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The Crystal Structure Analyses of Molecular Compound N – allyl-2, 6 - di (ortho-chloro-phenyl) – 4 - methoxyphenyl pyridinium perchlorate using X-ray Diffraction Data

Mouayed M. Mahmoud[1], Saad N. Ibrahim[2]

[1] Professor Assistant, Al-Mustansiriyah University, College of Science, Department of Physics.

[2] Assistant Lecturer, Al-Mustansiriyah University, College of Science, Department of Physics

Abstract: The crystal structure of the N-allyl-2,6-di (ortho-chloro-phenyl)-4-methoxyphenyl pyridinium perchlorate molecular compound was studied by X-ray diffraction. The structure analyses were carried out from diffraction data collected from single crystal of appropriate size. The lattice parameters are a=10.947A°, b=10.996A°, c=11.380A°, α=104.17°, β=100.76°, γ=84.27°, monoclinic system, the space group is $P\bar{1}$. The structure was solved by direct methods and refined by full matrix least-square calculation to a final discrepancy index R of 0,048 for 2721 reflections. The co-ordinates, the thermal parameters, the bond lengths and angles of all atoms were measured.

Keywords: X-ray, Molecular Compound, Crystal Structure

1. Unit Cell Constant and Space Group

The crystal was mounted on a goniometer head oscillation, zero, and first layers Weissenberg photographs were obtained with the crystal rotating about the b-axis (parallel to the long edge) using Cukα radiation λ=1.542 A°. From the symmetry of these photographs the crystal system was seen to be monoclinic. The unit cell dimensions were calculated from high order reflections on the zero layerWeissenberg photograph and from an oscillation photograph, and were later refined on the four-circle differactometer. The complex $C_{18}H_{20}N_1O_2Cl_3$ is monoclinic, space group $P\bar{1}$ with a=10.947A°, b=10.996A°, c=11.380A°, α=104.17°, β=100.76°, γ=84.27°, the volume of unit cell is M_r= molecular weight is 546.84

From the photographs showed the lattice is Centro symmetric and the related symmetric x,y,z, -x,-y,-z

Intensity data with the range θ≤25° were collected with diffractometer using a scintillation counter was recorded for 3060 out of 2721 reflections measured. Three standard reflections were measured every 100 at regular intervals confirmed that was no detectable decomposition, since there was no significant fall in their intensities, [reflections to monitor decomposition of the crystal and setting during data collection]. No absorption correction was made [in view of the small size of the spacemen]. The intensities were corrected for Lorentz and polarization factors. The reflections whose intensities were less than 3 times their standard deviation were regarded as unobserved.

2. Refinement of the Structure

The structure was solved by direct method A preliminary factors and an overall temperature factors were obtained using all the data. A program was used to convert the F's (Structure factors) to E's (Electronic map). The map showed the majority of non-hydrogen's atoms.

After two cycles of full matrix, least square refinement of positions and isotropic thermal parameters for Cl, O, N, and C atoms were carried out using unit weights. A three-dimensional Fourier electron density distribution computed from the resulting structure factors confirmed the structure, by showing no unusual features after two further cycles of isotropic least square R fell to 0.062. In subsequent refinement the Cl, O, N, and C. atoms were assumed to vibrate an isotropic and after fourcycles of refinement R dropped to 0.053.

The structure factor+, F(hkl), is the transform of the infinite crystal sampled at a reciprocal lattice point (hkl). F(hkl) can be defined as:

$$F(hkl) = \sum_{j=1}^{N} g_j exp2\pi i(hx_j + ky_j + lz_j)$$

Which is valid for all space group.
Where

$$g_j = f_j \exp\left(-\frac{B_i Sin^2\theta}{\lambda^2}\right)$$

If the atoms are harmonic isotropic oscillators.
Where f_j the scattering factor of the jth atom is dependent on $sin\theta/\lambda$, N is the number of atoms per unit cell.
B(in A^{o2}) , the isotropic temperature factor, is a measure of the root-mean-square amplitude of the thermal oscillations.

It is clear that isotropic motion is a poor approximation for atoms in most crystals, because the enviroments of these atoms are far from isotropic. Thus the motion is better described by the six parameters of a general ellipsoid rather than by the single parameter characteristic of sphere.

Three of these six parameters may be considered to define the size of the ellipsoid parallel to the three crystallographic axes describing the amount of motion in these directions, and three to define the orientation of these ellipsoidal axes relative to these crystal axes.

A Fourier difference map computed showed the majority of the H-atoms approximately were expected. It was decided however to calculate the position of the hydrogen atoms assuming regular geometry C-H, N-H and O-H distance of 1.0 A°. [Because of inherent difficulty of the x-ray diffraction technique in providing accurate positional parameters for H-atoms were Cl atoms are presented]. A comparison between the observed and calculated positions of the hydrogen atoms showed good agreement for most of the atoms. The calculated positions of the hydrogen atoms were included in further least square refinement with isotropic temperature factors. The refinement converged after a few cycles and R had dropped to the final value of 0.048

The agreement is measured by an R-residual factor defined as:

$$R = \frac{\Sigma(|F_o| - |F_c|)}{\Sigma|F_o|}$$

Where $|F_o|$: observed structure amplitude.

$|F_C|$: calculated structure amplitude.

R will typically be ≈ 0.05 if the structure correct.

[Atomic scattering factors were taken from international tables for x-ray crystallography volume IV.]

Table 1: The final co-ordinates are given in table below together with U for non-hydrogen H-atoms (where U is mean square vibration amplitude in (A°))

ATOM	X/a	Y/b	Z/c	U(ISO)
N(1)	0.9842(3)	0.7202(3)	0.1620(3)	0.0450
C(1)	1.1107(4)	0.7080(4)	0.2012(4)	0.0497
C(2)	1.1437(4)	0.6050(4)	0.2699(4)	0.0503
C(3)	1.0514(4)	0.5078(4)	0.3004(4)	0.0463
C(4)	0.9245(4)	0.5225(4)	0.2559(4)	0.0502
C(5)	0.8916(4)	0.6263(4)	0.1870(4)	0.0467
C(6)	1.0885(4)	0.3963(4)	0.3732(4)	0.0452
C(7)	1.2099(4)	0.3677(4)	0.3892(4)	0.0508
C(8)	1.2443(4)	0.2615(4)	0.4533(4)	0.0538
C(9)	1.1543(5)	0.1806(4)	0.5035(4)	0.0532
C(10)	1.0328(5)	0.2080(5)	0.4904(5)	0.0604
C(11)	0.9999(4)	0.3137(4)	0.4257(5)	0.0541
C(12)	1.3013(7)	0.0420(6)	0.5895(7)	0.0804
C(13)	1.2091(4)	0.8093(4)	0.1695(5)	0.0536
C(14)	1.2289(5)	0.8299(5)	0.0531(6)	0.0650
C(15)	1.3143(7)	0.9254(7)	0.0205(8)	0.0839
C(16)	1.3855(7)	1.0040(8)	0.109(1)	0.0921
C(17)	1.3727(6)	0.9848(6)	0.229(1)	0.0917
C(18)	1.2848(5)	0.8872(5)	0.2604(6)	0.0746
C(19)	0.9505(4)	0.8414(4)	0.1026(4)	0.0516
C(20)	0.9444(6)	0.9261(5)	0.1949(5)	0.0673
C(21)	1.0223(8)	1.0331(6)	0.2148(6)	0.0861
C(22)	0.7549(4)	0.6386(4)	0.1411(5)	0.0520
C(23)	0.6950(5)	0.6344(5)	0.0172(5)	0.0657
C(24)	0.5694(6)	0.6478(6)	-0.0245(7)	0.0742
C(25)	0.5005(6)	0.6632(6)	0.054(1)	0.0858
C(26)	0.5537(7)	0.6629(6)	0.177(1)	0.0872
C(27)	0.6820(5)	0.6502(5)	0.2234(6)	0.0718
O(1)	0.5573(9)	0.1921(7)	0.4465(8)	0.1223
O(2)	0.6487(7)	0.2709(7)	0.2803(6)	0.1113
O(3)	0.5068(7)	0.3680(9)	0.3416(8)	0.1139
O(4)	0.7154(7)	0.370(1)	0.4523(9)	0.1295
O(5)	1.1772(4)	0.0724(3)	0.5671(4)	0.0666
CL(1)	0.6086(1)	0.3023(2)	0.3885(2)	0.0805
CL(2)	1.1428(2)	0.7296(2)	-0.0578(1)	0.0886
CL(3)	0.7819(2)	0.6106(2)	-0.0837(1)	0.0972
O(11)	0.634(6)	0.413(5)	0.354(5)	0.21(2)
O(12)	0.5794	0.3311	0.4943	0.19(2)
O(13)	0.707(3)	0.242(3)	0.474(2)	0.108(9)
O(14)	0.503(3)	0.227(3)	0.355(3)	0.12(1)
H(2)	1.2311	0.5883	0.2833	0.0500
H(4)	0.8500	0.4547	0.2700	0.0500
H(7)	1.2847	0.4239	0.3503	0.0500
H(8)	1.3411	0.2525	0.4597	0.0500
H(10)	0.9675	0.1517	0.5236	0.0500
H(11)	0.9061	0.3286	0.4211	0.0500
H(121)	1.3628	0.1031	0.6261	0.0500
H(122)	1.3353	0.0458	0.5122	0.0500
H(123)	1.3083	-0.0247	0.6428	0.0500
H(15)	1.3300	0.9400	-0.0775	0.0500
H(16)	1.4686	1.0928	0.1033	0.0500
H(17)	1.4408	1.0536	0.2797	0.0500
H(18)	1.2675	0.8594	0.3592	0.0500
H(191)	0.8611	0.8131	0.0425	0.0500
H(192)	1.0178	0.8728	0.0486	0.0500
H(20)	0.8542	0.9156	0.2297	0.0500
H(211)	1.1044	1.0683	0.1619	0.0500
H(212)	1.0042	1.0569	0.3011	0.0500
H(24)	0.5247	0.6317	-0.1303	0.0500
H(25)	0.3992	0.6778	0.0200	0.0500
H(26)	0.5050	0.6592	0.2383	0.0500
H(27)	0.7292	0.6364	0.3300	0.0500

Table 2: Final thermal parameters are given in table below for non-hydrogen atoms

$$Temperature factor = exp[-2\pi^2(h^2U_{11}a^{*2} + k^2U_{22}b^{*2} + l^2U_{33}c^{*2} + 2hkU_{12}a^*b^* + 2hlU_{13}a^*c^* + 2klU_{23}b^*c^*)]$$

ATOM	U(11)	U(22)	U(33)	U(23)	U(13)	U(12)
C(1)	0.043(1)	0.051(1)	0.047(2)	0.002(1)	0.030(1)	-0.001(1)
C(2)	0.053(2)	0.051(2)	0.057(2)	0.004(1)	0.040(1)	0.000(1)
C(3)	0.045(1)	0.062(2)	0.048(1)	0.007(1)	0.034(1)	0.005(1)
C(4)	0.064(2)	0.063(2)	0.057(2)	-0.006(2)	0.043(2)	-0.003(2)
C(5)	0.077(2)	0.054(2)	0.061(2)	-0.004(2)	0.045(2)	-0.011(2)
C(6)	0.059(2)	0.052(2)	0.051(2)	0.005(1)	0.037(2)	-0.002(1)
C(7)	0.056(2)	0.051(2)	0.053(2)	0.000(1)	0.039(1)	-0.005(1)
N(8)	0.050(1)	0.053(1)	0.050(1)	-0.001(1)	0.036(1)	-0.004(1)
C(9)	0.052(2)	0.046(1)	0.044(1)	0.001(1)	0.034(1)	-0.004(1)
C(10)	0.053(2)	0.047(1)	0.054(2)	0.000(1)	0.039(1)	-0.003(1)
C(11)	0.070(2)	0.060(2)	0.063(2)	-0.005(2)	0.050(2)	-0.001(2)
C(12)	0.068(2)	0.064(2)	0.050(2)	-0.012(2)	0.038(2)	-0.012(2)
C(13)	0.056(2)	0.069(2)	0.051(2)	-0.003(2)	0.034(1)	-0.011(2)
C(14)	0.053(2)	0.066(2)	0.053(2)	0.001(1)	0.038(1)	-0.007(1)
C(15)	0.059(2)	0.115(4)	0.074(3)	-0.013(3)	0.049(2)	-0.009(2)
C(16)	0.054(2)	0.065(2)	0.074(2)	-0.003(2)	0.045(2)	-0.002(2)
C(17)	0.081(3)	0.118(4)	0.134(5)	-0.035(4)	0.085(4)	-0.017(3)
C(18)	0.065(2)	0.075(2)	0.093(3)	-0.014(2)	0.058(2)	-0.019(2)
C(19)	0.054(2)	0.097(4)	0.094(3)	0.025(3)	0.039(2)	0.018(2)
N(20)	0.077(2)	0.084(2)	0.065(2)	0.015(2)	0.054(2)	0.012(2)
O(21)	0.154(3)	0.112(2)	0.078(2)	-0.000(2)	0.090(2)	-0.000(2)
O(22)	0.117(2)	0.088(2)	0.088(2)	0.021(2)	0.076(2)	-0.002(2)

Table 3: Final bond lengths (Ao) and bond angles (°)

N1	C1	1.37536		1 0. 0. 0.
N1	C5	1.36629		1 0. 0. 0.
N1	C19	1.50037		1 0. 0. 0.
C1	N1	C5	119.79	
C1	N1	C19	119.14	
C5	N1	C19	120.81	
C1	N1	1.37536		1 0. 0. 0.
C1	C2	1.36417		1 0. 0. 0.
C1	C13	1.48188		1 0. 0. 0.
N1	C1	C2	119.89	
N1	C1	C13	118.79	
C2	C1	C13	121.31	
C2	C1	1.36417		1 0. 0. 0.
C2	C3	1.40718		1 0. 0. 0.
C2	H2	0.98025		1 0. 0. 0.
C1	C2	C3	121.97	
C1	C2	H2	119.45	
C3	C2	H2	117.12	
C3	C2	1.40718		1 0. 0. 0.
C3	C4	1.39090		1 0. 0. 0.
C3	C6	1.47408		1 0. 0. 0.
C2	C3	C4	116.14	
C2	C3	C6	121.30	
C4	C3	C6	122.55	
C4	C3	1.39090		1 0. 0. 0.
C4	C5	1.37153		1 0. 0. 0.
C4	H4	1.03026		1 0. 0. 0.
C3	C4	C5	121.77	
C3	C4	H4	121.87	
C5	C4	H4	116.31	
C5	N1	1.36629		1 0. 0. 0.
C5	C4	1.37153		1 0. 0. 0.
C5	C22	1.48612		1 0. 0. 0.
N1	C5	C4	120.36	
N1	C5	C22	119.43	
C4	C5	C22	120.19	
C6	C3	1.47408		1 0. 0. 0.
C6	C7	1.38709		1 0. 0. 0.
C6	C11	1.40321		1 0. 0. 0.
C3	C6	C7	121.46	
C3	C6	C11	120.55	
C7	C6	C11	117.96	
C7	C6	1.38709		1 0. 0. 0.
C7	C8	1.37650		1 0. 0. 0.
C7	H7	1.08830		1 0. 0. 0.
C6	C7	C8	122.04	
C6	C7	H7	123.89	
C8	C7	H7	114.03	
C8	C7	1.37650		1 0. 0. 0.
C8	C9	1.39226		1 0. 0. 0.
C8	H8	1.06671		1 0. 0. 0.
C7	C8	C9	118.84	
C7	C8	H8	113085	
C9	C8	H8	127.29	
C9	C8	1.39226		1 0. 0. 0.
C9	C10	1.38708		1 0. 0. 0.
C9	O5	1.36095		1 0. 0. 0.
C8	C9	C10	120.23	
C8	C9	O5	123.88	
C10	C9	O5	115.89	
C10	C9	1.38708		1 0. 0. 0.
C10	C11	1.36974		1 0. 0. 0.
C10	H10	0.97983		1 0. 0. 0.
C9	C10	C11	120.15	
C9	C10	H10	121.57	
C11	C10	H10	118.23	
C12	O5	1.41614		1 0. 0. 0.
C12	H21	0.90955		1 0. 0. 0.
C12	H22	1.03045		1 0. 0. 0.
C12	H23	0.90576		1 0. 0. 0.
O5	C12	H21	112.45	
O5	C12	H22	113.33	
O5	C12	H23	110.50	
H21	C12	H22	95.47	
H21	C12	H23	104.37	
H22	C12	H23	119.30	
C13	C1	1.48188		1 0. 0. 0.
C13	C14	1.38291		1 0. 0. 0.
C13	C18	1.40813		1 0. 0. 0.
C1	C13	C14	121.95	
C1	C13	C18	119.07	
C14	C13	C18	118.98	
C14	C13	1.38291		1 0. 0. 0.
C14	C15	1.35725		1 0. 0. 0.
C14	CL2	1.73411		1 0. 0. 0.
C13	C14	C15	123.05	
C13	C14	CL2	119.53	
C15	C14	CL2	117.43	
C15	C14	1.35725		1 0. 0. 0.
C15	C16	1.38259		1 0. 0. 0.
C15	H15	1.16151		1 0. 0. 0.
C14	C15	C16	117.80	
C14	C15	H15	121.77	
C16	C15	H15	120.40	
C16	C15	1.38259		1 0. 0. 0.
C16	C17	1.39507		1 0. 0. 0.
C16	H16	1.21316		1 0. 0. 0.
C15	C16	C17	121.42	
C15	C16	H16	130.06	

C17	C16	H16	108.45	
C17	C16	1.39507		1 0. 0. 0.
C17	C18	1.38376		1 0. 0. 0.
C17	H17	1.06327		1 0. 0. 0.
C16	C17	C18	119.97	
C16	C17	H17	106.99	
C18	C17	H17	133.01	
C18	C13	1.40813		1 0. 0. 0.
C18	C17	1.38376		1 0. 0. 0.
C18	H18	1.18580		1 0. 0. 0.
C13	C18	C17	118.70	
C13	C18	H18	115.06	
C17	C18	H18	126.22	
C19	N1	1.50037		1 0. 0. 0.
C19	C20	1.49198		1 0. 0. 0.
C19	H91	1.06054		1 0. 0. 0.
C19	H92	1.06011		1 0. 0. 0.
N1	C19	C20	110.85	
N1	C19	H91	102.11	
N1	C19	H92	104.78	
C20	C19	H91	113.66	
C20	C19	H92	116.96	
H91	C19	H92	107.11	
C20	C19	1.49198		1 0. 0. 0.
C20	C21	1.32193		1 0. 0. 0.
C20	H20	1.13458		1 0. 0. 0.
C19	C20	C21	122.92	
C19	C20	H20	119.29	
C21	C20	H20	115.21	
C21	C20	1.32193		1 0. 0. 0.
C21	H31	1.18693		1 0. 0. 0.
C21	H32	1.11379		1 0. 0. 0.
C20	C21	H31	122.47	
C20	C21	H32	101.37	
H31	C21	H32	134.10	
C22	C5	1.48612		1 0. 0. 0.
C22	C23	1.40286		1 0. 0. 0.
C22	C27	1.39931		1 0. 0. 0.
C5	C22	C23	121.84	
C5	C22	C27	119.51	
C23	C22	C27	118.60	
C23	C22	1.40286		1 0. 0. 0.
C23	C24	1.36994		1 0. 0. 0.
C23	CL3	1.72741		1 0. 0. 0.
C22	C23	C24	121.47	
C22	C23	CL3	118.81	
C24	C23	CL3	119.72	
C24	C23	1.36994		1 0. 0. 0.
C24	C25	1.34651		1 0. 0. 0.
C24	H24	1.19996		1 0. 0. 0.
C23	C24	C25	119.88	
C23	C24	H24	115.84	
C25	C24	H24	123.81	
C25	C24	1.34651		1 0. 0. 0.
C25	C26	1.37942		1 0. 0. 0.
C25	H25	1.11731		1 0. 0. 0.
C24	C25	C26	120.77	
C24	C25	H25	120.00	
C26	C25	H25	119.22	
C26	C25	1.37942		1 0. 0. 0.
C26	C27	1.40151		1 0. 0. 0.
C26	H26	0.96760		1 0. 0. 0.
C25	C26	C27	120.94	
C25	C26	H26	123.84	
C27	C26	H26	114.63	
C27	C22	1.39931		1 0. 0. 0.
C27	C26	1.40151		1 0. 0. 0.
C27	H27	1.20333		1 0. 0. 0.
C22	C27	C26	118.25	
C22	C27	H27	118.01	
C26	C27	H27	123.32	
O1	CL1	1.41171		1 0. 0. 0.
02	CL1	1.49673		1 0. 0. 0.
O3	CL1	1.40917		1 0. 0. 0.
O4	CL1	1.34408		1 0. 0. 0.
O5	C9	1.36095		1 0. 0. 0.
O5	C12	1.41614		1 0. 0. 0.
C9	O5		118.79	
CL1	O1	1.41171		1 0. 0. 0.
CL1	O2	1.49673		1 0. 0. 0.
CL1	O3	1.40917		1 0. 0. 0.
CL1	O4	1.34408		1 0. 0. 0.
O1	CL1	O2	109.07	
O1	CL1	O3	106.25	
O1	CL1	O4	116.50	
O2	CL1	O3	105.51	
O2	CL1	O4	103.41	
O3	CL1	O4	115.43	

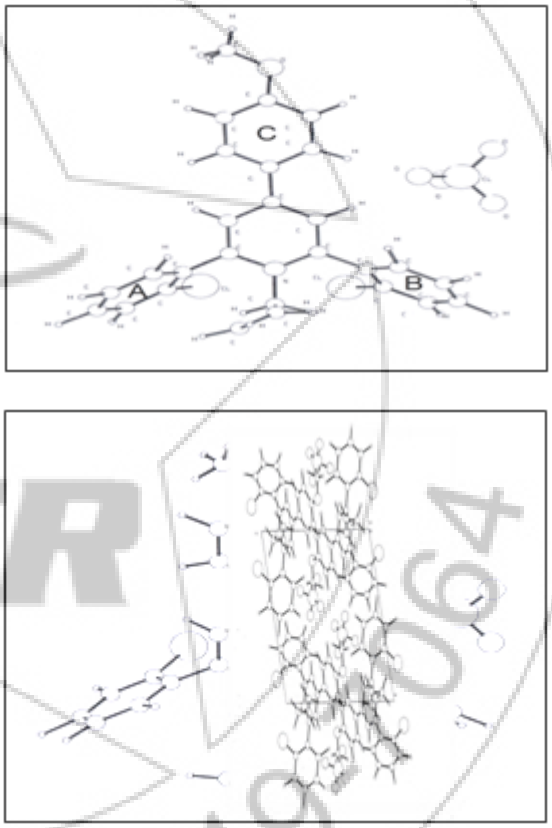

Figure 1: View of projected down the b- axis of the unit cell

3. Descriptions

Figure (1) shows that the crystal structure pointed out that The vinyl group B, A deflector on the level of Alberadiom ring at an angle of (106.6°) and (112°), respectively, and deviate vinyl group (C) at an angle of (16.0°) from the level of loop Alberadinyum. the group of Almitoxa located to the side of phenyl ring (A), either at the two chlorine atoms ortho - to two phenylalanine (B, A) be a cantilever to the bottom while it is set to top Alvinal high above the ring Alberadinyum.

A group of Alvinal encircled by the two sides of the vinyl (B, A). That this group closer to the phenyl ring (A) then to

(B). The focus of this group parallels the axis loop phenylalanine (A), which could lead to a rapprochement (due to the gravitational forces of the weak van der wall's force).

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