H8	106.6(2)	H9 -C9 -O20	105.8(2)
H9 -C9 -C5	110.6(2)	H9 -C9 -C8	115.4(2)
H10 -C10 -O21	112.5(2)	H10 -C10 -C8	108.7(2)

C11 -C10 -H10	107.6(2)	H11a-C11 -O22	109.8(2)
H11a-C11 -C10	110.1(2)	H11b-C11 -O22	110.1(2)
H11b-C11 -C10	109.8(2)	H11b-C11 -H11a	109.5(0)
H13 -C13 -C12	118.8(2)	C14 -C13 -H13	119.5(3)
H14 -C14 -C13	120.4(3)	C15 -C14 -H14	120.6(3)
H16 -C16 -C15	120.4(2)	C17 -C16 -H16	120.2(3)
H17 -C17 -C12	119.0(3)	H17 -C17 -C16	119.4(3)

Selected bond lengths for Conocarpin at room temperature			
C2 -O1	1.340(3)	C5 -O1	1.464(3)
C6 -O7	1.346(3)	C8 -O7	1.467(3)
C2 -O18	1.202(3)	C6 -O19	1.183(3)
C9 -O20	1.411(3)	C10 -O21	1.422(3)
C11 -O22	1.433(3)	C15 -O23	1.375(3)
C3 -C2	1.487(3)	C4 -C3	1.526(4)
C5 -C4	1.546(3)	C12 -C4	1.530(3)
C6 -C5	1.530(4)	C9 -C5	1.524(3)
C9 -C8	1.537(3)	C10 -C8	1.570(3)
C11 -C10	1.515(3)	C13 -C12	1.390(4)
C17 -C12	1.389(4)	C14 -C13	1.392(4)
C15 -C14	1.387(4)	C16 -C15	1.380(4)
C17 -C16	1.388(3)		
H20 -O20	1.080(0)	H21 -O21	1.080(0)
H22 -O22	1.080(0)	H23 -O23	1.080(0)
H3b -C3	1.080(0)	H3a -C3	1.080(0)
H4 -C4	1.080(0)	H8 -C8	1.080(0)
H9 -C9	1.080(0)	H10 -C10	1.080(0)
H11a-C11	1.080(0)	H11b-C11	1.080(0)
H13 -C13	1.080(0)	H14 -C14	1.080(0)

H16 -C16	1.080(0)	H17 -C17	1.080(0)

Selected bond angles for Conocarpin at room temperature			
C5 -O1 -C2	109.9(?)	C8 -O7 -C6	112.0(2)
O18 -C2 -O1	120.3(3)	C3 -C2 -O1	110.7(2)
C3 -C2 -O18	129.0(3)	C4 -C3 -C2	102.2(2)
C5 -C4 -C3	100.7(2)	C12 -C4 -C3	117.3(2)
C12 -C4 -C5	117.2(2)	C4 -C5 -O1	102.7(2)
C6 -C5 -O1	105.3(2)	C6 -C5 -C4	112.3(2)
C9 -C5 -O1	109.9(2)	C9 -C5 -C4	120.7(2)
C9 -C5 -C6	105.1(2)	O19 -C6 -O7	123.6(3)
C5 -C6 -O7	109.6(2)	C5 -C6 -O19	126.8(3)
C9 -C8 -O7	106.4(2)	C10 -C8 -O7	106.8(2)
C10 -C8 -C9	113.5(2)	C5 -C9 -O20	111.9(2)
C8 -C9 -O20	113.1(2)	C8 -C9 -C5	104.0(2)
C8 -C10 -O21	108.8(2)	C11 -C10 -O21	111.4(2)
C11 -C10 -C8	111.1(2)	C10 -C11 -O22	111.2(2)
C13 -C12 -C4	121.2(3)	C17 -C12 -C4	120.1(3)
C17 -C12 -C13	118.8(2)	C14 -C13 -C12	120.4(3)
C15 -C14 -C13	119.9(3)	C14 -C15 -O23	117.9(3)
C16 -C15 -O23	121.9(3)	C16 -C15 -C14	120.2(2)
C17 -C16 -C15	119.6(3)	C16 -C17 -C12	121.1(3)
C9 -O20 -H20	133.9(1)	C10 -O21 -H21	163.5(1)
C11 -O22 -H22	135.6(2)	C15 -O23 -H23	123.9(2)
H3b -C3 -C2	111.2(1)	H3a -C3 -C2	111.5(2)
H3a -C3 -H3b	109.5(0)	C4 -C3 -H3b	111.3(1)
C4 -C3 -H3a	111.1(1)	H4 -C4 -C3	113.1(1)
C5 -C4 -H4	113.8(2)	C12 -C4 -H4	95.5(1)
H8 -C8 -O7	114.0(1)	C9 -C8 -H8	108.3(1)
C10 -C8 -H8	107.9(1)	H9 -C9 -O20	103.2(1)

H9 -C9 -C5	113.2(1)	H9 -C9 -C8	111.8(1)
H10 -C10 -O21	108.9(1)	H10 -C10 -C8	109.6(1)
C11 -C10 -H10	107.0(1)	H11a-C11 -O22	109.1(1)
H11a-C11 -C10	109.1(1)	H11b-C11 -O22	109.0(2)
H11b-C11 -C10	108.9(1)	H11b-C11 -H11a	109.5(0)
H13 -C13 -C12	119.9(2)	C14 -C13 -H13	119.7(2)
H14 -C14 -C13	120.2(2)	C15 -C14 -H14	119.8(2)
H16 -C16 -C15	120.0(2)	C17 -C16 -H16	120.4(2)
H17 -C17 -C12	119.5(2)	H17 -C17 -C16	119.4(2)

Appendix 3

Selected Bond lengths for VI at room temperature.			
C6 -BR	1.959(5)	C5 -O1	1.391(6)
N1 -O2	1.225(7)	N1 -O3	1.157(7)
C3 -N1	1.494(7)	C9 -N2	1.134(7)
C2 -C1	1.521(7)	C6 -C1	1.530(7)
C7 -C1	1.535(8)	C3 -C2	1.314(7)
C9 -C2	1.433(7)	C4 -C3	1.488(7)
C5 -C4	1.557(7)	C8 -C4	1.518(7)
C6 -C5	1.523(7)	C8 -C7	1.555(8)
HO1 -O1	1.080(0)	H1 -C1	1.080(0)
H4 -C4	1.080(0)	H5 -C5	1.080(0)
H6 -C6	1.080(0)	H71 -C7	1.080(0)
H71 -C7	1.080(0)	H81 -C8	1.080(0)
H82 -C8	1.080(0)		

Selected Bond angles for VI at room temperature.			
O3 -N1 -O2	127.0(6)	C3 -N1 -O2	115.0(5)
C3 -N1 -O3	117.9(5)	C6 -C1 -C2	103.5(4)
C7 -C1 -C2	108.7(4)	C7 -C1 -C6	110.8(4)
C3 -C2 -C1	112.1(4)	C9 -C2 -C1	118.6(4)
C9 -C2 -C3	129.2(5)	C2 -C3 -N1	121.1(4)
C4 -C3 -N1	121.9(4)	C4 -C3 -C2	117.0(4)
C5 -C4 -C3	106.2(4)	C8 -C4 -C3	108.5(4)
C8 -C4 -C5	107.0(4)	C4 -C5 -O1	112.0(4)
C6 -C5 -O1	107.4(4)	C6 -C5 -C4	107.6(4)
C1 -C6 -BR	109.2(4)	C5 -C6 -BR	112.4(3)
C5 -C6 -C1	111.6(5)	C8 -C7 -C1	108.7(4)

C7 -C8 -C4	110.0(4)	C2 -C9 -N2	171.5(6)
C5 -O1 -HO1	166.4(3)	C2 -C1 -H1	114.2(3)
C6 -C1 -H1	112.3(3)	C7 -C1 -H1	107.4(3)
H4 -C4 -C3	111.5(3)	C5 -C4 -H4	112.5(2)
C8 -C4 -H4	109.8(3)	H5 -C5 -O1	108.3(3)
H5 -C5 -C4	108.5(2)	C6 -C5 -H5	113.0(3)
H6 -C6 -BR	108.5(2)	H6 -C6 -C1	109.1(3)
H6 -C6 -C5	105.8(3)	H71 -C7 -C1	109.7(3)
H71 -C7 -C1	109.6(3)	H71 -C7 -H71	109.5(0)
C8 -C7 -H71	109.7(3)	C8 -C7 -H71	109.7(3)
H81 -C8 -C4	109.3(3)	H81 -C8 -C7	109.3(3)
H82 -C8 -C4	109.5(3)	H82 -C8 -C7	109.3(3)
H82 -C8 -H81	109.5(0)		

Selected Bond lengths for VI at 100K.			
C6 -BR	1.951(4)	C5 -O1	1.410(5)
N1 -O2	1.223(4)	N1 -O3	1.158(5)
C3 -N1	1.526(5)	C9 -N2	1.150(4)
C2 -C1	1.519(5)	C6 -C1	1.542(5)
C7 -C1	1.537(5)	C3 -C2	1.347(5)
C9 -C2	1.417(5)	C4 -C3	1.497(5)
C5 -C4	1.547(5)	C8 -C4	1.539(5)
C6 -C5	1.538(5)	C8 -C7	1.539(5)
H0 -O1	.944(32)	H1 -C1	1.270(32)
H4 -C4	1.069(32)	H5 -C5	1.146(37)
H6 -C6	1.032(37)	H71 -C7	1.017(37)
H72 -C7	1.010(31)	H81 -C8	1.036(34)
H82 -C8	.659(37)		

Selected Bond angles for VI at 100K.			
O3 -N1 -O2	130.7(4)	C3 -N1 -O2	112.5(3)
C3 -N1 -O3	116.7(3)	C6 -C1 -C2	104.1(3)
C7 -C1 -C2	108.0(3)	C7 -C1 -C6	110.5(3)
C3 -C2 -C1	112.6(3)	C9 -C2 -C1	118.6(3)
C9 -C2 -C3	128.8(3)	C2 -C3 -N1	121.2(3)
C4 -C3 -N1	123.2(3)	C4 -C3 -C2	115.6(3)
C5 -C4 -C3	106.8(3)	C8 -C4 -C3	108.8(3)
C8 -C4 -C5	107.8(3)	C4 -C5 -O1	112.2(3)
C6 -C5 -O1	107.6(3)	C6 -C5 -C4	108.3(3)
C1 -C6 -BR	109.7(3)	C5 -C6 -BR	113.0(2)
C5 -C6 -C1	110.8(3)	C8 -C7 -C1	110.1(3)
C7 -C8 -C4	109.4(3)	C2 -C9 -N2	172.1(4)
H0 -O1 -C5	109.7(25)	H1 -C1 -C2	73.8(17)
H1 -C1 -C6	127.4(16)	H1 -C1 -C7	120.2(17)
H4 -C4 -C3	110.0(20)	H4 -C4 -C5	110.4(18)
H4 -C4 -C8	112.8(20)	H5 -C5 -O1	122.9(16)
H5 -C5 -C4	93.9(17)	H5 -C5 -C6	110.8(17)
H6 -C6 -BR	107.5(21)	H6 -C6 -C1	101.0(18)
H6 -C6 -C5	114.1(19)	H71 -C7 -C1	111.7(20)
H71 -C7 -C8	102.5(18)	H72 -C7 -C1	108.9(21)
H72 -C7 -C8	117.6(19)	H72 -C7 -H71	106.9(29)
H81 -C8 -C4	112.7(23)	H81 -C8 -C7	109.1(18)
H82 -C8 -C4	112.5(31)	H82 -C8 -C7	81.9(29)
H82 -C8 -H81	125.8(40)		

Appendix 4

Programs used

1. SHELX76, Sheldrick, 1978
2. SHELXS86, Sheldrick, 1985
3. NRCVAX, Gabe, Lee and Le Page, 1985
4. SCHAKAL, Keller, 1989
5. BRISK, written during this project.
6. CENTER, written during this project.

Appendix 5

Program listings

Brisk

Structure determination and refinement was carried out using the SHELX [Sheldrick, 1985] set of programs. The program BRISK was written to facilitate the transfer of structural information, such as atomic coordinates, thermal parameters and esd's to the NRCVAX [Gabe, Lee and Le Page, 1985] system of programs. The latter was then used for geometrical calculations and for producing ORTEP [Johnson, 1965] diagrams etc. Brisk also calculates the equivalent isotropic temperature factor for anisotropically refined atoms by the method of Hamilton (1959).

$$U_{eq}=\frac{1}{3}\sum_{i=1}^{3}\sum_{j=1}^{3}U_{ij}a^*_i a^*_j(a_i a_j)$$

The error in U_{eq} is evaluated according to the method suggested by Schomaker and Marsh (1983). Finally BRISK compiles tables of fractional coordinates, anisotropic thermal parameters and selected bond lengths and angles. The latter having been calculated using the SHELX BOND instruction.

```
      PROGRAM BRISK
      CHARACTER*12 CH
      CHARACTER*4 KEYW(7)
      INTEGER*1 MULT
      integer type,crdmul,adpmul
      real x(3),ex(3),tf(6),etf(6),sof,sf,fv21,fv31
      real dum(4),ax(3),axs(3),alf(3),calf(3,3)
      CHARACTER NAME*4,ES*2,inf*120,flag*1,code*4
      DATA KEYW /'WGHT','AFIX','    ','temp','BLOC','II.SF','ANIS'/
c Comments written to the screen
      print '(a40)',' Program --- BRISK--- version 3.20'
      print '(a26)','    written by D.G.Billing'
      print '(a26)','              23-November-1990    '
```

```
        print '(a1)',' '
        print '(a49)',' This program merges PUNCH file data (SHELX) with'
        print '(a35)','CD file data from NRCVAX, in ASCII'
        print '(a1)',' '
        print '(a50)',' Need a cd file created by "CDFILE" and converted'
        print '(a26)',' to ascii using "BINASC".'
        print '(a49)',' NB the atom types should be entered with cdfile'
        print '(a50)',' in the same order as the SHELX sfac instruction.'
        print '(a1)',' '

      write(*,'(A\)')'$Do you want to continue? (Y):'
      read(*,'(A1)') flag
       call yesno(flag)
       if (flag.eq.'N') goto 1000


       call fopen(code)

       write(*,'(a\)')'$Coordinates in tables to be multiplied by 10E'
       read(*,100)crdmul
100    format(i3)
       write(*,'(a\)')'$Adp''s in tables to be multiplied by 10E'
       read(*,100)adpmul
       write(3,110)crdmul
       write(3,115)adpmul
110    format(3x,'Coordinates have been multiplied by 10E',i1)
       write(11,115)adpmul
115    format(3x,'Adp''s have been multiplied by 10E',i1)

C INITIALIZE       ATOM COUNTER
       NATOM = 0
      WRITE(*,'(A\)')' Enter Z, the no of molecules in unit cell: '
       READ(*,2)MULT
2      FORMAT(I3)

c Read cell info from cd file

       read(7,5) inf
       read(7,5) inf
       read(7,5) inf
5      format(a120)
       read(7,7) ax(1),ax(2),ax(3),dum(1),dum(2)
       read(7,7) dum(1),dum(2),dum(3),dum(4),alf(1)
       read(7,7) alf(2),alf(3),axs(1),axs(2),axs(3)
7      format(5e14.6)
       do 8 n=1,3
8      calf(n,n)=1.0
       calf(1,2) = cos(alf(3))
```

```
      calf(1,3) = cos(alf(2))
      calf(2,3) = cos(alf(1))
      calf(2,1) = cos(alf(3))
      calf(3,1) = cos(alf(2))
      calf(3,2) = cos(alf(1))

      call cdprep


C START MAIN LOOP

18    READ (1,19)CH
19    FORMAT(A12)
      DO 22,N=1,7
      IF (CH(1:4).EQ.KEYW(N)) GOTO 18
22    CONTINUE
      IF (CH(1:4).EQ.'FVAR ) then
            backspace(1)
            read(1,20)name,sf,fv21,fv31
20          format(a4,3f10.5)
            goto 18
            endif

     IF (CH(1:3).EQ.'END') GOTO 37
      IF (CH(6:8).EQ.'---'.AND.CH(1:1).EQ.' ')THEN
            CALL BOND
            GOTO 18
      ENDIF
      IF (CH(6:8).EQ.'...'.AND.CH(1:1).EQ.' ') GOTO 18
      IF (CH(6:6).EQ.'-'.AND.CH(11:11).eq.'-'.AND.CH(1:1).EQ.' ')THEN
            CALL ANGLE
            GOTO 18
      ENDIF
      call pchread(name,x,ex,tf,etf,fv21,fv31,sof,type,es)
      CALL ATOM(name,x,ex,tf,etf,ax,axs,calf,crdmul,adpmul,es)
      CALL CDWRITE(name,x,ex,tf,etf,sof,type,es,mult)
      GOTO 18

37    continue
      call cdend
      call tab1(code)
      call tab2(code)
      call tab3(code)
      call tab4(code)

      print '(a1)',' '
      write(*,40)code
40    format(' Next run BINASC, using ',a4,'nucd.asc as input file')
```

```
      write(*,50)
50    format( ' GOOD LUCK!!, Hope the tables are satisfactory!')
      print '(a1)',' '
      print '(a1)',' '


1000  continue
c     close files
      close(1)
      close(2)
      close(3)
      close(4)
      close(10)
      close(11)
      close(7)
      close(8)
      close(9)
      close(12)
      close(13)
      close(14)

      STOP
      END

      subroutine cdprep
c Copies data generated by CDFILE, convened to ASCII by BINASC,
c in codeCD.ASC TO codeNUCD.ASC
      character*120 inf

      rewind(7)
5     read(7,20) inf
      if(inf(1:6).eq.'ENDCOD') goto 40
      write(2,20) inf
      goto 5
20    format(a120)
40    return
      end

      subroutine cdend
      character*120 inf

      backspace(7)

5     read(7,20,end=40) inf
      write(2,20) inf
20    format(a120)
      goto 5
40    return
```

```
      end

      subroutine pchread(name,x,ex,tf,etf,fv21,fv31,sof,type,es)
c reads in data from a SHELX punch file run with BOND and LIST cards
      integer type
      REAL sof,temp,fv21,fv31

      real x(3),ex(3),tf(6),etf(6)
      CHARACTER name*4,es*2


     BACKSPACE(1)

      READ(1,44)name,type,(x(i),i=1,3),sof,tf(1),tf(2),es
44    FORMAT(A4,I5,6F10.5,A2)
      IF (ES.EQ.' =') THEN
            READ(1,47)tf(3),tf(4),tf(5),tf(6)
47          FORMAT(5X,4F10.5)
      else
            tf(2) = 0.0
            tf(3) = 0.0
            tf(4) = 0.0
            tf(5) = 0.0
            tf(6) = 0.0
            if(tf(1).eq.21.0) tf(1) = fv21
            if(tf(1).eq.31.0) tf(1) = fv31
      endif

      READ(1,54) (ex(i),i=1,3),(etf(i),i=1,6)
54    FORMAT(4X,9F8.5)

c swap temperature factors to get them in the more conventional order
      temp = tf(4)
      tf(4) = tf(6)
      tf(6) = temp
      temp = etf(4)
      etf(4) = etf(6)
      etf(6) = temp

      return
      end



     SUBROUTINE CDWRITE(name,x,ex,tf,etf,sof,type,es,mult)
C Adds coordinates etc. to cd file of NRCVAX generated with BINASC
      INTEGER SITESYM,SYMCONST,FIXSOF,BLOCKNO,ANISFL
      INTEGER*1 MULT
```

```
      real sigocc,occ
      integer type
      real x(3),ex(3),tf(6),etf(6),sof
      CHARACTER NAME*4,ES*2


      SITESYM = 1
      SYMCONST = 0
      FIXSOF = 10
      BLOCKNO = 0
      SIGOCC = 0.0


     WRITE(2,50)name,SITESYM,SYMCONST
50    FORMAT(A4,2X,2I9)
      OCC = SOF - FIXSOF
      WRITE(2,55)OCC,(x(i),i=1,3),tf(1)
     WRITE(2,55)(tf(i),i=2,6)
      WRITE(2,55)SIGOCC,(ex(i),i=1,3),etf(1)
      WRITE(2,55)(etf(i),i=2,6)
55    FORMAT(5E14.6)

      IF(ES.EQ.' =') ANISFL = -1
      IF(ES.NE.' =') ANISFL = 0
      WRITE(2,60)ANISFL,TYPE,MULT
60    FORMAT(2I6,'  4096',5X,'0',5X,'1',I6,9X,'0')
     WRITE(2,70)
     WRITE(2,70)
     WRITE(2,70)
     WRITE(2,70)
70    FORMAT(3X,'.000000E+00',3X,'.000000E+00',3X,'.000000E+00',
    & 3X,'.000000E+00',3X,'.000000E+00')
      WRITE(2,80)
80    FORMAT(5X,'0',5X,'0',5X,'0',5X,'0',5X,'0',5X,'0',
    & 3X,'.000000E+00')
      WRITE(2,*)

     RETURN
     END

     SUBROUTINE BOND
c write bondlenghts found in punch file to a scratch file
      character*4 at1,at2
      real bl,ebl
      integer iebl,unit

      backspace(1)
      unit=4
```

```
      read(1,10) at1,at2,bl,ebl
10    format(1x,a4,3x,a4,f8.3,2x,f6.3)

      iebl = nint(1000*ebl)

      if (at1(1:1).eq.'H') unit=12
      if (at2(1:1).eq.'H') unit=12

      write(unit,30)at1,at2,bl,iebl
30    format(10x,a4,'-',a4,5x,f8.3,'(',i3,')')

      RETURN
      END


      SUBROUTINE ANGLE
c writes bondangles found in punch file to a scratch file
      character*4,at1,at2,at3
      real as,eas
      integer ieas,unit

      backspace(1)

      read(1,10)at1,at2,at3,as,eas
10    format(1x,a4,1x,a4,1x,a4,1x,f6.1,2x,f4.1)
      unit=10

      ieas = nint(10*eas)
      if(at1(1:1).eq.'H') unit=13
      if(at3(1:1).eq.'H') unit=13

      write(unit,30)at1,at2,at3,as,ieas
30    format(10x,a4,'-',a4,'-',a4,5x,f6.1,'(',i2,')')

      RETURN
      END

      SUBROUTINE ATOM(name,x,ex,tf,etf,ax,axs,calf,crdmul,adpmul,es)

      integer type
      real x(3),ex(3),tf(6),etf(6),sof
      real ax(3),axs(3),calf(3,3),a(3,3)
      CHARACTER name*4,E  *2
      integer ix(3),iex(3),itf(6),ietf(6)
      real ueq,eueq
      integer iueq,ieueq,crdmul,adpmul
```

```
      do 3,i=i,3
      call mult(x(i),crdmul,ix(i))
      call mult(ex(i),crdmul,iex(i))
3     continue

      if(ES.eq.' =') then
c insert ueq calc here

            a(1,1)=tf(1)
            a(2,2)=tf(2)
            a(3,3)=tf(3)
            a(1,2)=tf(4)
            a(1,3)=tf(5)
            a(2,3)=tf(6)
            a(3,2)=a(2,3)
            a(3,1)=a(1,3)
            a(2,1)=a(1,2)
            ueq = 0
            do 5 n1=1,3
            do 5 n2=1,3
5     ueq=ueq+a(n1,n2)*axs(n1)*axs(n2)*ax(n1)*ax(n2)*calf(n1,n2)
            ueq = ueq/3.0
            eueq = etf(1)*etf(1)+etf(2)*etf(2)+etf(3)*etf(3)
            eueq = sqrt(eueq/18)


c to here

            call mult(ueq,adpmul,iueq)
            call mult(eueq,adpmul,ieueq)

            write(3,10)name,(ix(i),iex(i),i=1,3),iueq,ieueq




            do 15,i=1,6
            call mult(tf(i),adpmul,itf(i))
            call mult(etf(i),adpmul,ietf(i))
15          continue



            write(11,20)name,(itf(i),ietf(i),i=1,6)

20    format(5x,a4,6(1x,i5,'(',i3,')'))
```

```
      else
            call mult(tf(1),3,iueq)
            call mult(etf(1),3,ieueq)


            if(name(1:1).eq.'H') then
            write(9,10)name,(ix(i),iex(i),i=1,3),iueq,ieueq
            else
            write(3,40)name,(ix(i),iex(i),i=1,3),iueq,ieueq
            endif


10    format(2x,a4,3(3x,i7,'(',i3,')'),4x,i5,'(',i3,') ')

      format(2x,a4,3(3x,i7,'(',i3,')'),4x,i5,'(',i3,')*')
      endif

      RETURN
      END


      subroutine yesno(flag)
      character flag*1
10    if (flag.eq.' ') flag='Y'
      if (flag.eq.'y') flag='Y'
      if (flag.eq.'n') flag='N'
      if (flag.eq.'Y') return
      if (flag.eq.'N') return
      write(*,'(A\)')'$Shithead "y","Y","n","N" or " " only Try again:'
      read(*,'(A1)') flag
      goto 10
      return
      end

      subroutine mult(val,mul,ival)
      real val,temp
      integer mul,ival

      if(mul.eq.0) temp=val*1
      if(mul.eq.1) temp=val*10
      if(mul.eq.2) temp=val*100
      if(mul.eq.3) temp=val*1000
      if(mul.eq.4) temp=val*10000
      if(mul.eq.5) temp=val*100000
      if(mul.eq.6) temp=val*1000000
      ival=nint(temp)
      return
      end
```

```
      subroutine fopen(code)

C OPEN THE FILES
      character*12 file1,file2,file3,file4,file5,file6,file7
      character*12 file8,file9
      character code*4,flag*1

      write(*,'(a\)') ' Enter crystal code (4 Characters): '
      read(*,'(a)') code

      file1 = code//'.pch'
      write(*,10) file1
10    format('$The input PCH file is ',a12)
     write(*,'(A\)')'$Is this OK? (Y):'
     read(*,'(A1)') flag
      call yesno(flag)
      if (flag.eq.'N') then
         write(*,'(A\)')'$Enter PCH filename: '
         read(*,'(A12)') file1
         endif
      open(1,file=file1,status='old')

      file2 = code//'nucd.asc'
      write(*,11) file2
11    format('$The OUTPUT file is ',a12)
     write(*,'(A\)')'$Is this OK? (Y):'
     read(*,'(A1)') flag
      call yesno(flag)
      if (flag.eq.'N') then
         write(*,'(A\)')'$Enter OUTPUT filename: '
         read(*,'(A12)') file2
         endif
      open(2,file=file2,status='unknown')


      file3 = code//'crds.tab'
      OPEN(3,STATUS = 'scratch')
      file4 = code//'bond.tab'
      OPEN(4,STATUS = 'scratch')
      file5 = code//'angl.tab'
      OPEN(10,STATUS = 'scratch')
      file6 = code//'adp.tab'
      OPEN(11,status = 'scratch')
      file9 = code//'hs.tab'
      open(9,status='scratch')
c  unit 12 - H bonds
      open(12,status='scratch')
```

```
c   unit 13 - H angles
        open(13,status='scratch')

        file7 = code//'cd.asc'
        write(*,12) file7
12      format('$The ASCII CD INPUT file is ',a12)
       write(*,'(A\)')'$Is this OK? (Y):'
       read(*,'(A1)') flag
        call yesno(flag)
        if (flag.eq.'N') then
           write(*,'(A\)')'$Enter ASCII CD INPUT filename: '
           read(*,'(A12)') file7
           endif
        open(7,file=file7,status='old')
        file8 = code//'.tab'
        write(*,15) file8
15      format('$The final TABLE file is ',a12)
       write(*,'(A\)')'$Is this OK? (Y):'
       read(*,'(A1)') flag
        call yesno(flag)
        if (flag.eq.'N') then
           write(*,'(A\)')'$Enter final TABLE filename: '
           read(*,'(A12)') file8
           endif
        open(8,file = file8,status = 'unknown')
        open(14,file = 'dave.wp',status = 'unknown')


        return
        end

      subroutine tab1(code)
c reads brisk table files and writes it neatly

      integer crds(3),scrds(3),ueq,eueq,nol,crdmul,adpmul,unit
      character name*4,flag*1,tof*1,code*4,fl*1,tab
      character*3 car(3),careq

        tof=char(12)
        tab=char(9)

        rewind(3)
        rewind(9)

        unit=3

        read(3,2)crdmul
2       format(42x,i1)
```

```
      read(3,3)adpmul
3     format(36x,i1)

      write(8,16) tof
      write(14,16) tof
16     format(a1)


     write(8,20)crdmul
20    format(5x,'                                    ',i1)
     write(8,22)
22    format(5x,'TABLE  : Fractional atomic coordinates (x10 ) and equiv
    &alent')
23    write(8,24)
24    format(' ')
     write(8,25)adpmul
25    format(5x,'                                 o2   ',i1)


26    write(8,28)code
28    format(5x,'         isotropic temperature factors (A x10 ) for '
    &  ,a4)


      write(8,24)
      write(8,24)
      write(8,24)

      write(8,30)
30    format(15x,'x/a',11x,'y/b',11x,'z/c',12x,'U')
      write(8,32)
32    format(59x,'eq')

      write(8,24)
      write(8,24)


      nol=0
5    read(unit,10,end = 1000) name,(crds(i),scrds(i),i=1,3),ueq,eueq,fl
10    format(2x,a4,3(3x,f7,1x,i3,1x),4x,i5,1x,i3,1x,a1)
      nol=nol+1
      if(nol.ge.50) then
         nol=0
         call newpage
         endif

      do 50,i=1,3
      call space(scrds(i),car(i))
```

```
50      continue
        call space(eueq,careq)

        write(8,100) name,(crds(i),car(i),i=1,3),ueq,careq,fl
100     format(5x,a4,3(3x,i7,'(',a3),5x,i5,'(',a3,a1)
        write(14,101) name,tab,(crds(i),car(i),tab,i=1,3),ueq,careq,fl
101     format(a4,a1,3(i6,'(',a3,a1),i5,'(',a3,a1)

        goto 5

1000    continue

        if(unit.eq.3) then
           unit=9
           noi=0
           write(*,'(A\)')'$Write H coords in separate table? (Y):'
           read(*,'(A1)') flag
           call yesno(flag)
           if (flag.eq.'Y') then

           write(8,24)
           write(8,110)
110     format(5x,'* isotropic temperature factors')


           write(14,16)tof
           write(8,16)tof

     write(8,120)crdmul
120     format(5x,'                                    ',i1)
     write(8,122)
122     format(5x,'TABLE  : Hydrogen atomic coordinates (x10 ) and')

           goto 23

           else
           unit=9
           goto 5
           endif


        endif




        return
```

```
      end

      subroutine newpage
      character tof*1
      tof=char(12)
      write(8,10)
10    format(' ')
      write(8,15)
15    format('Data continued on next page')
      write(8,20) tof
20    format(a1)
      return
      end

      subroutine tab2(code)

c reads brisk adp.tab file and writes it neatly

      integer u(6),su(6),nol,adpmul
      character name*4,tof*1,flag*1,code*4,tab*1
      character*3 cu(6)

      tof=char(12)
      tab=char(9)

      rewind(11)
      read(11,3)adpmul
3     format(36x,i1)


c write table heading

      write(8,5) tof
      write(14,5) tof
5     format(a1)

      write(8,10)adpmul
10    format(3x,'                                    o2    ',i1)
      write(8,15)code
15    format(5x,'TABLE  : Anisotropic temperature factors (A ,x10 )',
     & ' for ',a4)
      write(8,20)
20    format(' ')
      write(8,20)
      write(8,20)
      write(8,25)
25    format(9x,6('U',10x))
      write(8,30)
```

```
30      format(9x,' 11',' 22',' 33',' 12'
     &  ,' 13',' 23')
        write(8,20)
        write(8,20)

        nol=0
55      read(11,50,end=100) name,(u(i),su(i),i=1,6)
50      format(5x,a4,6(1x,i5,1x,i3,1x))
        nol=nol+1
        if(nol.ge.50) then
           nol=0
           call newpage
           endif

        do 200,i=1,6
        call space(su(i),cu(i))
200     continue


        write(8,60) name,(u(i),cu(i),i=1,6)
60      format(2x,a4,6(1x,i5,'(',a3,' '))
        write(14,61) name,(u(i),cu(i),i=1,6)
61      format(2x,a4,6(1x,i5,'(',a3,' '))


        goto 55

100     continue
        return
        end

        subroutine tab3(code)
c brisk bond lenghts printed neatly

        character atm1(2)*4,atm2(2)*4,tof*1,code*4,flag*1,tab*1
        integer sbl(2),nol,unit
        character*3 csbl(2)
        real bl(2)

        rewind(4)
        rewind(12)
        unit=4

        tof=char(12)
        tab=char(9)
        write(8,5)tof
        write(14,5)tof
5       format(a1)
```

```
      write(8,10)
10    format(5x,'                              o')
      write(8,15)code
15    format(5x,'TABLE  : Selected bond lenghts (A) for ',a4)
      write(8,20)
20    format(' ')
      write(8,20)

      nol=0

40    do 60,i=1,2
      read(unit,50,end=100)atm1(i),atm2(i),bl(i),sbl(i)
50    format(10x,a4,1x,a4,5x,f8.3,1x,i3,1x)
      call space(sbl(i),csbl(i))

60    continue
      write(8,70)(atm1(i),atm2(i),bl(i),csbl(i),i=1,2)
70    format(2(10x,a4,'-',a4,5x,f8.3,'(',a3,' '))
      write(14,71)(atm1(i),atm2(i),tab,bl(i),csbl(i),tab,i=1,2)
71    format(2(a4,'-',a4,a1,f8.3,'(',a3,' ',a1))


      nol=nol+1
      if(nol.ge.50) then
         nol=0
         call newpage
         endif

      goto 40
100   continue
      if(i.eq.2) then
         write(8,110)atm1(1),atm2(1),bl(1),csbl(1)
110   format(10x,a4,'-',a4,5x,f8.3,'(',a3,' ')
         write(14,111)atm1(1),atm2(1),tab,bl(1),csbl(1)
111   format(a4,'-',a4,a1,f8.3,'(',a3,' ')
         endif



      if(unit.eq.4) then
         unit=12
         nol=0
         write(*,'(A\)')'$Write H bonds in separate table? (Y):'
         read(*,'(A1)') flag
         call yesno(flag)
         if (flag.eq.'Y') then
```

```
            write(8,5)tof
            write(14,5)tof
          write(8,210)
210       format(5x,'                                o')
          write(8,215)code
215       format(5x,'TABLE  : Hydrogen bond lenghts (A) for ',a4)
          write(8,220)
220       format(' ')
          write(8,20)


            goto 40

            else
            unit=12
            goto 40
            endif
          endif

          return
          end

          subroutine tab4(code)
c writes brisk bond angles neatly
          character*4 atm1(2),atm2(2),atm3(2),code*4,flag*1
          character tof*1,tab*1
          integer nol,san(2),unit
          real as(2)
          character*3 csan(2)

          rewind(10)
          rewind(13)
          unit=10

          tof=char(12)
          tab=char(9)
          write(8,5)tof
          write(14,5)tof
5         format(a1)
          write(8,10)
10        format(5x,'                              o')
          write(8,15)code
15        format(5x,'TABLE  : Selected bond angles ( ) for ',a4)
          write(8 20)
20        format(' ')
          write(8,20)

          nol = 0
```

```
40      do 60,i=1,2
        read(unit,50,end=100)atm1(i),atm2(i),atm3(i),as(i),san(i)
50      format(10x,a4,1x,a4,1x,a4,5x,f6.1,1x,i2)
        call space(san(i),csan(i))

60      continue
        write(8,70)(atm1(i),atm2(i),atm3(i),as(i),csan(i),i=1,2)
70      format(2(10x,a4,'-',a4,'-',a4,5x,f6.1,'(',a3))
        write(14,71)(atm1(i),atm2(i),atm3(i),tab,as(i),csan(i),tab,i=1,2)
71      format(2(a4,'-',a4,'-',a4,a1,f6.1,'(',a3,a1))


        nol=nol+1
        if(nol.ge.50) then
           nol=0
           call newpage
           endif

        goto 40
100     continue
        if(i.eq.2) then
           write(8,110)atm1(1),atm2(1),atm3(1),as(1),csan(1)
110     format(10x,a4,'-',a4,'-',a4,5x,f6.1,'(',a3)
           write(14,111)atm1(1),atm2(1),atm3(1),tab,as(1),csan(1)
111     format(a4,'-',a4,'-',a4,a1,f6.1,'(',a3)

           endif

        if(unit.eq.10) then
           unit=13
           nol=0
           write(*,'(A\)')'$Write H bond angles in separate table? (Y):'
           read(*,'(A1)') flag
           call yesno(flag)
           if (flag.eq.'Y') then



           write(8,5)tof
           write(14,5)tof

        write(8,210)
210      format(5x,'                              o')
        write(8,215)code
215      format(5x,'TABLE  : Hydrogen bond angles ( ) for ',a4)
        write(8,220)
220      format(' ')
        write(8,20)
```

```
      goto 40

      else
      unit=13
      goto 40
      endif
   endif


      write(8,5)tof
      write(14,5)tof


   return
   end


   subroutine space(iin,cout)
   integer iin,ione,itwo
   character*3 cout


   if(iin.le.9) then
      cout(1:1)=char(iin+48)
      cout(2:3)=') '
      endif
   if(iin.le.99.and.iin.gt.9) then
      ione=iin/10
      itwo=iin-(ione*10)
      cout(1:1)=char(ione+48)
      cout(2:2)=char(itwo+48)
      cout(3:3)=')'
      endif


   return
   end
```

Center

The program CENTER calculates the errors in the centring of the crystal, using the ideas put forward in chapter 6.

```
      PROGRAM CENTER
      REAL R  RS,TH,CHIM,THX,THY
      REAL CHI1,CHI2,CHI3,CHI4,TH1,TH2,TH3,TH4
      REAL DH,DX,DY,DZ,VAR1,VAR2,PI
      CHARACTER*12 FILE1,FLAG
      CHARACTER*80 COM

C OPEN OUTPUT FILE

      write(*,'(A\)')' Enter OUTPUT file name :'
     read(*,'(A)') FILE1
      open(1,file = FILE1,status = 'unknown')

C STANDARD DISTANCES FOR CAD4

      PI=3.141592654
      RS=140
      RC=173

      WRITE(*,'(A\)') '$ENTER DE  TOR HEIGHT ERROR (DETER):'
      READ(*,*) DH
C *3.02 WITHOUT EXTENDER, SEE MANUAL
      DH = DH*3.02


C INPUT THETA VALUES

10    WRITE(*,*)
      WRITE(*,'(A\)') '$ENTER THETA VALUE FOR SETTING ONE: '
      READ(*,*) TH1
      TH1 = TH1*PI/180
      WRITE(*,'(A\)') '$ENTER THETA VALUE FOR SETTING TWO: '
      READ(*,*) TH2
      TH2 = TH2*PI/180
      WRITE(*,'(A\)') '$ENTER THETA VALUE FOR SETTING THREE: '
      READ(*,*) TH3
      TH3 = TH3*PI/180
      WRITE(*,'(A\)') '$ENTER THETA VALUE FOR SETTING FOUR: '
      READ(*,*) TH4
      TH4 = TH4*PI/180
```

```
C INPUT CHI VALUES

      WRITE(*,'(A\)') '$ENTER CHI VALUE FOR SETTING ONE: '
      READ(*,*) CHI1
      CHI1 = CHI1*PI/180
      WRITE(*,'(A\)') '$ENTER CHI VALUE FOR SETTING TWO: '
      READ(*,*) CHI2
      CHI2 = CHI2*PI/180
      WRITE(*,'(A\)') '$ENTER CHI VALUE FOR SETTING THREE: '
      READ(*,*) CHI3
      CHI3 = CHI3*PI/180
      WRITE(*,'(A\)') '$ENTER CHI VALUE FOR SETTING FOUR: '
      READ(*,*) CHI4
      CHI4 = CHI4*PI/180

C CALCULATE AVERAGE THETA VALUE

      TH = (ABS(TH1)+ABS(TH2)+ABS(TH3)+ABS(TH4))/4

      WRITE(*,'(A\)') '$AVERAGE VALUE OF THETA ='
      WRITE(*,100) TH*180/PI

C ERROR IN Z

      CHIM = CHI1+CHI2-CHI3-CHI4
      VAR1 = DH/(2*RC*SIN(TH))
      VAR2 = 2*SIN(TH)/(1/RS+1/RC)

      DZ = (4*CHIM-VAR1)*VAR2
      WRITE(*,'(A\)') '$ERROR IN Z = '
      WRITE(*,110) DZ

C ERROR IN X

      THX = TH1-TH2+TH3-TH4
      DX = THX/(4*SIN(TH)*(1/RS+1/RC))
      WRITE(*,'(A\)') '$ERROR IN X = '
      WRITE(*,110) DX

C ERROR IN Y

      THY = TH2+TH3-TH1-TH4
      DY = THY/(4*COS(TH)*(1/RS-1/RC))
      WRITE(*,'(A\)') '$ERROR IN Y = '
      WRITE(*,110) DY

      WRITE(*,*)
      WRITE(*,'(A\)') '$COMMENT: '
```

```
      READ(*,'(A)') COM

C OUTPUT TO FILE

      WRITE(1,120) DH
      WRITE(1,130) TH1*180/PI,TH2*180/PI,TH3*180/PI,TH4*180/PI
      WRITE(1,140) CHI1*180/PI,CHI2*180/PI,CHI3*180/PI,CHI4*180/PI
      WRITE(1,150)
      WRITE(1,160) DZ
      WRITE(1,170) DX
      WRITE(1,180) DY
      WRITE(1,'(A)') COM
      WRITE(1,*)

      WRITE(*,*)
      WRITE(*,'(A\)') '$REPEAT? (Y/N) '
      READ(*,'(A)') FLAG
      IF (FLAG.EQ.'Y') GOTO 10

100   FORMAT(F7.3,' degrees')
110   FORMAT(F7.4,'mm')
120   FORMAT('DETECTOR ERROR= ',F7.4,' (MULTIPLIED BY 3.02)')
130   FORMAT('TH1=',F7.3,' TH2=',F7.3,' TH3=',F7.3,' TH4=',F7.3)
140   FORMAT('CHI1=',F7.3,' CHI2=',F7.3,' CHI3=',F7.3,' CHI4=',F7.3)
150   FORMAT('CRYSTAL OFFSETS')
160   FORMAT('DZ = ',F7.4,'mm')
170   FORMAT('DX = ',F7.4,'mm')
180   FORMAT('DY = ',F7.4,'mm')

      CLOSE(1)
      STOP
      END
```

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TABLE : Anisotropic temperature factors (A^2, $*10^3$) for or67

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
N	56(2)	29(1)	49(2)	-2(1)	-1(1)	7(1)
O	105(2)	30(1)	65(2)	4(1)	1(1)	12(1)
C1	52(2)	39(2)	53(2)	2(2)	5(2)	5(2)
C2	77(2)	41(2)	50(2)	5(2)	1(2)	-3(2)
C3	80(3)	33(2)	75(2)	4(2)	0(2)	-5(2)
C4	77(2)	23(2)	64(2)	-2(2)	-10(2)	4(2)
C5	44(2)	35(2)	48(2)	2(2)	-4(2)	2(1)
C6	55(2)	39(2)	58(2)	-5(2)	0(2)	3(2)
C7	65(2)	54(2)	66(2)	-1(2)	-10(2)	-10(2)
C8	73(2)	75(3)	53(2)	15(2)	-12(2)	-15(2)
C9	80(3)	60(3)	53(2)	4(2)	2(2)	14(2)
C10	67(2)	47(2)	49(2)	-3(2)	0(2)	4(2)

TABLE : Anisotropic temperature factors (A^{o2}, $*10^{3}$) for lt0102

	U 11	U 22	U 33	U 12	U 13	U 23
N	27(1)	16(0)	18(1)	-1(0)	-2(0)	2(0)
O	45(1)	17(0)	29(1)	2(1)	0(1)	6(0)
C1	24(1)	20(1)	23(1)	1(1)	2(1)	2(1)
C2	33(1)	23(1)	24(1)	2(1)	2(1)	0(1)
C3	33(1)	18(1)	33(1)	2(1)	0(1)	-2(1)
C4	31(1)	15(1)	27(1)	-3(1)	-4(1)	4(1)
C5	21(1)	21(1)	21(1)	1(1)	-2(1)	3(1)
C6	27(1)	22(1)	26(1)	-2(1)	1(1)	0(1)
C7	30(1)	28(1)	31(1)	0(1)	-4(1)	-5(1)
C8	35(1)	39(1)	23(1)	7(1)	-6(1)	-6(1)
C9	37(1)	38(1)	21(1)	2(1)	1(1)	6(1)
C10	29(1)	26(1)	23(1)	-2(1)	-1(1)	4(1)

TABLE : Anisotropic temperature factors ($Å^2$,$*10^3$) for lt03

	U 11	U 22	U 33	U 12	U 13	U 23
N	22(1)	17(1)	32(1)	0(1)	0(1)	2(1)
O	33(1)	16(1)	41(1)	1(1)	1(1)	3(1)
C1	17(1)	26(1)	33(1)	-2(1)	2(1)	1(1)
C2	26(1)	22(1)	32(1)	1(1)	1(1)	1(1)
C3	29(1)	16(1)	38(2)	-1(1)	-3(1)	0(1)
C4	27(1)	14(1)	39(2)	-3(1)	-4(1)	2(1)
C5	18(1)	20(1)	34(1)	4(1)	-3(1)	1(1)
C6	20(1)	22(1)	35(1)	1(1)	-1(1)	3(1)
C7	23(1)	24(1)	40(2)	2(1)	-3(1)	-3(1)
C8	25(1)	28(1)	36(1)	7(1)	-3(1)	-3(1)
C9	23(1)	29(1)	34(2)	4(1)	-1(1)	6(1)
C10	23(1)	22(1)	34(1)	0(1)	-3(1)	3(1)

TABLE : Anisotropic temperature factors (A^{o2}, $*10^{3}$) for lt01

	U 11	U 22	U 33	U 12	U 13	U 23
N	14(1)	22(3)	14(1)	2(1)	0(1)	3(1)
O	21(1)	14(2)	19(1)	1(1)	1(1)	0(1)
C1	10(1)	14(3)	14(1)	0(1)	2(1)	-4(1)
C2	16(1)	23(3)	16(1)	1(1)	1(1)	2(1)
C3	17(1)	19(3)	19(1)	-1(1)	1(1)	-3(2)
C4	16(1)	19(3)	17(1)	-1(1)	-1(1)	6(2)
C5	10(1)	18(3)	12(1)	3(1)	-2(1)	1(1)
C6	11(1)	27(3)	18(1)	2(1)	0(1)	6(2)
C7	14(1)	18(3)	21(1)	1(1)	-2(1)	-3(2)
C8	15(1)	28(3)	15(1)	5(1)	-2(1)	-2(2)
C9	15(1)	26(3)	12(1)	3(1)	1(1)	4(1)
C10	12(1)	24(3)	17(1)	-3(1)	-1(1)	1(2)

TABLE : Anisotropic temperature factors (A^{o2}, $*10^{3}$) for Leucodrin

	U 11	U 22	U 33	U 12	U 13	U 23
O1	28(2)	23(1)	24(1)	3(2)	-3(1)	4(1)
O7	20(1)	15(1)	38(2)	-3(1)	-1(1)	-1(1)
O18	28(2)	27(2)	49(2)	2(2)	-5(2)	14(1)
O19	29(2)	19(1)	46(2)	-6(2)	2(2)	5(1)
O20	21(2)	22(1)	34(2)	-8(1)	1(1)	2(1)
O21	18(1)	25(2)	56(2)	5(1)	-7(2)	-12(1)
O22	39(2)	19(1)	35(2)	4(2)	-1(2)	-7(1)
O23	50(2)	41(2)	34(2)	-5(2)	-20(2)	8(1)
C2	18(2)	20(2)	34(2)	-4(2)	-1(2)	4(2)
C3	34(3)	19(2)	30(2)	5(2)	-4(2)	-1(2)
C4	21(2)	18(2)	22(2)	1(2)	1(2)	1(2)
C5	18(2)	17(2)	20(2)	0(2)	-1(2)	2(2)
C6	26(2)	20(2)	25(2)	1(2)	1(2)	-1(2)
C8	18(2)	14(2)	27(2)	-5(2)	2(2)	-2(2)
C9	17(2)	16(2)	24(2)	-2(2)	-2(2)	-1(2)
C10	20(2)	17(2)	26(2)	1(2)	-2(2)	0(2)
C11	28(2)	20(2)	41(2)	-2(2)	3(2)	-10(2)
C12	27(2)	20(2)	21(2)	-2(2)	0(2)	-1(2)
C13	38(2)	28(2)	29(2)	-6(2)	-3(2)	0(2)
C14	39(3)	34(2)	30(2)	-4(3)	-8(2)	1(2)
C15	42(3)	26(2)	26(2)	1(2)	-9(2)	1(2)
C16	45(3)	31(2)	29(2)	-2(3)	-2(2)	7(2)
C17	30(2)	26(2)	33(2)	-2(2)	-1(2)	4(2)

TABLE : Anisotropic temperature factors (A^2, $\times 10^4$) for Leucodrin at 100K

	U 11	U 22	U 33	U 12	U 13	U 23
O1	98(3)	96(3)	103(3)	20(3)	-14(3)	11(2)
O7	77(3)	78(2)	153(3)	-6(2)	-4(3)	-3(3)
O18	99(3)	110(3)	177(3)	8(3)	-16(3)	43(3)
O19	111(3)	95(3)	173(3)	-18(3)	8(3)	14(3)
O20	89(3)	117(3)	129(3)	-34(3)	1(3)	12(3)
O21	81(3)	111(3)	192(3)	24(3)	-33(3)	-40(3)
O22	142(3)	96(3)	140(3)	15(3)	-7(3)	-17(3)
O23	167(4)	168(3)	141(3)	-12(3)	-59(3)	39(3)
C2	77(3)	95(3)	133(4)	-6(3)	1(3)	16(3)
C3	125(4)	86(3)	128(4)	15(3)	-6(3)	-3(3)
C4	94(3)	90(3)	99(3)	1(3)	-4(3)	3(3)
C5	78(3)	84(3)	90(3)	3(3)	-6(3)	2(3)
C6	86(3)	93(3)	101(3)	1(3)	-2(3)	0(3)
C8	80(3)	75(3)	114(3)	-5(3)	-5(3)	-3(3)
C9	73(3)	90(3)	99(3)	-4(3)	-1(3)	3(3)
C10	81(3)	83(3)	110(3)	4(3)	-4(3)	-4(3)
C11	97(4)	97(3)	147(4)	-13(3)	0(3)	-29(3)
C12	107(4)	100(3)	95(3)	-3(3)	-2(3)	-5(3)
C13	136(4)	118(3)	108(3)	-24(4)	-9(3)	12(3)
C14	128(4)	137(4)	127(4)	-27(4)	-26(3)	2(3)
C15	143(4)	121(4)	100(3)	10(4)	-23(4)	-4(3)
C16	147(4)	133(4)	122(4)	-14(4)	-10(4)	30(3)
C17	122(4)	123(4)	125(4)	-15(4)	-1(3)	12(3)

TABLE : Anisotropic temperature factors (A^2 ,x10^3) for Conocarpin

	U 11	U 22	U 33	U 12	U 13	U 23
O1	39(1)	28(1)	35(1)	-6(1)	0(1)	4(1)
O7	24(1)	55(1)	35(1)	-2(1)	1(1)	-13(1)
O18	60(1)	30(1)	56(1)	-21(1)	2(1)	-1(1)
O19	33(1)	53(1)	64(1)	12(1)	2(1)	-9(1)
O20	33(1)	44(1)	41(1)	13(1)	-7(1)	-5(1)
O21	34(1)	30(1)	37(1)	7(1)	2(1)	8(1)
O22	43(1)	70(1)	32(1)	11(1)	-13(1)	-17(1)
O23	48(1)	30(1)	57(1)	-4(1)	-16(1)	13(1)
C2	33(2)	32(1)	40(2)	-2(1)	6(1)	-4(1)
C3	43(2)	29(1)	32(1)	-12(1)	-3(1)	-1(1)
C4	33(2)	28(1)	25(1)	-3(1)	3(1)	0(1)
C5	26(1)	24(1)	29(1)	-1(1)	0(1)	1(1)
C6	30(2)	38(1)	32(1)	-2(1)	4(1)	3(1)
C8	28(1)	32(1)	30(1)	2(1)	0(1)	-3(1)
C9	25(1)	36(1)	25(1)	0(1)	0(1)	-3(1)
C10	32(1)	27(1)	27(1)	3(1)	-1(1)	-2(1)
C11	35(2)	50(2)	33(1)	-3(2)	-6(1)	-11(1)
C12	34(1)	27(1)	27(1)	-3(1)	1(1)	1(1)
C13	43(2)	36(1)	44(2)	-8(2)	-11(2)	3(1)
C14	43(2)	39(2)	46(2)	-11(2)	-17(2)	11(1)
C15	43(2)	28(1)	35(1)	-3(1)	-1(1)	1(1)
C16	34(2)	33(1)	44(2)	-5(1)	-6(1)	1(1)
C17	36(2)	34(1)	39(2)	-4(1)	-5(1)	5(1)

TABLE : Anisotropic temperature factors (A^2, $\times 10^3$) for or83

	U 11	U 22	U 33	U 12	U 13	U 23
BR	52(0)	66(1)	94(1)	7(0)	22(0)	48(0)
O1	28(2)	52(3)	54(2)	0(2)	0(2)	-14(2)
O2	62(3)	69(3)	59(3)	-4(3)	-15(2)	22(3)
O3	84(4)	101(5)	79(3)	25(4)	39(3)	52(3)
N1	41(3)	30(3)	45(3)	-2(2)	-1(2)	3(2)
N2	30(2)	55(3)	66(3)	-4(2)	12(2)	-5(3)
C1	24(2)	39(3)	37(3)	4(2)	2(2)	8(2)
C2	24(2)	27(3)	36(2)	-1(2)	6(2)	-1(2)
C3	32(3)	29(3)	30(2)	-2(2)	-1(2)	1(2)
C4	23(2)	30(3)	37(2)	8(2)	5(2)	2(2)
C5	21(2)	35(3)	40(3)	2(2)	6(2)	2(2)
C6	27(2)	36(3)	48(3)	3(2)	12(2)	5(3)
C7	41(3)	44(3)	33(3)	6(3)	-2(2)	-2(2)
C8	37(3)	40(3)	41(3)	3(3)	7(2)	-4(2)
C9	31(3)	35(3)	35(2)	-4(2)	5(2)	-5(2)

TABLE : Anisotropic temperature factors (A^{o2} ,$x10^{3}$) for 1t06

	U 11	U 22	U 33	U 12	U 13	U 23
BR	16(0)	24(0)	31(0)	2(0)	7(0)	16(0)
O1	12(1)	20(1)	20(1)	2(1)	0(1)	-6(1)
O2	19(1)	29(1)	24(1)	0(1)	-5(1)	8(1)
O3	26(1)	32(2)	30(2)	3(1)	13(1)	10(1)
N1	12(1)	7(1)	38(2)	1(1)	-10(1)	-7(1)
N2	11(1)	24(1)	22(2)	0(1)	4(1)	-4(1)
C1	9(1)	16(1)	14(1)	0(1)	1(1)	1(1)
C2	9(1)	15(1)	13(1)	-1(1)	2(1)	-1(1)
C3	12(1)	14(1)	16(2)	0(1)	3(1)	0(1)
C4	11(1)	15(1)	16(2)	1(1)	3(1)	0(1)
C5	8(1)	15(1)	17(2)	0(1)	1(1)	0(1)
C6	10(1)	13(1)	18(2)	1(1)	5(1)	3(1)
C7	15(1)	23(1)	15(2)	2(1)	0(1)	-1(1)
C8	15(1)	15(1)	15(2)	-1(1)	3(1)	-1(1)
C9	9(1)	16(1)	16(1)	-1(1)	3(1)	1(1)

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR OR67 DAVE C10H11ON SP.GR. PBCA NO

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
2	0	0	14	-13	4	1	1	52	55	7	5	1	5	-4	3	2	2	6	7	4	7	2	7	-6
4	0	0	326	-346	5	1	1	36	38	9	5	1	6	6	4	2	2	14	14	8	7	2	6	7
6	0	0	17	18	6	1	1	42	44	11	5	1	4	3	5	2	2	22	-22	12	7	2	4	-3
8	0	0	79	78	7	1	1	44	-43	13	5	1	4	-4	6	2	2	43	-43	1	8	2	5	-6
10	0	0	10	-10	8	1	1	24	-24	0	6	1	12	11	7	2	2	15	-16	3	8	2	5	-5
12	0	0	15	-15	9	1	1	12	-12	1	6	1	24	-23	8	2	2	3	-2	1	9	2	5	-5
14	0	0	7	8	10	1	1	7	-7	3	6	1	19	-20	9	2	2	19	19	4	9	2	[illegible]	7
16	0	0	6	5	11	1	1	22	22	4	6	1	10	-11	10	2	2	14	14	7	9	2	6	-7
18	0	0	5	-5	15	1	1	11	-10	5	6	1	15	16	11	2	2	11	12	1	1	3	10	-10
2	1	0	16	16	19	1	1	3	4	7	6	1	9	10	13	2	2	10	-10	2	1	3	10	10
4	1	0	50	51	0	2	1	5	5	8	6	1	8	8	15	2	2	6	-6	3	1	3	40	40
8	1	0	16	-15	1	2	1	13	-13	9	6	1	7	-8	17	2	2	3	4	4	1	3	35	-34
10	1	0	15	-13	2	2	1	8	7	3	7	1	5	5	18	2	2	4	-4	5	1	3	18	-18
14	1	0	9	7	3	2	1	20	-18	7	7	1	6	-6	1	3	2	9	8	6	1	3	16	17
16	1	0	4	3	4	2	1	12	-12	11	7	1	5	5	2	3	2	17	17	7	1	3	21	-19
2	2	0	51	-47	5	2	1	6	-4	0	8	1	5	5	4	3	2	11	11	8	1	3	14	13
4	2	0	15	13	6	2	1	10	-11	0	0	2	26	28	5	3	2	4	-3	9	1	3	13	13
6	2	0	59	60	7	2	1	4	4	2	0	2	7	-7	6	3	2	4	3	10	1	3	16	-16
8	2	0	6	6	8	2	1	12	12	3	0	2	83	87	7	3	2	5	-5	11	1	3	8	7
10	2	0	22	-24	9	2	1	7	7	4	0	2	49	-50	8	3	2	10	-11	13	1	3	6	-6
12	2	0	9	-9	10	2	1	3	3	5	0	2	24	-24	9	3	2	4	-4	14	1	3	8	8
16	2	0	6	7	12	2	1	6	-6	6	0	2	16	18	10	3	2	13	-13	16	1	3	4	-3
4	3	0	5	-5	13	2	1	5	-5	7	0	2	36	-36	11	3	2	5	5	0	2	3	59	54
6	3	0	21	20	1	3	1	20	-17	8	0	2	53	53	12	3	2	4	5	1	2	3	34	-32
10	3	0	5	5	2	3	1	46	-43	9	0	2	11	12	14	3	2	7	7	2	2	3	9	-9
14	3	0	6	-5	3	3	1	5	-6	10	0	2	11	-12	0	4	2	33	-30	3	2	3	23	-22
0	4	0	48	42	4	3	1	16	16	11	0	2	11	10	1	4	2	16	15	4	2	3	16	-16
4	4	0	30	-31	5	3	1	24	24	12	0	2	27	-25	2	4	2	2	-2	5	2	3	25	26
8	4	0	10	10	6	3	1	13	14	13	0	2	9	-8	3	4	2	19	18	6	2	3	9	8
2	5	0	7	8	8	3	1	11	-11	14	0	2	6	6	4	4	2	15	14	7	2	3	4	4
4	5	0	12	12	9	3	1	18	-18	16	0	2	5	6	5	4	2	9	-10	8	2	3	9	-8
8	5	0	8	-8	12	3	1	3	4	17	0	2	7	6	6	4	2	6	5	9	2	3	10	-10
0	6	0	25	24	13	3	1	10	9	1	1	2	19	18	7	4	2	12	-12	10	2	3	4	-4
4	6	0	17	-18	15	3	1	4	-3	2	1	2	98	-101	9	4	2	8	8	12	2	3	7	7
8	6	0	8	9	0	4	1	47	-40	3	1	2	37	-37	10	4	2	5	-4	16	2	3	4	-3
2	7	0	16	17	1	4	1	45	40	4	1	2	71	-72	11	4	2	5	4	1	3	3	9	9
4	7	0	5	-6	2	4	1	5	6	5	1	2	10	-10	13	4	2	6	-6	2	3	3	27	-25
6	7	0	11	-11	3	4	1	40	38	6	1	2	28	28	17	4	2	3	4	3	3	3	28	-28
8	7	0	6	7	4	4	1	27	25	7	1	2	25	26	1	5	2	7	8	5	3	3	4	-3
10	7	0	5	5	5	4	1	20	-21	8	1	2	39	39	2	5	2	4	5	6	3	3	18	19
0	8	0	16	-17	6	4	1	7	-7	9	1	2	11	11	3	5	2	11	-11	7	3	3	27	28
4	8	0	10	12	7	4	1	15	-16	10	1	2	5	-4	4	5	2	5	-6	9	3	3	5	4
6	8	0	4	4	8	4	1	8	-8	11	1	2	11	-11	7	5	2	9	8	10	3	3	16	-17
2	9	0	5	5	9	4	1	3	4	12	1	2	9	-9	0	6	2	23	22	11	3	3	17	-18
6	9	0	5	-5	11	4	1	3	4	13	1	2	9	-8	1	6	2	7	-7	13	3	3	4	-5
1	1	1	69	-66	2	5	1	8	-9	17	1	2	4	4	4	6	2	14	-14	14	3	3	8	8
2	1	1	121	-123	3	5	1	3	4	0	2	2	34	-31	5	6	2	7	7	15	3	3	8	7
3	1	1	73	74	6	5	1	6	6	2	2	2	35	31	9	6	2	3	-3	17	3	3	4	3

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
0	4	3	41	-36	5	1	4	18	-18	9	5	4	7	8	8	3	5	6	7	4	0	6	46	-46
1	4	3	17	15	6	1	4	18	-18	10	5	4	8	8	9	3	5	14	-14	5	0	6	10	10
3	4	3	14	14	7	1	4	22	-22	11	5	4	7	-7	13	3	5	8	7	7	0	6	4	3
4	4	3	28	28	9	1	4	6	5	0	6	4	24	24	0	4	5	15	-15	8	0	6	23	23
5	4	3	13	-13	10	1	4	5	4	3	6	4	4	-4	2	4	5	13	13	9	0	6	8	-9
6	4	3	8	8	11	1	4	9	9	4	6	4	20	-21	4	4	5	12	12	11	0	6	4	-4
7	4	3	10	-10	12	1	4	6	-5	7	6	4	7	7	5	4	5	8	9	12	0	6	6	-6
8	4	3	15	-16	16	1	4	4	3	8	6	4	12	13	6	4	5	21	-22	13	0	6	4	4
9	4	3	9	9	0	2	4	29	-28	11	6	4	4	-4	7	4	5	11	11	15	0	6	4	3
11	4	3	8	9	2	2	4	13	-13	12	6	4	5	-5	8	4	5	9	-8	1	1	6	23	-24
12	4	3	8	8	3	2	4	7	-7	13	6	4	4	3	9	4	5	13	-15	2	1	6	68	69
1	5	3	6	-5	4	2	4	19	20	2	7	4	3	2	10	4	5	9	10	3	1	6	22	23
2	5	3	6	6	5	2	4	13	13	8	7	4	5	6	11	4	5	11	-11	4	1	6	28	-28
3	5	3	22	22	6	2	4	20	21	12	7	4	5	-4	12	4	5	5	5	5	1	6	12	12
5	5	3	9	-10	7	2	4	13	13	5	8	4	5	6	13	4	5	12	12	6	1	6	38	-37
6	5	3	7	-8	8	2	4	9	-9	9	8	4	5	-6	15	4	5	6	6	7	1	6	11	-12
7	5	3	20	-21	9	2	4	13	-13	2	9	4	5	-6	17	4	5	6	-5	8	1	6	16	15
9	5	3	7	8	10	2	4	10	-11	6	9	4	5	5	1	5	5	18	-17	10	1	6	15	15
10	5	3	8	9	11	2	4	11	-11	1	1	5	3	3	2	5	5	16	15	11	1	6	7	7
11	5	3	10	10	12	2	4	2	2	3	1	5	16	14	3	5	5	16	14	12	1	6	6	-6
14	5	3	6	-5	13	2	4	6	6	3	1	5	4	4	4	5	5	9	10	13	1	6	3	-3
0	6	3	10	10	15	2	4	6	6	4	1	5	20	20	5	5	5	15	15	14	1	6	6	-6
1	6	3	12	-12	1	3	4	7	-8	5	1	5	4	-4	6	5	5	3	-4	15	1	6	4	-4
4	6	3	5	-6	2	3	4	20	-19	6	1	5	7	-7	7	5	5	11	-12	0	2	6	8	-8
5	6	3	13	14	4	3	4	12	12	7	1	5	5	-5	8	5	5	9	9	2	2	6	7	-7
9	6	3	8	-9	5	3	4	3	-2	8	1	5	13	-13	9	5	5	8	-7	4	2	6	3	3
1	7	3	7	-7	6	3	4	5	5	9	1	5	4	4	10	5	5	4	-4	6	2	6	5	5
2	7	3	3	3	7	3	4	6	5	10	1	5	7	7	11	5	5	10	10	7	2	6	5	-5
3	7	3	5	6	8	3	4	8	-9	11	1	5	6	6	12	5	5	3	4	8	2	6	4	4
0	0	4	112	125	9	3	4	9	9	13	1	5	4	-3	15	5	5	6	-6	9	2	6	7	7
1	0	4	42	44	10	3	4	6	-6	15	1	5	3	-4	0	6	5	14	-14	10	2	6	4	-4
2	0	4	8	-8	11	3	4	6	-6	0	2	5	9	-8	1	6	5	8	-8	11	2	6	5	4
3	0	4	16	17	13	3	4	7	-7	1	2	5	19	-19	2	6	5	3	-3	12	2	6	7	-6
4	0	4	55	-56	0	4	4	5	-3	2	2	5	10	-10	3	6	5	10	-10	13	2	6	5	-6
5	0	4	51	-51	1	4	4	20	18	3	2	5	36	-34	4	6	5	7	8	14	2	6	3	4
6	0	4	19	21	4	4	4	3	4	6	2	5	10	10	6	6	5	6	6	16	2	6	5	4
7	0	4	7	-7	5	4	4	19	-19	7	2	5	20	19	7	6	5	5	6	1	3	6	4	-2
9	0	4	25	25	7	4	4	6	6	8	2	5	11	11	4	7	5	9	-10	2	3	6	15	14
10	0	4	9	-9	9	4	4	9	9	10	2	5	4	-5	8	7	5	7	8	3	3	6	3	-3
11	0	4	8	8	11	4	4	4	-4	11	2	5	6	-6	9	7	5	4	-5	4	3	6	4	-4
12	0	4	14	14	1	5	4	13	13	12	2	5	7	-8	11	7	5	5	5	5	3	6	10	10
13	0	4	8	-7	2	5	4	13	14	1	3	5	12	11	0	8	5	6	5	6	3	6	6	-6
14	0	4	3	2	3	5	4	11	-11	2	3	5	31	-30	1	9	5	7	-7	9	3	6	10	-10
15	0	4	6	-6	4	5	4	8	9	3	3	5	24	24	5	9	5	7	8	11	3	6	5	6
16	0	4	8	-7	5	5	4	11	-12	4	3	5	10	-9	0	0	6	59	59	15	3	6	4	-4
1	1	4	3	3	6	5	4	11	-12	5	3	5	11	10	1	0	6	16	-16	0	4	6	16	15
2	1	4	33	34	7	5	4	9	9	6	3	5	10	10	2	0	6	3	4	1	4	6	7	6
3	1	4	28	27	8	5	4	5	-5	7	3	5	7	8	3	0	6	8	-9	2	4	6	3	-4

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
3	4	6	9	9	2	3	7	4	4	3	0	8	11	11	4	5	8	9	9	15	3	9	4	3
4	4	6	11	-12	4	3	7	19	19	4	0	8	3	-2	5	5	8	10	10	0	4	9	12	12
5	4	6	6	-5	6	3	7	6	6	5	0	8	11	11	7	5	8	7	-7	1	4	9	29	29
6	4	6	5	4	7	3	7	4	4	6	0	8	4	-3	8	5	8	7	-7	2	4	9	9	9
7	4	6	8	-8	8	3	7	15	-15	7	0	8	7	-7	9	5	8	5	-5	3	4	9	15	16
8	4	6	6	7	10	3	7	9	-9	8	0	8	6	-7	11	5	8	3	3	4	4	9	12	-12
9	4	6	7	7	11	3	7	3	-3	9	0	8	4	-5	1	6	8	18	19	5	4	9	27	-27
13	4	6	6	-6	12	3	7	5	5	12	0	8	5	5	3	6	8	12	12	6	4	9	15	-15
2	5	6	25	25	14	3	7	6	5	1	1	8	53	-51	5	6	8	16	-16	7	4	9	5	6
5	5	6	6	6	0	4	7	5	-6	2	1	8	10	-9	7	6	8	5	-4	8	4	9	8	9
6	5	6	16	-17	2	4	7	10	-10	3	1	8	55	55	9	6	8	8	8	9	4	9	14	14
9	5	6	7	-6	3	4	7	6	7	4	1	8	7	-7	1	7	8	4	-5	10	4	9	7	7
10	5	6	8	8	5	4	7	6	6	5	1	8	19	19	2	7	8	5	-6	11	4	9	5	4
0	6	6	40	41	6	4	7	17	18	6	1	8	6	5	3	7	8	6	7	12	4	9	5	5
3	6	6	6	6	7	4	7	6	-6	7	1	8	26	-26	7	7	8	5	-5	13	4	9	4	-4
4	6	6	30	-31	8	4	7	7	8	8	1	8	9	8	11	8	8	4	4	1	5	9	14	14
6	6	6	5	5	9	4	7	4	-5	9	1	8	5	-5	0	8	8	7	-9	2	5	9	6	-6
7	6	6	8	-8	10	4	7	7	-8	11	1	8	10	10	1	8	8	5	5	3	5	9	7	-7
8	6	6	15	15	12	4	7	9	-8	12	1	8	6	-6	4	8	8	5	6	4	5	9	7	7
10	6	6	4	-4	16	4	7	5	5	15	1	8	4	-3	5	8	8	4	-5	5	5	9	10	-10
2	7	6	9	10	1	5	7	20	-21	0	2	8	23	-21	1	1	9	4	4	6	5	9	13	12
4	7	6	5	-5	2	5	7	55	-56	1	2	8	11	10	2	1	9	18	18	8	5	9	8	-8
6	7	6	6	-7	3	5	7	27	28	3	2	8	4	4	3	1	9	8	8	9	5	9	4	3
8	7	6	5	6	5	5	7	6	6	4	2	8	10	10	4	1	9	4	5	10	5	9	13	-13
0	8	6	3	2	6	5	7	29	30	5	2	8	15	-13	5	1	9	12	-11	12	5	9	4	4
2	9	6	3	-5	7	5	7	18	-19	7	2	8	4	-3	6	1	9	6	-6	14	5	9	8	7
1	1	7	3	-2	10	5	7	8	-9	9	2	8	14	13	7	1	9	6	-6	0	6	9	13	-14
2	1	7	32	-33	11	5	7	9	8	10	2	8	3	2	8	1	9	2	-2	4	6	9	8	8
3	1	7	12	13	0	6	7	25	25	11	2	8	4	3	9	1	9	8	8	6	6	9	4	4
4	1	7	5	5	1	6	7	8	-9	13	2	8	10	-9	0	2	9	6	6	1	7	9	4	4
5	1	7	3	3	3	6	7	6	-6	1	3	8	13	-11	2	2	9	5	-4	3	7	9	8	-8
6	1	7	12	12	4	6	7	16	-16	2	3	8	12	-11	3	2	9	4	-4	4	7	9	8	-8
7	1	7	13	-13	5	6	7	5	6	3	3	8	20	18	4	2	9	11	-11	7	7	9	8	7
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H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
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15	5	[illegible]	8	-8	2	2	4	12	-13	15	5	4	13	15	5	3	5	21	21	4	0	6	56	-55
16	5	[illegible]	7	-7	3	2	4	6	-6	16	5	4	6	5	6	3	5	15	14	5	0	6	15	16
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8	1	6	29	29	16	4	6	7	6	4	3	7	31	31	13	0	8	6	3	11	3	8	8	9
10	1	6	[illegible]	32	6	5	6	23	-24	6	3	7	8	7	14	0	8	4	5	14	3	8	4	-6
11	1	6	[illegible]	-9	7	5	6	7	6	7	3	7	7	5	15	0	8	11	10	17	3	8	7	-7
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H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
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H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
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H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
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OBSERVED AND CALCULATED STRUCTURE FACTORS FOR LEUCODRIN

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
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6	0	0	14	14	4	8	0	5	5	6	5	1	10	10	3	3	2	37	34	0	1	3	11	11
2	1	0	41	39	1	9	0	10	10	0	6	1	4	5	4	3	2	20	18	1	1	3	38	38
3	1	0	46	42	2	9	0	9	8	1	6	1	29	29	5	3	2	23	23	2	1	3	29	27
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2	2	0	70	71	4	0	1	19	18	2	7	1	19	20	5	4	2	12	13	1	2	3	20	20
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H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
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CREATED BY
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