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 dipenteneplatinum(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.

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

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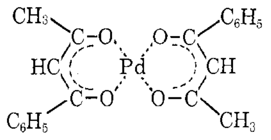
Received October 14, 1966

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.

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However, the powder patterns indicated all these crystals had the same structure regardless of their colors and external shapes. Microanalysis showed: C, 55.82% (calcd 55.99%); H, 4.11% (calcd 4.23%).

Preliminary cell parameters obtained from a rod-shaped crystal with rotation along the needle axis were: $a' = 11.0$ A, $b' = 10.5$ A, $c' = 15.2$ A, $\alpha \approx \beta \approx \gamma \approx 90°$. The diamond-shaped crystal rotating along one of the diagonal axes yielded monoclinic parameters as follows: $a = 9.367$ A, $b = 10.518$ A, $c = 9.454$ A; $\beta = 108.00°$. A close examination revealed that the a' and c' axes are just the diagonals of a parallelogram formed by the a and c axes and are not precisely perpendicular. The crystal belongs to a monoclinic space group (see below).

The diamond-shaped crystal was used for the subsequent experimental work, and the analysis used the monoclinic primitive cell. The crystal used in this study had the dimensions (along the external edges) 0.11 × 0.30 × 0.32 mm. Both molybdenum and copper radiations were employed to determine the unit cell parameters as mentioned above. The precession and Weissenberg cameras were calibrated with a sodium chloride crystal. Based on 2 molecules per unit cell, the calculated density was 1.608 g/ml, in excellent agreement with the experimental value, 1.609 g/ml, determined by flotation in potassium iodide solution. Systematic extinctions revealed the space group to be $P2_1/c$: $h0l$ absent for odd l, $0k0$ for odd k. The space group symmetry and the number of molecules per unit cell show the molecule to have the *trans* configuration with the palladium atom at a center of symmetry.

The diffraction zones ($h0l$) to ($h7l$) were recorded on multiple films by an integrating Weissenberg camera using the equiinclination geometry and Cu Kα radiation. Intensities of the 1377 observed reflections were measured on a modified Nonius microdensitometer utilizing the peak maximization method and were corrected in the usual way for Lorentz and polarization factors.

Structure Determination and Refinement

Since there are only two molecules per unit cell to occupy four general positions of the space group $P2_1/c$ the palladium atoms must be in special positions (0, 0, 0) and (0, $^1/_2$, $^1/_2$) or their equivalents. First we assumed all signs positive to calculate a two-dimensional Fourier projection from the corrected ($h0l$) reflections. Few of the oxygen and carbon peaks were clearly resolved in this projection. Next, calculation was shifted to the single atom (palladium), three-dimensional Fourier synthesis. There were a few extra peaks appearing in the resulting map. By setting up a three-dimensional model with reference to the expected molecular configuration one could easily determine the correct oxygen and carbon positions. Successive Fourier syntheses using these atomic coordinates showed large improvements of the R factor and confirmed the structure to be correct. After putting the structure factors of all layers on the same relative scale (the structure factor program summed the observed structure factors and scaled to the sum of the calculated structure factors for each layer), we made the refinements by least-squares techniques. The final R factor, $(\Sigma||F_o| - |F_c||)/\Sigma|F_o|$, was 0.097 for 1377 reflections, all equally weighted. All atoms were anisotropically refined.

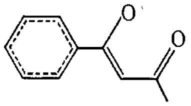
The atomic scattering factors were taken from Hanson, *et al.*,[4] for palladium and hydrogen, and from the International Tables for X-Ray Crystallography[5] for oxygen and carbon. The least-squares programs were from Gantzel, *et al.*[6] All calculations were performed by the IBM 7094 computer at the University of Illinois.

A stereoscopic view was constructed on a Calcomp plotter by the computer program ORTEP.[7]

Results and Discussion

The final observed and calculated structure factors are listed in Table I. Table II lists the atomic coordinates and the temperature factors. The bond lengths and bond angles are shown in Figure 1 and listed with the estimated standard deviations in Table III. Figures 2, 3, and 4 show the molecular skeletons projected along three different axes. Figure 5 is a stereoscopic perspective view of the crystal looking along the a^* axis.

The palladium and the four oxygens which form a parallelogram centered on the palladium are ocmpletely planar because a center of symmetry exists at the palladium atom. The difference between the two metal–oxygen bond lengths which appear in the vanadyl complex[2] and to a smaller extent in the copper complex[3] is not seen here. However, the pattern of bond lengths around the chelate ring is generally similar to that in the copper chelate and might be described roughly by the following single—and double—bond arrangement



Although the errors in this determination are such that the difference between the two carbon–oxygen bonds and the variation in the carbon–carbon bonds could well be exaggerated, the agreement between the corresponding copper[3] and palladium compounds increases our confidence. Averaging the two cases, the C_2–O_1 length is 1.238 ± 0.018 A, and the C_4–O_2 length is 1.295 ± 0.015 A. Moreover, the corresponding C–O distances in the analogous copper chelate in which the methyl group is replaced by phenyl are 1.30 ± 0.02 A.[8] Thus there is some indication that the carbon–oxygen bond adjacent to the methyl group has more double-bond character than the one adjacent to the phenyl group. As in the copper chelate, here the phenyl group is joined to the chelate ring by a bond somewhat shorter than the usual carbon–carbon single bond and is rotated about this linkage by an angle which is not large enough to destroy effective conjugation between phenyl and chelate rings. The angle of 23° in this case (Table IV) may be compared with 19° and 6.5° in the vanadyl chelate,[2] 14.3° in the copper chelate,[3]

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TABLE I

OBSERVED (F_o) AND CALCULATED (F_c) STRUCTURE FACTORS

h	$\lvert F_o \rvert$	F_c
k = 0 l = 0		
1	154.08	113.91
2	74.65	82.47
3	29.01	29.88
4	55.39	46.52
5	30.22	28.62
6	51.79	46.16
7	54.12	48.98
8	47.76	44.19
9	31.88	31.95
10	18.58	19.22
11	8.16	10.41
k = 0 l = -2		
11	9.02	10.12
10	15.61	15.28
9	20.68	19.25
8	40.44	37.66
7	30.52	29.45
6	17.24	16.88
5	43.47	43.27
4	65.55	66.72
3	102.94	107.01
2	92.24	97.35
1	206.31	183.80
k = 0 l = 2		
0	56.04	54.82
1	61.17	55.51
2	24.46	19.91
3	45.22	50.89
4	45.97	46.12
5	66.96	66.17
6	58.20	53.40
7	69.32	64.17
8	20.73	22.76
9	11.77	13.24
10	9.14	12.70
k = 0 l = -4		
11	7.75	8.38
10	16.32	15.07
9	21.09	20.44
8	25.87	23.71
7	28.78	29.44
6	45.66	45.90
5	30.41	28.09
4	31.64	32.08
3	39.62	41.83
2	43.65	50.52
1	78.14	76.09
k = 0 l = 4		
0	37.71	41.35
1	18.64	22.13
2	64.38	59.96
3	68.40	67.11
4	48.30	49.94
5	19.40	20.86
6	42.00	40.07
7	10.50	12.76
8	15.20	14.86
9	10.20	12.28
k = 0 l = -6		
11	12.36	14.34
10	14.28	14.51
9	18.93	18.78
8	16.83	17.58
7	20.62	19.64
6	4.67	4.95
5	22.36	22.81
4	38.75	35.30
3	35.93	32.00
2	60.36	57.01
1	48.17	46.67
k = 0 l = 6		
0	42.58	41.25
1	42.70	41.93
2	25.70	26.75
3	15.32	15.57
4	16.49	18.77
5	26.11	25.38
6	19.11	17.26
7	15.67	13.68
8	9.85	10.07
k = 0 l = -8		
10	6.87	6.23
9	8.10	7.67
8	14.51	14.27
7	14.73	11.54
6	17.24	15.81
5	24.25	21.17
4	38.34	35.69
3	37.77	34.48
2	29.48	25.63
1	17.12	15.17
k = 0 l = 8		
0	19.52	18.83
1	14.97	13.96
2	22.20	20.99
3	23.13	23.65
4	15.79	12.92
5	22.89	19.65
6	15.73	14.66
k = 0 l = -10		
9	6.41	8.67
8	10.32	10.77
7	8.45	9.08
6	11.71	11.65
5	18.87	19.69
4	10.77	8.74
3	10.02	7.66
2	13.34	10.57
1	20.55	18.49
k = 0 l = 10		
0	14.61	12.41
1	16.20	16.67
2	15.61	15.78
3	12.69	12.69
k = 0 l = -12		
5	6.18	6.18
4	5.47	6.25
3	6.18	7.08
2	6.00	9.94
k = 1 l = 0		
1	36.11	-34.60
2	21.14	19.81
3	1.06	3.17
4	7.73	-5.14
5	4.67	5.35
6	11.85	11.18
7	8.36	-8.56
8	9.61	-9.06
9	11.22	-12.82
10	3.24	-4.40
11	2.55	1.76
k = 1 l = -1		
11	10.85	12.48
10	15.34	15.87
9	25.07	26.00
8	31.39	29.20
7	28.07	27.23
6	47.53	44.11
5	25.14	25.48
4	46.04	49.34
3	41.80	48.55
2	122.37	151.04
1	84.15	95.04
k = 1 l = 1		
0	94.82	80.36
1	109.74	112.03
2	40.74	42.62
3	21.21	26.40
4	26.81	31.56
5	55.51	54.79
6	50.78	50.96
7	37.23	33.76
8	25.44	24.85
9	20.52	20.10
10	9.92	10.77
k = 1 l = -2		
11	6.87	-9.10
10	7.67	-9.18
9	6.18	-8.01
8	1.94	-2.09
7	11.04	9.41
6	8.30	7.25
5	8.87	9.58
4	6.92	11.58
3	33.51	-33.31
2	119.95	-119.50
1	38.06	-37.21
k = 1 l = 2		
0	.80	-1.36
1	31.31	-29.16
2	1.12	-4.87
3	7.49	4.60
4	1.49	2.17
5	1.69	.20
6	9.61	10.01
7	11.59	11.65
8	1.94	-2.59
9	1.69	1.46
10	1.31	1.43
k = 1 l = -3		
11	9.92	10.92
10	16.91	16.68
9	21.34	20.84
8	21.95	21.42
7	38.30	36.11
6	33.94	34.79
5	49.58	52.32
4	52.28	54.92
3	82.71	89.98
2	83.72	81.37
1	60.89	56.61
k = 1 l = 3		
0	35.85	36.98
1	70.05	68.04
2	42.23	41.69
3	61.00	60.62
4	46.41	48.67
5	58.26	61.33
6	30.50	29.15
7	19.77	19.38
8	20.72	20.54
9	10.79	11.11
10	8.18	10.24
k = 1 l = -4		
11	1.43	-2.43
10	3.55	-4.41
9	6.43	5.30
8	10.73	9.64
7	7.87	7.10
6	3.43	-2.60
5	13.53	-13.47
4	39.06	-36.86
3	27.07	-30.21
2	4.49	2.46
1	7.04	3.91
k = 1 l = 4		
0	35.75	-34.98
1	17.91	-16.20
2	3.92	-.34
3	10.73	9.94
4	15.91	16.30
5	27.38	29.25
6	2.00	4.38
7	6.24	-5.78
8	1.63	.41
k = 1 l = -5		
11	7.04	8.89
10	14.60	15.48
9	18.09	19.75
8	24.32	23.90
7	23.58	24.79
6	32.99	32.47
5	42.61	40.11
4	32.56	34.06
3	49.65	50.15
2	52.97	50.01
1	40.66	36.16
k = 1 l = 5		
0	73.67	68.43
1	46.15	44.37
2	51.16	51.78
3	34.06	32.41
4	44.36	46.26
5	23.27	24.20
6	17.34	18.20
7	17.34	17.30
8	17.22	16.54
k = 1 l = -6		
11	1.31	-.58
10	3.32	4.03
9	1.86	-.39
8	1.94	-3.63
7	1.94	.49
6	7.04	3.46
5	1.80	-4.11
4	5.67	1.09
3	1.63	-1.55
2	18.65	16.84
1	25.32	22.76
k = 1 l = 6		
0	1.69	.97
1	17.03	18.62
2	9.24	9.36
3	1.94	3.74
4	3.43	4.96
5	7.92	-6.58
6	1.75	-.73
7	4.06	-2.93
k = 1 l = -7		
11	6.73	8.75
10	9.73	12.21
9	13.16	13.99
8	10.53	11.64
7	14.34	15.04
6	22.03	22.18
5	24.64	22.98
4	31.25	31.82
3	40.30	39.27
2	32.19	32.39
1	37.43	36.21
k = 1 l = 7		
0	27.64	26.88
1	30.62	29.23
2	23.83	24.47
3	24.32	24.70
4	20.89	22.91
5	20.15	20.37
6	14.10	14.19
7	16.59	16.31
k = 1 l = -8		
10	1.31	.22
9	1.63	.89
8	5.12	4.16
7	1.94	3.24
6	1.94	2.01
5	2.00	1.12
4	1.94	-1.55
3	5.49	5.59
2	1.94	-4.47
1	5.49	-7.63
k = 1 l = 9		
0	14.91	15.65
1	19.08	20.17
2	15.10	16.31
3	17.60	19.52
4	13.91	15.14
3	8.79	9.98
2	6.73	7.81
1	9.30	10.84
k = 1 l = 8		
0	1.94	.85
1	10.04	-7.70
2	4.75	-.31
3	3.69	-2.70
4	3.32	-3.43
5	1.37	-1.73
6	2.75	-3.19
k = 1 l = -9		
10	4.86	7.77
9	5.43	5.61
8	6.49	8.06
7	11.53	12.80
6	19.21	19.99
5	14.42	15.33
4	19.66	19.96
3	17.28	18.83
2	16.59	17.12
1	12.42	13.34
k = 1 l = -10		
7	1.55	2.15
6	4.43	-4.71
5	4.61	-4.42
4	1.80	1.37
3	1.80	1.03
2	1.80	.76
1	3.86	2.63
k = 1 l = 10		
0	1.69	-1.46
1	1.49	-.50
k = 1 l = -11		
7	8.04	11.25
6	5.55	6.85
5	6.92	8.74
4	7.18	8.80
k = 1 l = 11		
0	10.85	13.39
1	9.55	11.96
k = 2 l = 0		
1	11.32	5.07
2	92.95	100.68
3	55.36	63.41
4	68.73	69.51
5	45.69	43.30
6	35.70	35.13
7	34.20	32.18
8	22.01	20.26
9	8.73	7.19
10	16.62	15.38
11	8.34	10.86
k = 2 l = -1		
11	1.16	1.30
10	2.65	2.54
9	3.51	-4.09
8	1.79	-2.39
7	4.49	-3.86
6	9.65	9.09
5	35.35	32.66
4	22.54	20.79
3	17.70	-15.97
2	3.96	2.83
1	31.39	28.04
k = 2 l = 1		
0	50.06	46.44
1	.69	2.93
2	3.73	-.01
3	22.25	-22.98
4	11.55	10.45
5	9.65	-8.01
6	1.61	-1.81
7	5.57	-5.84
8	1.79	.93
9	1.67	3.73
10	1.31	-.64
k = 2 l = -2		
11	15.40	17.06
10	17.42	18.73
9	18.79	18.98
8	21.89	20.92
7	34.08	31.40
6	42.83	41.50
5	51.32	55.19
4	45.57	48.43
3	84.67	90.86
2	10.51	13.19
1	88.50	86.45
k = 2 l = 2		
0	83.97	71.49
1	51.61	59.78
2	52.59	54.06
3	77.01	83.80
4	48.74	55.93
5	38.27	39.91
6	22.82	20.51
7	20.82	18.89
8	10.75	10.31
9	21.03	20.39
10	10.01	11.77
k = 2 l = -3		
10	1.61	-2.46
9	1.79	1.93
8	1.79	4.89
7	15.99	16.89
6	13.61	14.73
5	6.73	7.28
4	29.60	-29.52
3	3.79	-8.05
2	18.97	-17.65
1	4.20	-.40
k = 2 l = 3		
0	27.70	-22.87
1	33.86	-29.23
2	45.35	-43.35
3	3.28	2.03
4	11.50	12.38
5	5.24	6.19
6	10.34	-10.77
7	1.79	2.92
8	1.67	-1.36
9	1.31	-3.95
k = 2 l = -4		
11	1.26	14.70
10	10.92	12.29
9	13.10	13.12
8	27.42	26.65
7	24.03	24.14
6	35.06	32.91
5	46.67	48.25
4	83.32	87.40
3	49.43	49.30
2	49.02	48.51
1	51.61	51.87
k = 2 l = -7		
0	1.37	-.69
9	1.61	1.98
8	1.73	-.00
7	8.04	-8.16
6	1.79	-2.96
5	1.73	.10
4	8.40	9.56
3	6.26	9.08
2	1.67	-.32
1	1.67	.42
k = 2 l = 7		
0	10.57	8.32
1	9.14	8.25
2	14.02	13.25
3	6.20	-5.10
4	3.45	-5.24
5	7.87	-9.09
k = 7 l = -10		
4	4.47	5.80
k = 2 l = 4		
0	58.96	56.22
1	66.92	64.15
2	60.99	60.47
3	35.91	37.26
4	43.52	46.94
5	26.09	25.84
6	32.13	29.85
7	18.85	19.30
8	18.28	17.71
9	12.06	13.43
k = 2 l = -5		
11	1.22	2.09
10	1.55	-.31
9	1.73	1.74
8	3.63	6.75
7	1.79	.89
6	11.73	-9.21
5	17.99	-18.58
4	9.26	-9.57
3	1.37	-2.53
2	31.15	31.63
1	19.71	19.80
k = 2 l = 5		
0	10.87	-14.29
1	1.43	-1.92
2	10.63	8.83
3	23.23	23.54
4	1.79	1.75
5	7.81	-7.31
6	1.73	-3.36
7	5.98	-7.21
k = 2 l = -6		
11	4.59	7.74
10	5.12	6.07
9	14.14	15.13
8	17.70	18.23
7	29.37	29.62
6	30.98	32.18
5	34.21	38.44
4	35.70	35.99
3	29.43	31.16
2	33.17	33.42
1	41.85	40.81
k = 2 l = 6		
0	37.25	35.20
1	36.51	35.21
2	41.66	39.36
3	47.59	50.10
4	21.44	21.37
5	21.67	21.29
6	20.52	21.61
7	16.89	16.99
k = 2 l = -8		
10	6.79	10.10
9	7.92	10.55
8	11.20	13.45
7	10.75	12.39
6	14.77	17.12
5	23.28	24.81
4	18.10	17.79
3	13.34	13.01
2	25.23	28.59
1	25.81	28.09
k = 2 l = 8		
0	25.41	26.25
1	21.67	22.97
2	22.99	24.73
3	15.70	18.13
4	15.99	17.67
5	13.51	15.20
k = 2 l = -9		
9	1.22	2.48
8	1.43	-1.19
7	1.61	-1.49
6	1.73	-1.17
5	1.73	4.73
4	1.79	-2.49
3	1.79	-1.04
2	1.79	.28
1	1.79	-2.33
k = 2 l = 9		
0	1.73	4.51
1	1.67	-1.17
2	1.55	-4.02
3	1.37	-1.59
k = 2 l = -10		
9	2.12	5.60
8	3.90	6.58
7	5.81	8.67
6	10.51	11.69
5	11.61	13.05
4	11.26	13.25
3	14.14	17.79
2	9.65	12.06
1	9.65	12.30
k = 2 l = 10		
0	9.02	12.39
1	10.45	13.45
2	11.61	14.37
3	6.90	9.25
k = 3 l = 0		
1	14.30	-7.86
2	10.48	9.85
3	26.11	24.05
4	9.83	7.14
5	18.26	-18.30
6	1.65	-2.39
7	5.20	-4.24
8	4.08	4.50
9	5.73	4.94
10	5.00	6.03
k = 3 l = -1		
11	8.49	10.13
10	13.04	14.56
9	20.11	22.09
8	22.82	21.97
7	33.48	32.70
6	49.23	47.50
5	63.68	62.68
4	78.63	82.37
3	52.39	53.19
2	47.19	51.32
1	77.44	85.43
k = 3 l = 1		
0	51.74	49.99
1	83.90	75.27
2	92.43	95.89
3	99.66	104.58
4	74.50	75.99
5	47.73	45.25
6	30.39	31.64
7	21.76	19.79
8	32.90	32.65
9	19.44	20.42
10	13.97	15.50
k = 3 l = -2		
11	3.24	4.39
10	2.43	3.06
9	4.47	-.78
8	2.90	-2.77
7	1.71	.95
6	7.45	6.05
5	3.10	2.18
4	2.71	-2.80
3	34.41	32.62
2	10.02	10.03
1	12.46	9.60
k = 3 l = 2		
0	20.55	16.29
1	23.60	-17.45
2	13.51	13.76
3	10.55	-11.58
4	15.81	-16.53
5	6.98	5.53
6	1.79	-1.32
7	1.84	-3.68
8	3.88	-3.88
9	1.51	2.34
k = 3 l = -3		
11	8.71	9.62
10	12.71	14.08
9	23.46	23.40
8	16.34	16.19
7	24.52	23.17
6	34.66	35.78
5	57.34	59.28
4	26.36	30.93
3	30.05	33.99
2	65.12	70.74
1	52.25	49.19
k = 3 l = 3		
0	85.35	81.59
1	85.68	80.63
2	61.49	62.59
3	51.94	53.49
4	30.58	31.41
5	25.83	26.59
6	26.84	25.61
7	31.05	29.15
8	22.29	22.45
9	18.99	20.64
k = 3 l = -4		
11	1.18	-.39
10	1.59	2.01
9	2.77	2.70
8	1.84	-2.77
7	6.79	7.15
6	1.65	.93
5	1.51	-2.97
4	4.36	5.93
3	4.94	-7.27
2	1.18	.14
1	11.92	8.23
k = 3 l = 4		
0	29.13	-29.23
1	6.06	6.84
2	7.98	8.24
3	4.94	6.04
4	7.57	-8.55
5	2.83	-5.06
6	6.40	-4.87
7	1.71	-2.05
8	3.24	-4.41
k = 3 l = -5		
11	6.06	7.12
10	14.24	13.76
9	8.63	7.60
8	17.66	15.61
7	27.15	26.56
6	39.35	38.87
5	26.75	25.73
4	33.35	33.11
3	43.32	42.26
2	47.79	47.71
1	36.83	34.27
k = 3 l = 5		
0	40.02	39.36
1	40.60	39.82
2	50.69	50.71
3	22.87	21.77
4	18.59	18.25
5	30.78	29.70
6	23.93	22.52
7	21.95	20.70
8	14.69	15.66
k = 3 l = -6		
11	1.06	1.97
10	1.45	3.32
9	1.65	1.42
8	1.79	2.89
7	6.06	-4.34
6	12.20	-10.31
5	13.32	-10.27
4	3.16	4.72
3	12.65	11.36
2	11.20	9.81
1	3.49	-2.22
k = 3 l = 6		
0	1.59	-1.49
1	8.57	5.71
2	13.12	10.55
3	4.02	-3.41
4	1.79	-.30
5	6.40	-5.65
6	1.51	-3.57
7	3.55	-4.38
k = 3 l = -7		
11	6.12	8.50
10	7.45	7.83
9	12.20	11.70
8	17.54	17.82
7	23.46	24.53
6	24.85	22.95
5	21.95	19.04
4	13.85	12.21
3	36.65	32.61
2	24.52	20.64
1	34.14	31.54
k = 3 l = 7		
0	33.54	31.93
1	40.73	38.47
2	19.05	16.92
3	20.03	19.38
4	23.27	22.77
5	15.70	14.56
6	13.77	13.33
k = 3 l = -8		
9	4.75	-4.53
8	4.02	-3.84
7	1.71	-1.95
6	1.79	-2.95
5	1.84	2.24
4	6.40	5.59
3	6.40	7.64
2	1.79	-1.60
1	1.84	2.65
k = 3 l = 8		
0	12.85	9.93
1	11.01	8.91
2	1.71	-1.64
3	4.08	-5.55
4	3.96	-2.84
k = 3 l = -9		
9	8.63	9.93
8	10.94	12.30
7	12.06	10.66
6	10.28	7.72
5	8.91	8.97
4	25.63	23.08
3	19.38	16.31
2	21.76	20.00
1	22.60	21.62
k = 3 l = 11		
0	7.32	8.24
k = 4 l = 0		
1	44.82	54.08
2	64.41	74.97
3	67.08	69.11
4	62.40	61.13
5	50.46	48.26
6	38.57	37.23
7	23.28	22.00
8	24.62	22.73
9	18.79	17.81
10	20.93	21.40
k = 3 l = 9		
0	25.19	24.20
1	15.42	14.08
2	11.48	12.00
3	9.75	8.36
4	14.97	13.84
k = 3 l = -10		
7	1.31	-.10
6	1.45	.83
5	4.22	4.08
4	1.59	2.57
3	1.59	-2.20
2	3.96	-3.08
1	1.51	-2.33
k = 3 l = 10		
0	1.45	-1.08
1	1.31	.82
2	1.06	-.86
k = 3 l = -11		
7	4.75	5.66
6	6.40	7.45
5	9.75	10.70
4	9.69	9.78
3	11.14	11.61
2	10.48	11.73
1	8.30	9.84
k = 4 l = -1		
10	1.41	-4.51
9	1.67	-1.37
8	1.80	.53
7	2.83	-2.92
6	17.99	-16.18
5	13.89	-13.62
4	1.28	-1.30
3	11.81	12.32
2	.86	-3.41
1	16.97	-10.64
k = 4 l = 1		
0	4.22	4.72
1	21.46	16.29
2	12.89	10.03
3	19.38	-17.84
4	14.16	12.10
5	7.98	-5.16
6	1.75	-3.83
7	1.80	5.09
8	1.75	3.62
9	1.55	-2.29
10	1.14	-1.07
k = 4 l = -2		
11	7.57	11.66
10	10.20	9.86
9	16.36	16.41
8	25.28	24.40
7	44.08	41.63
6	36.16	32.65
5	52.45	51.60
4	39.12	39.24
3	72.99	74.33
2	27.03	26.99
1	56.50	55.82
k = 4 l = 2		
0	63.40	60.75
1	91.72	92.52
2	81.45	85.93
3	40.86	44.37
4	35.96	36.19
5	30.32	28.54
6	30.13	28.50
7	31.93	30.38
8	23.90	22.60
9	23.21	22.13
10	10.26	14.50
k = 4 l = -3		
11	1.08	-.61
10	1.47	-3.45
9	5.85	-4.42
8	1.80	-.75
7	7.65	-8.57
6	15.63	-9.60
5	17.38	18.28
4	4.43	2.56
3	24.95	-21.45
2	10.87	-5.82
1	6.71	3.31
k = 4 l = 3		
0	17.99	16.20
1	26.03	23.67
2	5.43	-4.93
3	8.85	5.46
4	6.98	-5.24
5	4.77	4.50
6	11.06	11.18
7	1.75	3.43
8	1.55	-.14
9	3.22	1.56
k = 4 l = -4		
11	8.65	9.76
10	12.55	11.76
9	16.97	16.92
8	30.66	28.52
7	27.31	25.91
6	35.15	37.68
5	17.30	17.83
4	44.82	42.81
3	20.79	21.78
2	51.79	53.92
1	56.89	55.75
k = 4 l = 4		
0	73.51	73.98
1	49.31	47.81
2	40.66	40.92
3	31.13	30.53
4	29.92	31.13
5	22.07	20.52
6	27.24	25.57
7	25.09	24.61
8	22.15	21.28
9	.75	14.79
k = 4 l = -5		
11	1.00	-3.23
10	1.41	-.71
9	8.18	6.53
8	1.80	1.67
7	4.96	-5.96
6	8.73	7.90
5	5.49	5.17
4	3.02	-3.93
3	7.57	-6.37
2	6.24	-6.33
1	1.41	.83
k = 4 l = 5		
0	1.47	2.76
1	1.55	.50
2	8.26	-8.02
3	1.75	-.67
4	5.37	7.69
5	1.80	1.82
6	1.67	-1.97
7	3.55	4.49
8	4.02	4.57
k = 4 l = -6		
11	5.37	6.90
10	9.53	10.88
9	16.30	16.89
8	16.24	16.91
7	16.83	17.81
6	17.24	16.88
5	28.50	30.25
4	31.13	27.29
3	29.05	27.01
2	34.15	29.56
1	48.85	51.79
k = 4 l = 6		
0	38.23	36.93
1	37.65	38.62
2	15.22	13.64
3	27.44	26.63
4	17.18	17.54
5	23.67	23.80
6	18.91	18.14
7	17.18	15.96
k = 4 l = -7		
10	1.22	2.54
9	1.47	.14
8	1.67	-1.69
7	1.80	-.04
6	2.88	3.94
5	1.80	.61
4	1.75	-4.63
3	1.75	1.24
2	16.97	12.87
1	4.43	.17
k = 4 l = 7		
0	8.73	11.25
1	11.95	-10.08
2	6.71	-5.70
3	1.75	2.12
4	1.61	1.30
5	1.41	.44
k = 4 l = -8		
10	6.24	8.04
9	10.06	11.70
8	12.89	13.25
7	15.30	15.73
6	14.50	14.46
5	18.44	16.33
4	14.75	14.17
3	22.54	21.40
2	30.72	28.34
1	25.62	23.59
k = 4 l = 8		
0	23.81	24.80
1	15.22	14.67
2	21.60	21.60
3	12.20	12.60
4	15.09	13.26
5	12.55	12.35
k = 4 l = -9		
8	1.33	-2.52
7	1.47	3.51
6	4.43	4.74
5	1.67	.73
4	1.75	-.64
3	1.75	2.17
2	1.75	-1.90
1	1.75	-3.52
k = 4 l = 9		
0	4.57	-4.90
1	1.55	-2.06
2	1.41	.06
3	1.22	.32
k = 4 l = -10		
8	5.90	8.10
7	5.57	8.12
6	7.12	8.67
5	6.90	6.74
4	9.79	9.46
3	15.30	14.22
2	18.85	19.23
1	15.36	15.81
k = 4 l = 10		
0	10.34	11.10
1	7.57	8.70
2	8.26	10.25
k = 5 l = 0		
1	16.49	19.96
2	7.26	6.84
3	6.63	6.27
4	13.04	-14.86
5	6.49	3.21
6	1.51	5.43
7	1.59	-2.41
8	3.45	-2.63
9	2.49	1.45
10	1.86	1.28
k = 5 l = -1		
10	10.85	11.48
9	15.95	15.21
8	20.03	16.57
7	24.58	21.46
6	34.73	31.25
5	49.02	48.32
4	47.93	51.16
3	49.37	56.53
2	39.91	41.74
1	27.13	27.59
k = 5 l = 1		
0	29.21	36.13
1	70.42	75.85
2	45.09	50.10
3	45.43	49.65
4	42.53	46.36
5	26.93	25.74
6	32.25	30.14
7	18.09	14.12
8	16.91	14.38
9	15.26	13.81
10	13.53	15.14
k = 5 l = -2		
10	1.18	2.04
9	1.45	1.57
8	7.81	-5.30
7	1.51	-.88
6	10.77	9.87
5	3.37	-2.49
4	1.18	3.41
3	4.28	8.16
2	.82	.62
1	24.52	-18.53
k = 5 l = 2		
0	16.63	15.38
1	6.28	-6.46
2	3.53	-1.57
3	5.10	-5.55
4	10.85	-11.59
5	1.45	-.05
6	1.59	1.04
7	1.51	3.47
8	6.77	4.85
9	1.10	-.25
k = 5 l = -3		
11	7.45	11.19
10	11.04	12.02
9	19.26	16.42
8	20.93	20.84
7	44.80	41.18
6	33.29	33.27
5	35.49	34.10
4	40.86	42.15
3	42.06	39.08
2	49.16	50.03
1	48.88	48.57
k = 5 l = -4		
11	.76	1.97
10	1.18	.50
9	1.45	-5.61
8	1.51	-2.57
7	8.55	8.07
6	3.86	3.14
5	11.53	-10.37
4	5.81	8.31
3	6.98	7.85
2	4.69	-5.13
1	19.95	-22.80
k = 7 l = 9		
0	13.73	13.82
1	13.44	14.64
2	11.40	13.97
k = 5 l = 3		
0	61.52	61.65
1	44.05	46.78
2	58.06	59.52
3	39.15	42.94
4	24.58	25.95
5	30.17	29.50
6	22.65	20.82
7	23.07	21.32
8	20.09	19.77
9	12.16	12.30
k = 5 l = 4		
0	2.14	1.96
1	17.54	-18.36
2	7.94	-7.65
3	7.67	-9.27
4	1.51	3.28
5	4.96	4.39
6	5.18	5.42
7	4.28	2.56
8	1.04	-1.68
k = 5 l = -5		
11	5.45	10.24
10	12.50	14.65
9	12.85	14.99
8	20.79	23.31
7	14.16	14.20
6	26.52	27.36
5	33.43	32.12
4	34.38	35.34
3	37.85	35.75
2	45.78	46.00
1	41.92	44.35
k = 5 l = 5		
0	50.14	50.04
1	39.29	34.85
2	27.40	26.31
3	32.67	33.21
4	31.08	32.55
5	26.11	25.84
6	19.89	18.99
7	18.30	19.16
k = 5 l = -6		
10	3.04	-3.29
9	3.81	-5.29
8	1.45	.69
7	6.98	7.21
6	1.59	-.18
5	5.94	5.42
4	1.51	3.41
3	1.45	.12
2	13.46	-15.13
1	4.63	-3.54
k = 5 l = 6		
0	13.26	-11.84
1	4.83	-3.61
2	1.59	1.03
3	1.59	-4.51
4	1.51	-.84
5	5.40	5.47
6	4.49	4.93
k = 5 l = -7		
10	4.63	6.68
9	7.59	10.48
8	10.14	10.69
7	15.81	17.21
6	17.26	16.76
5	23.62	27.35
4	24.30	25.62
3	30.45	30.72
2	15.75	17.32
1	32.31	30.93
k = 5 l = 7		
0	19.95	16.79
1	28.31	27.53
2	26.79	28.71
3	19.13	20.63
4	16.58	17.71
5	17.47	18.46
6	14.71	16.68
k = 5 l = -8		
9	1.04	1.20
8	5.10	5.07
7	1.37	2.36
6	1.45	.59
5	1.51	3.78
4	1.51	1.38
3	7.53	-10.40
2	8.42	-10.17
1	5.67	3.92
k = 5 l = 8		
0	11.53	-11.08
1	1.51	-.97
2	2.55	-2.89
3	1.24	1.16
4	1.04	.80
k = 5 l = -9		
9	4.00	7.79
8	7.53	10.13
7	8.28	11.55
6	11.12	16.22
5	7.04	10.10
4	7.67	10.88
3	9.59	10.92
2	17.61	21.53
1	12.08	11.99
k = 5 l = 9		
0	16.63	18.05
1	11.95	14.54
2	11.73	15.92
3	10.50	13.36
k = 5 l = -10		
7	.90	.43
6	1.10	.50
5	1.18	-2.43
4	3.10	-3.61
3	2.77	-3.21
2	2.69	2.58
1	5.86	6.49
k = 5 l = 10		
0	1.04	-1.53
1	1.65	-1.05
k = 5 l = -11		
5	3.73	7.20
4	6.14	9.64
3	6.00	10.02
2	5.67	8.85
k = 6 l = 0		
1	21.95	33.17
2	48.73	53.50
3	53.32	53.66
4	60.07	58.37
5	53.19	43.44
6	38.53	32.64
7	23.54	21.10
8	22.01	21.74
9	1.33	12.30
10	.92	10.66
k = 6 l = -1		
7	7.89	2.90
6	10.34	-11.93
5	14.46	11.86
4	10.34	10.07
3	1.12	-4.89
2	15.12	17.45
1	11.87	13.08
k = 6 l = 1		
0	5.77	-5.37
1	17.83	18.37
2	3.59	5.98
3	1.20	3.08
4	7.36	-6.72
5	13.53	-14.01
k = 6 l = -2		
10	10.42	11.18
9	18.50	19.20
8	22.29	18.55
7	28.64	24.24
6	29.78	29.70
5	49.86	49.63
4	29.24	25.96
3	33.22	34.45
2	36.60	36.24
1	71.15	68.53
k = 6 l = 2		
0	50.26	46.75
1	55.78	54.66
2	51.79	52.59
3	45.61	50.51
4	41.85	43.21
5	29.90	30.54
6	19.03	18.29
7	23.48	23.40
8	15.46	16.47
9	12.73	14.30
k = 6 l = -3		
8	8.55	6.40
7	7.89	-8.41
6	1.59	-3.20
5	1.53	1.19
4	12.20	11.73
3	13.32	12.81
2	11.06	10.38
1	1.12	-3.89
k = 6 l = 3		
0	3.98	-1.70
1	6.96	8.27
2	15.12	-12.33
3	12.60	-9.63
4	1.59	.22
5	10.01	-10.50
6	3.65	-4.45
7	1.53	-.33
8	3.85	3.97
k = 6 l = -4		
10	11.73	14.74
9	15.71	15.01
8	13.93	13.21
7	11.53	10.98
6	31.96	30.61
5	24.60	20.53
4	40.51	37.71
3	44.74	41.53
2	58.33	59.63
1	39.26	39.64
k = 6 l = 4		
0	38.92	37.11
1	31.37	29.54
2	34.35	33.91
3	28.25	26.19
4	27.77	29.28
5	25.52	26.52
6	28.50	27.72
7	14.26	15.45
8	10.75	12.05
k = 6 l = -5		
9	4.37	-3.46
8	2.51	-4.66
7	6.16	-6.58
6	1.65	2.32
5	9.55	11.78
4	1.33	.63
3	1.53	4.53
2	1.45	1.87
1	8.36	7.76
k = 6 l = 5		
0	20.29	21.44
1	2.45	-1.93
2	9.22	-7.47
3	4.57	-5.55
4	1.65	-5.94
5	1.59	.96
k = 6 l = -6		
10	5.57	8.62
9	5.43	6.72
8	8.89	7.99
7	18.69	21.19
6	23.27	21.05
5	27.58	27.26
4	28.50	29.47
3	101.83	41.28
2	26.52	26.04
1	16.44	13.45
k = 6 l = 6		
0	11.81	8.20
1	35.13	31.54
2	28.91	25.98
3	29.44	31.03
4	22.01	25.05
5	21.62	22.30
6	7.89	12.22
k = 6 l = -7		
6	1.59	7.58
5	1.65	4.83
4	1.65	-1.27
3	7.89	-6.96
2	5.90	-5.39
1	6.43	-5.71
k = 6 l = -8		
9	4.90	5.92
8	8.28	10.47
7	13.26	15.32
6	14.38	15.73
5	14.99	15.89
4	16.38	19.57
3	15.51	14.79
2	10.94	10.54
1	9.22	11.73
k = 6 l = 8		
0	25.72	26.65
1	18.97	19.24
2	17.70	21.60
3	14.07	17.86
4	12.60	16.24
k = 6 l = -9		
5	1.39	-4.35
4	1.45	-3.14
3	1.45	-1.32
2	4.04	5.76
1	1.45	-1.76
k = 6 l = 9		
0	1.39	-1.22
1	3.65	-5.86
k = 6 l = -10		
7	3.71	5.68
6	6.04	7.70
5	7.22	12.02
4	8.83	13.10
3	7.69	9.29
2	7.43	8.51
1	8.94	13.17
k = 6 l = 10		
0	8.89	13.67
k = 7 l = 0		
1	11.40	-13.23
2	4.20	-7.58
3	11.20	12.31
4	12.32	-14.70
5	1.86	-2.84
6	1.96	-1.40
7	1.86	1.97
8	1.69	.84
9	1.39	.05
k = 7 l = 8		
0	1.77	-4.71
1	1.69	.98
2	1.49	-1.04
3	1.22	1.88
k = 7 l = -1		
10	9.99	11.85
9	11.20	13.32
8	25.48	21.98
7	43.33	37.94
6	48.28	38.56
5	21.95	21.58
4	30.45	30.13
3	28.66	30.62
2	45.84	45.99
1	55.93	56.29
k = 7 l = 1		
0	54.06	58.16
1	50.98	50.25
2	42.96	49.04
3	39.41	46.24
4	30.54	33.06
5	11.20	31.05
6	34.92	31.38
7	30.54	25.67
8	21.66	19.65
9	15.22	14.22
k = 7 l = -2		
10	1.02	1.34
9	1.49	1.76
8	1.77	-.53
7	1.96	-3.50
6	7.75	-8.84
5	21.21	-22.51
4	1.59	4.08
3	5.79	4.73
2	10.28	8.41
1	13.73	11.15
k = 7 l = 2		
0	9.99	-10.27
1	6.36	3.53
2	10.55	16.23
3	8.77	13.40
4	1.77	-.63
5	1.96	.59
6	1.86	1.20
7	1.77	-3.32
8	1.49	-3.12
k = 7 l = -3		
10	11.57	12.20
9	21.38	18.03
8	23.34	23.75
7	25.41	20.81
6	11.87	9.09
5	18.20	17.45
4	27.37	27.63
3	42.49	39.46
2	48.65	44.11
1	60.05	57.69
k = 7 l = 3		
0	61.81	60.57
1	42.58	43.77
2	17.36	14.70
3	20.73	22.21
4	24.74	24.60
5	32.77	31.45
6	26.62	26.57
7	21.48	20.39
8	16.91	14.51
k = 7 l = -4		
10	1.02	2.27
9	1.49	.15
8	1.77	-3.87
7	9.81	-8.35
6	1.96	-2.82
5	1.86	3.33
4	11.20	12.00
3	6.81	7.60
2	7.75	7.24
1	6.53	-2.27
k = 7 l = 4		
0	8.96	10.41
1	8.87	12.86
2	19.24	22.44
3	9.53	-9.51
4	8.22	-7.03
5	1.86	-3.77
6	1.69	-2.17
7	1.39	-1.69
k = 7 l = -5		
10	.94	8.56
9	11.20	10.75
8	17.18	16.60
7	17.93	11.76
6	14.10	10.16
5	24.83	22.27
4	39.12	39.55
3	39.41	38.71
2	30.99	33.89
1	28.01	29.81
k = 7 l = 5		
0	20.54	20.59
1	10.28	8.82
2	26.05	25.81
3	25.11	24.55
4	27.73	26.72
5	22.50	23.55
6	22.03	20.78
7	12.99	13.06
k = 7 l = -6		
9	1.31	.44
8	1.59	-3.09
7	1.77	3.94
6	1.86	-.65
5	1.96	6.31
4	1.96	-.14
3	1.86	1.38
2	4.94	-6.67
1	1.86	-3.67
k = 6 l = 6		
0	1.96	.84
1	1.96	1.34
2	4.30	-8.32
3	1.86	-4.20
4	1.69	-1.84
5	1.39	-1.31
k = 7 l = -7		
9	9.53	11.97
8	12.24	9.81
7	13.26	10.96
6	17.36	16.73
5	25.41	24.61
4	18.10	15.40
3	18.03	14.19
2	18.10	18.33
1	26.25	23.11
k = 7 l = 7		
0	18.87	15.35
1	20.64	17.82
2	22.50	22.72
3	26.15	26.18
4	18.03	17.91
5	8.87	9.08
k = 7 l = -8		
7	1.49	.08
6	1.59	-.56
5	1.69	-5.52
4	1.77	.15
3	4.67	4.61
2	4.67	6.08
1	1.77	-.12
k = 7 l = -9		
7	5.61	5.26
6	11.30	11.28
5	11.67	10.89
4	15.77	14.58
3	13.26	10.87
2	15.32	16.51
1	15.50	14.37

TABLE II

FINAL ATOMIC COORDINATES AND ANISOTROPIC TEMPERATURE FACTORS[a]

Atom	x	y	z	$10^4 \times$ β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Pd	0.0000	0.0000	0.0000	102	47	114	1	18	7
				2	3	2	4	3	4
O_1	−0.1207	0.1005	−0.1703	150	0	146	44	16	24
	13	14	13	18	18	18	26	27	26
O_2	0.1395	0.1419	0.0818	122	21	132	0	20	−28
	12	13	13	16	18	17	24	25	27
C_1	−0.2112	0.2792	−0.3246	128	71	126	−3	−2	102
	19	20	19	25	28	25	40	39	39
C_2	−0.1018	0.2151	−0.1851	117	40	116	70	73	80
	18	23	18	23	30	22	38	35	37
C_3	0.0120	0.3002	−0.0892	48	120	267	17	−44	3
	18	23	27	18	33	44	39	45	58
C_4	0.1291	0.2574	0.0285	98	−14	158	−9	41	−2
	17	18	19	21	24	26	30	36	37
C_5	0.2484	0.3394	0.1137	117	24	118	−43	87	−28
	18	18	18	22	24	22	33	35	34
C_6	0.2850	0.4462	0.0496	137	36	146	−22	65	−35
	20	19	21	26	27	27	36	42	37
C_7	0.4081	0.5259	0.1252	163	71	188	6	27	−25
	23	22	24	30	30	32	47	50	48
C_8	0.4978	0.4944	0.2695	183	34	180	−17	84	−18
	22	24	22	29	31	30	49	46	50
C_9	0.4581	0.3880	0.3340	161	88	137	30	−1	−88
	23	23	22	30	34	28	47	46	45
C_{10}	0.3333	0.3053	0.2595	120	137	124	−2	−11	21
	20	23	21	25	34	26	45	41	44

[a] The estimated standard deviation is shown below each parameter. The form of the anisotropic temperature factor is $\exp(-\beta_{11}h^2 - \beta_{22}k^2 - \beta_{33}l^2 - \beta_{12}hk - \beta_{13}hl - \beta_{23}kl)$. Note that these parameters are computed in an analysis scheme employing individual scaling for each level and must thus be regarded as fitting parameters which do not yield the correct thermal ellipsoids. See E. C. Lingafelter and J. Donohue, *Acta Cryst.*, **20,** 321 (1966).

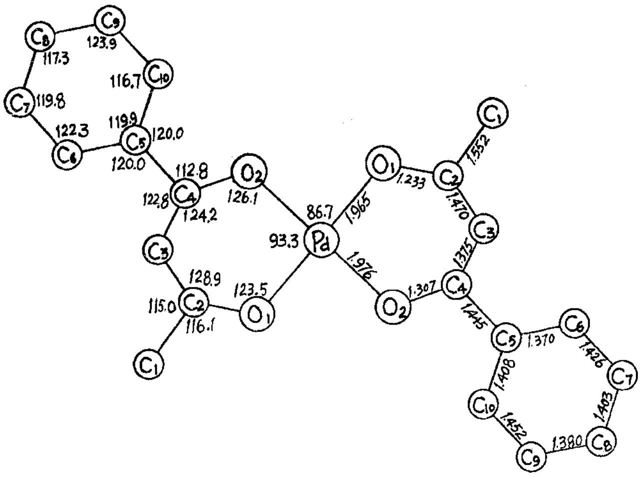

Figure 1.—Bond lengths and bond angles. For a list of estimated standard deviations see Table III. The metal–oxygen bonds have deviations of about 0.015 A; the others of about 0.025 A.

TABLE III

BOND LENGTHS AND BOND ANGLES AND THEIR ESTIMATED STANDARD DEVIATIONS

	Length, A	Esd, A		Angle, deg	Esd, deg
Pd–O_1	1.965	0.015	O_1–Pd–O_2	93.3	0.5
Pd–O_2	1.976	0.014	O_1–Pd–O_2'	86.7	0.5
O_1–C_2	1.233	0.028	Pd–O_1–C_2	123.5	1.2
O_2–C_4	1.307	0.023	Pd–O_2–C_4	126.1	0.8
C_2–C_3	1.470	0.030	O_1–C_2–C_1	116.1	1.9
C_3–C_4	1.375	0.028	O_2–C_4–C_5	112.8	0.9
C_1–C_2	1.552	0.026	O_1–C_2–C_3	128.9	2.0
C_4–C_5	1.445	0.024	O_2–C_4–C_3	124.2	1.5
C_5–C_6	1.370	0.026	C_2–C_3–C_4	123.0	2.0
C_6–C_7	1.426	0.029			
C_7–C_8	1.403	0.031	C_1–C_2–C_3	115.0	1.6
C_8–C_9	1.380	0.033	C_3–C_4–C_5	122.8	1.8
C_{10}–C_5	1.408	0.026	C_4–C_5–C_6	120.0	1.2
			C_4–C_5–C_{10}	120.0	1.2
			C_6–C_5–C_{10}	119.9	1.2
			C_5–C_6–C_7	122.3	1.9
			C_6–C_7–C_8	119.8	1.3
			C_7–C_8–C_9	117.3	2.0
			C_8–C_9–C_{10}	123.9	1.8
			C_9–C_{10}–C_5	116.7	2.0

and 9.3° and 4.6° in the copper chelate of dibenzoylmethane.[8]

The considerable difference in packing between this compound and the reported[3] crystal form of copper benzoylacetonate is most interesting. Here, the metal of one molecule in the unit cell is in contact (3.75 A) with a methyl group of the other molecule; the molecules so connected form a crinkled sheet in the (100) plane (see Figure 2). The sheets are joined by van der Waals contacts between the projecting phenyl side groups, as can be seen in Figures 3 and 4. On the other hand, in the copper chelate,[3] the fifth and sixth octahedral positions are occupied by C_3 atoms at *ca.* 3.1 A, which could be sufficiently close to cause observable axial coordination effects. Therefore, we have

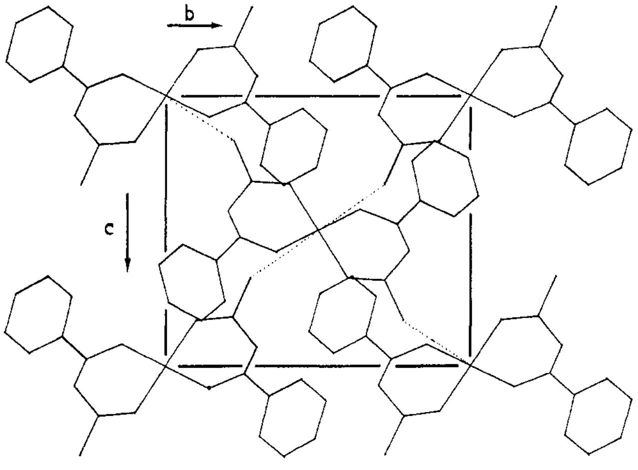

Figure 2.—Projection of molecular skeletons along [100]. This is a nonorthogonal projection along the **a** axis onto the **bc** plane. The dotted lines show the van der Waals contact between the palladium atoms and the methyl groups located 3.75 A away in axial positions.

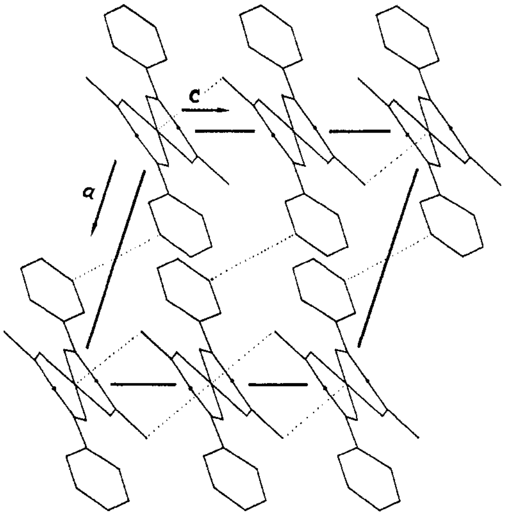

Figure 3.—Projection of molecular skeletons along [010]. This is an orthogonal projection along the **b** axis onto the **ac** plane. Some intermolecular contacts are indicated.

begun a comparison of some of the properties of copper benzoylacetonate as a guest in the palladium benzoylacetonate host crystal, where axial coordination effects should be virtually negligible, with those of pure crystal-

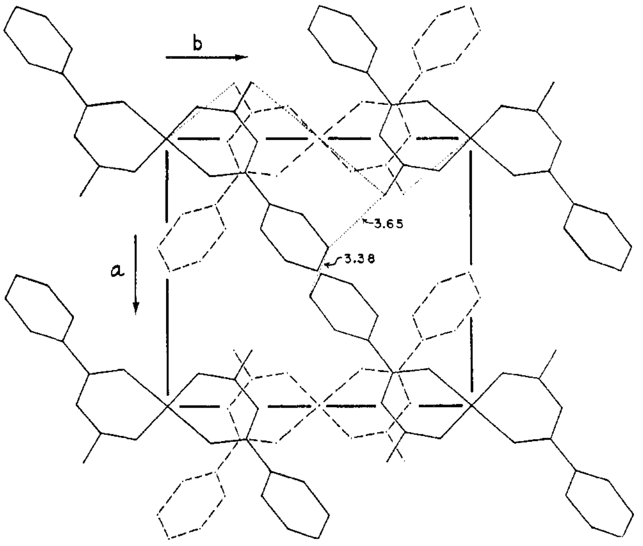

Figure 4.—Projection of molecular skeletons along [001]. This is a nonorthogonal projection along the **c** axis onto the **ab** plane. Some of the intermolecular contacts are noted on this diagram.

TABLE IV

DEVIATIONS OF ATOMS FROM THE MEAN PLANES (A)

Atom	Plane 1	Plane 2	Atom	Plane 1	Plane 2
Pd	−0.150	0.836	C_5	0.126	0.006[a]
O_1	0.007[a]	0.340	C_6	0.649	0.000[a]
O_2	−0.026[a]	0.550	C_7	0.831	−0.009[a]
C_1	0.099	−0.481	C_8	0.490	0.013[a]
C_2	0.010[a]	−0.109	C_9	−0.062	−0.008[a]
C_3	−0.038[a]	−0.321	C_{10}	−0.258	−0.001[a]
C_4	0.048[a]	0.104			

[a] The corresponding atoms used in calculation of the mean planes. Equations of the planes: (1) $0.7879X - 0.20504Y - 0.56559Z + 0.14995 = 0$ (chelate ring); (2) $0.75377X - 0.54890Y - 0.36131Z - 0.83561 = 0$ (phenyl ring). The angle between these two planes is 23° 13′.

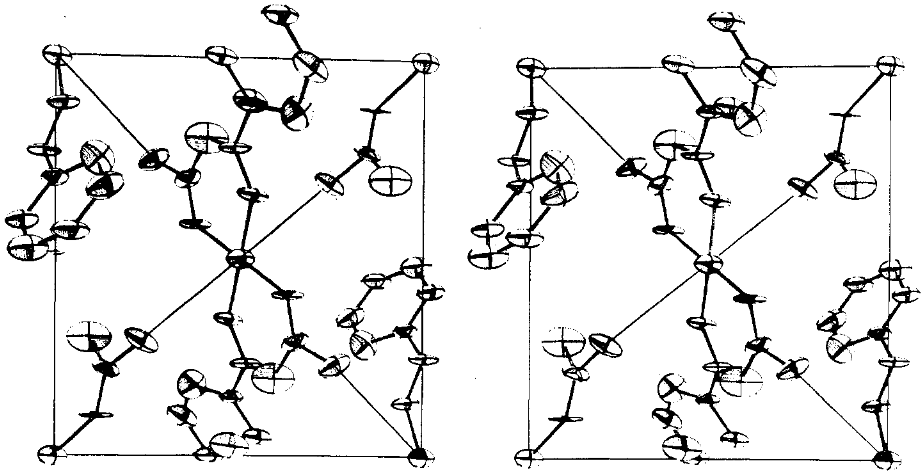

Figure 5.—Stereoscopic view of $Pd(C_6H_5COCHCOCH_3)_2$ crystal. The view direction is the *a** axis. Note the methyl groups completing a distorted octahedron about Pd. The atoms are represented by the computed thermal ellipsoids at the 50% probability level.[7] To view this figure, use a simple stereoscopic viewing aid. (Alternatively, the reader with obedient optic muscles can see this three-dimensional view with the aid of merely a stiff piece of cardboard.) Note that the computed ellipsoids were used for convenience. See footnote *a* of Table II.

line copper benzoylacetonate, to gauge the effect of weak axial coordination of copper to the π orbital of an aromatic carbon.

At this juncture, one cannot say why the copper compound packs in one way and the palladium one in another. However, we emphasize that the geometries of the two chelate rings are, within experimental error, identical. Furthermore, the difference in the twist of the phenyl ring out of the mean chelate plane is probably determined by the basic difference in mode of packing. Therefore, we are inclined to attribute the packing difference to a special stability of z axis coordination to the copper ion. This opinion is influenced by the fact that the same sort of packing, involving close axial approach of an aromatic carbon atom, recurs in other crystalline copper chelates—*e.g.*, copper acetylacetonate and copper dibenzoylmethanate.[8]

Acknowledgment.—We thank Mrs. Kam Chan and Miss Connie Go for their help in measuring some of the intensities and performing certain of the calculations, Professor I. C. Paul for helpful discussion, and Professor G. Stucky for providing the modified ORTEP computer program.

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Oxovanadium(IV) Tartrates. Characterization and Evidence for Intermetallic Coupling in Anionic Species[1]

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Received November 4, 1966

A study of physical properties of VO^{2+} with active and racemic tartaric acids in 1:1 aqueous solutions shows that these two acids give 1:1 complex anions, in the region of pH 7, differing in stability, optical spectra, and electron spin resonance spectra. The data indicate that each anionic complex is a dimer containing two VO^{2+} ions bridged by two tetranegative tartrate groups. The first electron spin resonance spectrum to show hyperfine splitting by two equivalent vanadium nuclei is reported for the racemic complex. Magnetic susceptibilities of two of the several solid salts isolated imply that the V–V exchange coupling is less than 50 cm^{-1}, as one would expect for the proposed geometries.

Introduction

The vanadyl(IV)[2] tartrate system has been extensively studied[3] although little information has been recorded on the structures of the species involved. In solution, vanadyl(IV) ion coordinates with tartrate anion to give various complexes depending upon the pH of the solution and the relative amounts of vanadyl(IV) and tartrate present.

Of particular interest is the anionic species formed in solutions containing a 1:1 ratio of vanadyl(IV) and tartrate ions at pH 7. The formation of this complex is indicated by a sudden change in color and, with optically active tartrate, the appearance of a four-peak optical spectrum[4] as the pH of the vanadyl(IV) tartrate solution is increased. Ducret's studies[5] indicate that with racemic tartrate the anionic species is a 1:1 complex containing a tetranegative tartrate ion in which all alcoholic and carboxylic hydrogens are removed. Jørgensen[6] has assumed, as have most authors implicitly, that this complex is mononuclear consisting of a vanadyl(IV) ion surrounded by four coordinating oxygens of the tetranegative tartrate ion. Our molecular models, however, indicate that such a system would be highly strained for any reasonable geometry about the vanadyl(IV) ion. Jones and Larsen,[7] on the other hand, have assumed that this system contains polynuclear species by analogy to the behavior of basic copper tartrate solutions.

The work reported here confirms that the principal anionic complex of vanadyl(IV) tartrate formed in higher pH solutions is not mononuclear. In a recent paper,[8] Dunlop, *et al.*, have also arrived at this conclusion based on one of the observations that we have made—*i.e.*, vanadyl(IV) tartrate solutions at higher pH containing optically active tartaric acid have different spectral properties than those containing the racemic acid. They, however, conclude that several polymeric species are present whereas our data imply that there is primarily one complex present—a tetranegative anion consisting of two vanadyl(IV) ions bridged by two tetranegative tartrate anions. From our work, we infer that with *d*- (or *l*-) tartrate, a dimer is formed having two *d*- (or *l*-) tartrate bridges while with the racemic tartrate, dimers are formed having one *d*- and one *l*-tartrate bridge.

Finally, we present electron spin resonance spectra

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[1] Supported by ARPA Contract SD-131 through the Materials Research Laboratory at the University of Illinois and also by the U. S. Public Health Service, Grant GM-10907, and a predoctoral fellowship awarded to R. E. T., through the Institute of General Medical Sciences.

[2] Vanadyl(IV) and oxovanadium(IV) both denote the VO^{2+} ion.

[3] See J. Selbin, *Chem. Rev.*, **65**, 158 (1965), for a list of references.

[4] J. Selbin and L. Morpurgo, *J. Inorg. Nucl. Chem.*, **27**, 673 (1965).

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[8] J. H. Dunlop, D. F. Evans, R. D. Gillard, and G. Wilkinson, *J. Chem. Soc., Sect. A*, 1260 (1966).