898. *Molecular Complexes. Part II.* The Crystal Structure of the 1 : 1 Complex of Bis-8-hydroxyquinolinatopalladium*(II) *and Chloranil*

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The crystal structure of the 1 : 1 complex of bis-8-hydroxyquinolinatopalladium(II) and chloranil has been determined by three-dimensional *X*-ray diffraction measurements. It is a plane-to-plane π-complex in which the quinoline system of the ligand is the electron donor and chloranil the acceptor. The structure differs from that predicted by the "overlap and orientation principle" because of a specific interaction between the chlorine atoms of the chloranil and the palladium atom, and the requirements of crystal packing.

RECENTLY, Bailey, Williams, and Wright [1] reported a number of what appeared to be "charge-transfer" or π-complexes of palladium, nickel, and copper 8-hydroxyquinolinates with a variety of organic electron-acceptor molecules. In the solid state, these fall into two stoicheiometric groups, those with two acceptor molecules to one molecule of the metal complex, *e.g.*, complexes with *sym*-trinitrobenzene, picryl azide, 1,2,4,5-tetracyanobenzene, and benzotrifurazan; and those with one acceptor molecule to one molecule

* Part I, B. Kamenar and C. K. Prout, preceding Paper.

[1] A. S. Bailey, R. J. P. Williams, and J. D. Wright, *J.*, 1965, 2579.

of the metal complex, *e.g.*, with chloranil, tetracyanoquinodimethane,[2] and benzotrifuroxan.

Whilst the physicochemical measurements of Bailey, Williams, and Wright [1] point to the complex form, the stoicheiometries do not allow the possibility of co-ordination of functional groups of the electron-acceptor molecules with the metal atom to be discounted.

X-Ray structural analyses have been carried out on some of these complexes to establish the complex form, to examine the effect of the presence of a metal atom on the structure of such complexes, and to define the co-ordination sphere around the metal atom. Examples have been chosen to take into consideration the interest of the structure of the electron-acceptor molecule and the usefulness of the structure determination to colleagues examining the single-crystal optical and electronic properties of the complexes.

The crystals of the bis-8-hydroxyquinolinatopalladium(II) complex with chloranil are green-black triclinic needles stable in air and in the *X*-ray beam. They are remarkable in that the intensity and extent of the Bragg scattering is such as might be expected from an ionic, or highly hydrogen-bonded crystal.

Crystal Data.—$C_{24}H_{12}Cl_4N_2O_4Pd$, $M = 640{\cdot}6$. Triclinic pinacoidal, $a = 8{\cdot}17 \pm 0{\cdot}02$, $b = 8{\cdot}18 \pm 0{\cdot}2$, $c = 9{\cdot}69 \pm 0{\cdot}02$ Å, $\alpha = 99{\cdot}5 \pm 0{\cdot}2$, $\beta = 77{\cdot}8 \pm 0{\cdot}2$, $\gamma = 66{\cdot}0 \pm 0{\cdot}02°$; $U = 546{\cdot}5$ Å^3, $D_m = 1{\cdot}931$, $Z = 1$, $D_c = 1{\cdot}946$, $F(000) = 316$. Space group $P\bar{1}$ (C_i^1, No. 2), Cu-K_α radiation, $\mu = 119{\cdot}2$ cm.$^{-1}$; single-crystal oscillation and Weissenberg photographs: optically biaxial.

Since there is only one molecule of the complex to the unit cell, and since there is no reason to suppose otherwise than that both constituent molecules will have centres of symmetry, it is reasonable to assume that the space group is $P\bar{1}$, and that the palladium atom is at the cell origin. This palladium atom is sufficiently heavy to dominate the phases of the structure factors. Therefore, the three-dimensional electron-density distribution in the crystal was computed on the assumption that all the structure amplitudes have positive signs. There were seventeen significant maxima in this distribution, corresponding to the seventeen independent atoms, excluding hydrogen, of the two centric molecules.

Preliminary atomic positions were improved by five cycles of least-squares refinement, by using a block-diagonal approximation to the normal matrix. Unit weights were used in the first two cycles and then the weighting scheme

$$\omega = \sqrt{\frac{1}{1 + \left(\frac{|F_o| - b}{a}\right)^2}}$$

where $a = 120$ and $b = 120$ on the same scale as Table 1. The final reliability index of observed reflections was 0·107.

Table 1 lists the observed structure amplitudes and calculated structure factors based on the atomic co-ordinates in Table 2 and thermal parameters in Table 3. The standard deviations are minimum values computed from the block-diagonalised normal matrix. The atomic co-ordinates (in Å) in Table 4 are referred to a set of orthogonal axes, X', Y', and Z', with their origin at the cell origin and defining the least-squares best plane of the bis-8-hydroxyquinolinatopalladium(II) molecule. The transformation matrix from the orthogonal axes, a, b', and c^*, where c^* is the reciprocal c axis and b' is on the same side of the ac^* plane, to these co-ordinates is

$$\begin{pmatrix} 0{\cdot}4270 & 0{\cdot}0762 & 0{\cdot}9010 \\ 0{\cdot}4906 & -0{\cdot}8566 & -0{\cdot}1600 \\ 0{\cdot}7597 & 0{\cdot}5104 & -0{\cdot}4032 \end{pmatrix}$$

[2] L. R. Melby, R. J. Harder, W. R. Hertler, W. Mahler, R. E. Benson, and W. E. Mochel, *J. Amer. Chem. Soc.*, 1962, **84**, 3374.

TABLE 1

Observed structure amplitudes and calculated structure factors for (*hkl*)

h	*k*	*l*	5|F_o|	5F_c
0	−7	1	143	21
		2	176	181
		3	157	152
		4	156	149
		5	48	−23
		6	67	63
		7	90	94
		8	112	126
		9	115	139
		10	95	109
0	−6	1	72	62
		2	38	47
		3	133	103
		4	258	255
		5	253	266
		6	176	197
		7	84	83
		8	85	85
		9	49	51
		10	100	111
		11	79	76
0	−5	1	220	204
		2	198	170
		3	195	164
		4	235	233
		5	216	190
		6	142	134
		7	69	71
		9	53	58
		10	99	99
		11	128	163
0	−4	1	46	−14
		2	169	163
		3	85	63
		4	45	55
		5	195	188
		6	203	211
		7	247	249
		8	180	170
		9	129	110
		10	71	74
		11	65	70
		12	101	117
0	−3	1	140	100
		2	260	334
		3	261	307
		4	164	158
		5	112	86
		7	164	157
		8	155	146
		9	264	279
		10	45	51
		11	100	112
0	−2	1	42	−8
		2	174	180
		3	131	106
		4	65	48
		5	36	32
		6	109	86
		7	180	149
		8	274	288
		9	298	300
		10	180	171
		11	101	104
		12	39	57
0	−1	2	67	36
		3	170	161
		4	249	292
		5	258	310
		6	155	128
		7	58	48
		8	68	51
		9	196	165
		10	179	175
		11	114	123
0	0	2	146	121
		3	111	−72
		4	220	188
		5	184	132
		6	79	61
		7	132	105
		8	132	100
		9	110	99
		10	138	166
		11	132	155
0	1	1	185	163
		2	171	178
		3	191	159
		4	257	276
		6	288	231
		7	201	233
		7	240	233
		8	40	33
		9	27	−8
		10	55	58
0	2	0	167	137
		1	244	300
		2	313	417
		3	279	311
		4	121	96
		5	114	96
		6	125	104
		7	156	133
		8	107	92
		9	124	132
		10	93	106
0	3	0	198	−192
		2	114	111
		3	196	168
		4	220	189
		5	250	248
		6	255	213
		7	199	192
		8	168	169
		9	136	144
		10	87	86
0	4	0	117	79
		1	176	172
		2	207	207
		3	223	218
		4	151	128
		5	278	304
		6	118	111
		7	77	86
		8	30	44
0	5	0	290	272
		1	160	134
		2	85	78
		3	124	−87
		4	119	110
		5	171	173
		6	141	159
		7	115	121
		8	89	102
0	6	0	143	129
		1	156	132
		2	157	134
		3	173	169
		4	139	120
		5	97	90
		6	99	88
0	7	0	155	145
		1	133	143
		2	147	166
		3	75	83
		5	44	−29
0	8	0	34	40
		1	44	57
		2	75	82
		3	136	183
0	9	0	122	143
1	−9	2	46	52
		3	56	63
		4	95	112
		5	105	135
1	−8	−2	32	43
		−1	34	39
		0	67	76
		1	87	85
		2	119	117
		3	169	175
		4	138	156
		5	103	106
		8	99	103
1	−7	−4	169	186
		−3	153	157
		−2	128	141
		−1	201	214
		0	164	153
		2	53	51
		3	31	35
		4	114	96
		5	30	37
		6	143	140
		7	148	147
		8	100	106
		9	97	112
		10	112	128
1	−6	−6	110	141
		−5	168	190
		−4	145	167
		−3	96	101
		−2	44	54
		−1	94	82
		0	144	120
		1	169	172

TABLE 1 (*Continued*)

h	*k*	*l*	$5\|F_o\|$	$5F_c$
		2	168	179
		3	171	148
		4	63	47
		5	32	26
		6	206	210
		7	75	81
		9	46	−32
		10	123	139
		11	147	169
1	−5	−7	46	65
		−6	68	75
		−5	49	60
		−4	106	101
		−3	141	127
		−2	165	133
		−1	219	197
		0	238	212
		1	294	261
		2	198	186
		3	50	52
		4	128	−78
		5	30	−5
		6	135	113
		7	206	210
		8	149	140
		9	97	101
		10	90	110
		11	93	105
1	−4	−8	49	59
		−7	155	164
		−6	110	117
		−5	177	154
		−4	31	17
		−3	189	167
		−2	190	185
		−1	60	54
		1	119	96
		2	242	228
		3	270	265
		4	250	233
		5	163	153
		6	137	124
		7	147	132
		8	167	159
		9	115	108
		11	40	54
		12	74	97
1	−3	−9	107	115
		−8	196	191
		−7	184	190
		−6	144	127
		−5	69	−43
		−4	166	−144
		−3	195	187
		−2	208	174
		−1	302	303
		0	141	118
		1	294	264
		2	238	225
		3	238	214
		4	278	314
		5	228	194
		6	67	−26
		7	66	69
		8	182	174
		9	284	258
		10	93	103
		11	61	83
		12	51	61
1	−2	−10	84	93
		−9	68	86
		−8	116	119
		−7	117	114
		−6	181	164
		−5	210	166
		−4	213	187
		−3	196	157
		−2	282	287
		−1	265	254
		0	177	156
		1	37	−15
		2	118	−79
		3	144	147
		4	360	472
		5	260	295
		6	239	233
		7	103	99
		8	202	195
		9	118	100
		10	30	15
		11	119	119
		12	63	79
1	−1	−10	153	172
		−9	163	164
		−8	142	124
		−7	166	137
		−6	268	239
		−5	292	285
		−4	164	151
		−3	41	30
		−2	47	41
		−1	254	264
		0	237	270
		1	213	236
		2	229	336
		3	167	158
		4	132	128
		5	127	104
		6	230	203
		7	165	121
		8	123	105
		9	32	5
		10	170	168
		11	166	179
		12	167	199
1	0	−11	131	150
		−10	206	221
		−10	200	221
		−9	89	101
		−9	102	191
		−8	70	71
		−8	77	71
		−7	53	56
		−7	53	56
		−6	93	76
		−5	294	264
		−4	353	404
		−3	262	264
		−2	236	283
		2	136	161
		3	72	−64
		4	46	−12
		5	146	124
		6	326	309
		7	266	225
		8	181	146
		9	147	140
		10	112	120
		11	114	117
		12	73	120
1	1	−10	146	109
		−10	113	109
		−9	122	125
		−9	126	125
		−8	118	162
		−8	175	162
		−7	207	183
		−6	141	109
		−5	272	257
		−4	181	152
		−3	250	269
		−2	165	−150
		2	251	450
		3	250	323
		4	320	359
		5	18	76
		6	100	80
		7	84	68
		8	147	121
		9	187	165
		10	165	161
		11	96	108
		11	116	108
1	2	−11	121	136
		−10	152	104
		−10	92	104
		−9	103	86
		−9	82	86
		−8	163	138
		−7	205	183
		−6	60	57
		−5	34	−2
		−4	46	39
		−3	244	244
		−2	251	290
		−1	340	521
		1	222	190
		2	59	43
		3	295	309
		4	230	231
		5	109	92
		6	111	91
		7	158	120
		8	191	164
		9	292	270
		10	128	143

TABLE 1 (*Continued*)

h	*k*	*l*	5|F_o|	5F_c
		11	95	115
1	3	−11	93	100
		−10	39	−1
		−9	93	88
		−8	113	102
		−7	237	221
		−6	240	219
		−5	256	249
		−4	165	141
		−3	149	123
		−2	58	48
		−1	200	183
		0	270	249
		1	135	89
		2	160	140
		3	251	264
		4	303	350
		5	274	250
		6	150	121
		7	83	71
		8	100	97
		9	98	100
		10	93	107
1	4	−11	37	57
		−10	190	195
		−9	198	200
		−7	122	108
		−6	186	171
		−5	284	271
		−5	75	271
		−4	88	84
		−2	151	127
		−1	277	251
		0	357	330
		1	324	317
		2	190	141
		3	70	60
		5	92	83
		6	154	135
		7	177	173
		8	123	126
		9	131	122
1	5	−11	58	66
		−10	179	190
		−9	129	121
		−8	70	70
		−6	244	197
		−5	286	273
		−4	268	237
		−3	260	244
		−2	175	146
		−1	88	−50
		0	138	115
		1	121	104
		2	213	191
		3	99	−79
		4	88	78
		5	199	205
		6	225	236
		7	168	175
		8	96	93
1	6	−11	49	53
		−10	125	115
		−9	166	181
		−8	170	179
		−7	125	122
		−6	70	59
		−5	69	61
		−4	128	115
		−3	172	152
		−2	124	97
		−1	117	89
		0	174	139
		1	258	246
		2	184	158
		3	32	30
		4	131	133
		5	58	67
		6	91	103
		7	20	42
1	7	−10	31	38
		−9	53	66
		−8	119	122
		−7	84	85
		−4	137	129
		−3	235	212
		−2	249	243
		−1	191	174
		0	118	111
		3	97	103
		4	106	126
		5	148	151
		6	104	110
1	8	−9	66	73
		−8	104	107
		−7	178	200
		−6	48	56
		−5	77	71
		−4	67	73
		−3	67	67
		−2	90	84
		−1	126	125
		0	142	129
		1	127	127
		2	140	159
		3	179	189
		4	148	156
1	9	−7	35	50
		−5	123	116
		−4	102	100
		−3	112	117
		−2	80	89
		−1	147	153
		0	150	156
		1	83	72
		2	76	93
2	−10	−3	5	98
		−2	4	85
		−1	3	98
2	−8	0	140	159
		1	131	134
		2	69	60
		4	60	60
		5	79	72
		6	90	92
		7	45	57
2	−7	−3	120	136
		−2	76	67
		−1	47	49
		0	44	51
		1	47	48
		2	79	72
		3	63	50
		4	80	72
		5	162	152
		6	164	177
		7	131	138
		8	58	68
2	−6	−4	117	122
		−3	55	62
		−2	78	84
		−1	94	93
		0	86	70
		1	194	188
		2	221	214
		3	156	129
		5	54	−18
		6	152	138
		7	125	121
		8	145	140
		9	69	83
		10	86	90
2	−5	−5	101	87
		−4	164	166
		−3	215	226
		−2	165	169
		−1	133	117
		0	54	39
		1	91	86
		2	96	95
		3	205	189
		4	74	55
		5	190	181
		6	150	138
		7	165	153
		8	161	140
		9	116	115
		10	92	95
2	−4	−7	135	129
		−6	130	118
		−5	113	106
		−4	98	89
		−2	114	101
		−1	198	185
		0	190	167
		1	168	147
		2	204	189
		3	278	297
		4	264	264
		5	149	146
		9	135	141
		10	145	146
		11	117	129
		12	76	89
2	−3	−8	65	62
		−7	107	106

TABLE 1 (*Continued*)

h	*k*	*l*	5|F_o|	5F_c
		−6	171	174
		−5	140	129
		−3	186	182
		−2	228	211
		−1	263	286
		0	188	161
		1	133	115
		2	82	66
		3	128	129
		4	140	127
		5	189	186
		6	191	190
		7	126	126
		8	88	78
		9	98	86
		10	169	166
		11	123	129
		12	38	55
2	−2	−9	70	64
		−8	134	144
		−7	170	179
		−6	291	311
		−5	119	99
		−4	203	194
		−3	111	110
		−2	34	25
		−1	122	−94
		0	40	−1
		1	274	324
		2	221	235
		3	141	153
		4	221	228
		5	169	186
		6	209	220
		7	143	128
		8	123	96
		9	62	56
		10	84	79
		11	135	139
		11	140	139
		12	138	135
		12	129	135
2	−1	−10	129	140
		−9	157	161
		−8	160	157
		−7	18	129
		−6	111	98
		−5	86	82
		−4	189	169
		−3	216	211
		−2	172	147
		−1	84	−52
		0	204	182
		1	243	308
		2	168	195
		3	97	92
		4	58	17
		5	38	−0
		6	183	175
		7	263	259
		8	361	413
		9	99	95
		10	107	113
		11	76	69
		11	67	69
		12	147	163
		12	140	163
2	0	−10	45	60
		−9	60	65
		−8	146	150
		−7	159	154
		−6	157	160
		−5	150	155
		−4	58	265
		−3	250	288
		−2	165	179
		−1	74	64
		3	293	271
		4	181	230
		5	186	176
		6	146	131
		7	268	270
		8	255	235
		9	150	130
		10	83	77
		10	66	77
		11	65	69
		11	55	69
		12	125	142
		12	121	142
2	1	−10	40	44
		−9	81	85
		−8	197	195
		−7	169	165
		−6	95	88
		−5	178	168
		−4	22	22
		−3	125	123
		−2	228	291
		3	185	190
		4	162	180
		5	68	54
		6	45	41
		7	77	65
		8	210	186
		9	215	208
		10	176	179
		11	160	148
		12	28	94
2	2	−11	129	145
		−10	75	82
		−9	51	47
		−8	31	42
		−7	91	62
		−6	247	227
		−5	252	269
		−4	107	104
		−3	161	157
		−2	244	315
		2	130	−93
		3	173	181
		4	173	151
		5	199	192
		6	248	206
		7	145	108
		8	31	37
		9	127	110
		10	137	129
		11	121	122
2	3	−11	159	170
		−10	186	186
		−9	126	113
		−8	113	100
		−7	139	123
		−6	207	188
		−5	152	130
		−4	84	79
		−2	193	202
		−1	191	214
		1	332	347
		2	278	284
		3	222	218
		5	143	141
		6	156	150
		7	31	−13
		8	31	34
		9	118	108
		10	154	161
2	4	−11	131	133
		−11	126	133
		−10	243	216
		−9	184	177
		−8	94	92
		−6	41	−1
		−5	93	81
		−4	228	227
		−3	283	274
		−2	107	91
		−1	99	76
		0	177	144
		1	231	215
		2	164	183
		3	103	89
		4	160	151
		5	170	159
		6	192	170
		7	164	150
		8	174	178
		9	116	105
		10	81	90
2	5	−10	216	194
		−9	131	130
		−8	187	186
		−7	108	111
		−6	142	119
		−5	118	98
		−4	160	145
		−3	153	129
		−2	35	22
		−1	111	−78
		0	118	102
		1	196	171
		2	251	255
		3	267	286
		4	264	278
		5	156	145

TABLE 1 (*Continued*)

h	*k*	*l*	5|F_o|	5F_c
		6	102	98
		7	99	94
		8	127	112
		9	76	81
2	6	−10	100	99
		−9	99	109
		−8	136	133
		−7	142	130
		−6	171	135
		−5	61	58
		−4	89	75
		−3	189	176
		−2	147	112
		−1	229	202
		0	196	168
		1	138	126
		2	91	78
		3	69	56
		4	214	221
		5	105	104
		6	129	125
		7	113	102
		8	158	167
2	7	−10	47	51
		−9	106	51
		−8	72	73
		−7	170	168
		−6	237	236
		−5	252	233
		−4	143	128
		−3	76	68
		−2	76	74
		−1	155	129
		0	88	84
		1	108	96
		3	164	166
		4	174	180
		5	169	166
		6	108	102
		7	39	68
2	8	−9	102	112
		−8	67	74
		−7	112	112
		−6	140	130
		−5	153	145
		−4	91	76
		−3	81	78
		−2	119	97
		−1	173	156
		0	165	163
		1	137	139
		2	115	113
		5	55	47
2	9	−8	85	94
		−7	54	55
		−5	148	160
		−4	155	172
		−3	159	158
		−2	59	73
		−1	44	55
		0	42	51
		1	90	95
		2	74	76
		4	4	49
2	10	0	66	112
3	−7	−1	27	−5
		0	53	53
		1	134	118
		2	145	144
		3	190	188
		4	140	152
		5	92	83
		6	62	66
		7	104	92
		8	95	105
3	−6	−3	99	97
		−2	126	124
		−1	130	134
		0	80	85
		1	59	62
		2	97	96
		3	102	99
		4	86	88
		5	60	61
		6	64	70
		7	148	149
		8	166	180
		9	174	164
		10	79	87
3	−5	−5	178	208
		−4	126	121
		−3	93	84
		−2	130	135
		−1	79	81
		1	90	92
		2	90	83
		3	151	130
		4	90	73
		5	208	194
		6	139	138
		7	48	52
		9	107	107
		10	158	150
		11	93	107
3	−4	−6	110	113
		−5	96	107
		−4	94	97
		−3	93	91
		−2	149	136
		−1	184	170
		0	239	245
		1	200	196
		2	89	83
		3	136	−102
		4	98	−58
		5	191	191
		6	126	114
		7	99	99
		9	171	186
		10	185	181
		11	157	163
3	−3	−7	82	74
		−6	86	85
		−5	150	147
		−4	173	181
		−3	150	136
		−2	132	119
		−1	104	97
		0	190	160
		1	155	141
		2	105	85
		4	124	105
		5	179	163
		6	222	230
		7	163	140
		8	95	99
		9	49	59
		10	51	56
		11	125	128
		12	179	187
3	−2	−8	162	163
		−7	82	83
		−6	67	64
		−5	95	−62
		−4	119	110
		−3	148	150
		−2	141	134
		−1	147	123
		0	212	202
		1	264	273
		2	269	314
		3	235	260
		4	117	95
		6	43	42
		7	154	156
		8	163	158
		9	90	96
		10	96	94
		11	102	110
		12	157	155
3	−1	−9	114	121
		−8	102	113
		−7	104	114
		−5	28	−19
		−4	231	227
		−3	224	226
		−2	288	309
		−1	107	105
		0	144	129
		1	57	55
		2	154	155
		3	267	365
		4	238	274
		5	73	75
		6	136	133
		7	190	177
		8	209	203
		9	64	62
		10	66	65
		12	79	83
3	0	−9	126	130
		−8	133	135
		−7	207	196
		−6	180	168
		−5	142	125
		−4	42	36

TABLE 1 (*Continued*)

h	*k*	*l*	5|F_o|	5F_c
		−3	120	126
		−2	141	133
		−1	161	156
		3	275	442
		4	193	210
		5	190	198
		6	40	38
		7	134	130
		9	40	52
		10	205	179
		11	142	134
		12	109	122
3	1	−10	138	137
		−9	80	86
		−8	67	74
		−7	164	150
		−6	207	194
		−5	176	156
		−4	156	132
		−3	145	144
		−2	180	195
		−1	123	159
		3	62	−26
		4	104	86
		5	276	273
		6	258	238
		7	212	186
		8	63	52
		9	197	172
		10	150	141
		11	171	165
3	2	−10	57	78
		−9	105	109
		−8	125	117
		−7	131	110
		−6	246	229
		−5	275	286
		−4	186	186
		−3	97	91
		−2	116	−85
		2	50	121
		3	151	158
		4	180	178
		5	260	252
		6	180	147
		7	147	118
		8	164	137
		9	104	105
		10	118	115
		11	125	135
3	3	−10	90	109
		−9	152	146
		−8	143	131
		−7	57	50
		−6	130	128
		−5	78	74
		−4	188	195
		−3	64	58
		−2	250	299
		−1	187	199
		0	102	99
		1	216	235
		2	206	203
		3	218	224
		4	30	15
		6	99	87
		7	222	198
		8	266	242
		9	191	190
		10	104	105
		11	78	103
3	4	−9	88	84
		−8	163	152
		−7	140	136
		−6	131	120
		−5	122	115
		−4	187	163
		−3	203	200
		−2	275	310
		−1	87	87
		0	65	61
		2	281	287
		3	262	264
		4	178	165
		5	135	135
		6	126	102
		7	128	107
		8	185	183
		9	77	91
		10	93	98
3	5	−10	114	109
		−9	88	88
		−8	167	158
		−7	183	161
		−6	180	159
		−5	60	56
		−4	43	44
		−3	78	73
		−2	219	217
		−1	266	283
		0	208	193
		1	203	178
		2	183	169
		3	188	165
		4	124	113
		5	63	67
		6	64	53
		7	115	95
		8	116	107
		9	157	146
3	6	−10	132	126
		−8	46	50
		−7	129	127
		−6	227	203
		−5	146	144
		−4	133	117
		−3	144	137
		−2	149	125
		−1	179	162
		0	162	166
		1	83	79
		3	49	41
		4	140	135
		5	181	200
		6	197	197
		7	116	116
		8	86	87
3	7	−9	111	111
		−8	53	67
		−7	141	128
		−6	137	131
		−5	128	120
		−4	151	139
		−3	63	55
		−2	69	−34
		−1	137	122
		0	169	165
		1	215	220
		3	120	118
		4	134	144
		5	130	142
		6	101	91
		7	73	79
3	8	−9	110	116
		−8	69	66
		−5	105	110
		−4	171	163
		−3	149	137
		−2	139	130
		−1	120	11
		0	138	136
		1	39	40
		3	4	129
		4	89	94
		5	116	123
3	9	−7	72	79
		−6	59	73
		−5	85	89
		−4	102	103
		−3	121	121
		−2	106	103
		−1	82	81
		0	42	38
		1	82	85
		2	142	157
		3	147	156
3	10	−5	61	65
		−4	79	75
		−3	103	102
		−2	132	146
		−1	133	129
		0	100	107
4	−7	1	6	72
		2	83	81
		3	78	105
4	−6	−1	93	96
		−1	93	96
		0	77	77
		0	77	77
		3	84	82
		4	138	129
		5	149	160
		6	93	97
		7	40	54
		9	93	97
4	−5	−1	73	76

TABLE 1 (*Continued*)

h	*k*	*l*	5|F_o|	5F_c
		0	131	118
		1	158	146
		2	120	102
		3	91	75
		4	126	105
		5	125	113
		6	102	108
		7	77	79
		9	52	60
		10	108	111
4	−4	−5	133	136
		−4	128	112
		−3	132	134
		−2	114	109
		−1	53	36
		0	129	114
		1	126	133
		2	123	106
		5	189	174
		6	181	178
		7	201	196
		8	117	109
		9	70	71
		10	45	53
		11	107	107
4	−3	−6	129	109
		−5	133	129
		−4	134	140
		−3	96	102
		−2	81	74
		−1	38	38
		0	119	108
		1	163	154
		2	245	254
		3	154	117
		4	203	202
		5	73	75
		6	70	73
		7	89	63
		8	123	108
		9	135	121
		10	51	65
		11	88	93
		12	52	115
4	−2	−7	110	94
		−6	101	93
		−5	84	84
		−4	71	80
		−3	163	157
		−2	229	232
		−1	211	194
		0	145	125
		1	114	103
		2	150	135
		3	151	139
		4	118	114
		5	78	66
		6	56	59
		7	64	47
		8	181	173
		9	200	179
		10	137	139
		11	67	68
4	−1	−8	124	125
		−7	195	200
		−6	143	143
		−5	76	79
		−4	151	147
		−3	116	105
		−2	132	118
		−1	87	76
		0	136	123
		1	117	107
		2	141	145
		3	148	147
		4	212	224
		5	212	213
		6	100	99
		9	161	135
		10	137	130
		11	99	98
		12	60	74
4	0	−8	140	135
		−7	164	171
		−5	152	145
		−4	101	98
		−3	65	59
		−2	41	−4
		−1	148	140
		0	238	334
		0	239	334
		1	197	214
		2	82	91
		3	102	106
		4	30	57
		5	149	146
		6	150	151
		7	203	189
		8	130	118
		9	114	101
		10	135	124
		11	16	140
		12	117	131
4	1	−9	119	112
		−8	107	102
		−7	74	80
		−6	101	101
		−5	205	182
		−4	214	204
		−3	126	134
		−2	57	−9
		−1	81	76
		0	105	122
		3	117	142
		4	92	94
		5	184	184
		6	274	277
		7	330	359
		8	38	41
		9	38	54
		11	159	148
		12	83	97
4	2	−9	151	143
		−8	135	145
		−7	122	125
		−6	97	84
		−5	131	100
		−4	149	137
		−3	152	157
		−2	49	163
		−1	69	86
		0	101	151
		3	141	175
		4	94	91
		5	41	49
		6	211	194
		7	209	160
		8	175	140
		9	149	122
		10	125	120
		11	145	127
4	3	−10	51	60
		−9	118	105
		−8	100	102
		−7	120	101
		−6	168	160
		−5	71	69
		−4	154	156
		−3	234	254
		−2	289	396
		−1	171	204
		0	33	16
		1	65	−18
		2	88	83
		3	138	133
		4	151	146
		5	169	154
		6	136	111
		7	174	147
		8	178	155
		9	152	148
		10	128	116
		11	43	60
4	4	−10	67	70
		−9	60	67
		−8	83	86
		−7	208	201
		−6	194	184
		−5	76	73
		−4	57	58
		−3	134	137
		−2	303	351
		−1	49	72
		0	140	141
		1	93	81
		2	179	175
		3	133	120
		4	157	166
		5	139	137
		6	37	40
		7	27	4
		8	115	103
		9	136	143
		10	164	145
4	5	−10	94	97
		−9	71	71

TABLE 1 (*Continued*)

h	*k*	*l*	5|F_o|	5F_c
		−8	42	56
		−7	88	80
		−6	85	80
		−5	125	120
		−4	115	101
		−3	204	182
		−2	152	139
		−1	176	178
		0	198	207
		1	176	154
		2	115	90
		3	53	11
		4	118	116
		5	150	145
		6	81	60
		7	140	131
		8	147	137
		9	137	135
4	6	−9	97	99
		−8	47	55
		−7	26	48
		−6	89	94
		−5	190	170
		−4	190	155
		−3	25	44
		−1	76	74
		0	151	139
		1	210	190
		2	199	181
		3	214	201
		4	147	143
		5	120	102
		6	103	102
		7	125	127
		8	71	70
4	7	−9	132	127
		−8	81	84
		−7	87	81
		−6	37	45
		−5	71	71
		−4	104	98
		−3	110	97
		−2	103	79
		−1	158	146
		0	147	139
		1	179	169
		2	198	175
		3	170	159
		4	69	65
		5	60	72
		6	101	97
		7	130	128
4	8	−8	125	117
		−7	174	147
		−6	105	100
		−5	91	90
		−4	123	123
		−3	84	69
		−2	139	124
		−1	85	85
		0	65	76
		1	46	45
		2	81	81
		3	179	198
		4	120	137
		5	140	133
		6	86	86
4	9	−7	65	148
		−6	146	134
		−5	60	69
		−3	41	51
		−2	125	112
		−1	124	124
		0	128	129
		2	114	114
		3	95	91
		4	93	94
4	10	−5	99	101
		−4	100	101
		−3	88	83
		−2	82	93
		−1	75	84
		0	62	63
		1	53	57
5	−6	1	91	100
		2	104	110
		3	133	149
		4	100	87
		5	101	96
		6	108	107
		7	47	59
5	−5	−1	102	91
		0	129	115
		1	130	113
		2	150	140
		3	93	95
		4	34	45
		5	34	48
		6	128	103
		7	136	134
		8	137	128
		9	63	71
5	−4	−4	6	85
		−3	80	96
		1	80	77
		2	125	112
		3	147	132
		4	145	121
		5	102	93
		6	130	111
		7	120	113
		8	112	117
		9	73	74
5	−3	−5	58	65
		−4	54	57
		−3	118	113
		−2	105	97
		−1	106	92
		0	162	142
		1	92	79
		2	78	55
		3	27	12
		4	167	150
		5	114	106
		6	28	50
		7	28	46
		8	135	134
		9	168	176
		10	144	139
		11	71	81
5	−2	−6	114	114
		−5	139	132
		−4	121	114
		−3	124	108
		−2	107	96
		−1	162	161
		0	119	114
		2	136	−96
		4	216	224
		5	166	172
		6	153	145
		7	62	59
		8	132	137
		9	120	108
		10	133	131
		11	157	128
5	−1	−7	60	63
		−6	112	95
		−5	119	118
		−4	91	84
		−3	83	80
		−2	89	89
		−1	160	155
		0	175	169
		1	164	157
		2	114	95
		3	113	106
		4	82	75
		5	136	129
		6	112	99
		7	105	111
		8	73	72
		9	81	76
		10	151	145
		11	206	193
		12	30	115
5	0	−6	61	−24
		−5	144	131
		−4	175	178
		−3	174	193
		−2	139	137
		−1	129	130
		0	130	133
		0	130	133
		1	168	192
		2	162	189
		3	87	87
		4	34	37
		5	74	77
		6	155	172
		7	172	165
		8	117	105
		9	102	82
		10	61	70
		11	118	94
		12	83	108

TABLE 1 (*Continued*)

h	*k*	*l*	5|F_o|	5F_c
5	1	−8	133	133
		−7	47	46
		−6	27	35
		−5	154	145
		−4	137	135
		−3	166	172
		−2	49	74
		−1	98	104
		0	77	90
		0	78	90
		1	144	186
		2	188	322
		3	165	238
		4	87	94
		5	74	76
		6	68	70
		7	99	91
		8	39	50
		9	115	97
		10	78	69
		11	109	98
5	2	−8	136	129
		−7	107	103
		−6	91	89
		−5	48	47
		−4	122	112
		−3	116	121
		−2	145	161
		−1	126	156
		0	83	125
		3	64	89
		4	146	164
		5	92	80
		6	172	156
		7	46	44
		8	100	98
		9	202	165
		10	137	124
		11	95	92
5	3	−9	74	73
		−8	130	123
		−7	157	154
		−6	161	150
		−5	144	127
		−4	80	87
		−3	51	49
		−1	83	100
		0	110	155
		1	57	76
		2	63	67
		3	136	149
		4	185	243
		5	204	213
		6	169	150
		8	126	109
		9	106	89
		10	144	143
		11	76	85
5	4	−9	108	107
		−8	72	72
		−7	132	127
		−6	152	141
		−5	119	107
		−4	63	65
		−2	96	101
		−1	118	127
		0	154	196
		1	130	133
		2	117	129
		3	82	89
		4	113	117
		5	98	96
		6	161	142
		7	186	174
		8	141	127
		9	140	120
		10	119	118
5	5	−9	96	91
		−8	75	69
		−7	116	121
		−6	91	83
		−5	158	148
		−4	106	102
		−3	213	227
		−2	106	98
		0	108	81
		1	152	143
		2	189	178
		3	80	82
		4	86	85
		5	125	119
		6	175	171
		7	190	182
		8	123	127
		9	61	69
5	6	−9	116	125
		−8	151	133
		−7	109	106
		−6	66	68
		−5	62	67
		−4	122	119
		−3	202	196
		−2	112	103
		−1	97	99
		0	90	83
		1	210	205
		2	170	160
		3	11	103
		4	78	83
		5	39	43
		6	59	59
		7	138	135
		8	109	105
		9	78	117
5	7	−8	116	100
		−7	140	114
		−6	77	77
		−5	77	81
		−4	118	101
		−3	175	158
		−2	180	180
		−1	128	123
		0	91	88
		1	48	55
		2	68	65
		3	92	92
		4	108	103
		5	96	95
		6	116	113
		7	102	98
5	8	−7	145	127
		−6	100	108
		−5	87	87
		−4	59	69
		−3	47	42
		−2	110	98
		−1	140	131
		0	116	107
		1	67	64
		2	75	83
		3	105	118
		4	118	137
		5	117	116
		6	61	68
5	9	−6	78	76
		−5	127	114
		−4	75	72
		−3	41	40
		−2	117	118
		−1	120	124
		0	121	126
		2	88	90
		3	83	91
		4	90	100
5	10	−4	86	84
		−3	85	84
		−2	65	63
		−1	72	77
		1	70	72
6	−5	1	34	31
		2	75	73
		3	100	88
		4	61	51
		5	67	59
		6	48	42
		7	39	56
6	−4	−1	93	87
		0	84	65
		1	76	52
		2	117	98
		3	127	116
		4	128	124
		5	68	66
		8	89	79
		9	84	91
6	−3	−3	64	57
		−2	91	82
		−1	113	104
		0	121	113
		1	87	79
		2	51	52
		3	86	71
		4	106	95
		5	143	129
		6	140	128
		7	78	86

TABLE 1 (*Continued*)

h	*k*	*l*	5|F_o|	5F_c
		8	49	56
		9	93	76
		10	125	108
6	−2	−4	106	96
		−3	62	63
		−1	79	80
		0	121	125
		1	156	149
		2	113	89
		3	108	91
		4	151	143
		5	136	128
		6	144	131
		7	86	73
		8	44	54
		10	99	90
		11	86	98
6	−1	−5	100	95
		−4	100	98
		−3	112	94
		−2	93	84
		−1	123	118
		0	126	123
		1	176	155
		2	70	63
		3	138	143
		5	44	56
		6	79	57
		7	155	138
		8	163	152
		9	99	94
		10	75	72
		11	67	68
6	0	−6	106	99
		−5	95	86
		−4	119	109
		−3	146	136
		−2	112	112
		−1	72	70
		0	49	55
		1	75	69
		2	112	19
		3	141	149
		4	129	127
		5	82	76
		6	31	37
		7	26	117
		8	149	131
		9	122	113
		10	73	72
		11	66	45
6	1	−7	94	83
		−6	64	67
		−5	102	90
		−4	62	59
		−3	86	73
		−2	107	99
		−1	127	154
		0	111	126
		1	88	103
		2	63	80
		3	96	122
		4	113	128
		5	75	77
		6	43	59
		7	61	67
		8	153	135
		9	145	135
		10	126	121
		11	96	89
6	2	−7	35	20
		−7	35	20
		−6	139	139
		−5	120	115
		−4	93	86
		−2	105	105
		−1	143	172
		0	83	114
		3	33	60
		4	111	140
		5	176	178
		6	197	206
		7	113	98
		8	52	57
		9	73	66
		10	134	110
		11	102	113
6	3	−8	60	70
		−7	76	74
		−6	121	106
		−5	136	120
		−4	95	98
		−2	76	77
		−1	85	102
		0	87	141
		2	97	137
		3	55	68
		4	79	90
		5	163	174
		6	217	213
		7	23	28
		8	65	66
		9	70	58
		10	160	139
		11	26	89
6	4	−8	89	85
		−7	59	62
		−6	63	64
		−5	117	117
		−4	160	158
		−3	150	164
		−2	82	96
		−1	72	90
		0	70	99
		1	116	131
		2	97	96
		3	88	85
		4	76	82
		5	147	163
		6	138	114
		7	142	126
		8	127	114
		9	112	102
		10	69	74
6	5	−8	122	103
		−7	135	129
		−6	54	63
		−5	99	94
		−4	137	126
		−3	178	190
		−2	97	101
		0	26	38
		1	91	92
		2	125	128
		3	160	156
		3	146	156
		4	137	133
		4	133	133
		5	56	61
		5	56	61
		6	66	66
		6	66	66
		7	100	93
		7	95	93
		8	119	115
		8	117	115
		9	121	107
6	6	−8	128	116
		−6	57	65
		−5	46	51
		−4	99	102
		−3	198	187
		−2	77	88
		−1	132	130
		0	74	77
		1	91	91
		2	44	58
		3	94	103
		4	99	96
		5	57	60
		6	55	62
		7	124	123
		8	142	128
6	7	−7	80	74
		−6	118	109
		−5	93	89
		−4	119	111
		−3	114	58
		−2	81	82
		−1	116	104
		0	133	112
		1	104	100
		2	62	71
		3	116	116
		4	114	116
		5	68	69
		6	117	119
		7	90	92
6	8	−6	103	89
		−5	99	88
		−4	48	58
		−3	55	58
		−2	82	84
		−1	122	111
		0	154	148
		1	139	138

TABLE 1 (*Continued*)

h	*k*	*l*	5|F_o|	5F_c	*h*	*k*	*l*	5|F_o|	5F_c	*h*	*k*	*l*	5|F_o|	5F_c
		2	118	126			−3	86	79			3	77	74
		3	52	63			−2	85	91			4	32	38
		5	62	76			−1	57	72	6	10	−2	97	81
		6	98	106			0	81	87			−1	86	88
6	9	−5	91	84			1	112	103			0	33	53
		−4	90	87			2	128	113			1	49	75
												2	6	124

TABLE 2

Atomic co-ordinates (× 10^4) and standard deviations (× 10^4)

	x/a	σ(*x/a*)	*y/b*	σ(*y/b*)	*z/c*	σ(*z/c*)
Pd	0	0	0	0	0	0
C(1)	7662	25	3669	19	2407	14
C(2)	7186	31	4792	21	3859	18
C(3)	8340	30	4219	22	4687	15
C(4)	9948	27	2364	20	4178	14
C(5)	1091	29	1634	23	4945	15
C(6)	2568	33	9817	27	4408	17
C(7)	2920	28	8722	21	2939	16
C(8)	1767	23	9408	17	2101	13
C(9)	284	23	1318	16	2735	14
N(1)	9112	26	1931	19	1895	15
O(1)	2052	26	8532	21	743	16
C(10)	3811	26	1964	19	462	14
C(11)	6209	20	8994	15	767	12
C(12)	4896	19	1132	14	1366	10
O(2)	4869	25	2007	19	2522	13
Cl(1)	2527	10	4357	6	1062	5
Cl(2)	7501	8	8058	7	1820	5

TABLE 3

Thermal parameters (× 10^4). The temperature factor, T, is equal to $2^{-(b_{11}h^2 + b_{22}k^2 + b_{33}l^2 + b_{12}hk + b_{13}hl + b_{23}kl)}$

	b_{11}	b_{22}	b_{33}	b_{23}	b_{13}	b_{12}
Pd	95	23	14	−20	4	−28
C(1)	180	73	16	−56	54	27
C(2)	340	35	78	−57	−8	−31
C(3)	332	114	17	−24	−59	−73
C(4)	270	93	6	−20	−4	−120
C(5)	291	157	20	39	−33	−173
C(6)	339	205	43	1	−36	−187
C(7)	269	74	37	24	32	−115
C(8)	142	21	25	−21	66	81
C(9)	181	8	29	−59	11	31
N(1)	165	25	40	−20	−62	−3
O(1)	274	45	19	−44	−33	40
C(10)	192	48	45	−34	21	64
C(11)	179	106	44	71	33	−6
C(12)	252	50	17	−89	68	−10
O(2)	454	184	46	−17	−139	−229
Cl(1)	503	51	105	−3	−108	−31
Cl(2)	295	203	89	19	−140	−139

Interatomic distances, together with standard deviations derived from the formula of Cruickshank and Ahmed,[3] are listed in Table 5. The inter-bond angles are given in Figure 3.

The crystal is made up of planar bis-8-hydroxyquinolinatopalladium(II) molecules and planar chloranil molecules arranged in planes approximately parallel to the crystallographic (111) plane. Each such plane contains equal numbers of metal-complex and chloranil

[3] D. W. J. Cruickshank and F. R. Ahmed, *Acta Cryst.*, 1953, **6**, 385.

TABLE 4

Atomic co-ordinates (Å) referred to a set of orthogonal axes related to the least-squares best plane through the bis-8-hydroxyquinolinatopalladium(II) molecule

	X'	*Y'*	*Z'*		*X'*	*Y'*	*Z'*
Pd	0·0000	0·0000	0·0000	N(1)	1·715	−0·971	−0·054
C(1)	2·048	−2·240	0·042	O(1)	1·080	1·657	0·082
C(2)	3·392	−2·698	−0·045	C(10)	2·130	0·677	3·450
C(3)	4·413	−1·810	0·020	C(11)	0·843	0·899	3·256
C(4)	4·169	−0·358	0·046	C(12)	3·143	1·674	3·282
C(5)	5·086	0·569	−0·003	O(1)	2·691	−0·865	3·964
C(6)	4·763	1·927	−0·054	Cl(1)	−0·354	−0·259	3·414
C(7)	3·366	2·319	−0·021	Cl(2)	4·332	1·460	3·419
C(8)	2·358	1·376	−0·010				
C(9)	2·784	−0·010	0·062				

TABLE 5

Interatomic distances (in Å) with standard deviations

(*a*) *Bis-8-hydroxyquinolinatopalladium*(II)

Pd–N(1)	1·97(0·02)	C(4)–C(9)	1·42(0·02)
Pd–O(1)	1·98(0·02)	C(5)–C(6)	1·39(0·05)
C(1)–C(2)	1·42(0·03)	C(6)–C(7)	1·45(0·03)
C(1)–N(1)	1·31(0·04)	C(7)–C(8)	1·38(0·03)
C(2)–C(3)	1·35(0·03)	C(8)–C(9)	1·45(0·03)
C(3)–C(4)	1·46(0·04)	C(8)–O(1)	1·31(0·02)
C(4)–C(5)	1·33(0·03)	C(9)–N(1)	1·44(0·03)

(*b*) *Chloranil*

C(10)–C(11)	1·32(0·02)	C(11)–C(12)	1·55(0·03)
C(10)–C(12)	1·43(0·02)	C(11)–Cl(2)	1·67(0·02)
C(10)–Cl(1)	1·72(0·03)	C(12)–O(2)	1·22(0·01)

(*c*) *Between bis-8-hydroxyquinolinatopalladium*(II) *and chloranil molecules*

Pd–Cl(2)	3·44(0·01)	C(8)–C(11)	3·44
C(1)–O(2)	3·09	C(8)–C(12)	3·40
C(3)–O(2)	3·37	C(9)–C(10)	3·52
C(7)–C(11)	3·12		

(*d*)

C(4)–C(6) *	3·45

* C(6) is in a second bis-8-hydroxyquinolinatopalladium(II) molecule plane-to-plane with, and slightly overlapping, the first containing the atom C(4).

molecules. The planes are arranged with respect to each other in such a way that the donor and acceptor molecules are arranged in stacks characteristic of the sandwich arrangement of a π-complex. The stacks of molecules are staggered along the crystallographic *a* axis [Figure 1(a and b)].

The bis-8-hydroxyquinolinatopalladium(II) molecule has the expected *trans*-configuration. The palladium–oxygen (1·98 Å) and palladium–nitrogen distances (1·97 Å) are equal within experimental error. Also, none of the small anomalies in interatomic distance and angle in the bis-8-hydroxyquinolinatopalladium(II) molecule, or in the chloranil molecule, is outside the estimated experimental error.

Figure 2 shows the chloranil molecule projected parallel to, and perpendicular to the least-squares best plane of the metal complex. Since the palladium atom is at the centre of symmetry, it is clear that the next nearest neighbours of this atom, after the oxygen and nitrogen atoms of the ligands, are two chlorine atoms of two separate chloranil molecules at a distance of 3·44 Å. These chlorine atoms complete an elongated octahedron about the palladium atom. The palladium–chlorine distance of 3·44 Å is shorter than the non-bonded palladium–chlorine distance of 3·85 Å found in palladium chloride ($PdCl_2$),[4] but is very much longer than can be expected from any normal bonding interaction. The π-system of the chloranil molecule lies far from the ideal position of maximum overlap with the π-system of the ligand predicted by the " overlap and orientation

[4] A. F. Wells," Structural Inorganic Chemistry," 3rd edn., Oxford, 1961, p. 876.

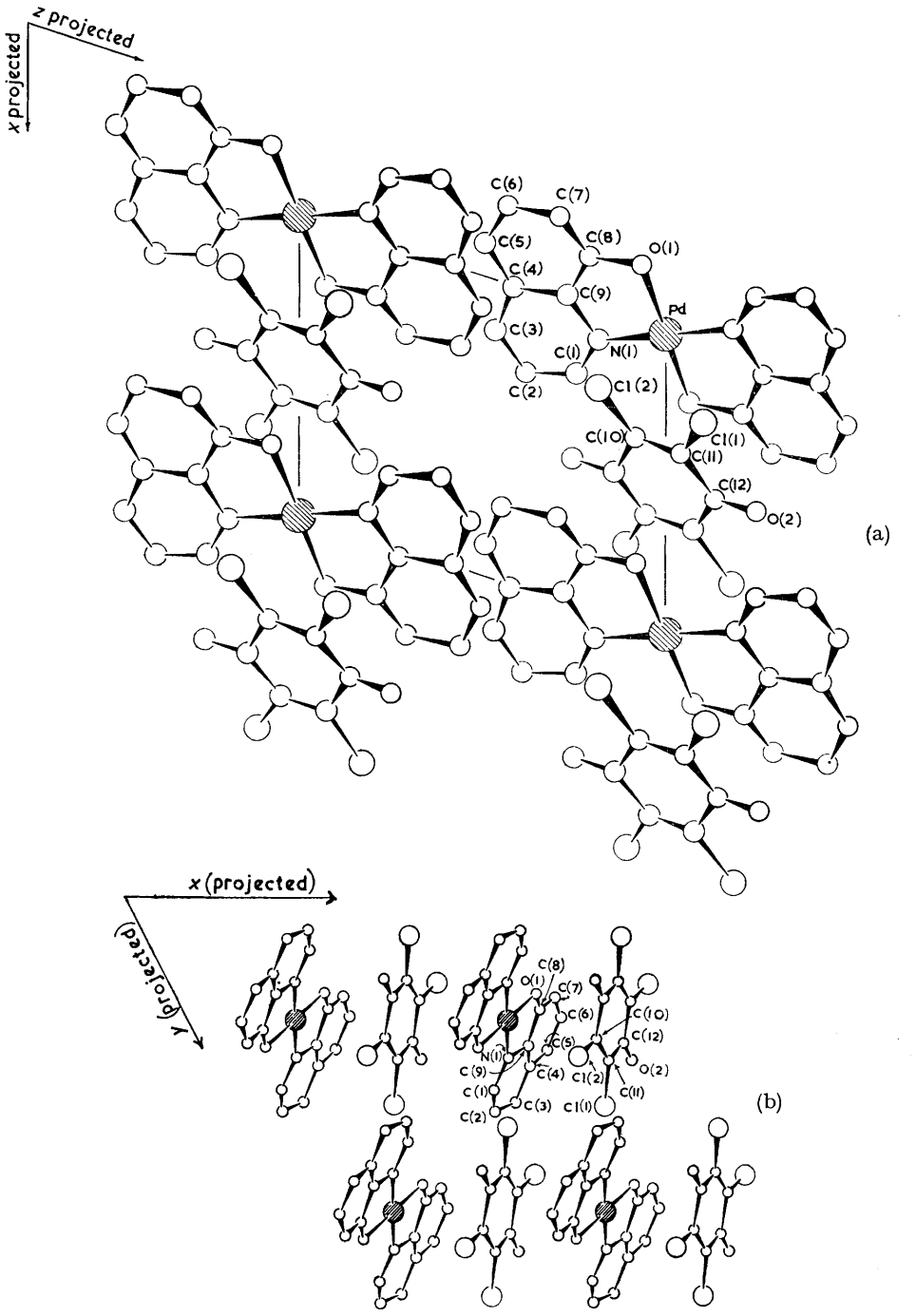

FIGURE 1. The molecular arrangement in the crystal of the bis-8-hydroxyquinolinato-palladium(II)–chloranil complex

(a) projected down the *b* axis; (b) projected down the *c* axis.

The palladium atoms are at the cell origin.

principle."[5] Further, the best plane of the chloranil molecule makes an angle of $15\frac{1}{2}°$ with the best plane of the metal complex molecule.

Since the peripheral atoms of the chloranil might be expected to carry partial negative charges, it is not surprising that one or other of them should be attracted to the palladium atom, which will tend to be positively charged. The Van der Waals interaction between

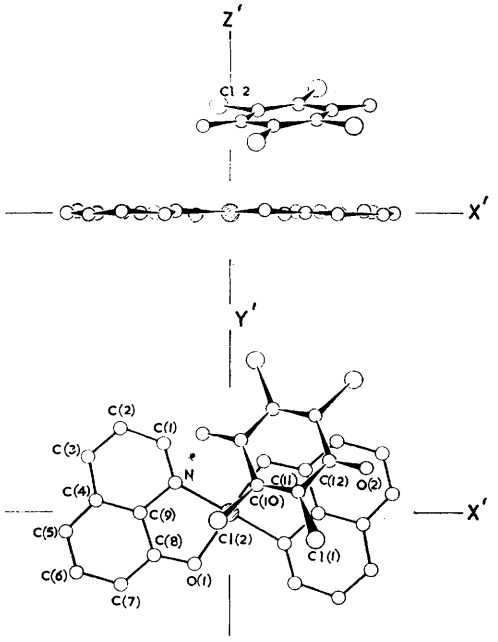

FIGURE 2. A chloranil molecule in the bis-8-hydroxyquinolinatopalladium(II) chloranil complex projected parallel to, and perpendicular to, the least-squares best plane of the bis-8-hydroxyquinolinatopalladium(II) molecule

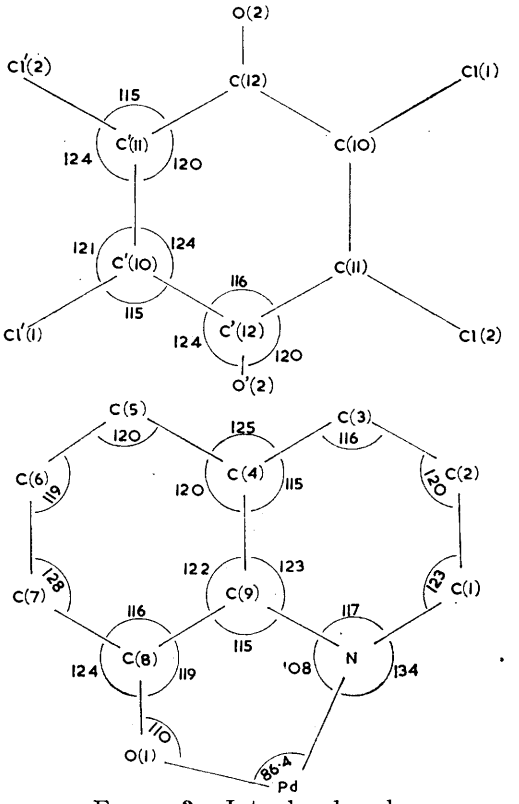

FIGURE 3. Inter-bond angles

chlorine and palladium is expected to be considerably greater than that between oxygen and palladium, because of the greater polarisability of the chlorine atom. It is therefore not surprising that the chlorine atoms should be associated with the palladium atom. Further, the octahedral position might be expected on the grounds that this minimises the repulsion between the chlorine atom and the ligand atoms. Given that a centrically related pair of chlorine atoms from a single chloranil molecule is attracted to two palladium atoms, and that plane-to-plane stacks are formed, the molecular packing is determined by a compromise between maximum π-overlap of donor and acceptor orbitals and a minimisation of the free space in the crystal. In practice, this leads to a tilting of the chloranil with respect to the 8-hydroxyquinolinato plane, because of repulsion between the peripheral atoms of the chloranil molecules and those of bis-8-hydroxyquinolinates in neighbouring stacks. The direction of the tilt is determined by the repulsion between the chloranil molecules and the bis-8-hydroxyquinolinates of its own stack.

EXPERIMENTAL

Preparation.—The complex was prepared by the method described by Bailey, Williams, and Wright.[1]

X-*Ray photography.*—The unit-cell dimensions were obtained from calibrated zero-layer

[5] R. S. Mulliken, *Rec. Trav. chim.*, 1956, **75**, 845.

Weissenberg photographs about [100], [010], and [121] axes. The X-ray intensities of 1880 independent reflections were estimated visually from sets of multiple-film Weissenberg photographs about the *a* axis. The results from individual layers were placed on a common scale by using the exposure time to X-rays from a stabilised source and carefully standardised development conditions. The intensities were corrected for Lorentz and polarisation effects but not for absorption or extinction.

Calculations.—These were carried out on a Ferranti Mercury computer. Structure factors and cycles of least-squares were computed by using Rollett's SFLS programme.[6] Atomic scattering factors for nitrogen, oxygen, carbon, and chlorine were as given by Berghius *et al.*,[7] and for palladium by Thomas and Umeda [8] with a correction for anomalous dispersion.[9] For Fourier syntheses, Mills's general Fourier programme OSM 4 [6] was used.

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[6] O. S. Mills and J. S. Rollett, "Computing Methods and the Phase Problem in X-ray Crystal Analysis," Pergamon Press, London, 1961, p. 107.
[7] J. Berghius, I. M. Haanapel, M. Potters, B. D. Loopstra, C. H. MacGillavry, and A. L. Veenendal, *Acta Cryst.*, 1955, **8**, 478.
[8] L. H. Thomas and K. Umeda, *J. Chem. Phys.*, 1957, **26**, 293.
[9] C. H. Dauben and D. H. Templeton, *Acta Cryst.*, 1955, **8**, 841.