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Crystal Structure of 1-(4-Pyridylmethyl)-2,4,6-Triphenyl Pyridinium Perchlorate

1-(4-pyridylmethyl)-2,4,6-triphenyl pyridinium perchlorate crystallizes in space group $P\bar{1}$ with 3 molecules per asymmetric unit. The cell parameters obtained are: $a = 12.142(3)$, $b = 16.972(4)$, $c = 20.229(7)$ Å, $\alpha = 83.66(4)$, $\beta = 74.83(3)$, and $\gamma = 69.47(2)°$. The crystal structure was determined using the direct methods technique. Anisotropic refinement was undertaken using block diagonal least squares. In the final cycle of the refinement the reliability factor was found to be $R_0 = 0.084$ for 3982 reflections with $I \geq 3\sigma(I)$. All rings are planar within experimental errors. Rings B, C, D, and E are twisted with respect to the central plane.

Introduction

As it has been mentioned in the previous papers (FARAG et al. 1990) that pyridinium compounds have been known for wide variety of biological activities, therefore for a series of these compounds X-ray analyses were undertaken in order to give more details of the conformation of the molecules.

Experimental

The compound of $C_{29}H_{23}N_2ClO_4$ was prepared by reaction of 2,4,6-triphenyl pyridinium perchlorate with 1-(4-pyridylmethylamine) in ethanol at room temperature and the reaction mixture was worked up according to the reported methods (KATRITZKY et al. Recrystalization from acetone yield good prismatic crystals. A crystal of dimensions $0.3 \times 0.3 \times 0.3$ mm was chosen for X-ray analysis. The crystal was mounted on glass fiber in random orientation. Accurate cell parameters and an orientation matrix for data collection were obtained from least squares refinement, using the setting angle of 25 reflections. The cell parameters obtained are: $a = 12.142(3)$, $b = 16.975(4)$, $c = 20.229(7)$ Å, $\alpha = 83.66(2)$, $\beta = 74.83(2)$, and $\gamma = 69.47(2)°$. The density of the crystals measured by floatation methods was found to be 1.33 g/cm^3, the calculated density was 1.32 g/cm^3. This corresponds to $Z = 6$ molecules of $C_{29}H_{23}N_2ClO_4$ in the unit cell of space group $P\bar{1}$.

Data collection was carried out with MoKα($\lambda = 0.71073$ Å) radiation on an ENRAF-Nonius CAD4 diffractometer with a graphite crystal, incident beam monochromator. The data were collected at temperature of 23°(1) using the omega scanning technique. Data were collected to a maximum 2θ of 50°. A total of 3982 independent reflections were collected with $I > 3\sigma(I)$. The usual Lorentz polarization corrections were made but no attempt was made for absorption correction ($\mu = 1.9$ cm^{-1}).

The structure was solved by direct methods technique using SDP plus (FRENZ) on an SM 1420 computer. 1700 reflections with E > 1.4 were used. A total of 15 phase sets was calculated; the one with the best figure of merit was selected and an E-map was computed which revealed 30 non-hydrogen atoms. The remaining atoms were located in a succeeding Fourier synthesis. Several cycles of isotropic refinement were carried out followed by anisotropic block diagonal least squares, after several cycles R_0 had converged to 0.084. Unit weights are used during the refinement. In the final cycle the refinement included 487 variable parameters. The Fourier map showed no significant residual electron density.

Scattering factors were taken from CROMER and WABER (1979). Anomalous dispersion effects were included in F_c (Ibers, Hamilton).

The final fractional coordinates for the non-hydrogen atoms with their estimated standard of deviations are listed in Table 1.

Table 1

Atom	x	y	z	B_j(Å^2)	Atom	x	y	z	B_j(Å^2)
Cl1	0.9300(3)	0.1754(2)	0.3179(2)	4.52(8)	C141	0.3680(9)	0.1829(6)	0.3418(5)	3.4(3)
Cl2	0.5721(3)	0.3516(2)	0.1393(2)	5.80(9)	C142	0.376(1)	0.1486(9)	0.4058(7)	7.4(4)
Cl3	0.1894(3)	0.3272(2)	0.6869(2)	6.5(1)	C144	0.562(1)	0.1629(8)	0.3918(7)	6.6(4)
O11	0.8613(8)	0.1796(6)	0.2697(5)	6.9(2)*	C145	0.5620(9)	0.1947(7)	0.3286(6)	5.1(3)
O12	0.9789(8)	0.2401(6)	0.3016(5)	7.3(3)*	C146	0.4617(8)	0.2057(7)	0.3024(5)	4.0(3)
O13	0.854(1)	0.1855(8)	0.3844(7)	11.0(4)*	C211	0.3402(9)	0.5568(6)	0.6661(5)	3.8(3)
O14	1.0279(8)	0.0971(6)	0.3136(5)	6.9(2)*	C212	0.300(1)	0.5397(8)	0.6141(6)	5.8(4)
O21	0.455(1)	0.3642(9)	0.1887(8)	13.5(5)*	C213	0.342(1)	0.566(1)	0.5465(6)	7.3(4)
O22	0.581(1)	0.4311(7)	0.1267(6)	9.0(3)*	C214	0.427(1)	0.6061(9)	0.5338(6)	6.9(4)
O23	0.655(1)	0.2976 (8)	0.1756(6)	10.1(3)*	C215	0.464(1)	0.6240(8)	0.5852(6)	6.5(4)
O24	0.564(1)	0.3158(9)	0.0862(8)	13.3(5)*	C216	0.422(1)	0.6004(7)	0.6517(6)	5.7(3)
O31	0.211(1)	0.258(1)	0.7385(9)	15.8(6)*	C221	0.0514(8)	0.6422(6)	0.8910(5)	3.0(2)
O32	0.186(1)	0.3904(9)	0.7233(7)	12.7(4)*	C222	−0.0325(8)	0.6200(6)	0.9429(5)	3.5(3)
O33	0.285(1)	0.3149(8)	0.6267(7)	11.4(4)*	C223	−0.1275(9)	0.6802(7)	0.9833(6)	4.8(3)
O34	0.088(1)	0.327(1)	0.6761(8)	13.7(5)*	C224	−0.139(1)	0.7656(7)	0.9723(6)	4.7(3)
N11	0.2253(7)	0.2648(5)	0.2715(4)	3.2(2)	C225	−0.054(1)	0.7868(7)	0.9208(6)	5.0(3)
N21	0.3464(7)	0.4577(5)	0.7646(4)	3.2(2)	C226	0.0389(9)	0.7266(7)	0.8803(5)	4.2(3)
N31	0.2226(7)	0.0471(6)	0.6965(5)	3.8(2)	C231	0.3677(8)	0.3595(6)	0.8651(5)	3.4(3)
N143	0.469(1)	0.1412(9)	0.4327(6)	9.3(4)	C232	0.4285(9)	0.3637(6)	0.9143(5)	4.1(3)
N243	0.755(1)	0.390(1)	0.6268(7)	14.0(5)	C233	0.483(1)	0.2890(7)	0.9470(6)	4.9(3)
N343	−0.150(1)	0.0870(8)	0.8639(7)	9.7(4)	C234	0.476(1)	0.2147(8)	0.9333(6)	5.9(4)
C12	0.2623(8)	0.2547(6)	0.2024(5)	2.7(3)	C235	0.416(1)	0.2122(7)	0.8849(7)	5.6(4)
C13	0.2409(9)	0.3237(7)	0.1592(5)	3.5(3)	C236	0.3605(9)	0.2836(7)	0.8513(6)	4.4(3)
C14	0.1779(9)	0.4041(7)	0.1841(5)	3.1(3)	C240	0.4430(9)	0.3945(6)	0.7198(5)	4.0(3)
C15	0.1364(9)	0.4122(7)	0.2537(6)	3.6(3)	C241	0.5662(9)	0.4049(6)	0.7080(5)	3.6(3)
C16	0.1634(9)	0.3421(6)	0.2976(5)	3.1(3)	C242	0.642(1)	0.387(1)	0.6456(7)	9.0(5)
C23	0.1965(9)	0.5948(6)	0.7780(6)	3.4(3)	C244	0.793(1)	0.410(1)	0.6741(9)	9.2(5)
C24	0.1515(8)	0.5782(6)	0.8481(5)	3.0(3)	C245	0.726(1)	0.4303(9)	0.7382(7)	7.9(4)
C25	0.2098(9)	0.4991(6)	0.8726(6)	3.2(3)	C246	0.606(1)	0.4317(8)	0.7552(6)	6.0(4)
C26	0.3048(8)	0.4406(6)	0.8332(5)	2.9(3)	C311	0.1983(9)	0.0833(7)	0.5780(6)	4.5(3)
C32	0.268(1)	0.0750(7)	0.6315(6)	4.1(3)	C312	0.114(1)	0.1563(8)	0.5680(7)	6.2(4)
C33	0.371(1)	0.0947(8)	0.6160(6)	4.5(3)	C313	0.050(1)	0.1632(9)	0.5181(7)	8.4(4)
C34	0.437(1)	0.0875(8)	0.6653(6)	4.4(3)	C314	0.071(1)	0.0976(9)	0.4807(7)	8.9(4)
C35	0.389(1)	0.0586(8)	0.7298(6)	4.6(3)	C315	0.157(2)	0.026(1)	0.4896(8)	11.0(5)
C36	0.2833(9)	0.0392(7)	0.7457(6)	4.3(3)	C316	0.222(1)	0.0181(9)	0.5377(7)	9.2(5)
C111	0.3257(9)	0.1695(6)	0.1727(5)	3.3(3)	C321	0.5455(9)	0.1117(8)	0.6511(6)	5.7(4)
C112	0.266(1)	0.1129(7)	0.1759(7)	4.5(3)	C322	0.625(1)	0.0802(9)	0.6935(7)	7.9(4)
C113	0.322(1)	0.0404(7)	0.1400(7)	5.5(4)	C323	0.727(1)	0.105(1)	0.6810(8)	11.5(6)
C114	0.442(1)	0.0218(8)	0.1003(7)	5.3(4)	C324	0.750(1)	0.158(1)	0.6272(8)	8.7(5)
C115	0.500(1)	0.0782(8)	0.0981(6)	4.9(4)	C325	0.675(1)	0.184(1)	0.5827(9)	8.7(5)
C116	0.444(1)	0.1520(7)	0.1330(6)	4.1(3)	C326	0.569(1)	0.1633(9)	0.5963(7)	7.2(4)
C121	0.1618(9)	0.4809(7)	0.1388(5)	3.3(3)	C331	0.2380(8)	0.0099(6)	0.8159(5)	3.6(3)
C122	0.057(1)	0.5495(7)	0.1536(6)	4.6(3)	C332	0.188(1)	0.0688(7)	0.8689(6)	5.3(3)
C123	0.049(1)	0.6211(8)	0.1113(7)	6.1(4)	C333	0.150(1)	0.0425(8)	0.9340(6)	5.5(4)
C124	0.137(1)	0.6253(8)	0.0561(6)	6.0(4)	C334	0.158(1)	−0.0392(9)	0.9489(6)	6.3(4)
C125	0.240(1)	0.5556(8)	0.0420(6)	5.7(4)	C335	0.206(1)	−0.0990(8)	0.8985(6)	5.5(4)
C126	0.256(1)	0.4820(8)	0.0822(6)	4.9(4)	C336	0.243(1)	−0.0716(7)	0.8308(6)	4.6(3)
C131	0.131(1)	0.3570(7)	0.3727(6)	4.0(3)	C340	0.1094(9)	0.0253(6)	0.7125(6)	4.4(3)
C132	0.210(1)	0.3713(9)	0.4026(7)	6.2(4)	C341	0.0096(8)	0.0823(6)	0.7653(5)	3.9(3)
C133	0.180(1)	0.388(1)	0.4719(7)	7.9(5)	C342	−0.057(1)	0.0466(8)	0.8169(6)	5.9(3)
C134	0.070(1)	0.3919(9)	0.5095(7)	7.3(5)	C344	−0.175(1)	0.170(1)	0.8635(8)	9.8(5)
C135	−0.013(1)	0.380(1)	0.4806(7)	7.8(5)	C345	−0.115(1)	0.2137(9)	0.8139(8)	7.8(5)
C136	0.018(1)	0.3609(9)	0.4118(7)	6.2(4)	C346	−0.021(1)	0.1670(7)	0.7634(6)	5.5(3)
C140	0.2557(9)	0.1927(6)	0.3198(5)	3.6(3)					

Results and discussion

Crystals of $C_{29}H_{23}N_2ClO_4$ crystallize in the space group $P\bar{1}$ with 3 molecules per asymmetric unit. The overall structures of the three independent molecules as well the numbering schemes are shown in Figure 1.

Bond lengths and angles for the three molecules are listed in Tables 2 and 3.

It was noticed as expected (EL-SHORA et al.) that the perchlorate group is disordered, therefore the temperature factors for all the oxygen atoms were refined isotropically.

The average C–C bond lengths in the phenyl rings C, D, and E of molecules (I), (II) are found to be in the range of 1.37 to 1.38 Å which is in agreement with that value found in similar structures (EL-SHORA et al. 1989, 1990 A, B). In molecule (III) the corresponding values range from 1.35 to 1.39 Å. For rings A and B which contain the nitrogen atoms the average bond lengths in the three molecules range from 1.35 to 1.38 Å and C–N bond

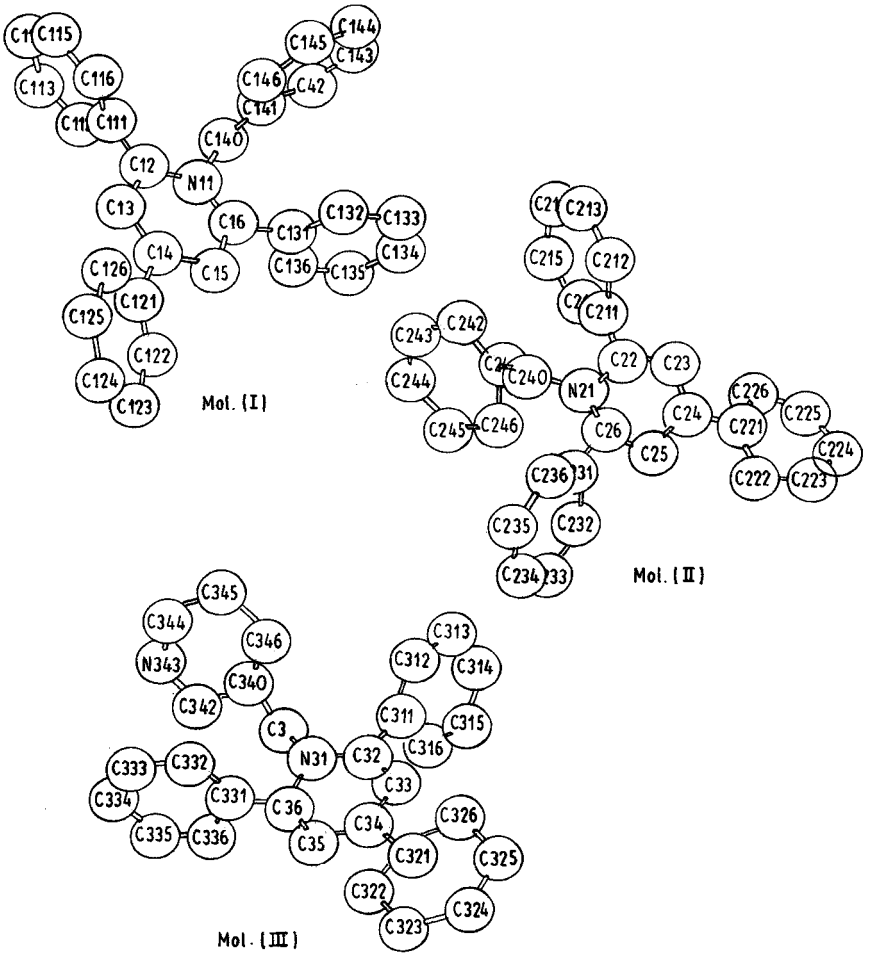

Fig. 1. Structures of the three independent molecules with their numbering

Table 2
Bond lengths for the nonhydrogen atoms of molecules (I), (II) and (III) in Å

atom 1	atom 2	distance	atom 1	atom 2	distance	atom 1	atom 2	distance
N11	C12	1.36	C14	C121	1.49	C136	C131	1.38
N21	C22	1.37	C24	C121	1.46	C236	C231	1.38
N31	C32	1.39	C34	C131	1.47	C336	C331	1.37
C12	C13	1.37	C121	C122	1.38	N11	C140	1.48
C22	C23	1.37	C221	C222	1.38	N21	C240	1.46
C32	C33	1.36	C321	C322	1.39	N31	C340	1.49
C13	C14	1.39	C122	C123	1.40	C140	C141	1.49
C23	C24	1.42	C222	C223	1.38	C240	C241	1.52
C33	C34	1.40	C322	C323	1.41	C340	C341	1.51
C14	C15	1.37	C123	C124	1.35	C141	C142	1.37
C24	C25	1.38	C223	C224	1.40	C241	C242	1.35
C34	C35	1.39	C323	C324	1.36	C341	C342	1.37
C15	C16	1.40	C124	C125	1.38	C142	N143	1.33
C25	C26	1.36	C224	C225	1.38	C242	N243	1.34
C35	C36	1.38	C324	C325	1.37	C342	N343	1.30
C16	N11	1.35	C125	C126	1.40	N143	C144	1.36
C26	N21	1.38	C225	C226	1.37	N243	C244	1.29
C36	N31	1.36	C325	C326	1.41	N343	C344	1.34
C12	C111	1.49	C126	C121	1.39	C144	C145	1.33
C22	C211	1.49	C226	C221	1.39	C244	C245	1.35
C32	C311	1.50	C326	C321	1.37	C344	C345	1.38
C111	C112	1.38	C16	C131	1.49	C145	C146	1.40
C211	C212	1.36	C26	C231	1.49	C245	C246	1.40
C311	C312	1.34	C36	C331	1.48	C345	C346	1.38
C112	C113	1.36	C131	C132	1.35	C146	C141	1.36
C212	C213	1.41	C231	C232	1.41	C246	C241	1.36
C312	C313	1.39	C331	C332	1.41	C346	C341	1.35
C113	C114	1.40	C132	C133	1.39	Cl1	O11	1.42
C213	C214	1.37	C232	C233	1.39	Cl1	O12	1.39
C313	C314	1.33	C332	C333	1.36	Cl1	O13	1.41
C114	C115	1.37	C133	C134	1.34	Cl1	O14	1.43
C214	C215	1.34	C233	C234	1.36	Cl2	O21	1.47
C314	C315	1.35	C333	C334	1.36	Cl2	O22	1.38
C115	C116	1.37	C134	C135	1.37	Cl2	O23	1.41
C215	C216	1.38	C234	C235	1.37	Cl2	O24	1.33
C315	C316	1.38	C334	C335	1.38	Cl3	O31	1.48
C116	C111	1.39	C135	C136	1.39	Cl3	O32	1.35
C216	C211	1.39	C235	C236	1.37	Cl3	O33	1.42
C316	C311	1.35	C335	C336	1.41	Cl3	O34	1.30

Table 3
Bond angles for the nonhydrogen atoms of molecules (I), (II), and (III)

atom 1	atom 2	atom 3	angle	atom 1	atom 2	atom 3	angle	atom 1	atom 2	atom 3	angle
C12	N11	C16	120.2	C212	C213	C214	119	C324	C325	C326	120
C22	N21	C26	118.3	C312	C313	C314	121	C125	C126	C121	117.3
C32	N31	C36	119	C113	C114	C115	119	C225	C226	C221	121
C13	C12	N11	119.9	C213	C214	C215	121	C325	C326	C321	120
C23	C22	N21	121.8	C313	C314	C315	119	N11	C16	C131	122
C33	C32	N31	122	C114	C115	C116	121	N21	C26	C231	120.1
C12	C13	C14	121	C214	C215	C216	121	N31	C36	C331	121
C22	C23	C24	120.8	C314	C315	C316	121	C15	C16	C131	117.8
C32	C33	C34	121	C115	C116	C111	120	C25	C26	C231	119.6

Table 3 (Fortsetzung)

atom 1	atom 2	atom 3	angle	atom 1	atom 2	atom 3	angle	atom 1	atom 2	atom 3	angle
C13	C14	C15	117.5	C215	C216	C211	120	C35	C36	C331	120
C23	C24	C25	115.6	C312	C316	C311	121	C16	C131	C132	121
C33	C34	C35	115	C13	C14	C121	122.7	C26	C231	C232	116.9
C14	C15	C16	120.7	C23	C24	C221	121	C36	C331	C332	118
C24	C25	C26	123.3	C33	C34	C321	123	C16	C131	C136	120
C34	C35	C36	123	C15	C14	C121	119.7	C26	C231	C236	122
C15	C16	N11	117.8	C25	C24	C221	123.3	C36	C331	C336	122
C25	C26	N21	119.6	C35	C34	C321	122	C132	C131	C136	119
C35	C36	N31	120	C14	C121	C122	120.6	C232	C231	C236	121
N11	C12	C111	121	C24	C221	C222	120.9	C332	C331	C336	119.3
N21	C22	C211	119	C34	C321	C322	119	C131	C132	C133	122
N31	C32	C311	118	C14	C121	C126	118.4	C231	C232	C233	118
C13	C12	C111	119.1	C24	C221	C226	120.5	C331	C332	C333	119
C23	C22	C211	119.2	C34	C321	C326	121	C132	C133	C134	119
C33	C32	C311	121	C126	C121	C122	121	C232	C233	C234	121
C12	C111	C112	121.8	C226	C221	C222	118.6	C332	C333	C334	121
C22	C211	C212	121	C326	C321	C322	121	C133	C134	C135	121
C32	C311	C312	121	C121	C122	C123	118	C233	C234	C235	120
C12	C111	C116	118	C221	C222	C223	121	C333	C334	C335	122
C22	C211	C216	119	C321	C322	C323	118	C134	C135	C136	120
C32	C311	C316	121	C122	C123	C124	123	C234	C235	C236	121
C112	C111	C116	120	C222	C223	C224	120	C334	C335	C336	117
C212	C211	C216	119	C322	C323	C324	121	C135	C136	C131	119
C312	C311	C316	119	C123	C124	C125	118	C235	C236	C231	119
C111	C112	C113	120	C223	C224	C225	118.3	C335	C336	C331	121
C211	C212	C213	120	C323	C324	C325	121	N11	C140	C141	113
C311	C312	C313	120	C124	C125	C126	123	N21	C240	C241	113
C112	C113	C114	121	C224	C225	C226	121	C31	C340	C341	112.1
C140	C141	C142	117.4	C240	C241	C242	118	C340	C341	C342	118
C140	C141	C146	124	C240	C241	C246	125.4	C340	C341	C346	124
C142	C141	C146	119	C242	C241	C246	117	C342	C341	C346	117
C141	C142	C143	123	C241	C242	N243	126	C341	C342	N343	126
C142	N143	C144	117	C242	N243	C244	116	C342	N343	C344	115
N143	C144	C145	123	N243	C244	C245	124	N343	C344	C345	124
C144	C145	C146	119	C244	C245	C246	119	C344	C345	C346	117
C145	C146	C141	119.2	C245	C246	C241	118	C345	C346	C341	120
O11	Cl1	O12	107.7	O21	Cl2	O22	104.8	O31	Cl3	O32	99
O12	Cl1	O13	110.3	O22	Cl2	O23	113.9	O32	Cl3	O33	109.9
O13	Cl1	O14	110.3	O23	Cl2	O24	113.8	O33	Cl3	O34	113.5

distances in the three molecules in ring A vary from 1.36 to 1.39 but those in rings B show shorter values and range from 1.29 to 1.38 Å. This is probably due to steric repulsion between the molecules.

The average bond angles in rings A, B, C, D, and D in the three molecules are found to be within the expected values of such structures which is 120°. The cation moiety of each molecule contains five planes of atoms. The dihedral angles between plane A (the central plane) and rings B, C, D, and E are listed in the following table.

Mol./plane	A/B	A/C	A/D	A/E
Molecule I	67	38	92	86
Molecule II	94	35	112	101
Molecule III	88	20	108	93

These values showed as have been expected that rings B, C, D, and E are twisted with respect of the central plane. This is probably due two the steric repulsion between the molecules.

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