

labeled drawing of the dimer; crystal packing is shown in Fig. 2.

**Discussion.** The dimer is joined across an inversion center by an  $(\eta^5, \eta^1)$ -Cp\* group which bridges the two symmetry-related Sc atoms. The  $\eta^1$ -methylene (C6) has been formed as the result of C—H activation of a Cp\* methyl group. A somewhat surprising result is that, in all other regards, the bonding is very similar to that determined for  $\text{Cp}_2^*\text{ScCH}_3$  (Thompson *et al.*, 1987). The  $(\eta^5, \eta^1)$ -ring remains planar with a maximum deviation of only 0.22 Å from the least-squares plane calculated for C1 through C10. The geometry about C6 is essentially tetrahedral; the angles C1—C6—Sc', C1—C6—H6A, C1—C6—H6B and H6A—C6—H6B are 118.9, 107.3, 104.8 and 98.1°, respectively, giving an average value of 107.3 (8.6)°. The H6A—C6—H6B angle is slightly compressed (98.1°) while the C1—C6—Sc' angle is somewhat opened (118.9°). The Sc'...H6A and Sc'...H6B distances are 2.76 (3) and 2.79 (3) Å indicating that there is no agostic interaction for the  $\alpha$ -H atoms with the Sc center. The Cp\*—Sc—Cp\* angle (141.8°) is approximately the same as for  $\text{Cp}_2^*$

$\text{ScCH}_3$  (144.7°) suggesting that formal replacement of a methyl ligand with a bulkier  $(\eta^5, \eta^1)$ -Cp\* group as the alkyl does not significantly perturb the Sc—Cp\* bonding.

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## A Silicon-Bridged Bis(substituted Cp) Yttrium Complex

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**Abstract.** *rac*-Bis(tetrahydrofuran)lithium [bis(2-trimethylsilyl-4-*tert*-butyl- $\eta^5$ -cyclopentadienyl)dimethylsilane]dichloroyttrate,  $[\text{Li}(\text{C}_4\text{H}_8\text{O})_2][\text{Y}(\text{C}_{26}\text{H}_{48}\text{Si}_3\text{Cl}_2)]$ ,  $M_r = 755.87$ , triclinic,  $P\bar{1}$ ,  $a = 13.110$  (8),  $b = 17.163$  (15),  $c = 20.623$  (14) Å,  $\alpha = 104.02$  (7),  $\beta = 99.38$  (5),  $\gamma = 100.24$  (6)°,  $V = 4326$  (6) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.16$  g cm<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71073$  Å,  $\mu = 15.86$  cm<sup>-1</sup>,  $F(000) = 1608$ , room temperature,  $R = 0.056$  for 6136 reflections with  $F_o^2 > 3\sigma(F_o^2)$ . There are two virtually identical molecules in the asymmetric unit. In each, the Y atom is tetrahedrally coordinated to a substituted Si-bridged bis(cyclopentadienyl) ligand and to two Cl ions in the cleft. The Li atom is 2.35 Å from each Cl ion, and two molecules of tetrahydrofuran are connected to the Li, completing its tetrahedral coordination.

\* Contribution No. 8539.

**Introduction.** The Ziegler–Natta polymerization of olefins has occupied the attention of chemists for nearly four decades. Recently, the development of homogeneous transition-metal catalyst systems has afforded the possibility of mechanistic investigations into various key steps of polymer initiation, propagation and chain termination. Brintzinger and co-workers have developed a series of *ansa*-zirconocene catalysts which possess a  $C_2$  symmetric ligand arrangement about the metal center (Roll, Brintzinger, Rieger & Zolk, 1990; Wiesenfeldt, Reinmuth, Barsties, Evertz & Brintzinger, 1989). Moreover, activation of these *ansa*-zirconocene catalysts with methylalumoxane results in production of highly isospecific polymers. Work in this laboratory has centered on the study of well defined single-component Ziegler–Natta-type catalyst systems (Piers, Shapiro, Bunel & BercaW, 1990). We report

Table 1. Final heavy-atom coordinates ( $\times 10^4$ ) and equivalent isotropic displacement parameters ( $\text{\AA}^2 \times 10^4$ )

$$U_{\text{eq}} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	$U_{\text{eq}}$
Ya	1810 (.4)	7217 (.3)	4009 (.3)	432 (2)
Cl1a	3262 (1)	6960 (1)	3292 (1)	719 (5)
Cl2a	2931 (1)	8756 (1)	4442 (1)	708 (5)
Si1a	-400 (1)	6062 (1)	4245 (1)	611 (5)
Si2a	1161 (2)	8070 (1)	5831 (1)	813 (6)
Si3a	372 (1)	5333 (1)	2407 (1)	632 (5)
C1a	-1428 (5)	6446 (4)	4683 (3)	976 (23)
C2a	-793 (5)	4914 (4)	3970 (3)	903 (21)
C3a	978 (4)	6434 (4)	4793 (3)	531 (16)
C4a	1549 (4)	7246 (3)	5231 (3)	525 (17)
C5a	2638 (5)	7228 (4)	5301 (3)	587 (18)
C6a	2789 (5)	6479 (4)	4926 (3)	563 (17)
C7a	1748 (5)	5989 (3)	4610 (3)	571 (18)
C8a	567 (6)	7615 (5)	6447 (3)	1259 (29)
C9a	245 (6)	8639 (4)	5464 (4)	1368 (30)
Cl0a	2392 (6)	8882 (4)	6303 (3)	1038 (26)
Cl1a	3807 (5)	6180 (4)	4988 (4)	696 (21)
Cl2a	4745 (5)	6882 (4)	5048 (4)	1049 (26)
Cl3a	3780 (5)	5484 (5)	4385 (4)	1252 (29)
Cl4a	3948 (5)	5889 (4)	5634 (4)	1177 (26)
Cl5a	-204 (4)	6555 (3)	3552 (3)	475 (17)
Cl6a	280 (4)	6335 (3)	2977 (3)	450 (15)
Cl7a	585 (4)	7071 (4)	2782 (3)	508 (16)
Cl8a	295 (4)	7738 (3)	3200 (3)	523 (18)
Cl9a	-156 (4)	7421 (4)	3682 (3)	529 (17)
C20a	1199 (5)	4727 (3)	2801 (3)	954 (23)
C21a	-985 (5)	4682 (4)	2047 (4)	1240 (29)
C22a	965 (5)	5552 (3)	1698 (3)	902 (23)
C23a	317 (5)	8576 (4)	3081 (3)	679 (20)
C24a	1242 (6)	8841 (4)	2763 (3)	960 (23)
C25a	372 (6)	9235 (4)	3732 (4)	1043 (26)
C26a	-726 (6)	8500 (4)	2585 (4)	1278 (27)
Yb	3053 (.4)	2441 (.3)	867 (.3)	450 (2)
Cl1b	2749 (1)	3843 (1)	1553 (1)	642 (4)
Cl2b	4415 (1)	3364 (1)	406 (1)	751 (5)
Si1b	1996 (1)	449 (1)	726 (1)	536 (5)
Si2b	2974 (1)	900 (1)	-942 (1)	725 (6)
Si3b	2180 (1)	2113 (1)	2530 (1)	632 (5)
C1b	2456 (5)	-477 (3)	297 (3)	765 (19)
C2b	829 (4)	83 (3)	1066 (3)	627 (18)
C3b	1682 (4)	1105 (3)	156 (3)	441 (16)
C4b	2233 (4)	1382 (3)	-330 (3)	535 (18)
C5b	1854 (4)	2088 (4)	-425 (3)	578 (18)
C6b	1106 (4)	2267 (3)	-28 (3)	496 (17)
C7b	1026 (4)	1661 (3)	330 (3)	463 (16)
C8b	4245 (5)	652 (4)	-590 (3)	997 (22)
C9b	3356 (5)	1625 (4)	-1452 (3)	1007 (23)
Cl0b	2094 (5)	-67 (4)	-1534 (3)	1039 (25)
Cl1b	402 (4)	2873 (3)	-76 (3)	518 (17)
Cl2b	1022 (5)	3688 (4)	-149 (3)	797 (20)
Cl3b	-476 (5)	2469 (4)	-728 (3)	795 (20)
Cl4b	-114 (5)	3050 (4)	525 (3)	769 (19)
Cl5b	3123 (4)	1210 (3)	1392 (3)	465 (16)
Cl6b	3090 (4)	1899 (3)	1949 (3)	464 (17)
Cl7b	4127 (5)	2438 (3)	2100 (3)	548 (18)
Cl8b	4772 (5)	2123 (4)	1669 (3)	534 (18)
Cl9b	4131 (5)	1370 (3)	1230 (3)	555 (18)
C20b	872 (4)	2259 (3)	2133 (3)	699 (18)
C21b	2823 (5)	3081 (4)	3217 (3)	892 (22)
C22b	1952 (6)	1256 (4)	2931 (3)	1061 (23)
C23b	5959 (5)	2444 (4)	1766 (4)	767 (25)
C24b	6521 (5)	2046 (4)	2261 (4)	1257 (32)
C25b	6284 (4)	3375 (4)	2056 (4)	1076 (30)
C26b	6324 (5)	2187 (4)	1100 (4)	1068 (29)
Lia	4084 (8)	8380 (6)	3729 (5)	776 (34)
O1a	4148 (4)	8946 (4)	3036 (3)	1111 (18)
C27a	4386 (7)	9815 (7)	3176 (6)	1758 (44)
C28a	4541 (11)	9997 (11)	2520 (11)	2677 (77)
C29a	3822 (11)	9301 (14)	2042 (8)	2627 (79)
C30a	3910 (8)	8591 (7)	2320 (6)	1723 (44)
O2a	5548 (4)	8631 (3)	4200 (3)	1040 (19)
C31a	6340 (10)	8477 (9)	3881 (7)	2243 (63)
C32a	7242 (17)	8569 (16)	4334 (10)	3091 (105)
C33a	7130 (14)	8873 (11)	4969 (9)	2576 (83)
C34a	6003 (9)	8994 (5)	4901 (6)	1534 (43)
Lib	4128 (8)	4571 (6)	1161 (6)	792 (33)
O1b	5229 (4)	5223 (3)	1958 (3)	1035 (19)
C27b	6245 (8)	5647 (6)	1925 (5)	1492 (40)

Table 1 (cont.)

	x	y	z	$U_{\text{eq}}$
C28b	6714 (13)	6188 (10)	2621 (10)	2485 (81)
C29b	6127 (15)	5857 (11)	3055 (8)	2715 (90)
C30b	5115 (7)	5376 (6)	2646 (5)	1452 (39)
O2b	3806 (4)	5367 (3)	713 (3)	1018 (17)
C31b	3480 (7)	5239 (5)	-5 (5)	1193 (31)
C32b	2784 (9)	5833 (8)	-63 (7)	1821 (51)
C33b	3178 (10)	6511 (7)	561 (8)	1849 (51)
C34b	3759 (8)	6177 (7)	1041 (5)	1634 (42)

Table 2. Selected bond distances ( $\text{\AA}$ ) and angles ( $^\circ$ )

Ya—Cp1a	2.383	Yb—Cp1b	2.396
Ya—Cp2a	2.385	Yb—Cp2b	2.389
Ya—Cl1a	2.628 (2)	Yb—Cl1b	2.614 (1)
Ya—Cl2a	2.642 (2)	Yb—Cl2b	2.623 (2)
Lia—Cl1a	2.366 (11)	Lib—Cl1b	2.377 (11)
Lia—Cl2a	2.340 (11)	Lib—Cl2b	2.401 (11)
Lia—O1a	1.917 (12)	Lib—O1b	1.950 (12)
Lia—O2a	1.925 (12)	Lib—O2b	1.893 (12)
Si1a—C1a	1.869 (7)	Si1b—C1b	1.872 (6)
Si1a—C2a	1.863 (6)	Si1b—C2b	1.858 (6)
Si1a—C3a	1.878 (6)	Si1b—C3b	1.862 (5)
Si1a—C15a	1.858 (6)	Si1b—C15b	1.887 (6)
Si2a—C4a	1.845 (6)	Si2b—C4b	1.843 (6)
Si2a—C8a	1.850 (8)	Si2b—C8b	1.869 (7)
Si2a—C9a	1.856 (8)	Si2b—C9b	1.868 (7)
Si2a—C10a	1.878 (7)	Si2b—C10b	1.859 (7)
Cp1a—Ya—Cp2a	122.1	Cp1b—Yb—Cp2b	122.5
Cp1a—Ya—Cl1a	114.2	Cp1b—Yb—Cl1b	112.4
Cp1a—Ya—Cl2a	107.0	Cp1b—Yb—Cl2b	108.6
Cp2a—Ya—Cl1a	108.6	Cp2b—Yb—Cl1b	108.1
Cp2a—Ya—Cl2a	114.2	Cp2b—Yb—Cl2b	114.3
Cl1a—Ya—Cl2a	84.6 (0)	Cl1b—Yb—Cl2b	84.6 (0)
Cl1a—Lia—Cl2a	97.8 (4)	Cl1b—Lib—Cl2b	95.1 (4)
Cl1a—Lia—O1a	113.7 (5)	Cl1b—Lib—O1b	107.9 (5)
Cl1a—Lia—O2a	115.5 (5)	Cl1b—Lib—O2b	117.9 (5)
Cl2a—Lia—O1a	114.3 (5)	Cl2b—Lib—O1b	120.3 (5)
Cl2a—Lia—O2a	113.9 (5)	Cl2b—Lib—O2b	113.3 (5)
O1a—Lia—O2a	102.4 (6)	O1b—Lib—O2b	103.0 (6)
Cl5a—Si1a—C3a	99.1 (3)	Cl5b—Si1b—C3b	99.5 (2)

herein the crystal structure of the  $C_2$  symmetric complex  $\text{rac}-(\text{CH}_3)_2\text{Si}[2-\text{Si}(\text{CH}_3)_3-4-\text{C}(\text{CH}_3)_3\text{C}_5\text{H}_2]_2-\text{YCl}_2\text{Li}(\text{OCH}_2\text{CH}_2\text{CH}_2\text{CH}_2)_2$ .

**Experimental.** A crystal with a most irregular triangular prism shape,  $0.63 \times 0.67 \times 1.18$  mm, was used for data collection on a CAD-4 diffractometer, with  $\omega$  scans. 25 reflections with  $26 < 2\theta < 29^\circ$  were used for the determination of the cell dimensions. An absorption correction was not applied:  $\psi$  scans of six high  $\chi$  reflections suggested corrections of  $\pm 7\%$  to  $I$ , but the corrections resulted in a poorer goodness of fit for merging the data. The crystal was somewhat obscured by grease in its capillary, so no analytical absorption correction could be made;  $(\sin\theta/\lambda)_{\text{max}} = 0.59 \text{ \AA}^{-1}$ ;  $h$  from  $-15$  to  $15$ ,  $k$  from  $-20$  to  $20$ ,  $l$  from  $0$  to  $24$ . Three standard reflections ( $1\bar{2}\bar{5}$ ,  $3\bar{2}4$  and  $15\bar{2}$ ) showed no variations greater than those predicted by counting statistics. 16018 reflections were measured, of which 15167 were independent. Goodness of fit for merging was 1.35, and  $R_{\text{int}}$  for 372 reflections with exactly two observations was 0.051. All reflections,  $F_o^2$  positive and negative, were used in solution and refinement of the structure. The structure was solved from a Patterson map, which gave coordinates of Y atoms; remaining atoms were

found by successive structure factor–Fourier calculations.  $F_o^2$  values were used in full-matrix least-squares refinement, with  $w = 1/\sigma^2(F_o^2)$ . H atoms were positioned by calculation (C–H = 0.95 Å) assuming staggered geometries on methyl groups; H-atom parameters were not refined, but the atoms were repositioned near the conclusion of the refinement.  $R$  on  $F = 0.134$  for 12 716 reflections with  $F_o^2 > 0$  ( $wR$  on  $F^2 = 0.010$ );  $R$  on  $F = 0.056$  for 6136 reflections with  $F_o^2 > 3\sigma(F_o^2)$  ( $wR$  on  $F^2 = 0.007$ ); and  $S = 1.33$  for 15 167 reflections and 775 parameters. Variances [ $\sigma^2(F_o^2)$ ] were derived from counting statistics plus an additional term,  $(0.014I)^2$ ; variances of the merged

data by propagation of e.s.d. plus another additional term,  $(0.014I)^2$ . In the final cycle, maximum shift/e.s.d. was 0.09 in the Y molecule, and 1.56 for  $y$  of C32 in thfa [several parameters of this thf group continued to shift (with no change in the residuals) for at least five full-matrix cycles after the remainder of the structure had settled down, presumably because of disorder]. In the final difference map one peak of  $1.01 \text{ e } \text{Å}^{-3}$  was found near Ya; other peaks were  $\pm 0.86 \text{ e } \text{Å}^{-3}$ . Scattering factors were taken from Cromer & Waber (1974) and dispersion corrections ( $f'$ ) from Cromer (1974). Programs used were those of the *CRYM* crystallographic computing system (Duchamp, 1964) and *ORTEPII* (Johnson, 1976). Final heavy-atom parameters are given in Table 1.\*

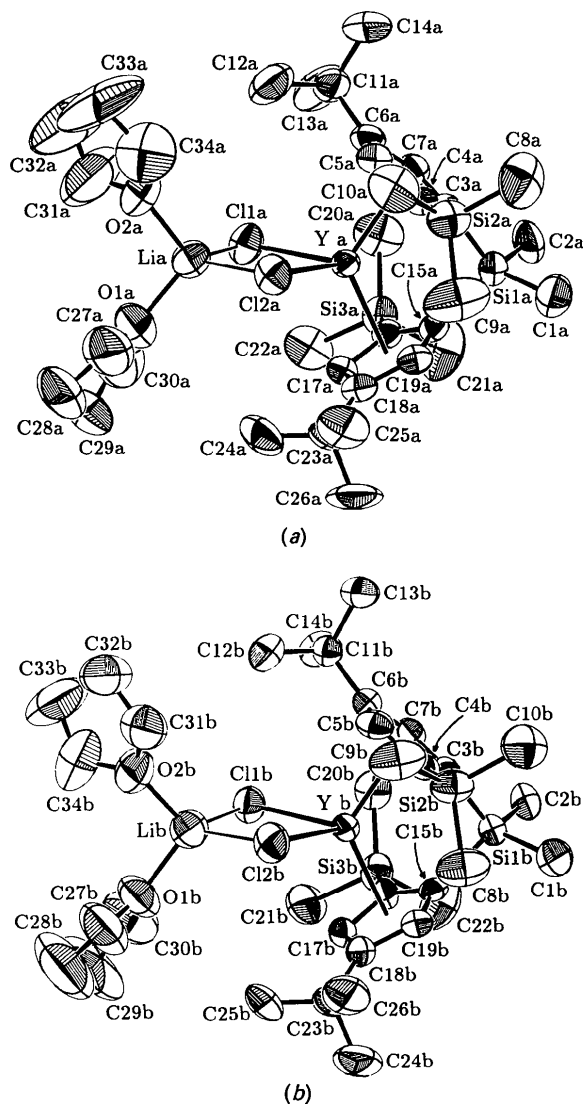


Fig. 1. An *ORTEPII* (Johnson, 1976) drawing showing the atomic numbering systems of (a) molecule *A* (C16a, between C15a and C17a, is not labeled) and (b) molecule *B* (C16b not labeled). Atoms are shown as 50% probability ellipsoids, with H atoms omitted.

**Discussion.** Fig. 1 shows labeled drawings of molecules *A* and *B*; selected distances and angles for these are given in Table 2. Because the two molecules are so similar, this discussion uses average distances with the scatter standard deviation given in square brackets. Fig. 2 shows the unit-cell packing. The

\* Lists of assigned H-atom parameters, anisotropic displacement parameters, complete distances and angles, and observed and calculated structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55177 (75 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HH0616]

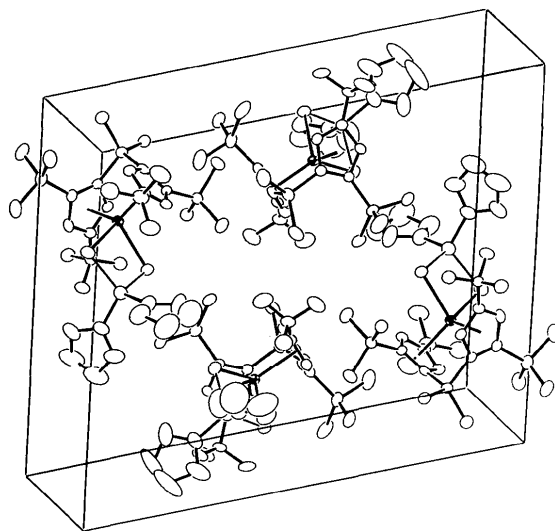


Fig. 2. An *ORTEPII* (Johnson, 1976) view of the contents of a unit cell, with one unit cell outlined. Y atoms have their principal ellipsoids outlined. Atoms are shown as 50% probability ellipsoids and H atoms are omitted. The  $c$  axis is horizontal and the view is perpendicular to the  $bc$  plane. The *A* molecules are closer to the center of the cell and the *B* molecules are in the upper-left and lower-right corners.

centroids of the Cp rings are 2.388 [6] Å from the Y atoms, but the Cp planes are not perpendicular to the Y—Cp vectors. Y—C(Cp ring) distances range from 2.585 (5) to 2.817 (6) Å, with the C atoms closest to the Si bridge being closest to Y. This effect is caused by the bridging Si atom; it 'pinches' the Cp rings together and the Y atom can no longer fit between them perfectly. This effect is also seen in the angle between the two Cp planes: this is 113 (4)°, whereas the Cp1—Y—Cp2 angle is 122.3°. {The Cp(C)—Si1—Cp(C) angle is 99.3 [3]°.} Other distances in the Y molecule are normal: Y—Cl 2.627 [12], Si—C 1.863 [12], C—C (Cp rings) 1.422 [17] Å. In each molecule there is a strain evidenced by two normal C(Cp)—Si—CH<sub>3</sub> angles of 108.4 [4]° and a third larger angle of 117.0[10]°, to the methyl C atom near the other Cp ring. This strain appears in all four independent Cp rings and reflects a steric crowding between the —Si(CH<sub>3</sub>)<sub>3</sub> group and the rest of the molecule: from the affected CH<sub>3</sub> group, C—C distances are 3.81 [4] Å to a methyl C atom on the Si bridge (C1 or C2), 4.02 [5] Å to a methyl C atom on the 'Bu group of the opposite bridge and only 3.68 [3] Å to a Cp C atom in the opposite Cp ring. The 'radius' of a —CH<sub>3</sub> group is taken as 2.0 Å; thus the first contact is 0.09 Å short while the second is at just the van der Waals distance. The 3.68 Å contact is short enough to imply the severe strain of bending a C—Si—C bond by 8° or so.

The Li(thf)<sub>2</sub><sup>+</sup> group is joined to the two Cl atoms of the Y molecule, with Li—Cl distances averaging

2.371 [25] Å, almost exactly the sum of the crystal radii for Li<sup>+</sup> and Cl<sup>-</sup> and shorter than the Li—C distance (2.57 Å) in LiCl (Wells, 1962). Coordination about the Li<sup>+</sup> atom is approximately tetrahedral (Fig. 1), with normal Li—O distances {1.92 [2] Å}. The thf molecules have large apparent thermal motions, indicating a moderate disorder.

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## A Bis(pyrazolyl)(bipyridyl)platinum Complex

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**Abstract.** (4,4'-Dimethyl-2,2'-bipyridyl)bis(3,5-dimethylpyrazolium)platinum(II) 0.5-tetrahydrofuran solvate monohydrate, [Pt(C<sub>5</sub>H<sub>7</sub>N<sub>2</sub>)<sub>2</sub>(C<sub>12</sub>H<sub>12</sub>N<sub>2</sub>)]·0.5C<sub>4</sub>H<sub>8</sub>O·H<sub>2</sub>O, *M<sub>r</sub>* = 623.65, monoclinic, *P*2<sub>1</sub>/*n*, *a* = 8.625 (2), *b* = 20.593 (8), *c* = 14.451 (4) Å, β = 90.32 (2)°, *V* = 2566.7 (14) Å<sup>3</sup>, *Z* = 4, *D<sub>x</sub>* =

1.61 g cm<sup>-3</sup>, λ(Mo *K*α) = 0.71073 Å, μ = 55.50 cm<sup>-1</sup>, *F*(000) = 1232, room temperature, *R* = 0.0387 for 2874 reflections with *F<sub>o</sub>*<sup>2</sup> > 3σ(*F<sub>o</sub>*<sup>2</sup>). The square-planar Pt complex has normal Pt—N-(bipyridyl) bonds [2.009 (8) Å] and slightly short Pt—N(pyrazolyl) bonds [1.983 (7) Å]. The ligand molecules have normal distances and angles; the planes of the pyrazolyl ligands are twisted by about

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