CHAPTER-3

MOLECULAR AND CRYSTAL STRUCTURE OF "5, 7, 4'-TRIHYDROXY-6, 3'-DIPRENYLISOFLAVONE" [C_{25} H_{26} O_5] MN-02

3.1 INTRODUCTION

5, 7, 4'-trihydroxy-6, 3'-diprenylisoflavone (MN-02) one of the most important isoflavonoids found in the plant fruits *Cudrania javanensis*[1] of Moraceae family of neuroprotective activity[2,3].Again prenylated flavonoid has more antioxidative effect than non-prenylated flavonoid[4].

Here we will explain the single crystal structure elucidation[5-10] of its (MN-02) The analysis of this structure is undertaken in order to get accurate bond distances of this derivative. The study of the title compound will help to understand the nature of molecular packing in crystalline space. It also aimed at finding the molecular geometry and molecular conformations of the crystal.

3.2 CRYSTAL GROWTH

For the growth of the single crystal of the compound (MN-02) toluene and ethyl acetate (95:05) as solvent. A glass tube stand is used to keep the tube undisturbed for growing the crystals. The compound (C_{25} H_{26} O_5) powder is dissolved in minimum volume of toluene until the solution becomes saturated and add a few drops of ethyl acetated in the ratio 95:05.The mouth of the tube is covered by a piece of aluminum foil to slow down the rate of evaporation. Precaution has been taken so that no foreign particles could enter into the solution. During the entire seeding period, solution was kept undisturbed. After a week some pale yellow crystalline solid of size 0.50 x 0.15 x 0.08 mm^3 are obtained. The crystals are taken out of the solution carefully and allowed

to dry in a glass plate inside a vacuum dessicator. The crystal growth took place at room temperature (29+3°c)

3.3 SELECTION OF THE CRYSTAL

A good crystal is selected by observing it under a polarizing microscope. Deformed or twinned crystals are rejected. The cross section of the instance region of x-ray is ordinarily about 1mm in diameter, care have been taken to select a crystal of this range. Stress is given on the surface morphology of the crystal so that it can be oriented about the desired axis.

3.4 EXPERIMENTAL

Compound (MN-02) have been isolated by chromatographic method in the laboratory of Institute of Advanced Studies in Science &Technology (IASST) Guwahati, Assam, India. from the fruits extract of *Cudrania javanensis* and colourless needle like crystal are grown in toluene for our further study.

Applying Oscillation and Weissenberg technique the preliminary crystal data are determined using CuKα radiation. The crystals are found to be monoclinic. The zero layer line Weissenberg photographs about different axes revealed that hkl have no condition but reflections (1).hkl: h+k = 2n; 0kl: h = 2n ;(2). h0l : h, l = 2n ; hk0 ; h+k =2n ; (3).h00 : h = 2n ; 0k0 : k = 2n; 00l : l = 2n. have been absent.

These absences clearly establish the space group is C_2/c with the general equivalent positions ±(x, y, z; x,-y, z+.5 ; x+.5, y+.5, z; x+.5,-y+.5 ; z+.5)

The number of molecules per unit cell is found to be 8; hence the asymmetric unit must consist of 2 molecules

For the collection of intensity data, a single crystal of size 0.50 x 0.15 x 0.08 mm^3 is

selected. Three dimensional intensity data are collected at 296(2) K temperature with Bruker 3-circle diffractometer (Bruker Nonius SMART APEX 2) equipped with CCD area detector, and using graphite monochromated MoK_{α} radiation (λ = 0.71073 Å) from 60W microfocus Siemens Microsource with glass polycapillary optics in Chemistry Department, Indian Institute of Technology Guwahati(IITG). Bruker SMART software[11] is used for data collection and also for indexing the reflections and the unit cell parameters. The collected data were integrated using SAINT software[11]. The structures are solved by direct methods and refined by full-matrix least squares calculation using SHELXTL software[13, 14]. Lattice parameters are determined from θ values in the range $1.61 < \theta < 26.66$. A total of 11024 reflections are recorded for the values of θ up to 26.66, out of which 3900 reflections are unique. Absorption corrections are not applied.

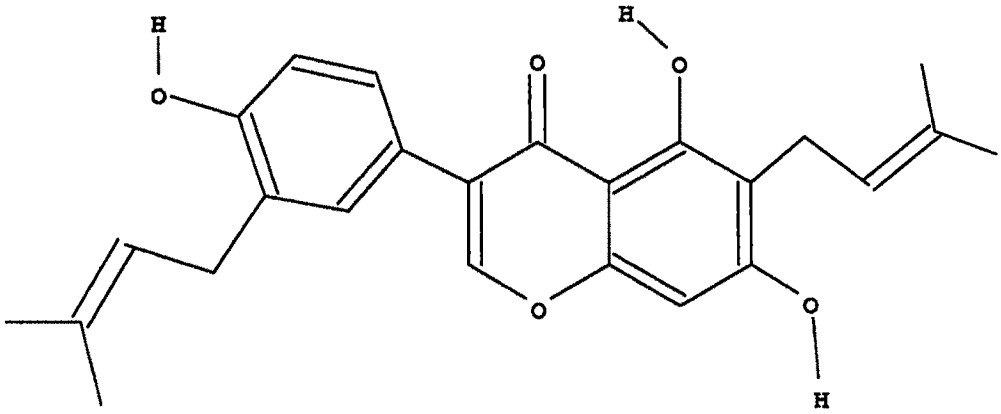

Figure 3.1: Structure of 5, 7, 4'-trihydroxy-6, 3'-diprenylisoflavone, (MN-02)

All the non-H atoms are refined in the anisotropic approximation against F^2 of all reflections. The H-atoms except those attached to N, O and F are placed at their calculated positions and refined in the isotopic approximations, those attached to heteroatom (N, O and F) are located from the difference Fourier maps, and refined with isotopic displacement co-efficient. The final refinement cycle converged R=0.0546, and wR (F^2) = 0.1204. Final cycle of refinement resulted in the residual electron density in the range and 0.354 and -0.166 e.$Å^{-3}$. Atomic scattering factors are taken from International Tables for X-ray crystallography[15]. The accurate cell parameters are determined by a least-squares refinement of the setting angles of 28 reflections lying in the range $2.5°\leq\theta\leq24.74°$.A total of 11024 independent reflections up to 80° in 2θ with I>2δ (I) are measured by the ω-2θ scan technique, the scan speed being 2.5°/min. The hkl range of measured reflections are -31<=h<=27, -9<=k<=9, -28<=l<=27.

This assured stability of the crystal at the time of data collection.Out of 11024 reflections measured, 3900 are unique and the value of R_{int} from merging equivalent 2 reflections is .0546 for 2789 having Fo > 4σ(Fo) are considered observed and used in the structure analysis. The intensities are corrected for the Lorenz and polarization factors but no absorption correction is made. The covalent structure of MN-02 is given in the figure 3.1.Crystal data and other experimental details are given in the table 3.1.

3.5 RESULTS AND DISCUTION

From the XRD data of single crystal is collected at 296k temperature with a Bruker 3-circle diffractometer. In the analysis of the crystal structure of compound MN-02, it has been observed that the values obtained for bond lengths and bond angles are on

the average; satisfy the requirement of the molecular valancy structure. The results of investigation reveal that the crystal structure comprises 8 molecules per asymmetric unit. The ORTEP diagram[16] of (MN-02) with the atomic numbering scheme is shown in the figure 3.2. table 3.3: anisotropic displacement parameters ($Å^2 x\ 10^3$) for (MN-02).The anisotropic displacement factor exponent takes the form: $-2\pi^2$ [$h^2 a^{*2} U^{11}$ + ... + 2 h k a* b* U^{12}]. Table 3.3. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2 x\ 10^3$) for (MN-02). The least of atomic co-ordinate and equivalent isotropic displacement parameters ($Å^2 x\ 10^3$) for MN-02. U (eq) is defined as one third of the traces of the orthogonalized Uij tensor are given in the table 3.4. The list of structure factors are also given in Appendix-II.

Table 3.1: Crystal data and structure refinement for MN-02

Identification code	MN-02	
Empirical formula	C25 H26 O5	
Formula weight	406.46	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	C_2/c	
Unit cell dimensions	a = 25.2732(12) Å	α= 90°.
	b = 7.4414(3) Å	β= 93.063(3)°.
	c = 22.3113(9) Å	γ = 90°.
Volume	4190.0(3) $Å^3$	
Z	8	

Density (calculated)	1.289 Mg/m^3
Absorption coefficient	0.089 mm^{-1}
F(000)	1728
Crystal size	0.50 x 0.15 x 0.08 mm^3
Theta range for data collection	1.61 to 26.66°.
Index ranges	-31<=h<=27, -9<=k<=9, -28<=l<=27
Reflections collected	11024
Independent reflections	3900 [R(int) = 0.0509]
Completeness to theta = 26.66°	88.1 %
Absorption correction	None
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3900 / 0 / 280
Goodness-of-fit on F^2	0.928
Final R indices [I>2sigma (I)]	R1 = 0.0817, wR2 = 0.2209
R indices (all data)	R1 = 0.1589, wR2 = 0.2623
Extinction coefficient	0.0002(4)
Largest diff. peak and hole	0.897 and -0.283 $e.Å^{-3}$

3.6 EVALUTION OF SYMMETRY ELEMENTS

Specimen-Single crystal of (MN-02) {5, 7, 4'-trihydroxy-6, 3'-diprenylisoflavone}[3] from the plant material of *Cudrania javanensis.*

X-ray target material = Mo

Applied voltage =50 KV

Applied current =30 mA

Exposure time =8(eight) hours

Wavelength of MoKα = 0.71073Å

Space group = C_2/c

Cell constants:	a = 25.2732(12) Å	α= 90°.
	b = 7.4414(3) Å	β= 93.063(3)°.
	c = 22.3113(9) Å	γ = 90°.
Volume	4190.0(3) $Å^3$	
Z	8	

Instrument used for data collection: Bruker Nonius SMART CCD 3-circle diffractometer

Equipped with graphite monochromator

Number of reflections measured: 11024

Temperature of crystal during data collection: 296(2) K

Crystal dimension =0.50 x 0.15 x 0.08 mm^3

Density calculated: 1.289 Mg/m^3

Absorption coefficient: 0.089 mm^{-1}

F (000) : 1728 .Theta range : 1.61 to 26.66°.

Index ranges : -31<=h<=27, -9<=k<=9, -28<=l<=27

Independent reflection: 3900 [R (int) = 0.0509]

Completeness to theta =26.66°. 88.1 %

Goodness of fit on F2 = 0.928

Refinement method: Full Matrix least- squares on F2

Largest diff.peak and hole: 0.897 and -0.283 $e.Å^{-3}$

3.7 DATA COLLECTION AND REDUCTION

The size of the crystal 0.50 x 0.15 x 0.08 mm^3 Symmetry C2/c is determines using a Bruker Nouius SMART CCD diffractometer. Three dimensional intensity data are collected on a bruker Nonius SMART CCD 3circle diffractometer with MoK_α radiation(λ=·71073Å) equipped with graphite monocromator.The Bruker SMART software[11] is used for data collection and also for indexing the reflections and determining the unit cell. The cell parameter are refined by least squares method on the basis of 3900 independent reflections with I>2σ (I)(-31<=h<=27, -9<=k<=9, -28<=l<=27) are measured by w/2θ scan technique, with scan speed of 2.5°min^{-1}.Lortentz and polarization correction applied but absorption correction is ignored as structure contained only light atoms.

3.8 STRUCTURE DETERMINATION

The structure is solved by direct method refined by full-matrix least-squares calculations using SHELXTL-97 software[14-15].In this program,E-values are calculated by a modified k-curve method (Karle, Hauptmann and Christ,1958)[17]The atoms are refined by full matrix least squres method first isotropically and then anisotropically using the program SHELXL-97. H-atoms are not refined .Altogether 280 paremeters are refined. The E-statistics $<|E^2-1|> = 0.937$ imply that the crystal is non-centrosymmetric. The values of R (int) = 0.0509 and R (sigma) = 0.1009 show that the quality of the data is satisfactory. 320 reflections and 1618. unique TPR (Triple phase relationships), for phase annealing, 581 Phases refined using 7973. unique TPR,

862 reflections and 15968. unique TPR for R_α

The final R value is 0.0538.The function is minimized with weight = 1 / [$sigma^2(Fo^2)$ + (0.1436 * P)^2 + 0.00 * P] where P = (Max (Fo^2, 0) + 2 * Fc^2) / 3

The final R=0.0817for 3900 reflection and goodness of fit=0.928.

The final R indices [I>2 sigma (I)] = R1 = 0.0538, wR2 = 0.1099

. R indices (all data) R1 = 0.1589, wR2 = 0.2623

.A final difference Fourier synthesis revealed the maximum and minimum electron density peaks of 0.897 and -0.283 e.$Å^{-3}$ respectively.

3.9 BOND ANGLES AND BOND LENGTHS (A)

Selected bond lengths and bond angles of the molecule (MN-02) involving non-hydrogen atoms are given in Table 3.5 and Table 3.6 and those involving hydrogen atoms are in table 3.7 and table 3.8

3.10 THE PHENYL RING

The phenolic ring deviates from the isoflavion ring with a torsion angles of C2-C3-C11-C12 = -39.46(54)°and C4-C3-C11-C16 =140.40(40)°.Proved the two plane in the isoflavone are near planer as C2-O1-C9-C10 is 1.71(48)°.

In the molecule {5,7,4'-trihydroxy-6,3'-diprenylisoflavone}[2]the C—C bond length are normal and are in good agreement with the standard value 1.326(2)Å[18].However, the angle C11—C12—C13 in the molecule is slightly lower than the normal value as also found in other molecules. The C11—C12—C13 angle in the molecule is122.94°.The exocyclic bond lengths is C3—C11=1.4789(50)Å which is shorter then the normal C3(sp^2)—C11(sp^2) single bond distance 1.517(5)Å indicates some conjugation between isoflavion group and the phenyl[19-21] ring .

3.11 CONFORMATION OF THE MOLECULE

The molecular conformation can be described by the torsion angles about the various bonds; and the calculations of the least squares planes for various sets of atom in the molecule, together with their e.s.d from the respective planes.

The torsion angles for the molecule MN-02 are listed in the table3.9.The dyhidral angle between the mean plane passing through the ring $C_3C_4C_{10}C_9O_1C_2$ and the phenyl ring $C_{11}C_{12}C_{13}C_{14}C_{15}C_{16}$ is 46(1)° which shows that the two rings are not planar. This can be confirmed by studying the torsion angles w.r.t the bond $C_3 - C_{11}$

Studying the torsion angle through the central bond $C_6 - C_5$ and $C_6 - C_7$ it is seen that C_{17} atom is coplanar with the C_5 C_6 $C_7C_8C_9C_{10}$ ring. After studying the torsion angle C_{22}-C_8-C_7-O_4 and C_9-C_8-C_7-C_6 the ring$C_8C_7C_6O_5C_{10}C_9C_8$ with the ring $C_8C_7O_4C_{24}C_{23}C_{22}$ are not co-planar.

3.12 MOLECULAR PACKING

The crystal structure is stabilized by a net work of intermolecular hydrogen bonds and normal van der Waals contacts. All the intermolecular contacts less than 4.00Å involving the non hydrogen atoms are listed in the table 3.10.

The packing of the molecules of mercury[22] diagram in the crystalline space as viewed down the a-axis, b-axis and c-axis of the unit cell is depicted in figures 3.3, 3.4 and 3.5 respectively.

The details of the hydrogen bond geometry of the present structure are as follows:

D-H....A	D–H	D...A	H...A	D–H...A
O3 -H3O...O2 (i)	0.811(3)	2.573(4)	1.852(3)	147.60(21)
C16 -H16...O2 (ii)	0.930(4)	2.929(5)	2.541(3)	105.43(24)
C17 -H17... O4 (i)	0.970(4)	2.780(5)	2.453(3)	99.28(25)
C17 -H17...O3 (i)	0.970(4)	2.795(5)	2.442(3)	101.05(25)

C22	-H22...O5 (i)	0.970(5)	2.943(5)	2.534(3)	105.37(27)
O4	-H4O...O5 (ii)	0.793(3)	2.741(4)	1.984(3)	159.36(22)
O5	-H5O...O2 (iii)	0.829(3)	2.764(4)	2.005(3)	151.87(20)

Symmetry codes:

(i) x, y, z

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

(iii) -x,-y+1,-z

The molecule contains four strong C–H····O interactions of C16–H16····O2, C17–H17····O4, C22–H22...05 and C17–H17····O3. Besides their some strong intermolecular D·····A interactions also observed inside the molecule with O3·····O2 = 2.573(4) Å, O5·····O2 = 2.764(4) Å and O4·····O5 = 2.741(4) Å

Moreover, it can be concluded that the structural features of the compound investigated fit into general pattern observed in all other isoflavion derivatives. The approximate correlations between the bond distances are indicative of extensive electron delocalization in the molecule. The structural activity of the compound may be estimated from the atomic charge density distribution using the three dimensional crystallographic parameters of the molecule.

3.13 CONCLUSION

The single crystal of (MN-02) {5, 7, 4'-trihydroxy-6, 3'-diprenylisoflavone} has a monoclinic cell with lattice parameters a = 25.2732(12) Å b = 7.4414(3) c = 22.3113(9) Å α =90° β= 93.063(3)°.° γ= 90° with space group C_2/c .

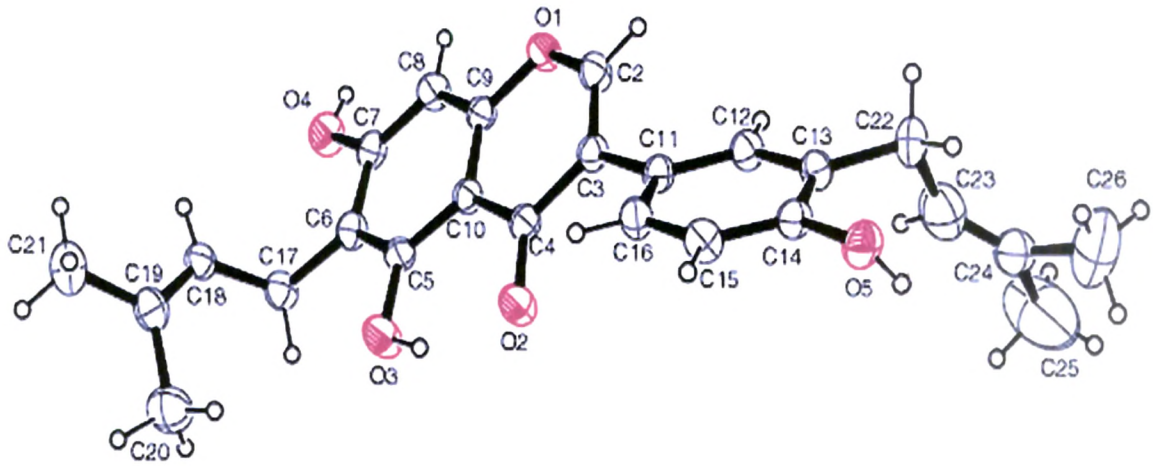

Figure 3.2. An ORTEP view of (MN-02) in 30% probability with atomic numbering scheme.

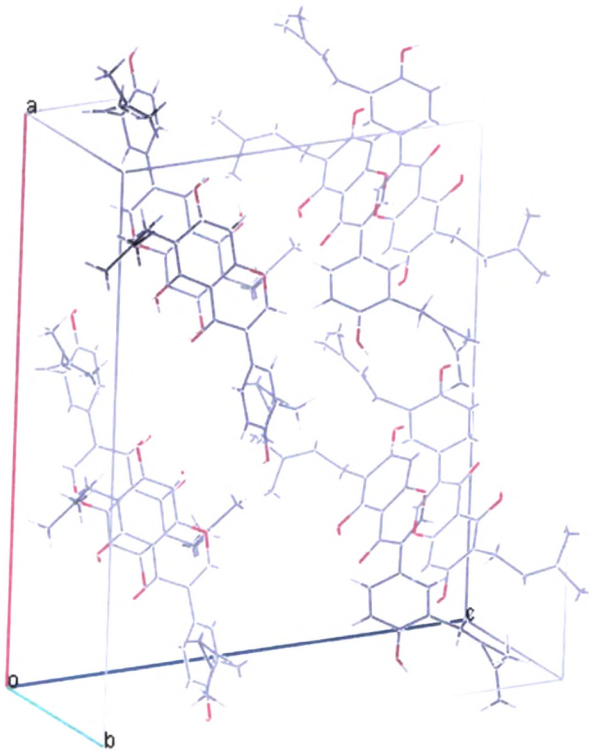

Figure 3.3: The packing of the molecules in the crystalline space as viewed down the a-axis of the unit cell.

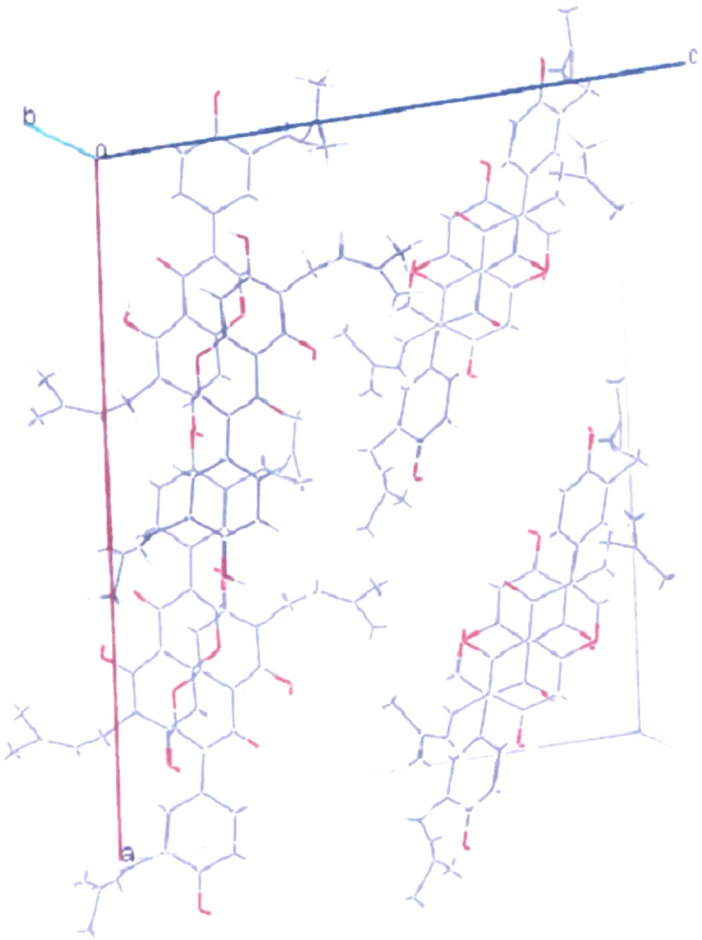

Figure 3.4: The packing of the molecules in the crystalline space as viewed down the b-axis of the unit cell

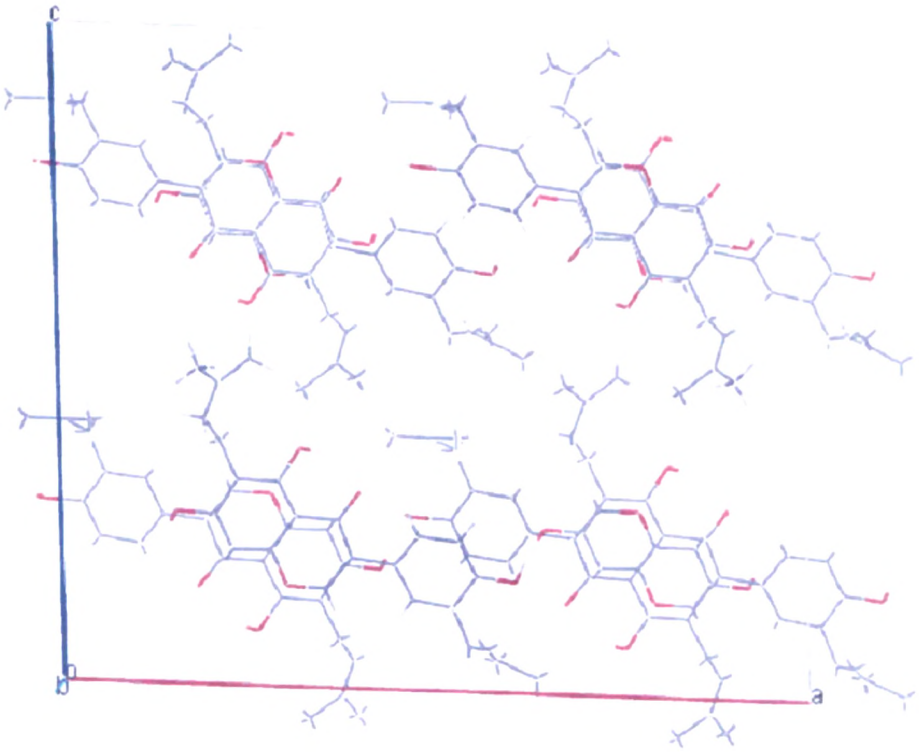

Figure 3.5: The packing of the molecules in the crystalline space as viewed down the c- axis of the unit cell

Table 3.2: Anisotropic displacement parameters ($Å^2 x\ 10^3$) for MN-02.

The anisotropic displacement factor exponent takes the form:

$$-2\pi^2[\ h^2 a^{*2} U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(50)	880(90)	640(70)	300(30)	-270(40)	-270(40)	660(70)
C(23)	520(40)	380(30)	40(7)	54(9)	-72(11)	-380(30)
C(51)	190(20)	800(70)	250(30)	360(40)	-43(17)	-200(30)
O(7)	40(4)	95(5)	44(4)	-12(3)	6(3)	-5(3)
O(2)	31(4)	94(5)	51(4)	-5(3)	-4(3)	15(3)
O(1)	43(4)	71(4)	32(4)	2(3)	-4(3)	5(3)
C(8)	72(7)	60(5)	39(6)	-7(4)	3(5)	-8(5)
C(10)	27(4)	44(5)	40(5)	9(4)	4(4)	3(3)
C(16)	39(5)	66(6)	45(6)	8(5)	15(4)	8(4)
C(2)	28(5)	62(6)	50(6)	-11(4)	4(4)	-1(4)
C(14)	32(5)	33(4)	66(6)	-2(4)	7(4)	1(4)
C(4)	26(5)	53(5)	38(5)	3(4)	-9(4)	-3(4)
C(3)	45(5)	40(5)	49(6)	5(4)	2(5)	8(4)
C(15)	57(6)	43(5)	86(7)	2(5)	17(5)	11(4)
C(18)	54(6)	56(5)	62(7)	-8(5)	36(5)	13(4)
C(13)	41(5)	46(5)	66(6)	2(5)	18(5)	9(4)

C(22)	82(7)	57(6)	81(8)	-3(5)	55(6)	-4(5)
C(9)	34(5)	54(5)	44(6)	3(4)	13(4)	9(4)
C(19)	62(7)	62(6)	47(7)	-9(5)	13(6)	7(5)
C(5)	25(5)	61(5)	40(6)	-12(4)	-10(4)	7(4)
C(6)	37(5)	49(6)	48(6)	-3(4)	1(4)	-2(4)
C(21)	162(12)	84(8)	76(8)	38(6)	23(7)	-24(7)
C(17)	43(6)	61(6)	61(6)	-14(5)	14(5)	6(4)
C(20)	67(7)	122(9)	50(6)	2(6)	12(5)	4(7)
C(7)	38(5)	47(6)	74(8)	5(5)	17(5)	6(4)
C(11)	37(5)	42(5)	51(6)	3(4)	12(5)	-3(4)
C(30)	46(6)	52(5)	42(6)	11(4)	5(4)	-8(4)
C(29)	45(6)	45(5)	54(6)	-3(4)	13(5)	-1(4)
O(6)	36(4)	69(4)	50(4)	-8(3)	0(3)	9(3)
C(31)	37(5)	44(5)	49(6)	3(4)	10(5)	12(4)
C(36)	31(5)	53(5)	46(6)	-4(4)	-5(4)	8(4)
C(34)	31(5)	37(4)	46(6)	-2(4)	-1(4)	-6(3)
C(27)	40(5)	49(5)	49(6)	2(4)	9(5)	9(4)
C(32)	22(4)	52(6)	50(6)	4(4)	0(4)	6(4)
C(38)	32(5)	72(6)	27(4)	2(4)	-12(3)	10(4)
C(28)	21(4)	54(6)	38(5)	0(4)	7(4)	-5(4)
C(39)	25(5)	71(6)	45(5)	-13(5)	-4(4)	7(4)
C(40)	47(6)	51(5)	37(5)	-8(4)	5(4)	-3(4)
C(35)	39(5)	52(5)	46(6)	-8(4)	7(4)	4(4)
C(37)	44(5)	50(5)	52(6)	4(4)	-1(4)	3(4)
C(41)	20(4)	76(6)	43(6)	5(4)	6(4)	8(4)

C(33)	11(4)	48(4)	58(6)	8(4)	-9(4)	10(3)
O(10)	32(3)	72(4)	63(4)	-7(3)	1(3)	11(3)
C(47)	44(5)	67(5)	49(6)	10(4)	-2(4)	3(4)
O(8)	49(4)	105(5)	39(4)	-3(4)	-6(3)	-2(4)
O(3)	40(4)	110(5)	58(5)	-16(4)	7(3)	24(4)
C(42)	53(6)	52(5)	57(6)	7(5)	8(5)	3(4)
C(12)	58(6)	35(4)	45(6)	0(4)	-2(4)	5(4)
O(4)	30(3)	83(5)	73(5)	-4(4)	10(3)	19(3)
O(5)	40(4)	86(5)	76(5)	-4(4)	11(3)	9(3)
O(9)	45(4)	68(4)	74(5)	6(4)	-2(3)	6(3)
C(24)	147(12)	67(7)	70(8)	-11(6)	-38(7)	-13(7)
C(49)	59(6)	76(7)	69(7)	1(5)	26(5)	-5(5)
C(43)	26(5)	70(6)	46(6)	-5(5)	-3(4)	3(4)
C(48)	68(4)	57(4)	80(5)	16(3)	36(4)	30(4)
C(44)	45(6)	69(7)	61(7)	8(5)	13(5)	7(5)
C(45)	54(5)	85(7)	63(6)	-6(5)	4(4)	5(4)
C(46)	102(10)	92(8)	96(9)	12(7)	-40(7)	-3(7)
C(25)	53(4)	92(5)	97(6)	1(4)	15(4)	-7(4)
C(26)	287(17)	60(5)	140(10)	-3(5)	49(11)	-96(8)

Table 3.3: Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for MN-02.

	x	y	z	U(eq)
H (3O)	1914	4574	-1272	79
H(4O)	4023	3442	-50	73
H(5O)	-759	6274	671	72
H(2)	1828	6314	1158	59
H(6)	3393	4577	564	57
H(12)	974	5061	1221	55
H(15)	-34	8166	-208	60
H(16)	848	7656	-348	59
H(17A)	3570	2723	-1395	63
H(17B)	3017	3072	-1726	63
H(19A)	2559	5487	-2492	101
H(19B)	2887	5845	-3057	101
H(19C)	2934	3991	-2725	101
H(21A)	3892	7989	-2230	120
H(21B)	3342	8621	-2514	120
H(21C)	3716	7435	-2889	120
H(22A)	-340	5821	1645	71
H(22B)	235	5648	1943	71

H(23)	250	2654	1434	130
H(26A)	-925	2462	2379	222
H(26B)	-1202	2246	1738	222
H(26C)	-947	4091	1933	222
H(25A)	-800	-47	1768	485
H(25C)	-217	-177	2041	485
H(25B)	-326	-131	1343	485
H(18)	3801(17)	5650(50)	-1558(18)	46(11)
H (50C)	5405	5377	3584	930
H (50A)	5365	4986	4271	930
H(50B)	5777	6391	4057	930
H(23A)	5506	1794	1018	376
H(23B)	6014	882	847	376
H(51A)	4562	5453	4114	624
H(51B)	4416	6885	3617	624
H(51C)	4401	7404	4297	624
H(6)	2089	325	1718	69
H(16)	4616	-2609	2638	59
H(2)	3623	-1437	1126	56
H(15)	5503	-3145	2509	73
H(18B)	1576	4	4093	67
H(18A)	1953	-1211	3745	67
H(22)	5404	-897	325	86
H(21C)	1734	-2573	5148	160
H(21B)	2079	-3712	4725	160

H(21A)	1539	-2890	4478	160
H(17A)	1896	2307	3675	66
H(17B)	2452	1936	4001	66
H(19A)	2908	-498	4776	119
H(19B)	2580	-856	5341	119
H(19C)	2533	998	5009	119
H(27)	7266	11242	3437	55
H(38)	5423	13139	2093	53
H(37)	6313	12689	1940	58
H(41)	6436	10061	3511	56
H(33)	8862	9472	2855	47
H(10O)	4707	11256	2961	84
H(47B)	5102	10812	3911	65
H(47A)	5673	10782	4232	65
H(8O)	7372	9432	1027	97
H(3O)	3541	261	3572	104
H(42B)	9047	7785	904	65
H(42A)	8497	8068	559	65
H(12)	4489	-60	1071	55
H(4O)	1414	1288	2326	93
H(8)	6177	-2767	1931	101
H(10)	9462	8171	2227	94
H(43)	9253	10786	739	57
H(45A)	9299	13143	63	101
H(45C)	8768	13492	-316	101

H(45B)	9217	12334	-584	101
H(46A)	8161	9433	-132	147
H(46B)	8490	9689	-701	147
H(46C)	8131	11254	-485	147
H(25B)	6671	2515	510	120
H(25A)	6376	2473	-126	120
H(25C)	6383	758	282	120
H(26C)	5536	5267	696	242
H(26B)	5943	5268	189	242
H(26A)	6146	5196	865	242

Table 3.4: Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for MN-02. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	2457(1)	5528(4)	765(1)	49(1)
O(2)	1433(1)	5135(4)	-703(1)	59(1)
O(3)	2222(1)	4440(4)	-1349(1)	66(1)
O(4)	3883(1)	3578(4)	-374(1)	61(1)
O(5)	-595(1)	7230(4)	636(1)	60(1)

C(2)	1942(2)	5939(5)	789(2)	49(1)
C(3)	1574(1)	5863(5)	333(2)	42(1)
C(4)	1753(1)	5314(5)	-248(2)	44(1)
C(5)	2531(1)	4482(5)	-838(2)	43(1)
C(6)	3059(2)	4055(5)	-865(2)	45(1)
C(7)	3374(1)	4063(5)	-328(2)	45(1)
C(8)	3175(1)	4555(5)	215(2)	48(1)
C(9)	2646(1)	5013(4)	226(2)	41(1)
C(10)	2304(1)	4971(5)	-290(2)	41(1)
C(11)	1009(1)	6262(5)	415(2)	42(1)
C(12)	769(1)	5677(5)	930(2)	46(1)
C(13)	235(2)	5977(5)	1028(2)	47(1)
C(14)	-61(1)	6876(5)	582(2)	45(1)
C(15)	169(2)	7521(5)	78(2)	50(1)
C(16)	698(2)	7217(5)	-6(2)	49(1)
C(17)	3290(2)	3603(5)	-1460(2)	52(1)
C(18)	3509(2)	5236(6)	-1753(2)	48(1)
C(19)	3340(2)	5983(6)	-2262(2)	58(1)
C(20)	2891(2)	5265(8)	-2661(2)	84(2)
C(21)	3595(2)	7660(7)	-2495(2)	80(2)
C(22)	7(2)	5293(6)	1600(2)	60(1)
C(24)	-405(2)	2234(7)	1729(2)	74(1)
C(23)	-41(3)	3243(10)	1581(3)	108(2)
C(26)	-915(3)	2809(12)	1965(4)	148(3)
C(25)	-440(7)	311(15)	1719(5)	323(15)

Table 3.5: Bond distances (Å) involving non-hydrogen atoms with e.s.d's in parenthesis.

O(1)-C(2)	1.342(4)	C(9)-C(10)	1.401(5)
O(1)-C(9)	1.372(4)	C(11)-C(16)	1.388(5)
O(2)-C(4)	1.270(5)	C(11)-C(12)	1.398(5)
O(3)-C(5)	1.347(5)	C(12)-C(13)	1.397(5)
O(4)-C(7)	1.345(4)	C(13)-C(14)	1.386(5)
O(5)-C(14)	1.385(4)	C(13)-C(22)	1.517(5)
C(2)-C(3)	1.341(6)	C(14)-C(15)	1.380(5)
C(3)-C(4)	1.456(5)	C(15)-C(16)	1.377(5)
C(3)-C(11)	1.479(5)	C(17)-C(18)	1.499(6)
C(4)-C(10)	1.424(5)	C(18)-C(19)	1.315(6)
C(5)-C(6)	1.375(5)	C(19)-C(20)	1.501(7)
C(5)-C(10)	1.427(5)	C(19)-C(21)	1.510(6)
C(6)-C(7)	1.403(6)	C(22)-C(23)	1.531(9)
C(6)-C(17)	1.518(5)	C(24)-C(23)	1.245(8)
C(7)-C(8)	1.385(5)	C(24)-C(25)	1.434(12)
C(8)-C(9)	1.383(5)	C(24)-C(26)	1.481(9)

Table 3.6: Bond angles (deg) involving non-hydrogen atoms, with e, s, d's in parenthesis for MN-02.

C(2)-O(1)-C(9)	118.8(3)	C(8)-C(9)-C(10)	122.3(3)
O(1)-C(2)-C(3)	126.6(3)	C(9)-C(10)-C(4)	120.4(3)
C(2)-C(3)-C(4)	116.9(3)	C(9)-C(10)-C(5)	116.8(3)
C(2)-C(3)-C(11)	122.2(3)	C(4)-C(10)-C(5)	122.8(3)
C(4)-C(3)-C(11)	120.9(4)	C(16)-C(11)-C(12)	117.5(3)
O(2)-C(4)-C(10)	120.9(3)	C(16)-C(11)-C(3)	122.4(3)
O(2)-C(4)-C(3)	121.8(3)	C(12)-C(11)-C(3)	120.2(3)
C(10)-C(4)-C(3)	117.3(3)	C(13)-C(12)-C(11)	122.9(4)
O(3)-C(5)-C(6)	118.6(3)	C(14)-C(13)-C(12)	117.0(3)
O(3)-C(5)-C(10)	119.3(3)	C(14)-C(13)-C(22)	123.5(3)
C(6)-C(5)-C(10)	122.2(3)	C(12)-C(13)-C(22)	119.5(4)
C(5)-C(6)-C(7)	118.0(3)	C(15)-C(14)-O(5)	117.5(3)
C(5)-C(6)-C(17)	120.6(4)	C(15)-C(14)-C(13)	121.2(3)
C(7)-C(6)-C(17)	121.3(3)	O(5)-C(14)-C(13)	121.3(3)
O(4)-C(7)-C(8)	122.0(4)	C(16)-C(15)-C(14)	120.6(4)
O(4)-C(7)-C(6)	115.8(3)	C(15)-C(16)-C(11)	120.7(3)
C(8)-C(7)-C(6)	122.1(3)	C(18)-C(17)-C(6)	111.7(3)
C(7)-C(8)-C(9)	118.5(4)	C(19)-C(18)-C(17)	127.5(5)
O(1)-C(9)-C(8)	117.8(3)	C(18)-C(19)-C(20)	124.2(4)
O(1)-C(9)-C(10)	119.9(3)	C(18)-C(19)-C(21)	121.4(4)

C(20)-C(19)-C(21)	114.4(4)	C(23)-C(24)-C(26)	126.1(6)
C(13)-C(22)-C(23)	110.1(4)	C(25)-C(24)-C(26)	103.9(7)
C(23)-C(24)-C(25)	130.1(8)	C(24)-C(23)-C(22)	130.7(6)

Table 3.7: Bond distances (Å) involving hydrogen atoms with e.s.d's in parenthesis.

O(3)-H(3O)	0.8107	C(20)-H(19B)	0.9832
O(4)-H(4O)	0.7934	C(20)-H(19C)	0.9654
O(5)-H(5O)	0.8287	C(21)-H(21A)	0.9600
C(2)-H(2)	0.9300	C(21)-H(21B)	0.9600
C(8)-H(6)	0.9300	C(21)-H(21C)	0.9600
C(12)-H(12)	0.9300	C(22)-H(22A)	0.9700
C(15)-H(15)	0.9300	C(22)-H(22B)	0.9700
C(16)-H(16)	0.9300	C(23)-H(23)	0.9300
C(17)-C(18)	1.499(6)	C(26)-H(26A)	0.9600
C(17)-H(17A)	0.9700	C(26)-H(26B)	0.9600
C(17)-H(17B)	0.9700	C(26)-H(26C)	0.9600
C(18)-H(18)	0.89(4)	C(25)-H(25A)	0.9949
C(20)-H(19A)	0.9521	C(25)-H(25C)	0.9962
		C(25)-H(25B)	0.9971

Table 3.8: Bond angles (deg) involving hydrogen atoms, with e, s, d's in parenthesis for MN-02

C(5)-O(3)-H(3O)	109.8	C(17)-C(18)-H(18)	112(2)
C(7)-O(4)-H(4O)	110.2	C(18)-C(19)-C(20)	124.2(4)
C(14)-O(5)-H(5O)	109.8	C(19)-C(20)-H(19A)	110.9
O(1)-C(2)-H(2)	116.7	C(19)-C(20)-H(19B)	110.3
C(3)-C(2)-H(2)	116.7	H(19A)-C(20)-H(19B)	108.2
C(7)-C(8)-H(6)	120.8	C(19)-C(20)-H(19C)	110.5
C(9)-C(8)-H(6)	120.8	H(19A)-C(20)-H(19C)	109.7
C(13)-C(12)-H(12)	118.5	H(19B)-C(20)-H(19C)	107.1
C(11)-C(12)-H(12)	118.5	C(19)-C(21)-H(21A)	109.5
C(16)-C(15)-H(15)	119.7	C(19)-C(21)-H(21B)	109.5
C(14)-C(15)-H(15)	119.7	H(21A)-C(21)-H(21B)	109.5
C(15)-C(16)-H(16)	119.6	C(19)-C(21)-H(21C)	109.5
C(11)-C(16)-H(16)	119.6	H(21A)-C(21)-H(21C)	109.5
C(18)-C(17)-H(17A)	109.3	H(21B)-C(21)-H(21C)	109.5
C(6)-C(17)-H(17A)	109.3	C(13)-C(22)-H(22A)	109.6
C(18)-C(17)-H(17B)	109.3	C(23)-C(22)-H(22A)	109.6
C(6)-C(17)-H(17B)	109.3	C(13)-C(22)-H(22B)	109.6
H(17A)-C(17)-H(17B)	107.9	C(23)-C(22)-H(22B)	109.7
C(19)-C(18)-H(18)	120(2)	H(22A)-C(22)-H(22B)	108.2

C(24)-C(23)-H(23)	114.7
C(22)-C(23)-H(23)	114.6
C(24)-C(26)-H(26A)	109.5
C(24)-C(26)-H(26B)	109.4
H(26A)-C(26)-H(26B)	109.5
C(24)-C(26)-H(26C)	109.5
H(26A)-C(26)-H(26C)	109.5
H(26B)-C(26)-H(26C)	109.5
C(24)-C(25)-H(25A)	112.9
C(24)-C(25)-H(25C)	113.3
H(25A)-C(25)-H(25C)	105.5

Table 3.9: Torsion angles (deg) for MN-02.

C9	-O1	-C2	-C3	1.68	0.56
C2	-O1	-C9	-C8	-178.51	0.33
C2	-O1	-C9	-C10	1.71	0.48
O1	-C2	-C3	-C4	-1.28	0.58
O1	-C2	-C3	-C11	176.93	0.34
C2	-C3	-C4	-O2	177.37	0.35
C2	-C3	-C4	-C10	-2.34	0.51
C11	-C3	-C4	-O2	-0.86	0.55
C11	-C3	-C4	-C10	179.43	0.33
C2	-C3	-C11	-C12	-39.46	0.54
C2	-C3	-C11	-C16	140.40	0.40
C4	-C3	-C11	-C12	138.67	0.37
C4	-C3	-C11	-C16	-41.47	0.53
O2	-C4	-C10	-C5	2.41	0.56
O2	-C4	-C10	-C9	-174.17	0.34
C3	-C4	-C10	-C5	-177.88	0.33
C3	-C4	-C10	-C9	5.53	0.51
O3	-C5	-C6	-C7	-178.50	0.33
O3	-C5	-C6	-C17	1.88	0.54
C10	-C5	-C6	-C7	1.69	0.55

C10	-C5	-C6	-C17	-177.92	0.34
O3	-C5	-C10	-C4	4.02	0.54
O3	-C5	-C10	-C9	-179.28	0.33
C6	-C5	-C10	-C4	-176.18	0.35
C6	-C5	-C10	-C9	0.52	0.53
C5	-C6	-C7	-O4	177.86	0.33
C5	-C6	-C7	-C8	-2.75	0.56
C17	-C6	-C7	-O4	-2.52	0.52
C17	-C6	-C7	-C8	176.87	0.35
C5	-C6	-C17	-C18	91.69	0.44
C7	-C6	-C17	-C18	-87.92	0.44
O4	-C7	-C8	-C9	-179.16	0.34
C6	-C7	-C8	-C9	1.49	0.56
C7	-C8	-C9	-O1	-178.88	0.32
C7	-C8	-C9	-C10	0.90	0.56
O1	-C9	-C10	-C4	-5.30	0.52
O1	-C9	-C10	-C5	177.92	0.31
C8	-C9	-C10	-C4	174.92	0.35
C8	-C9	-C10	-C5	-1.86	0.53
C3	-C11	-C12	-C13	-178.60	0.34
C16	-C11	-C12	-C13	1.54	0.55
C3	-C11	-C16	-C15	178.24	0.35
C12	-C11	-C16	-C15	-1.90	0.55

C11	-C12	-C13	-C14	0.91	0.55
C11	-C12	-C13	-C22	179.91	0.35
C12	-C13	-C14	-O5	179.73	0.33
C12	-C13	-C14	-C15	-3.08	0.55
C22	-C13	-C14	-O5	0.77	0.58
C22	-C13	-C14	-C15	177.96	0.37
C12	-C13	-C22	-C23	-71.67	0.51
C14	-C13	-C22	-C23	107.27	0.49
O5	-C14	-C15	-C16	-179.91	0.34
C13	-C14	-C15	-C16	2.80	0.59
C14	-C15	-C16	-C11	-0.19	0.59
C6	-C17	-C18	-C19	-115.26	0.48
C17	-C18	-C19	-C20	-1.91	0.74
C17	-C18	-C19	-C21	178.75	0.41
C13	-C22	-C23	-C24	-137.02	0.71
C13	-C22	-C23	-H23	42.99	0.76
C26	-C24	-C23	-C22	1.09	1.18
C25	-C24	-C23	-C22	-178.59	0.87

Table 3.10: Intermolecular contacts less than 4.00Å for the non-hydrogen atoms for MN-02

O1	...O3	-x+1/2,-y+1/2,-z	3.9883 0.0042
O1	...C4	-x+1/2,-y+1/2+1,-z	3.8912 0.0045
O1	...C5	-x+1/2,-y+1/2,-z	3.7318 0.0046
O1	...C5	-x+1/2,-y+1/2+1,-z	3.7168 0.0046
O1	...C9	-x+1/2,-y+1/2+1,-z	3.9884 0.0044
O1	...C17	-x+1/2,-y+1/2,-z	3.9661 0.0048
O2	...O4	-x+1/2,-y+1/2,-z	3.7755 0.0040
O2	...C7	-x+1/2,-y+1/2,-z	3.8951 0.0047
O2	...C8	-x+1/2,-y+1/2,-z	3.7727 0.0048
O2	...C14	-x,-y+1,-z	3.7985 0.0044
O2	...C24	-x,-y+1,-z	3.8960 0.0059
O3	...O1	-x+1/2,-y+1/2,-z	3.9883 0.0042
O3	...C20	-x+1/2,+y-1/2,-z-1/2	3.8144 0.0065
O5	...C3	-x,-y+1,-z	3.9409 0.0045
C2	...C6	-x+1/2,-y+1/2,-z	3.7200 0.0052
C2	...C6	-x+1/2,-y+1/2+1,-z	3.7292 0.0052
C2	...C7	-x+1/2,-y+1/2,-z	3.9312 0.0052
C2	...C7	-x+1/2,-y+1/2+1,-z	3.9297 0.0052
C2	...C9	-x+1/2,-y+1/2+1,-z	3.9422 0.0053

C2	...C10	-x+1/2,-y+1/2+1,-z	3.7903	0.0052
C2	...C17	-x+1/2,-y+1/2,-z	3.7552	0.0055
C2	...C18	-x+1/2,-y+1/2+1,-z	3.7801	0.0058
C3	...O5	-x,-y+1,-z	3.9409	0.0045
C3	...C6	-x+1/2,-y+1/2,-z	3.9429	0.0050
C3	...C7	-x+1/2,-y+1/2+1,-z	3.7784	0.0050
C3	...C9	-x+1/2,-y+1/2+1,-z	3.8897	0.0050
C4	...O1	-x+1/2,-y+1/2+1,-z	3.8912	0.0045
C4	...C8	-x+1/2,-y+1/2+1,-z	3.8236	0.0052
C4	...C9	-x+1/2,-y+1/2+1,-z	3.7951	0.0050
C5	...O1	-x+1/2,-y+1/2,-z	3.7318	0.0046
C5	...O1	-x+1/2,-y+1/2+1,-z	3.7168	0.0046
C5	...C8	-x+1/2,-y+1/2,-z	3.7963	0.0053
C6	...C2	-x+1/2,-y+1/2,-z	3.7200	0.0052
C6	...C2	-x+1/2,-y+1/2+1,-z	3.7292	0.0052
C6	...C3	-x+1/2,-y+1/2,-z	3.9429	0.0050
C6	...C9	-x+1/2,-y+1/2,-z	3.8256	0.0051
C7	...O2	-x+1/2,-y+1/2,-z	3.8951	0.0047
C7	...C2	-x+1/2,-y+1/2,-z	3.9312	0.0052
C7	...C2	-x+1/2,-y+1/2+1,-z	3.9297	0.0052
C7	...C3	-x+1/2,-y+1/2+1,-z	3.7784	0.0050
C7	...C9	-x+1/2,-y+1/2,-z	3.9936	0.0050
C7	...C10	-x+1/2,-y+1/2,-z	3.7544	0.0051

C7	...C11	-x+1/2,-y+1/2+1,-z	3.8220	0.0050
C8	...O2	-x+1/2,-y+1/2,-z	3.7727	0.0048
C8	...C4	-x+1/2,-y+1/2+1,-z	3.8236	0.0052
C8	...C5	-x+1/2,-y+1/2,-z	3.7963	0.0053
C8	...C16	-x+1/2,-y+1/2+1,-z	3.7739	0.0053
C9	...O1	-x+1/2,-y+1/2+1,-z	3.9884	0.0044
C9	...C2	-x+1/2,-y+1/2+1,-z	3.9422	0.0053
C9	...C3	-x+1/2,-y+1/2+1,-z	3.8897	0.0050
C9	...C4	-x+1/2,-y+1/2+1,-z	3.7951	0.0050
C9	...C6	-x+1/2,-y+1/2,-z	3.8256	0.0051
C9	...C7	-x+1/2,-y+1/2,-z	3.9936	0.0050
C9	...C9	-x+1/2,-y+1/2,-z	3.9323	0.0048
C9	...C9	-x+1/2,-y+1/2+1,-z	3.8960	0.0048
C9	...C10	-x+1/2,-y+1/2,-z	3.7135	0.0048
C9	...C10	-x+1/2,-y+1/2+1,-z	3.7372	0.0048
C10	...C2	-x+1/2,-y+1/2+1,-z	3.7903	0.0052
C10	...C7	-x+1/2,-y+1/2,-z	3.7544	0.0051
C10	...C9	-x+1/2,-y+1/2,-z	3.7135	0.0048
C10	...C9	-x+1/2,-y+1/2+1,-z	3.7372	0.0048
C11	...O4	-x+1/2,-y+1/2+1,-z	3.8511	0.0044
C11	...C7	-x+1/2,-y+1/2+1,-z	3.8220	0.0050
C11	...C14	-x,-y+1,-z	3.9451	0.0051
C12	...C15	-x,-y+1,-z	3.9723	0.0053

C12	...C18	-x+1/2,-y+1/2+1,-z	3.9486	0.0055
C12	...C21	-x+1/2,-y+1/2+1,-z	3.9619	0.0064
C13	...C16	-x,-y+1,-z	3.9785	0.0052
C13	...C25	x,+y+1,+z	3.9963	0.0134
C14	...O2	-x,-y+1,-z	3.7985	0.0044
C14	...C11	-x,-y+1,-z	3.9451	0.0051
C14	...C14	-x,-y+1,-z	3.8359	0.0054
C14	...C25	x,+y+1,+z	3.7621	0.0128
C15	...O4	-x+1/2,-y+1/2+1,-z	3.7994	0.0047
C15	...C12	-x,-y+1,-z	3.9723	0.0053
C15	...C15	-x,-y+1,-z	3.8595	0.0053
C15	...C15	-x,-y+2,-z	3.7997	0.0053
C15	...C23	-x,-y+1,-z	3.7406	0.0072
C16	...C8	-x+1/2,-y+1/2+1,-z	3.7739	0.0053
C16	...C13	-x,-y+1,-z	3.9785	0.0052
C16	...C24	-x,-y+1,-z	3.8968	0.0063
C16	...C23	-x,-y+1,-z	3.8200	0.0073
C17	...O1	-x+1/2,-y+1/2,-z	3.9661	0.0048
C17	...C2	-x+1/2,-y+1/2,-z	3.7552	0.0055
C18	...C2	-x+1/2,-y+1/2+1,-z	3.7801	0.0058
C18	...C12	-x+1/2,-y+1/2+1,-z	3.9486	0.0055
C18	...C26	x+1/2,-y+1/2, +z-1/2	3.9850	0.0100
C19	...C26	x+1/2,-y+1/2, +z-1/2	3.8508	0.0097

C20	...O3	-x+1/2, +y+1/2,-z-1/2	3.8144	0.0065
C20	...C26	x+1/2,-y+1/2, +z-1/2	3.9119	0.0100
C21	...C12	-x+1/2,-y+1/2+1,-z	3.9619	0.0064
C21	...C26	x+1/2,-y+1/2+1, +z-1/2	3.8095	0.0102
C21	...C25	x+1/2,-y+1/2, +z-1/2	3.7889	0.0155
C22	...C25	x, +y+1, +z	3.9139	0.0126
C24	...O2	-x,-y+1,-z	3.8960	0.0059
C24	...C16	-x,-y+1,-z	3.8968	0.0063
C24	...C24	-x, +y,-z+1/2	3.9086	0.0071
C24	...C23	-x, +y,-z+1/2	3.9500	0.0078
C23	...C15	-x,-y+1,-z	3.7406	0.0072
C23	...C16	-x,-y+1,-z	3.8200	0.0073
C23	...C24	-x, +y,-z+1/2	3.9500	0.0078
C23	...C26	-x, +y,-z+1/2	3.9528	0.0107
C26	...C18	x-1/2,-y+1/2, +z+1/2	3.9850	0.0100
C26	...C19	x-1/2,-y+1/2, +z+1/2	3.8508	0.0097
C26	...C20	x-1/2,-y+1/2, +z+1/2	3.9119	0.0100
C26	...C21	x-1/2,-y+1/2+1, +z+1/2	3.8095	0.0102
C26	...C23	-x, +y,-z+1/2	3.9528	0.0107
C25	...C13	x, +y-1, +z	3.9963	0.0134
C25	...C14	x, +y-1, +z	3.7621	0.0128
C25	...C21	x-1/2,-y+1/2, +z+1/2	3.7889	0.0155
C25	...C22	x, +y-1, +z	3.9139	0.0126

REFERENCES: 3

1. kanjilal U.N., Kanjilal P.C. & Das A, "*Flora of Assam* vol-2 pp-270-271,1934

2. Mahabusarakam, W.; Deachathai, S.; Phongpaichit, S.; Jansakul, C.; Taylor, W. C. *Phytochemistry (Elsevier)*, 65(8), 1185-1191, 2004

3. Shiao, Young-Ji; Wang, Chuen-Neu; Wang, Wan-Yu; Lin, Yun-Lian. *Planta Medica* 71(9), 835-840, 2005

4. Toda, Shizuo1; Shirataki, Yoshiaki., *Pharmaceutical Biology* (Formerly International Journal of Pharmacognosy), Volume 44, Number 4, June, pp. 271-273(3) 2006

5. Sarkhel S, Jain G K, Singh H, Subramanya S & Maulik P R, *Acta Cryst* C56, e253-e254, 2000

6. Glidewell C., Low J. N., Melguizo M and Quesada A. *Acta Cryst.* C59, 14-18, 2003

7. Glidewell C., Low J. N., Melguizo M and Quesada A. *Acta Cryst.* C59, 19-21, 2003

8. Jones N. A., Jenkinson S. F., Soengas R., Izumori K., Fleet G. W. J.and Watkin D. J. *Acta Cryst.* C63, 7-10, 2007

9. Tamuly Chandan, Sarma Rupam S., Batsnov Anderei S., Goeta Andres E and Baruah Jubaraj.B. *Acta Cryst.*.C61, 0324-0327, 2005

10. Simone M., Fleet G. W. J., Bream R and Watkin D. J. *Acta Cryst.* E63, 1409- 1411,2007

11. SMART-*NT, Data Collection Software, version 5.0*, Bruker Analytical X-ray Instruments Inc., Madison, Wisconsin, USA, 1999.

12. *SAINT-NT, Data Reduction Software, version 5.0*, Bruker Analytical X-ray

Instruments Inc., Madison, Wisconsin, USA, 1999.

13. G M SHELXS97, Program for the solution of crystal structure, University of Göttingen, Germany, 1997.
14. G M SHELXL97, Program for the refinement of crystal structure, University of Göttingen, Germany, 1997.
15. International Table for X-ray crystallography, Vol.1V.Kynoch Press, Biraingham (present distributor: Kluwer Academic Publisherrs: Dordrecht).
16. Johnson C K, *ORTEP32, Report ORNL-5138*, Oak Ridge National Laboratory, Tennessee, USA, 1976.
17. Karle, J., Hauptman, H.and Christ, C.L (1958).*Acta Cryst.* 11,757-761.
18. Allen,F.H.,Kennard,O.,Watson,D.G.,Brammer,L.,Orpen,A.G.and Taylor,R.*J.Chem.Soc.*Perkin Trans.H,S1-S19 (1987)
19. Siriwardane U., Reddy T. R and Biehl E. R. *Acta Cryst.* C45, 339-341,1989
20. Costa S., Boechat N., Wardell S. M. S. V., Ferreira V. F.,. Low J. N and Glidewell C. *Acta Cryst.* C63, 33-37, 2007
21. Allen, F.H., Kennard, O.and Taylor*Acc.Chem.Res.*16, 146-153, 1983
22. CCDC Mercury 1.4.1, 2005

****