under the *trans* effect of the amine group in [Pt(dien)-X]$^+$, and it would be desirable to see to what extent this behavior is changed when a strong *trans*-effect ligand is present.

Acknowledgment.—This work was supported by the Italian Consiglio Nazionale delle Ricerche (CNR, Rome). We wish to thank professor M. L. Tobe for reading the final manuscript.

CONTRIBUTION FROM THE DEPARTMENTS OF CHEMISTRY, UNIVERSITY OF SOUTH CAROLINA, COLUMBIA, SOUTH CAROLINA 29208, AND NORTHWESTERN UNIVERSITY, EVANSTON, ILLINOIS 60201

Crystal Structure of *cis*-Pt[P(CH_3)$_3$]$_2$$Cl_2$

BY G. G. MESSMER,[1] E. L. AMMA,[2] AND JAMES A. IBERS

Received October 3, 1966

The crystal structure of *cis*-Pt[P(CH_3)$_3$]$_2$$Cl_2$ has been determined from counter data by three-dimensional single-crystal X-ray diffraction techniques. Unit cell constants are: $a = 16.67 \pm 0.02$ A, $b = 11.93 \pm 0.02$ A, $c = 6.33 \pm 0.01$ A, $\beta = 91° 25' \pm 15'$. The space group is B2$_1$. The structure is made up of discrete molecular units with ordinary van der Waals separations. The asymmetric unit is one molecule with Pt–P distances of 2.256 and 2.239 A (both ±0.008 A) and Pt–Cl distances of 2.364 and 2.388 A (both ±0.008 A). The molecule is twisted toward a tetrahedral configuration with nearest neighbors of platinum displaced about 0.1 A out of the best least-squares plane defined by the platinum and its four nearest neighbors. These bond lengths are to be compared with Pt–P distances of 2.298 ± 0.018 and 2.315 ± 0.004 A and Pt–X distances of 2.294 ± 0.009 and 2.428 ± 0.002 A observed in *trans*-Pt[P(C_2H_5)$_3$]$_2$$Cl_2$ and *trans*-Pt[P(C_2H_5)$_3$]$_2$$Br_2$, respectively.

Introduction

We have completed crystal structure determinations of *trans*-Pt[P(C_2H_5)$_3$]$_2$$X_2$, X = Cl and Br, and found substantially shorter Pt–P bond lengths than would be predicted from a sum of covalent radii.[3,4] However, the Pt–X bonds were found to be normal single bonds, equal to the sum of the single-bond covalent radii. Hence, Pt–X (X = Cl, Br) and Pt–P distances have been carefully determined in the X–Pt–X and P–Pt–P atomic arrangements. To substantiate the thermodynamic results of Chatt and Wilkens[5] in terms of bond lengths, we undertook to solve the crystal structure of *cis*-Pt[P(CH_3)$_3$]$_2$$Cl_2$.

Experimental Section

Crystals of *cis*-Pt[P(C_2H_5)$_3$]$_2$$Cl_2$ and *cis*-Pt[P(CH_3)$_3$]$_2$$Cl_2$ were kindly provided to us by Professor J. Chatt. *cis*-Pt[P(C_2H_5)$_3$]$_2$-Cl_2 was found to have a large triclinic cell, and this structure determination was abandoned. Crystals of *cis*-Pt[P(CH_3)$_3$]$_2$-Cl_2 were found to be monoclinic with unit cell constants from calibrated precession photographs: $a = 16.67 \pm 0.02$ A, $b = 11.93 \pm 0.02$ A, $c = 6.33 \pm 0.01$ A, and $\beta = 91° 25' \pm 15'$ (Mo Kα, λ 0.7107 A). The systematic extinctions observed were: for hkl, $h + l = 2n + 1$; for $0k0$, $k = 2n + 1$. The possible space groups with these extinctions are B2$_1$ or B2$_1$/m. With a reorientation of the a and c axes, these would correspond to the more common P2$_1$ or P2$_1$/m.[6] The structure analysis established the correct space group as B2$_1$ or P2$_1$ (*vide infra*). We retained the use of the B-centered cell for the structure analysis because the crystals were most easily aligned about the c axis of the B-centered cell. All our intensity data were collected with this axis vertical on the diffractometer, and this orientation facilitated comparison with our previous photographic structure determination.[7] Further, the β angle for the B-centered cell is approximately 90°. With four molecular entities in the B-centered cell, the calculated density was found to be 2.16 g cm^{-3} compared with the observed of 2.18 g cm^{-3} obtained by flotation in carbon tetrachloride–bromoform mixtures. (This corresponds to two molecules in the primitive cell.)

A single crystal, 0.20 × 0.15 × 0.15 mm, mounted on a GE single-crystal orienter with a Picker diffractometer using Zr-filtered Mo Kα radiation was used to measure 2433 independent hkl intensity data by a scanning technique at room temperature. Background was estimated by stationary counting at ±0.8° 2θ from the peak maxima for 10 sec. The peak was then scanned for 40 sec with a 2θ scan. The integrated intensity was obtained by subtracting the background scaled to 20 sec from the 2θ scan. The value of the linear absorption coefficient (μ) for Mo Kα radiation is 132 cm^{-1}. No corrections were made for absorption and, consequently, no detailed physical interpretation should be made of the anisotropic temperature factors. Recently Srivastava and Lingafelter[8] have shown that absorption effects with μ = 191 cm^{-1} do not appreciably affect atomic coordinates; hence, we feel our estimates of error in atomic coordinates are realistic. Corrections were made for anomalous dispersion (see below).

Structure Determination

In space group B2$_1$ with four molecules per unit cell, all atoms would be in the general positions: $(0, 0, 0; \frac{1}{2}, 0, \frac{1}{2}) + (x, y, z; \bar{x}, \frac{1}{2} + y, \bar{z})$. In space group B2$_1$/m: (a) the Pt, 2P, 2Cl, and one of the three carbon atoms on each phosphorus could lie in the mirror plane at $y = \frac{1}{4}$: $(0, 0, 0; \frac{1}{2}, 0, \frac{1}{2}) + (x, \frac{1}{4}, z; \bar{x}, \frac{3}{4}, \bar{z})$ and the remaining carbon atoms in the general positions $(0, 0, 0; \frac{1}{2}, 0, \frac{1}{2}) + (x, y, z; \bar{x}, \bar{y}, \bar{z}; x, \frac{1}{2} - y, z; \bar{x}, \frac{1}{2} + y, \bar{z})$; (b) the Pt could lie in the mirror

(1) In partial fulfillment for the Ph.D. requirements, University of Pittsburgh.

(2) Address all correspondence to this author: Department of Chemistry, University of South Carolina, Columbia, S. C. 29208

(3) G. G. Messmer and E. L. Amma, *Inorg. Chem.*, **5**, 1775 (1966).

(4) For a more complete introduction and references, see preceding paper.[3]

(5) J. Chatt and R. G. Wilkens, *J. Chem. Soc.*, 273, 4300 (1952); 70 (1953); 525 (1956).

(6) "International Tables for X-ray Crystallography," Vol. I, The Kynoch Press, Birmingham, England, 1952, pp 79, 93.

(7) P. D. Carfagna and E. L. Amma, unpublished results.

(8) R. C. Srivastava and E. C. Lingafelter, *Acta Cryst.*, **20**, 918 (1966).

TABLE I

OBSERVED AND CALCULATED STRUCTURE FACTORS[a]

* H 0 0 *		
2	177	188
4	159	169
6	369	369
8	39	25
10	153	152
12	71	68
14	62	69
16	193	189
18	52	47
20	86	82
22	92	84
24	35	16
26	48	45
* H 1 0 *		
2	327	438
4	224	244
6	145	156
8	326	354
10	87	87
12	98	94
14	171	175
16	32	27
18	117	116
20	58	59
22	29	27
24	88	82
26	46	19
* H 2 0 *		
0	459	487
2	106	112
4	122	145
6	271	325
8	75	86
10	241	265
12	112	115
14	51	60
16	187	191
18	52	53
20	63	56
22	66	69
24	19	15
26	38	42
* H 3 0 *		
2	253	247
4	183	192
6	141	159
8	362	419
10	107	113
12	136	150
14	184	199
16	42	21
18	91	92
20	20	46
22	20	16
24	64	67
28	18	20
* H 4 0 *		
0	376	346
2	187	171
4	206	209
6	252	286
8	57	54
10	262	294
12	124	130
14	56	59
16	174	177
18	40	43
20	42	42
22	65	62
24	28	7
26	47	40
* H 5 0 *		
2	311	300
4	154	157
6	99	107
8	260	292
10	60	69
12	102	108
14	159	168
16	31	34
18	99	102
20	56	54
22	16	20
24	67	66
* H 6 0 *		
0	473	438
2	151	143
4	186	190
6	229	239
8	38	32
10	136	154
12	79	84
14	58	54
16	143	145
18	34	26
20	63	62
22	76	74
26	52	39
* H 7 0 *		
2	351	336
4	199	200
6	64	69
8	187	210
10	25	29
12	73	77
14	117	123
16	21	5
18	112	107
20	62	56
24	69	59
26	36	14
* H 8 0 *		
0	304	279
2	55	50
4	142	139
6	216	220
8	47	57
10	152	166
12	90	95
14	23	36
16	119	118
18	50	23
20	41	47
22	60	60
24	14	14
26	33	33
* H 9 0 *		
2	181	173
4	131	131
6	68	70
8	199	212
10	41	38
12	98	101
14	124	127
16	20	12
18	77	77
20	42	42
22	29	17
24	40	41
26	45	11
28	24	19
* H 10 0 *		
0	165	154
2	32	23
4	113	112
6	176	181
8	29	18
10	162	168
12	105	104
16	92	92
18	13	22
20	28	32
22	46	43
26	41	30
* H 11 0 *		
2	164	164
4	97	98
6	34	33
8	151	158
12	74	81
14	96	99
16	12	27
18	72	68
20	32	36
* H 12 0 *		
0	167	160
2	32	39
4	99	101
6	121	125
8	13	8
10	100	98
12	63	68
14	26	27
16	63	72
20	50	33
22	46	41
* H 13 0 *		
2	146	148
4	90	89
8	84	93
10	29	16
12	38	43
14	58	62
18	59	60
20	30	33
* H 14 0 *		
0	130	132
2	42	25
4	73	75
6	103	102
8	27	16
10	72	74
12	50	49
16	63	58
20	18	26
* H 15 0 *		
2	86	89
4	51	58
6	19	25
8	86	88
12	47	45
14	54	55
16	20	7
18	14	41
20	30	24
* H 16 0 *		
0	64	64
4	41	41
6	64	70
8	32	13
10	67	66
12	53	44
16	40	43
18	33	15
* H 17 0 *		
2	43	57
4	40	39
8	59	64
12	28	33
14	38	39
16	38	17
18	28	29
* H 18 0 *		
0	62	58
4	35	41
6	37	46
* H 19 0 *		
2	47	47
6	29	9
8	32	39
* H 20 0 *		
4	21	23
* H 0 1 *		
-1	113	127
1	366	396
-3	333	337
3	328	339
-5	24	38
5	135	118
-7	107	103
7	477	473
-9	326	328
9	201	203
-11	15	10
11	82	88
-13	139	138
13	176	168
-15	172	164
15	75	77
-17	62	55
17	84	82
-19	142	132
19	65	58
-21	44	36
21	17	13
-23	33	29
23	95	85
-25	64	62
25	25	34
-27	33	7
27	10	17
* H 1 1 *		
-1	544	551
1	213	249
-3	117	103
3	106	121
-5	199	174
5	429	478
-7	210	202
7	118	122
-9	79	83
9	163	180
-11	231	228
11	146	144
-13	74	75
13	32	25
-15	117	120
15	173	181
-17	179	175
17	65	66
-19	21	22
19	55	49
-21	64	62
21	98	94
-23	45	62
23	29	36
-25	45	11
25	54	47
-27	43	32
27	35	26
* H 2 1 *		
-1	282	237
1	282	273
-3	430	401
3	152	165
-5	142	131
5	29	38
-7	196	189
7	293	330
-9	368	365
9	165	182
-11	83	74
11	97	107
-13	118	116
13	208	212
-15	129	130
15	75	77
-17	34	25
17	110	108
-19	107	105
19	76	72
-21	42	32
-23	27	34
23	76	73
-25	58	64
-27	35	5
* H 3 1 *		
-1	498	463
1	201	177
-3	124	93
3	110	103
-5	353	315
5	224	265
-7	298	276
7	96	107
-9	96	86
9	180	202
-11	264	243
11	160	165
-13	72	66
13	29	37
-15	73	69
15	219	224
-17	140	130
17	83	79
19	44	33
-21	61	55
21	83	86
-23	61	56
23	20	31
-25	29	19
-27	31	40
* H 4 1 *		
-1	188	156
1	321	287
-3	429	380
3	161	164
-5	176	163
5	19	29
-7	235	212
7	268	304
-9	358	331
9	122	131
-11	25	27
11	103	108
-13	130	118
13	209	216
-15	130	120
15	66	69
-17	17	20
17	106	106
-19	102	95
19	82	82
-21	26	16
-23	48	32
23	66	63
-25	58	61
25	31	24
-27	14	1
27	15	15
* H 5 1 *		
-1	395	352
1	218	206
-3	143	113
3	110	114
-5	245	223
5	270	290
-7	237	212
7	122	132
-9	69	73
9	178	198
-11	216	199
11	121	127
-13	94	94
13	24	18
-15	78	77
15	163	166
-17	142	135
17	62	59
19	44	39
-21	71	58
21	94	84
-23	60	54
23	13	26
25	54	39
27	10	26
* H 6 1 *		
-1	119	103
1	313	290
-3	260	230
3	267	256
-5	87	77
-7	96	95
7	288	315
-9	225	201
9	104	114
-13	146	132
13	141	146
-15	142	128
15	48	46
-17	38	37
17	74	78
-19	114	109
19	68	68
-21	15	32
21	9	20
-23	40	26
23	68	65
-25	40	49
25	24	25
* H 7 1 *		
-1	332	303
1	162	148
-3	11	12
3	143	139
-5	154	137
5	308	315
-7	181	167
7	79	77
-9	61	53
9	161	175
-11	186	170
11	124	132
-13	66	62
13	44	48
-15	93	83
15	101	111
-17	141	134
17	41	39
-19	40	13
19	40	39
-21	63	55
21	64	70
-23	38	53
-25	22	9
25	42	41
27	15	29
* H 8 1 *		
-1	96	87
1	240	223
-3	255	238
3	199	191
-5	112	104
5	56	58
-7	93	92
7	201	210
-9	214	197
9	81	88
-11	51	36
11	68	70
-13	126	115
13	129	136
-15	118	108
15	31	30
-17	32	27
17	85	88
-19	89	83
19	65	64
-21	31	35
21	38	10
-23	38	30
23	44	54
-25	46	39
25	25	23
27	11	13
* H 9 1 *		
-1	225	212
1	94	81
-3	33	39
3	70	63
-5	192	176
5	156	159
-7	203	188
7	59	55
-9	57	52
9	131	141
-11	159	147
11	123	130
-13	59	54
13	40	49
-15	36	35
15	123	120
-17	91	84
17	61	52
-19	16	18
19	39	33
-21	56	43
21	57	55
-23	40	42
23	17	18
25	31	30
27	40	22
* H 10 1 *		
-1	47	40
1	151	150
-3	224	211
3	123	122
-5	89	86
7	140	142
-9	185	169
9	60	64
-11	15	17
11	57	63
-13	97	87
13	131	129
-15	88	80
15	24	36
-17	26	26
17	70	80
-19	64	60
19	63	59
21	10	20
23	38	40
25	10	16
* H 11 1 *		
-1	177	170
1	79	77
-3	50	36
3	73	74
-5	155	147
5	130	134
-7	154	146
7	41	40
-9	44	48
9	119	127
-11	126	124
11	97	96
-13	48	58
13	31	18
-15	35	38
15	85	89
-17	81	72
17	22	42
-19	31	13
19	31	30
-21	42	39
21	24	45
-23	27	34
* H 12 1 *		
-1	44	39
1	138	134
-3	145	137
3	133	137
-5	56	50
5	13	17
-7	50	46
7	126	131
-9	106	102
9	61	51
11	27	34
-13	85	76
13	78	79
-15	60	69
-17	32	24
17	52	57
-19	57	58
19	56	43
-21	28	24
21	33	17
-23	36	17
23	30	38
-25	43	28
-27	34	9
* H 13 1 *		
-1	124	121
1	61	64
-3	32	16
3	61	65
-5	71	68
5	128	135
-7	85	82
7	39	38
-9	46	24
9	79	92
-11	84	85
11	65	74
-13	46	35
13	38	20
-15	21	35
15	63	54
-17	70	68
17	32	29
19	37	21
-21	31	32
21	39	36
-23	30	30
23	15	12
25	22	24
27	24	18
* H 14 1 *		
-1	30	32
1	99	103
-3	105	104
3	98	100
-5	39	43
-7	47	38
7	101	102
-9	95	83
9	47	45
11	46	32
-13	64	67
13	57	61
-15	61	57
-17	10	20
17	42	45
-19	63	44
19	20	33
-21	46	25
21	41	8
23	24	30
* H 15 1 *		
-1	96	99
1	34	37
3	23	32
-5	80	72
5	73	80
-7	79	81
7	33	25
-9	28	27
9	56	65
-11	67	70
11	61	57
-13	30	29
-15	17	21
15	58	52
-17	58	44
17	18	26
-19	49	15
21	43	28
-23	29	20
23	19	12
-25	29	11
25	29	16
-27	42	17
* H 16 1 *		
-1	31	21
1	55	57
-3	75	79
3	49	49
-5	53	34
-7	42	38
7	57	61
-9	70	67
9	26	31
-11	27	13
11	32	25
-13	34	37
13	49	54
-15	35	35
15	34	18
17	31	35
19	30	26
23	45	20
* H 17 1 *		
-1	58	64
1	26	30
-3	26	5
3	28	25
-5	61	54
5	58	50
-7	50	54
7	36	18
9	52	48
-11	36	51
11	33	40
-13	29	23
-15	15	19
15	56	41
-17	37	31
19	11	12
21	15	20
* H 18 1 *		
1	44	51
-3	41	55
3	48	42
7	63	53
-9	40	45
9	33	21
-11	14	20
11	20	16
13	38	34
15	26	13
* H 19 1 *		
-1	54	44
-5	30	30
5	37	43
7	14	16
9	23	33
11	21	23
* H 0 2 *		
0	236	225
-2	326	322
2	67	66
-4	7	8
4	385	397
-6	161	173
6	258	259
-8	176	175
8	164	164
-10	176	184
10	227	214
-12	241	247
12	37	40
-14	21	27
14	169	164
-16	124	126
16	105	90
-18	137	142
18	30	21
-20	25	14
20	84	91
-22	51	40
22	58	51
-24	34	40
24	16	31
-26	33	21
26	58	33
28	15	12
* H 1 2 *		
0	236	209
-2	179	173
2	221	258
-4	377	352
4	89	96
-6	42	37
6	322	349
-8	180	175
8	232	242
-10	276	277
10	52	58
-12	58	56
12	219	211
-14	149	150
14	140	142
-16	94	89
16	74	74
-18	68	62
18	71	59
-20	97	96
20	28	26
-22	15	19
22	78	74
-24	38	39
24	34	38
-26	34	38
26	20	13
28	35	36
* H 2 2 *		
0	354	311
-2	410	380
2	130	134
-4	81	76
4	281	333
-6	237	278
6	197	220
-8	153	152
8	111	120
-10	148	142
10	149	157
-12	180	177
12	74	75
-14	38	30
14	164	168
-16	95	91
16	108	100
-18	126	126
18	26	26
-20	33	28
20	112	104
-22	55	52
22	61	54
-24	43	41
24	36	30
-26	17	25
-28	42	34
* H 3 2 *		
0	259	231
-2	335	288
2	210	216
-4	439	395
4	58	55
-6	44	36
6	234	276
-8	218	205
8	100	115
-10	242	226
10	42	48
-12	19	14
12	186	199
-14	100	104
14	120	124
-16	83	74
16	92	93
-18	52	54
18	108	98
-20	95	95
20	31	39
-22	13	10
22	50	67
24	36	41
-26	35	45
26	17	8
-28	15	4
28	21	27
* H 4 2 *		
0	314	279
-2	416	361
2	98	91
-4	101	92
4	262	277
-6	290	248
6	132	146
-8	193	185
8	128	141
-10	116	121
10	129	139
-12	179	162
12	37	45
-14	33	34
14	160	165
-16	84	80
16	104	99
-18	116	108
18	11	19
-20	39	32
20	109	98
-22	69	59
22	45	50
-24	56	40
24	47	26
-26	27	23
-28	23	35
28	21	13
* H 5 2 *		
0	209	189
-2	205	189
2	227	223
-4	272	255
4	136	140
-6	61	63
6	246	278
-8	170	164
8	148	160
-10	215	203
10	30	30
-12	26	40
12	172	183
-14	131	129
14	94	97
-16	81	74
16	84	79
-18	52	53
18	82	82
-20	88	84
20	43	37
22	57	57
-24	41	36
24	17	38
-26	57	39
* H 6 2 *		
0	164	150
-2	248	223
2	54	49
-4	73	64
4	272	278
-6	173	153
6	158	170
-8	176	160
8	141	155
-10	131	126
10	179	193
-12	196	181
12	86	57
-14	16	27
14	119	125
-16	99	97
16	77	76
-18	107	105
18	32	21
-20	34	16
20	67	68
-22	29	45
22	19	36
-24	43	36
24	45	34
-28	32	27
* H 7 2 *		
0	182	173
-2	104	97
2	209	199
-4	217	195
4	74	85
-6	49	41
6	220	234
-8	148	139
8	157	166
-10	206	190
10	38	45
-12	67	60
12	154	156
-14	141	136
14	75	75
-16	86	89
16	63	65
-18	49	52
18	67	67
-20	81	69
20	13	26
22	43	50
24	42	32
26	18	9
28	43	26
* H 8 2 *		
0	147	143
-2	238	226
2	72	69
-4	79	69
4	207	212
-6	177	167
6	114	119
-8	137	132
8	110	115
-10	90	86
10	141	147
-12	143	136
12	65	73
-14	22	31
14	96	104
-16	76	68
16	66	67
-18	94	84
18	27	26
20	67	60
-22	52	47
22	17	31
-24	45	30
24	38	33
-26	15	17
26	26	29
-28	19	26
28	45	13
* H 9 2 *		
0	162	156
-2	131	124
2	191	184
-4	222	204
4	62	63
-6	26	19
6	133	134
-8	118	112
8	93	97
-10	138	126
10	13	37
-12	51	37
12	105	108
-14	84	80
14	59	61
-16	67	70
16	81	75
-18	44	47
18	80	76
-20	69	59
20	28	30
22	47	47
-24	18	31
24	56	30
-26	33	31
* H 10 2 *		
0	151	143
-2	193	181
2	32	32
-4	88	80
4	131	134
-6	179	168
6	63	61
-8	119	111
8	94	107
-10	43	54
10	89	95
-12	100	100
12	50	47
-14	37	27
14	89	99
-16	42	46
16	61	66
-18	65	62
-20	21	22
20	46	55
-22	42	48
22	40	29
-24	25	26
24	23	25
-26	26	16
* H 11 2 *		
0	127	122
-2	89	89
2	155	149
-4	162	147
4	64	64
-6	50	51
6	117	120
-8	89	91
8	79	81
-10	112	107
-12	31	37
12	81	87
-14	80	76
14	44	46
-16	58	55
16	56	57
-18	34	37
18	68	59
-20	35	51
20	29	28
22	49	37
24	36	24
* H 12 2 *		
0	91	86
-2	110	104
2	21	27
-4	31	44
4	128	122
-6	104	103
6	71	74
-8	92	85
8	92	98
-10	53	53
10	93	96
-12	111	99
12	47	43
14	66	71
-16	42	50
16	44	45
-18	50	55
18	10	12
-20	28	14
20	32	36
-22	33	34
22	27	21
24	37	23
* H 13 2 *		
0	73	77
-2	51	45
2	104	103
-4	83	81
4	45	47
6	105	104
-8	65	67
8	80	86
-10	89	84
-12	33	38
12	71	74
-14	67	72
14	37	37
-16	43	51
16	27	38
18	14	41
-20	51	39
* H 14 2 *		
0	63	56
-2	94	94
4	99	95
-6	88	81
6	52	49
-8	63	65
8	63	64
-10	23	43
10	75	76
-12	71	76
12	37	34
14	66	51
-16	44	37
16	26	36
-18	39	43
-20	25	17
20	18	28
-22	11	26
* H 15 2 *		
0	66	70
-2	45	46
2	77	81
-4	86	88
4	34	31
6	65	65
-8	46	50
8	53	50
-10	61	63
10	9	11
12	51	50
-14	45	41
14	44	29
-16	30	38
16	52	33
18	33	35
-20	35	30
20	18	12
-22	36	13
* H 16 2 *		
0	47	54
-2	89	74
2	16	7
-4	46	36
4	58	61
-6	66	63
6	36	29
-8	36	43
8	23	37
-10	17	30
10	29	45
-12	35	44
14	39	42
-16	31	25
16	43	32
-18	42	28
20	22	25
22	29	15
* H 17 2 *		
0	36	44
-2	40	41
2	46	54
-4	54	59
4	33	22
-6	29	19
6	47	48
-8	24	36
8	45	32
-10	42	43
10	26	11
-12	18	20
14	25	21
* H 18 2 *		
0	37	37
-2	43	48
4	48	47
-6	47	42
6	28	27
-8	43	34
-10	10	25
10	38	33
* H 19 2 *		
0	18	28
2	37	33
-4	34	33
4	31	15
6	32	41
-8	34	28
-10	36	32
10	11	6
-12	18	14
-14	39	27
* H 20 2 *		
-2	33	28
2	10	5
4	18	32
-6	26	26
* H 0 3 *		
-1	91	87
1	161	164
-3	214	217
3	71	73
-5	289	298
5	204	207
-7	35	35
7	231	219
-9	244	258
9	8	9
-11	174	183
11	232	225
-13	90	87
13	210	194
-15	111	118
15	46	40
-17	64	59
17	83	78
-19	64	63
19	66	58
-21	61	55
21	57	53
-23	43	3
23	47	36
-25	17	39
-27	36	26
27	39	35
* H 1 3 *		
-1	223	202
1	60	53
-3	260	241
3	232	263
-5	171	156
5	250	271
-7	205	204
7	126	132
-9	63	64
9	188	187
-11	195	196
11	134	133
-13	180	187
13	135	131
15	129	120
-17	81	81
17	36	28
-19	81	90
19	96	84
-21	28	29
21	75	69
-23	31	43
23	16	17
25	31	34
* H 2 3 *		
-1	83	75
1	267	261
-3	308	277
3	145	159
-5	273	255
5	205	237
-7	54	43
7	181	198
-9	179	175
-11	152	144
11	183	185
-13	70	68
13	158	154
-15	120	116
15	40	41
-17	51	52
17	93	83
-19	80	85
19	59	61
-21	72	66
21	61	56
23	47	43
-25	44	41
-27	36	30
27	15	32
* H 3 3 *		
-1	370	323
1	60	54
-3	294	259
3	278	296
-5	132	128
5	237	275
-7	159	148
7	92	92
-9	105	103
9	108	116
-11	113	107
11	76	80
-13	137	135
13	107	111
-15	28	22
15	92	92
-17	103	99
-19	88	80
19	85	95
-21	44	46
21	85	76
-23	59	57
-25	34	18
25	25	39
-27	23	19
27	33	23
* H 4 3 *		
-1	135	122
1	280	268
-3	286	259
3	183	187
-5	255	236
5	182	201
-7	40	43
7	145	166
-9	142	139
9	32	43
-11	139	134
11	146	153
-13	64	59
13	109	112
-15	101	104
15	55	56
-17	38	35
17	100	89
-19	84	86
19	61	55
-21	65	68
21	53	54
-23	19	12
23	56	49
-25	49	39
25	32	12
-27	18	31
27	35	27
* H 5 3 *		
-1	218	194
1	34	34
-3	239	221
3	230	229
-5	147	134
5	189	203
-7	186	174
7	79	82
-9	106	98
9	155	169
-11	154	147
11	96	100
-13	140	133
13	104	108
-15	34	28
15	103	108
-17	55	85
-19	70	70
19	76	68
-21	45	31
21	55	60
-23	45	49
25	35	33
* H 6 3 *		
-1	112	101
1	169	158
-3	177	165
3	108	107
-5	209	197
5	153	162
-7	42	43
7	161	178
-9	198	194
9	37	46
-11	148	139
11	153	162
-13	90	86
13	129	133
-15	100	98
15	55	51
-17	51	54
17	89	81
-19	39	48
19	50	49
-21	36	48
21	50	41
-23	17	15
23	44	39
-25	45	30
-27	15	24
* H 7 3 *		
-1	140	131
-3	186	170
3	168	166
-5	144	127
5	131	134
-7	192	182
7	87	92
-9	82	78
9	158	170
-11	147	141
11	95	103
-13	133	128
13	92	93
-15	28	17
15	100	105
-17	75	68
17	35	26
-19	61	62
19	49	53
21	39	45
-23	26	38
23	34	24
* H 8 3 *		
-1	100	96
1	182	173
-3	150	141
3	89	92
-5	172	156
5	147	152
-7	36	37
7	139	150
-9	142	131
-11	122	115
11	104	106
-13	72	74
13	100	102
-15	85	90
15	52	46
-17	49	61
17	72	69
-19	50	52
19	42	47
21	38	39
23	28	35
* H 9 3 *		
-1	200	186
1	39	41
-3	148	130
3	160	156
-5	106	100
5	130	133
-7	132	122
7	85	81
-9	64	71
9	102	108
-11	66	70
11	61	72
-13	88	85
13	67	74
15	71	72
-17	67	71
-19	47	49
19	47	54
-21	24	33
21	28	40
-23	42	41
* H 10 3 *		
-1	89	85
1	184	172
-3	136	132
3	130	125
-5	128	117
5	109	107
7	112	111
-9	77	75
9	31	29
-11	83	82
11	61	65
-13	45	50
13	52	55
-15	68	71
15	52	49
-17	16	32
17	63	62
-19	54	50
19	41	38
-21	26	48
21	26	38
23	44	37
* H 11 3 *		
-1	137	131
-3	114	112
3	113	105
-5	53	63
5	100	102
-7	120	113
7	59	68
-9	68	67
9	88	90
-11	65	65
11	57	60
-13	64	72
13	66	72
-15	13	9
15	54	58
-17	47	61
17	41	15
-19	44	40
19	48	41
-21	14	26
21	34	31
* H 12 3 *		
-1	75	72
1	90	90
-3	88	86
3	83	75
-5	95	94
5	92	86
7	92	96
-9	99	96
9	28	24
-11	71	71
11	72	73
-13	43	53
13	32	59
-15	73	63
15	13	34
-17	32	31
17	59	50
-19	10	28
19	32	28
21	31	25
* H 13 3 *		
-1	75	74
-3	73	70
3	63	68
-5	61	63
5	59	64
-7	98	100
7	45	46
-9	38	38
9	83	87
-11	61	69
11	54	56
-13	67	62
13	51	54
-15	13	12
15	41	55
-17	37	39
19	41	27
* H 14 3 *		
-1	59	54
1	67	74
-3	65	60
3	63	41
-5	70	73
5	70	67
-7	20	15
7	66	71
-9	65	66
-11	42	59
11	59	50
-13	31	40
13	42	50
-15	26	46
15	31	24
-17	42	32
19	40	22
21	11	20
* H 15 3 *		
1	18	11
-1	57	70
-3	58	59
3	68	70
-5	48	45
5	37	47
-7	77	62
7	32	33
-9	31	33
9	56	56
-11	37	43
11	32	32
-13	48	47
13	28	33
-15	28	20
15	23	39
-17	25	34
* H 16 3 *		
-1	33	28
1	71	72
-3	56	55
3	41	46
-5	50	53
5	40	45
7	38	52
-9	37	34
9	24	7
11	10	30
-13	36	28
13	32	30
-15	36	32
15	27	20
-17	18	17
17	28	27
* H 17 3 *		
-1	43	53
-3	32	43
3	40	47
-5	34	29
5	40	44
-7	42	39
9	32	37
-11	34	30
11	14	20
13	29	27
15	26	27
* H 18 3 *		
-3	20	34
3	32	27
-5	51	40
7	37	35
-9	32	35
9	23	8
-11	18	29
11	31	29
-13	15	19
* H 19 3 *		
-3	34	30
3	15	28
-5	23	23
-7	52	34
-9	41	17
9	11	29
-11	37	29
* H 0 4 *		
0	63	59
-2	233	243
2	193	192
-4	131	135
4	205	211
-6	211	211
6	41	39
-8	177	191
8	133	119
10	157	151
-12	133	133
12	84	80
-14	104	111
14	135	118
16	35	36
-18	67	66
18	84	74
-20	33	34
20	97	85
-22	55	39
22	9	4
-24	42	41
24	37	31
-26	15	1
26	52	28
28	34	9
* H 1 4 *		
0	194	179
-2	36	33
2	149	177
-4	315	305
4	98	108
-6	191	184
6	148	156
-8	60	66
-10	166	173
10	155	155
-12	102	108
12	187	179
-14	86	83
-16	66	67
16	105	94
-18	19	20
18	46	84
-20	64	70
20	27	25
-22	34	42
22	41	44
-24	29	22
24	30	12
-26	15	30
-28	15	21
* H 2 4 *		
0	113	105
-2	221	197
2	194	209
-4	131	118
4	238	276
-6	155	149
-8	158	152
8	149	150
-10	50	52
10	148	150
-12	154	152
12	88	84
-14	108	110
14	105	98
-18	78	88
18	81	69
-20	15	38
20	98	81
-24	39	39
24	30	34
26	33	31
* H 3 4 *		
0	194	181
-2	65	59
2	218	217
-4	194	181
4	109	122
-6	139	131
8	69	78
-10	139	142
10	119	127
-12	73	78
12	144	146
-14	119	122
14	11	11
-16	93	92
16	75	67
18	65	61
-20	76	77
20	20	32
-22	45	50
22	46	48
-24	10	11
26	23	27
28	34	29
* H 4 4 *		
0	69	62
-2	191	165
2	194	192
-4	148	136
4	247	257
-6	118	108
6	31	16
-8	124	120
8	138	149
10	142	142
-12	155	151
12	55	61
-14	103	96
14	89	89
16	14	26
-18	89	94
18	68	58
-20	22	44
20	80	72
-22	28	33
24	35	34
-26	10	10
26	10	31
28	29	10
* H 5 4 *		
0	190	175
-2	92	86
2	164	159
-4	237	215
4	99	98
-6	159	143
6	154	167
-8	63	67
8	66	68
-10	152	145
10	116	118
-12	73	77
12	132	143
-14	88	92
14	37	22
-16	70	72
16	87	79
18	59	70
-20	32	60
-22	21	44
22	10	47
-26	30	26
26	41	20
* H 6 4 *		
0	73	73
-2	220	201
2	140	132
-4	122	109
4	144	147
-6	190	175
6	26	35
-8	153	150
8	111	116
10	102	114
-12	117	113
14	100	115
-16	14	16
16	15	39
-18	61	60
-20	21	34
20	73	65
-22	36	38
22	14	10
-24	25	33
* H 7 4 *		
0	156	148
2	118	114
-4	209	196
4	90	88
-6	142	133
6	126	129
-8	62	70
8	34	31
-10	133	125
10	82	84
-12	91	90
12	123	126
-14	64	60
14	14	21
-16	52	59
16	69	71
-18	25	28
18	84	68
-20	16	49
-22	45	32
22	46	40
* H 8 4 *		
0	92	82
-2	178	164
2	117	110
-4	84	79
4	150	146
-6	129	121
6	40	46
-8	128	122
8	105	111
-10	42	62
10	88	93
-12	99	99
12	68	63
-14	77	72
14	95	92
-16	30	28
16	44	39
-18	38	61
18	28	40
20	53	52
-24	23	28
24	30	26
26	49	21
* H 9 4 *		
0	142	127
-2	26	33
2	150	141
-4	119	112
4	86	83
-6	77	81
6	153	157
-8	36	44
8	55	59
-10	91	97
10	64	65
-12	56	65
12	78	86
-14	75	77
-16	62	68
16	47	49
-18	25	18
18	21	46
-20	46	47
20	33	27
-22	39	33
22	43	38
-24	21	10
-26	28	22
* H 10 4 *		
0	44	45
-2	125	115
2	96	87
-4	83	78
4	153	148
-6	63	64
-8	89	78
8	95	97
10	87	89
-12	88	92
12	51	45
-14	58	50
14	68	62
-16	24	34
-18	76	61
18	46	32
-20	38	31
20	28	36
-22	40	30
22	23	9
-24	18	24
* H 11 4 *		
0	97	92
-2	45	61
2	106	100
-4	99	93
4	64	68
-6	73	70
6	98	101
8	55	57
-10	88	90
10	47	56
-12	36	41
12	61	67
-14	46	58
-16	30	59
16	55	48
-18	28	21
-20	34	35
20	32	22
-22	23	32
22	28	35
24	22	14
* H 12 4 *		
0	42	42
-2	108	109
2	68	59
-4	66	62
4	79	79
-6	93	90
-8	86	82
8	66	66
10	75	63
-12	53	70
12	43	42
-14	26	40
14	72	66
-16	17	19
16	28	27
-18	24	38
18	40	27
20	29	32
-22	37	27
* H 13 4 *		
0	74	75
2	68	67
-4	100	97
4	60	51
-6	54	60
6	66	66
-8	55	41
8	32	29
-10	63	70
10	40	42
-12	28	39
12	64	59
-14	21	34
14	17	11
-16	34	39
16	42	40
18	40	33
-20	31	27
* H 14 4 *		
0	28	31
-2	83	80
2	45	46
-4	33	37
4	53	58
-6	67	64
-8	56	59
8	36	54
10	42	41
-12	32	48
12	19	28
-14	36	38
14	30	51
-16	29	18
16	27	21
-18	25	29
18	15	17
* H 15 4 *		
0	57	62
2	63	53
-4	52	53
4	31	36
-6	46	47
6	66	65
-8	33	26
8	36	20
-10	55	47
10	17	30
12	39	43
-14	20	35
14	25	10
18	18	24
20	29	15
22	27	19
* H 16 4 *		
2	47	39
-4	30	32
4	65	62
-18	22	26
-24	27	12
* H 0 5 *		
-1	134	139
1	229	229
-3	59	49
-5	226	225
5	143	137
-7	93	97
7	52	51
-9	60	60
9	87	87
-11	91	94
11	153	147
13	21	12
-15	71	81
15	86	73
-17	51	55
17	118	96
-19	28	17
21	56	46
* H 1 5 *		
-1	133	117
1	110	117
-3	186	184
3	205	226
-5	49	51
-7	159	155
7	108	109
-9	99	109
9	146	141
11	35	26
-13	116	121
13	93	91
-15	53	50
17	43	48
19	83	83
23	52	28
* H 2 5 *		
-1	138	116
1	201	196
-3	36	29
-5	217	204
5	136	151
-7	107	105
7	87	93
-9	85	87
9	81	83
-11	123	120
11	167	160
-13	20	30
-15	99	99
15	71	68
-17	57	57
17	84	82
-21	28	51
* H 3 5 *		
-1	66	59
1	107	100
-3	148	136
3	188	196
-5	50	52
5	20	33
-7	157	150
7	111	123
-9	105	106
9	169	172
-11	42	34
-13	143	147
13	102	97
15	45	50
-17	19	38
-19	35	63
19	81	67
* H 4 5 *		
-1	117	108
1	160	156
3	60	62
-5	185	174
5	137	149
-7	94	87
7	91	93
-9	92	90
9	62	66
-11	117	118
11	150	154
-15	91	93
15	39	55
-17	53	62
17	81	75
* H 5 5 *		
-1	101	97
1	106	95
-3	178	166
3	192	188
-5	36	37
5	18	32
-7	148	143
7	91	93
-9	90	88
-13	105	110
13	87	86
-15	47	47
-17	32	38
17	32	37
-19	31	59
* H 6 5 *		
-1	131	125
1	181	172
-3	40	42
-5	190	173
5	133	137
-7	78	68
7	57	59
-9	70	66
9	55	50
-11	91	87
11	112	115
-13	28	44
-15	64	67
15	36	54
-17	45	50
17	77	77
-21	64	51
21	52	43
* H 7 5 *		
-1	76	106
1	70	62
-3	161	149
3	164	160
-7	116	113
7	74	78
-9	77	82
9	87	96
-11	25	23
11	44	37
-13	74	83
13	88	83
-15	33	40
15	44	49
-19	25	49
19	66	61
-21	34	15
* H 8 5 *		
-1	104	89
1	138	131
3	40	30
-5	136	127
5	130	124
-7	66	73
7	65	70
9	57	46
-11	91	92
-15	78	66
15	40	44
-17	44	44
17	66	59
19	22	13
21	47	38
* H 9 5 *		
-1	65	60
1	47	45
-3	110	104
3	135	129
-5	42	50
-7	104	103
7	85	77
-9	68	73
9	93	106
-13	91	92
13	83	77
15	51	44
-17	40	40
-19	49	42
19	30	43
* H 10 5 *		
-1	70	67
1	91	87
3	47	45

-5	108	98	1	36	19	-8	152	149	18	65	55	-14	31	19	-5	54	63	9	47	32
5	100	101	-3	55	49	8	147	143	* H	7	6 *	14	10	18	5	38	35	11	47	40
-7	43	41	3	55	59	-10	46	51	0	144	134	-16	33	43	-7	43	49	-13	39	44
7	76	76	-7	40	45	10	38	25	-2	68	57	* H	12	6 *	7	96	100	13	45	47
-9	69	67	7	32	35	-12	11	38	2	39	34	2	66	79	-9	105	106	-15	51	49
9	43	36	9	47	44	12	90	78	-4	29	39	-2	73	63	-11	31	31	17	43	40
-11	67	88	-11	17	17	-14	89	99	4	101	94	-4	55	57	11	57	52	* H	8	7 *
11	97	87	11	23	13	14	72	57	-6	112	109	-6	16	18	-13	15	30	-1	90	88
-15	63	60	13	44	37	-16	35	21	6	85	86	* H	0	7 *	13	69	60	-3	26	29
15	48	38	* H	16	5 *	-18	28	43	10	88	89	-1	125	124	-15	48	55	3	68	59
-17	14	42	-1	20	34	18	76	66	-12	57	66	1	55	53	17	50	51	-5	45	43
17	40	41	1	44	38	* H	3	6 *	12	20	17	-5	26	36	-19	30	33	5	73	67
* H	11	5 *	-5	52	45	-2	81	78	14	16	33	5	114	106	19	27	23	-7	79	76
-1	58	54	5	48	49	2	19	16	16	62	59	-7	81	93	* H	4	7 *	9	71	66
1	49	48	* H	0	6 *	-4	68	56	-18	31	23	9	95	93	-1	124	119	-11	56	55
-3	96	92	-2	114	117	4	91	99	18	28	8	-11	51	66	1	50	42	11	42	37
3	98	94	2	194	189	6	84	85	-20	14	19	11	41	43	3	61	66	15	46	46
-5	36	36	-4	112	115	-10	96	99	20	45	35	-13	29	44	-5	68	75	* H	9	7 *
5	15	17	4	84	75	10	130	130	* H	8	6 *	13	30	21	5	72	72	1	76	78
-7	79	88	6	70	70	-12	87	83	0	35	19	-15	45	36	-7	104	103	-3	76	75
7	54	51	-8	111	118	12	42	42	-2	83	79	15	78	69	9	84	86	3	34	37
-9	49	51	8	146	134	-16	58	66	2	123	114	-17	55	64	-11	67	60	-5	66	55
9	68	75	-10	65	62	16	97	85	-4	101	98	-19	37	13	11	41	42	-7	40	37
-13	57	66	12	85	71	-22	30	38	4	33	40	21	29	29	-13	55	56	7	64	60
13	58	51	-14	91	93	* H	4	6 *	6	38	42	* H	1	7 *	13	18	20	-9	52	68
-15	39	33	14	50	42	0	54	43	-8	89	92	1	117	126	15	64	67	11	27	40
15	26	35	-16	34	15	-2	95	85	8	90	95	-3	87	85	17	26	24	13	17	44
17	17	16	-18	51	54	2	154	147	-10	53	46	3	66	69	-19	25	9	-15	45	43
* H	12	5 *	18	83	72	-4	114	104	-12	25	33	-5	67	68	* H	5	7 *	17	57	38
-1	72	69	20	14	16	6	60	69	12	74	63	5	22	30	1	111	105	* H	10	7 *
1	88	89	-22	28	2	-8	139	138	-14	57	65	-7	25	23	-3	89	84	-1	78	75
3	37	37	22	14	22	-12	44	40	14	60	49	7	129	123	3	59	57	3	44	50
-5	76	95	-24	29	39	12	74	75	-16	29	14	-9	96	97	-5	60	63	-5	51	52
5	78	75	24	53	40	-14	83	93	-18	36	37	9	59	47	7	105	101	5	53	48
-7	46	31	-26	26	15	14	66	64	* H	9	6 *	11	54	48	-9	89	91	-7	55	65
7	35	48	* H	1	6 *	18	61	59	0	97	89	-13	33	52	9	38	30	9	56	57
-9	34	40	0	203	186	-20	35	35	-2	48	53	13	67	53	-11	28	35	-11	37	45
-11	57	61	-2	96	88	* H	5	6 *	-4	23	34	-15	52	60	11	38	51	11	14	28
11	60	55	2	49	33	0	164	149	4	83	78	17	37	52	13	63	55	-13	37	36
-15	43	40	-4	62	55	-2	76	70	-6	75	107	-19	43	45	-15	65	56	-15	29	12
15	22	34	4	106	107	-4	58	51	6	36	66	-21	10	28	15	21	7	* H	11	7 *
* H	13	5 *	-6	152	148	4	113	115	-8	15	21	* H	2	7 *	17	50	45	1	50	60
-1	66	63	6	98	99	-6	161	130	10	88	85	-1	145	130	* H	6	7 *	-3	54	56
-3	93	79	-8	50	54	6	81	90	-12	9	63	1	42	46	-1	105	101	3	44	37
3	78	80	8	38	34	-8	31	26	12	13	29	-3	43	41	1	41	37	7	56	52
-5	25	25	-10	61	66	8	22	27	-16	47	52	3	75	78	-3	44	33	-9	49	52
-7	66	68	10	130	121	-10	68	71	16	20	49	-5	78	71	3	65	66	* H	12	7 *
7	47	42	-12	65	78	10	108	110	* H	10	6 *	5	75	81	5	91	91	-1	38	55
-9	29	41	12	18	26	-12	76	80	0	27	26	-7	105	107	-7	76	85	3	36	41
9	48	46	-14	22	9	14	43	33	-2	50	67	9	97	90	9	82	78	5	74	49
-11	45	19	-16	67	73	-16	54	67	2	85	84	-11	64	65	-11	59	61	-7	29	49
-13	46	44	16	103	85	16	84	71	-4	67	73	11	61	43	11	33	41	9	35	48
13	54	50	-18	37	33	* H	6	6 *	4	26	21	-13	54	52	-13	40	43	* H	13	7 *
-15	30	27	18	34	12	2	165	155	6	42	38	13	45	24	13	30	15	1	54	50
15	43	26	-20	44	26	-4	102	102	-8	79	88	-15	22	18	-15	30	28	-3	31	35
* H	14	5 *	20	38	38	4	58	53	8	73	74	15	82	73	15	48	50	3	26	35
-1	42	45	-22	18	46	-8	103	95	-14	63	56	-17	42	56	-17	54	54	-5	14	39
1	67	64	* H	2	6 *	8	101	109	14	62	46	17	24	29	* H	7	7 *	7	36	45
-5	52	61	0	51	47	-10	47	57	* H	11	6 *	21	44	27	1	105	96	9	23	15
5	63	62	-2	112	98	-12	32	23	0	79	78	* H	3	7 *	-3	61	43	* H	14	7 *
7	39	31	2	160	164	12	53	64	2	20	17	-1	26	19	3	67	60	-1	30	43
-9	10	26	-4	136	126	-14	76	75	4	81	73	1	119	110	-5	49	63	3	29	30
9	17	22	4	55	47	-16	9	19	6	38	55	-3	112	112	7	85	90	7	21	10
* H	15	5 *	6	7	74	-18	46	48	10	51	63	3	54	47	-9	45	68	9	33	35

[a] Far left column contains h followed by F_o then F_c. F_o = $10F$(Calcd). F(Calcd) on absolute scale. Reflections of zero observed intensity are not listed and were not included in refinement.

plane and the other atoms in the general positions; (c) all other special positions in this space group are centers of symmetry, and the asymmetric *cis* molecule could not be placed at a center of symmetry. The occurrence of a number of substantial peaks in the three-dimensional Patterson at $y \neq 0, {}^1/_2$ rules out possibility (a) in $B2_1/m$. The existence of a large peak at $y = 0$ that can be most readily assigned to a Pt-Cl(P) interaction in the Patterson rules out possibility (b) of $B2_1/m$, and therefore the correct space group is $B2_1$. The structure was solved and refined on this basis.

The Pt, P, and Cl atoms were located from the three-dimensional Patterson vector maps. The carbon atoms were located from three-dimensional electron density and difference electron density maps.[9] The structure was refined by full-matrix least squares including anisotropic temperature factors: (a) without dispersion corrections, (b) with only the real part of the dispersion correction, and (c) with real and imaginary dispersion corrections.[10-13] The function minimized was $\Sigma w \cdot (F_o - F_c)^2$. The observations were weighted on counting statistics and a 4% intensity factor.[14] Atomic coordinate shifts for the final least-squares cycle were less than 4×10^{-5} of a cell edge for Pt, P, and Cl and less than 4×10^{-4} for carbon. The final disagreement index R[15] and the weighted R for refinements a–c were found to be: 0.100, 0.120; 0.101, 0.120; and 0.097, 0.115, respectively. A final difference map appeared qualitatively clean and showed no unusual features. Final calculated and observed structure factors from the refinement, including dispersion corrections, are listed in Table I. Table II contains atomic parameters and errors, and Table III shows interatomic distances, angles, and errors,[16] as well as the best least-squares plane through Pt, P, and Cl.[17] We are disappointed that the disagreement index is not lower, but some improvement could be obtained by neglecting a number of reflections that are obviously in error due to extinction. This relatively high disagreement index may also reflect the neglect of absorption corrections.

It is readily shown that in an acentric space group where the effects of anomalous dispersion are important Friedel's law fails. In particular, in $P2_1$ ($B2_1$) $F^2(hkl)$ is no longer equal to $F^2(h\bar{k}l)$ and it is necessary to test both possibilities if a complete data set is not available. The test is equivalent to refining two structures: structure 1 has the coordinates as given in Table II; structure 2 has essentially the same x and z coordinates but with the y reversed in sign. We carried out both calculations and have rejected structure 2 on the following grounds: (A) The structure obtained does not make chemical sense in that two distinctly different Pt–P and Pt–Cl distances result (Pt–P, 2.201 ± 0.008, 2.244 ± 0.005 A; Pt–Cl, 2.370 ± 0.007, 2.423 ± 0.008 A). This arises from a shift of the Pt atom relative to the X-ray source. Templeton, Zalkin, and Ueki[18] have recently noted this effect in their study of thorium nitrate pentahydrate. (B) Structure 2 refines to a weighted R factor of 0.121. If this is tested using Hamilton's[19] R-factor test as a hypothesis of one degree of freedom, then structure 2 can be rejected at the 0.5% confidence level.

Results and Discussion

The crystal structure of *cis*-$Pt[P(CH_3)_3]_2Cl_2$ is made up of discrete molecular units separated by ordinary van der Waals distances (Figures 1 and 2). The average Pt–P distance of 2.247 A (2.256 ± 0.008, 2.239 ± 0.006 A) is significantly different from the 2.135 ± 0.004 and 2.300 ± 0.019 A Pt–P distances observed in *trans*-$Pt[P(C_2H_5)_3]_2Br_2$ and *trans*-$Pt[P(C_2H_5)_3]_2Cl_2$, respectively. However, a Pt–P distance of 2.267 ± 0.008 A has been observed in *trans*-$Pt[P(C_6H_5)_2C_2H_5]_2HCl$ by Eisenberg and Ibers.[20] These numbers indicate that Pt–P distances may be as sensitive to substituents on the phosphorus as they are to *trans* substituents on the metal

(9) Fourier programs are due to Sly, Shoemaker, and van den Hende for the IBM 7090.

(10) Least-squares refinement performed with ORFLS of W. Busing, K. O. Martin, and H. Levy as modified by Ibers for inclusion of anomalous dispersion. We are grateful to Professor L. Dahl and the University of Wisconsin for the CDC 1604 time to perform some of these calculations.

(11) Anomalous dispersion corrections made to F, as suggested by: J. A. Ibers and W. C. Hamilton, *Acta Cryst.*, **17**, 781 (1964).

(12) (a) Scattering factors for neutral Cl, P, and C from: J. A. Ibers, "International Tables for X-Ray Crystallography," Vol. III, The Kynoch Press, Birmingham, England, 1962, p 202; (b) scattering factor for neutral Pt from: D. T. Cromer and J. T. Waber, *Acta Cryst.*, **18**, 104 (1965).

(13) Real and imaginary dispersion corrections applied to Pt, P, and Cl from: D. T. Cromer, *ibid.*, **18**, 17 (1965).

(14) S. W. Peterson and H. A. Levy, *ibid.*, **10**, 70 (1957).

(15) $R = \Sigma||F_o| - |F_c||/\Sigma|F_o|$. Weighted $R = [\Sigma w(F_o - F_c)^2]^{1/2}/[\Sigma w F_o^2]^{1/2}$.

(16) Distances, angles, and errors were calculated with W. Busing, K. O. Martin, and H. A. Levy, ORFFE.

(17) W. C. Hamilton, *Acta Cryst.*, **14**, 185 (1961).

(18) T. Ueki, A. Zalkin, and D. H. Templeton, *ibid.*, **20**, 836 (1966).

(19) W. C. Hamilton, *ibid.*, **18**, 502 (1965).

(20) R. Eisenberg and J. A. Ibers, *Inorg. Chem.*, **4**, 773 (1965).

TABLE II

ATOM POSITIONAL AND TEMPERATURE PARAMETERS AND ERRORS;[a] $\sigma' = \sigma \times 10^5$

Atom	x/a	$\sigma'(x/a)$	y/b	$\sigma'(y/b)$	z/c	$\sigma'(z/c)$
Pt	0.4066	4	−0.5000	...[b]	0.7008	12
Cl_1	0.5351	43	−0.5359	68	0.8523	153
P_1	0.4521	39	−0.3451	65	0.5378	125
Cl_2	0.3578	55	−0.6533	77	0.9034	146
P_2	0.2825	27	−0.4950	102	0.5574	93
C_1	0.5556	224	−0.3032	353	0.5875	746
C_2	0.4514	209	−0.3507	344	0.2500	528
C_3	0.3961	249	−0.2194	238	0.6115	679
C_4	0.2543	180	−0.4076	275	0.3343	583
C_5	0.2039	106	−0.4606	221	0.7485	424
C_6	0.2557	71	−0.6306	177	0.4334	627

Thermal Parameters and Standard Deviations; Anisotropic Temperature Factors of the Form $\exp[-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + 2\beta_{12}hk + 2\beta_{13}hl + 2\beta_{23}kl)]$; $\sigma' = \sigma \times 10^5$

Atom	β_{11}	σ'	β_{22}	σ'	β_{33}	σ'	β_{12}	σ'	β_{13}	σ'	β_{23}	σ'
Pt	0.0021	2	0.0040	5	0.0152	20	0.0006	4	0.0001	4	−0.0010	16
Cl_1	0.0027	21	0.0067	49	0.0321	257	0.0010	26	−0.0031	62	−0.0012	87
P_1	0.0022	19	0.0052	45	0.0217	202	−0.0006	24	0.0014	47	−0.0011	77
Cl_2	0.0049	35	0.0064	55	0.0275	247	0.0002	36	0.0019	73	0.0054	97
P_2	0.0018	12	0.0040	31	0.0193	145	0.0004	35	−0.0008	32	−0.0034	129
C_1	0.0038	142	0.0077	280	0.0478	1556	−0.0017	167	−0.0050	404	0.0055	559
C_2	0.0048	127	0.0091	282	0.0166	815	0.0010	160	0.0042	254	0.0031	393
C_3	0.0073	182	0.0023	147	0.0386	1156	0.0006	131	0.0071	364	−0.0021	339
C_4	0.0032	97	0.0048	191	0.0354	1125	0.0005	112	−0.0046	276	0.0020	376
C_5	0.0012	46	0.0065	169	0.0251	709	0.0008	71	0.0040	149	−0.0040	278
C_6	0.0036	95	0.0021	101	0.0510	1229	−0.0012	77	0.0011	268	−0.0027	296

[a] With real and imaginary dispersion corrections. [b] Parameter fixed.

TABLE III

INTERATOMIC DISTANCES, ANGLES, AND ERRORS[a] FOR *cis*-$Pt[P(CH_3)_3]_2Cl_2$

I. Intramolecular

Bonded, A		Nonbonded, A		Nonbonded, A	
Pt–Cl_1	2.364 ± 0.008	P_1–Cl_1	3.304 ± 0.012	C_4–C_6	2.73 ± 0.04
Pt–Cl_2	2.388 ± 0.009	P_2–Cl_1	3.131 ± 0.013	C_5–C_6	2.99 ± 0.04
Pt–P_1	2.256 ± 0.008	P_1–Cl_2	4.639 ± 0.013		
Pt–P_2	2.239 ± 0.006	P_2–Cl_1	4.593 ± 0.008	C_1–Cl_1	3.27 ± 0.04
		P_1–P_2	3.350 ± 0.011	C_2–Cl_1	4.59 ± 0.03
P_1–C_1	1.818 ± 0.034	Cl_1–Cl_2	3.293 ± 0.013	C_6–Cl_2	3.40 ± 0.04
P_1–C_2	1.822 ± 0.035			C_5–Cl_2	3.56 ± 0.03
P_1–C_3	1.833 ± 0.029	C_1–C_2	2.78 ± 0.05		
P_2–C_4	1.809 ± 0.033	C_1–C_3	2.85 ± 0.06	C_3–P_2	3.81 ± 0.04
P_2–C_5	1.851 ± 0.019	C_2–C_3	2.94 ± 0.05	C_4–P_1	3.59 ± 0.03
P_2–C_6	1.849 ± 0.024	C_4–C_5	2.84 ± 0.05	C_5–P_1	4.59 ± 0.02

X–Pt–X angles, deg		Pt–P–C angles, deg		C–P–C angles, deg	
P_1–Pt–P_2	96.2 ± 0.4	Pt–P_1–C_1	118.4 ± 1.3	C_1–P_1–C_2	99.5 ± 2.0
Cl_1–Pt–Cl_2	87.7 ± 0.3	Pt–P_1–P_2	115.7 ± 0.4	C_1–P_1–C_3	102.5 ± 2.0
Cl_1–Pt–P_1	91.3 ± 0.3	Pt–P_1–C_3	112.0 ± 1.1	C_2–P_1–C_3	107.1 ± 1.9
Cl_2–Pt–P_2	85.1 ± 0.4	Pt–P_2–C_4	123.5 ± 1.1	C_4–P_2–C_5	102.0 ± 1.5
P_1–Pt–Cl_2	174.5 ± 0.3	Pt–P_2–C_5	113.7 ± 0.8	C_4–P_2–C_6	96.8 ± 1.8
P_2–Pt–Cl_1	171.1 ± 0.4	Pt–P_2–C_6	111.1 ± 1.1	C_5–P_2–C_6	107.7 ± 1.3

II. Intermolecular

Shortest Pt–Pt, 6.326 A

All intermolecular distances are equal to or greater than normal van der Waals distances with van der Waals radii of Cl = 1.80 A and CH_3 = 2.0 A.

Deviation of Pt, P, and Cl from Best Least-Squares Plane,[b] $aX + bY + cZ - d = 0$

$a = +4.717$	Pt, +0.0002 ± 0.006 A
$b = -6.971$	Cl_1, +0.1209 ± 0.009 A
$c = -4.857$	Cl_2, −0.1451 ± 0.010 A
$d = +2.000$	P_2, +0.0763 ± 0.007 A
	P_1, −0.0726 ± 0.008 A

[a] With real and imaginary dispersion corrections. [b] Positional standard deviations were used to provide weights for the least-squares plane. *X*, *Y*, and *Z* refer to the monoclinic coordinate system.

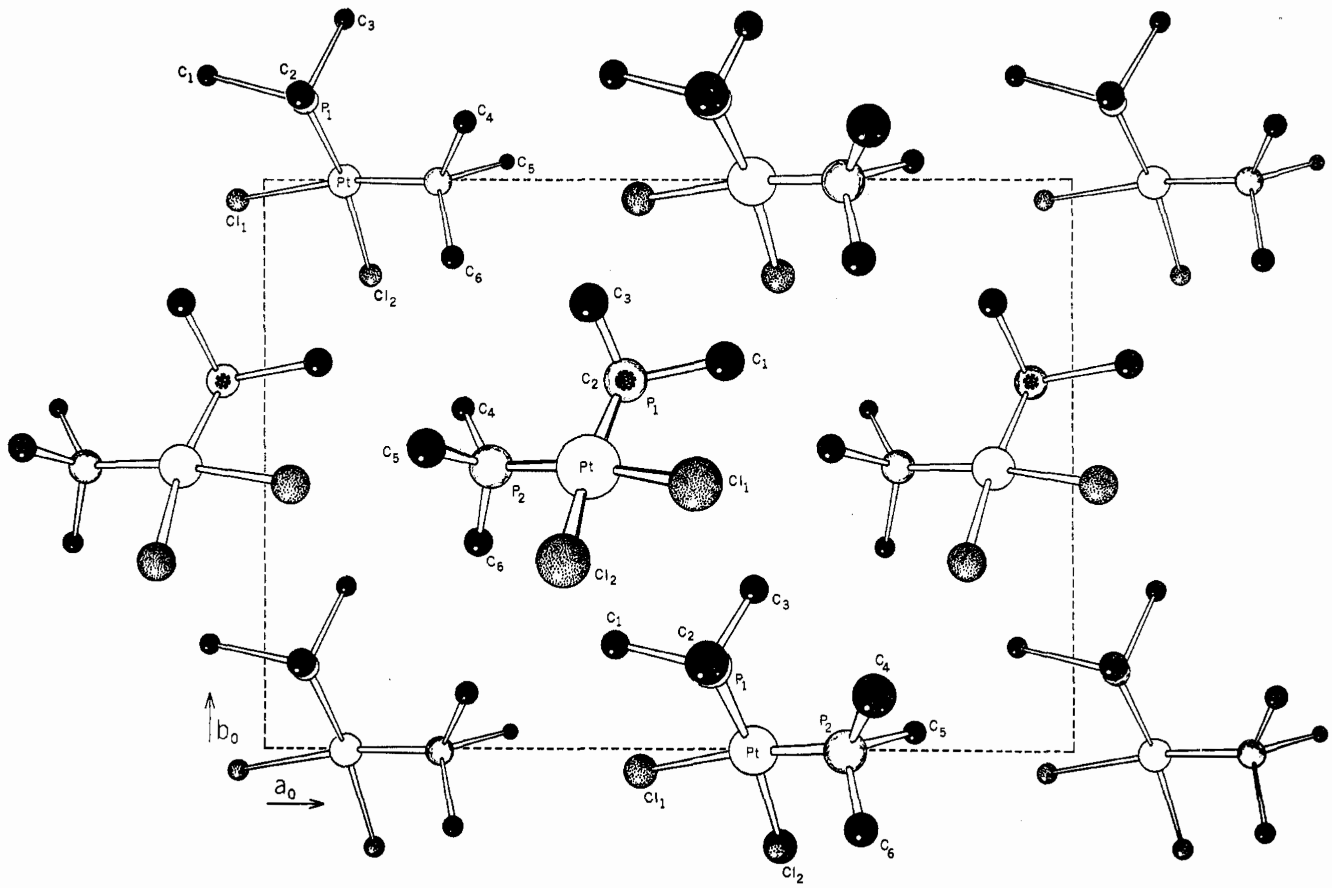


Figure 1.—Perspective view of the structure of *cis*-$Pt[P(CH_3)_3]_2Cl_2$ looking in the direction of negative *c*. C_2 is superposed in this view by P_1.

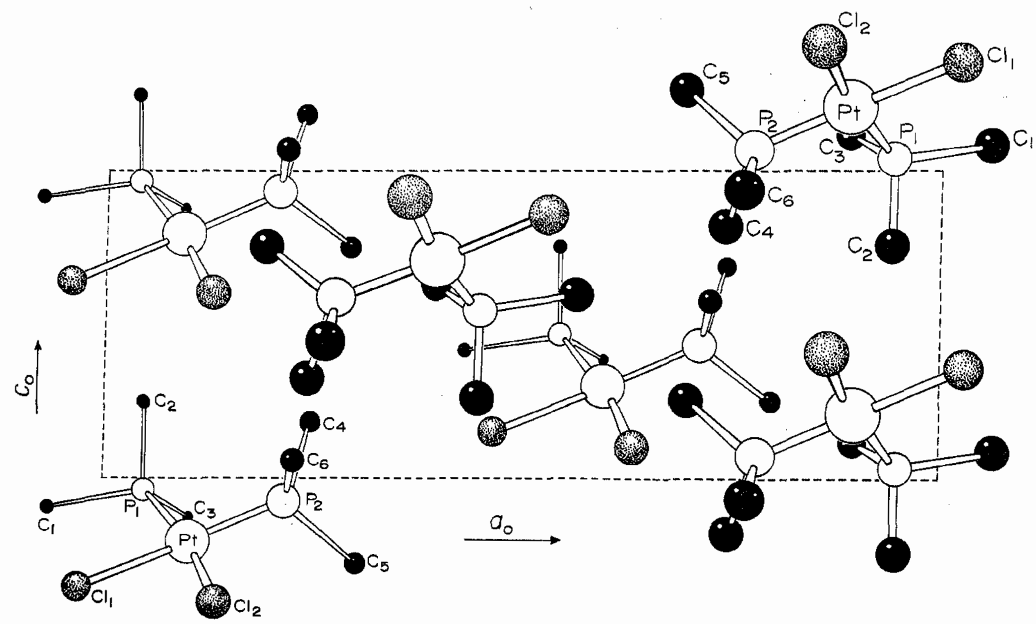


Figure 2.—Perspective view of the structure of *cis*-$Pt[P(CH_3)_3]_2Cl_2$ in the direction of positive *b*. For orientation purposes the "large" molecule approximately in the center in this view corresponds to the molecule approximately in the center of the cell in Figure 1.

atom. A Pt–P "normal" single-bond length[21] of 2.41 A would be expected from covalent radii. The average Pt–Cl distance of 2.376 A (2.364 ± 0.008, 2.388 ± 0.009 A) is significantly longer than the Pt–Cl distance of 2.294 ± 0.009 A in the *trans* isomer which is a "normal" single Pt–Cl bond. The Pt–Br distance of 2.428 ± 0.002 A in *trans*-$Pt[P(C_2H_5)_3]Br_2$ is also a "normal" Pt–X single bond. This 2.376 A distance is to be compared with the Pt–Cl distances of 2.422 ± 0.009 A observed by Eisenberg and Ibers[20] and 2.42 A by Wun-

(21) L. Pauling, "Nature of the Chemical Bond," 3rd ed, Cornell University Press, Ithaca, N. Y., 1960, p 246.

derlich and Mellor[22] in which the chlorine is *trans* to a strongly *trans* labilizing ligand, hydride and ethylene, respectively. However, Pt–Cl distances of 2.343 ± 0.013 and 2.316 ± 0.008 A were observed in dipentene-platinum(II) chloride.

The Pt, 2P, 2Cl best least-squares plane (Table III) indicates that Pt and its nearest neighbors do not lie in a plane and there exists a nonnegligible distortion toward a tetrahedral geometry. The P_1–Pt–P_2 angle is considerably greater than the ideal value of 90° (96.3 ± 0.3°). Further, Cl_1 is rather tightly packed to C_1 as is Cl_2 to C_5 and C_6. These facts suggest that the nonplanarity of the Pt, 2P, 2Cl entity is due primarily to steric effects. The general shortening of the Pt–P bond in going from the *trans* to the *cis* isomer is in line with the ideas of Chatt, Duncanson, and Venanzi,[23] as well as Craig, Maccoll, Nyholm, Orgel, and Sutton.[24]

The average value of 1.830 A for the phosphorus–carbon distances is nearly that predicted from covalent radii sums (with or without electronegativity corrections).

The C–C nonbonded intramolecular distances are as long or longer than those predicted by Bartell[25] based on a simple steric model assigning a nonbonded radius of 1.25 A to carbon atoms bonded to a common atom.

Although these results indicate a general shortening of Pt–P distances and lengthening of Pt–Cl distances from *trans*-Pt$[P(C_2H_5)_3]_2Cl_2$ and *trans*-Pt$[P(C_2H_5)_3]_2Br_2$ consistent with the thermodynamic results of Chatt and Wilkens[5] and the nuclear spin-coupling results of Pidcock, Richards, and Venanzi,[26] we do not feel it profitable in view of the molecular distortions to discuss the relative effects of π-bonding and steric factors to the *trans* effect without considerably more data on Pt–X bonds.

Acknowledgment.—We wish to acknowledge financial support from the National Institutes of Health, Grant No. GM-08344-04 and GM-13985-01.

(22) J. A. Wunderlich and D. P. Mellor, *Acta Cryst.*, **7**, 130 (1954).
(23) J. Chatt, L. A. Duncanson, and L. M. Venanzi, *J. Chem. Soc.*, 4456 (1955).
(24) D. P. Craig, A. Maccoll, R. S. Nyholm, L. E. Orgel, and L. E. Sutton, *ibid.*, 332 (1954).
(25) L. S. Bartell, *J. Chem. Phys.*, **32**, 827 (1960).
(26) A. Pidcock, R. E. Richards, and L. M. Venanzi, *Proc. Chem. Soc.*, 184 (1962).

CONTRIBUTION FROM THE NOYES CHEMICAL LABORATORY,
UNIVERSITY OF ILLINOIS, URBANA, ILLINOIS

Bis(1-phenyl-1,3-butanedionato)palladium(II). Crystal and Molecular Structure of the *trans* Form[1]

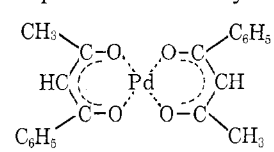
BY PING-KAY HON, C. E. PFLUGER, AND R. LINN BELFORD

Received October 14, 1966

The crystal structure of bis(1-phenyl-1,3-butanedionato)palladium(II) has been determined by three-dimensional Fourier methods and the atomic coordinates refined by three-dimensional least-squares methods with anisotropic temperature factors. The crystals are monoclinic, space group $P2_1/c$ with 2 molecules per unit cell. The cell dimensions are: a = 9.367 A, b = 10.518 A, c = 9.454 A; β = 108.00°. The structure can also be described with an end-centered unit cell having nearly orthogonal axes and 4 molecules. The molecules pack into layers parallel to the b axis. In each molecule the palladium and the four oxygens are exactly coplanar, as required by crystal symmetry, and the axial positions are occupied by two neighboring methyl groups 3.75 A from the palladium. The palladium–oxygen distance is 1.97 A. The carbon–oxygen distances are 1.23 and 1.31 A. The final residue R is 0.097 for 1377 reflections.

Introduction

Recently we reported crystal structures of the vanadyl[2] and copper[3] chelates of 1-phenyl-1,3-butanedione (benzoylacetone). Some differences in metal–oxygen bond lengths were tentatively explained in terms of the resonant and inductive effects of the phenyl group. To further explore this point, and also to study a known host crystal into which copper benzoylacetonate could be introduced for oriented crystal electron spin resonance studies, we have determined the crystal structure of bis(1-phenyl-1,3-butanedionato)palladium (commonly called palladium benzoylacetonate)



Experimental Section

The compound precipitated from an aqueous solution of palladium chloride in excess mixed with an ethanol solution of 1-phenyl-1,3-butanedione. The straw-yellow precipitate was washed with water and air dried. The powder dissolved readily in chloroform giving an orange-red solution which, when evaporated to dryness, gave orange crystals of rod and diamond shapes. The compound also dissolved moderately in acetone giving a yellow solution from which small yellow needlelike crystals resulted.

(1) Supported by the U. S. Public Health Service, under Institute of General Medical Sciences Grant GM-10907.
(2) P. K. Hon, R. L. Belford, and C. E. Pfluger, *J. Chem. Phys.*, **43**, 1323 (1965).
(3) P. K. Hon, C. E. Pfluger, and R. L. Belford, *Inorg. Chem.*, **5**, 516 (1966).