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PREPARATION OF THE FIRST η^2-OLEFIN COMPLEX OF A 4f-TRANSITION METAL, $(Me_5C_5)_2Yb(\mu\text{-}C_2H_4)Pt(PPh_3)_2$

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Preparation of the First η^2-Olefin Complex of a 4f-Transition Metal, $(Me_5C_5)_2Yb(\mu\text{-}C_2H_4)Pt(PPh_3)_2$

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Abstract

Reaction of $(Me_5C_5)_2Yb$ with the platinum olefin complex, $(Ph_3P)_2Pt(C_2H_4)$ gives the first olefin complex of a lanthanide metal, $(Me_5C_5)_2Yb(\mu\text{-}C_2H_4)Pt(PPh_3)_2$, as shown by X-ray crystallography.

Insertion of an olefin into a metal-carbon bond is a fundamental and a reasonably well-known process in d- and f-transition metal organometallic chemistry.[1] It is generally assumed that coordination of the olefin to the metal center is a prerequisite to insertion though no olefin complexes of an f-transition metal have been isolated, in contrast to the enormous number of olefin complexes of the d-transition metals as well as the monovalent coinage metals.[2] Recently the η^6-arene and η^2-acetylene complexes, $(Me_6C_6)Sm(AlCl_4)_3$[3a] and $(Me_5C_5)_2Yb(MeC{\equiv}CMe)$,[3b] have been characterized showing that π-complexes of the lanthanides can be isolated.

We have observed that the bent, base-free compound, $(Me_5C_5)_2Yb$,[4a] initiates the polymerization of ethylene, though not that of propylene nor styrene,[4b] the mechanism of which is unknown though it presumably involves an η^2-ethylene coordination complex. These studies encouraged us to try to isolate an olefin complex of an f-transition metal. In order to isolate such a complex, we postulated that an olefin with sterically small electron-donor groups rather than electron-withdrawing groups would maximize the Lewis basicity of the olefin donor orbitals. One way of creating such an olefin is to use a Pt(0) complex with ethylene, $(\eta^2\text{-}C_2H_4)Pt(PPh_3)_2$;[5] the olefin is electron-rich since Pt(0) is a good π-donor.

Addition of white $(\eta^2\text{-}C_2H_4)Pt(PPh_3)_2$ to a toluene solution of $(Me_5C_5)_2Yb$ gives a red solution from which deep red needles of the adduct (I) may be isolated, mp 178-180 °C. The infrared spectrum (Nujol mull) is not greatly different from the superposition of the spectra of the individual molecules and the solution NMR spectra are consistent with a chemically exchanging system. The ^1H NMR spectrum of $(Ph_3P)_2Pt(C_2H_4)$ in C_6D_6 (30 °C, 500 MHz) shows the ethylene protons at δ 2.63 and J Pt-H = 60 Hz. The ethylene protons of (I) are observed (C_6D_6, 30 °C, 90 MHz) at δ 2.18, J Pt-P = 56 Hz but the line

is broadened and no other coupling information is available. At 500 MHz, 30 °C, C_6D_6, the ethylene protons are not observable but at 50 °C a broadened triplet begins to appear at δ 2.23 which does not appreciably sharpen to 90 °C. At -70 °C in C_7D_8 (500 MHz) the Me_5C_5 resonance is broadened and overlaps with the ethylene resonance. This behavior is characteristic of a chemical exchange process that is rapid at high temperature, intermediate at 30 °C and apparently not stopped at -70 °C.[6]

Although the solution spectroscopy is ambiguous relative to structure, the solid state structure determined by X-ray diffraction at -80 °C is shown in the Figure.[7] The $(Ph_3P)_2Pt$ and $(Me_5C_5)_2Yb$ portions of (I) are only slightly perturbed relative to uncomplexed $(Ph_3P)_2Pt(\eta^2\text{-}C_2H_4)$[5b] and $(Me_5C_5)_2Yb(thf)$.[8] The averaged Pt-P and Pt-C distances are 2.268 ± 0.002 Å, 2.111 ± 0.003 Å and 2.275 ± 0.005 Å and 2.084 ± 0.001 Å in $(Ph_3P)_2(\eta^2\text{-}C_2H_4)$ and (I), respectively. The P-Pt-P and C-Pt-C angles in $(Ph_3P)_2Pt(\eta^2\text{-}C_2H_4)$ and (I) are 111.6(1)°, 39.7(4)° and 106.31(3)°, 40.3(1)°, respectively. The averaged Yb-C(Me_5C_5) and Yb-Me_5C_5 ring centroid distances and the Me_5C_5 ring centroid-Yb-Me_5C_5 ring centroid angle in $(Me_5C_5)_2Yb(thf)$ and (I) are 2.66 ± 0.01 Å, 2.37 Å, 143.5° and 2.67 ± 0.02 Å, 2.39 Å, 142.3°, respectively. The Yb-C(1,2) distances in (I) are 2.770(3) Å and 2.793(3) Å which average to 2.781 ± 0.006 Å and the C(1)-Yb-C(2) angle is 29.9(1)°. In $(Me_5C_5)_2Yb(\eta^2\text{-}MeC{\equiv}CMe)$ the equivalent parameters are 2.850 ± 0.010 Å and 23.4(1)°.[3b] The Yb-C (olefin) distance in (I) is similar to the divalent Yb to semi-bridging methyl distance in $Yb[N(SiMe_3)_2]_2(dmpe)$[9a] of 2.77 Å and 2.86 Å, in $Na[N(SiMe_3)_2]_3$[9b] of 2.86 Å and 2.91 Å, and in $Yb[N(SiMe_3)_2]_2(Me_3Al)_2$[9c] which range from 2.756(2) Å to 3.202(3) Å. In addition, the Yb to olefin distance is intermediate between the long and short bridging Lu-C distance of 2.756(9) Å and 2.440(9) Å, respectively, in $(Me_5C_5)_4Lu_2(Me)(\mu\text{-}Me)$,[1i] since Lu(III) is ca. 0.1 Å smaller than Yb(II) in a given coordination number.[9d]

The bridging olefin portion of (I) is the structural feature of principal interest. The hydrogen atoms on the olefin of (I) were located and refined isotropically whereas they were not located in $(Ph_3P)_2Pt(\eta^2\text{-}C_2H_4)$.[8a] The averaged C-H distance is 0.96 ± 0.06 Å, the averaged H-C-H, H-C-C, and H-C-Pt angles are 117.1 ± 0.3°, 117.8 ± 2.7°, and 112.2 ± 3.5°, respectively. The C-C distance in (I) of 1.436(5) Å is the same as that in $(Ph_3P)_2Pt(\eta^2\text{-}C_2H_4)$ of 1.43(1) Å. In addition α, the angle between the normals to the planes defined by the hydrogen atoms, is 52.7°.[2c] The intersection of the planes defined by H(1)C(1)H(2) and C(1)C(2)Pt, H(3)C(2)H(4) and C(1)C(2)Pt are 89.0 and 85.7°, respectively. Intersection of the PtC(1)C(2) and YbC(1)C(2) planes is 15.1°. This bending results in moving two of the four hydrogen atoms, H(1,3), towards the electropositive ytterbium atom, the averaged YbC(1)H(1) and YbC(2)H(3) angles being 69.5 ± 0.6° and the averaged YbC(1)H(2) and YbC(2)H(4) angles being 100.0 ± 1.4°. The bending results in two types of Yb...H contact distances, YbH(1) = 2.58(5) Å, YbH(3) = 2.64(3) Å, YbH(2) = 3.09(4) Å and YbH(4) = 3.15(3) Å. The shorter pair of distances is similar to those found in the semi-bridging methyl to divalent Yb distances in which two of the three hydrogens on each methyl group approach the ytterbium atom at distances of 2.53, 2.63, 2.72 and 3.13 Å in $Yb[N(SiMe_3)_2]_2(Me_3Al)_2$[9c] and the Yb...H contact distances of 2.77 and 2.86 Å in $Yb[N(SiMe_3)_2]_2(dmpe)$.[9a] The Yb...H distances are substantially longer than those found in the bridging hydrides, $Cp_4Lu_2(thf)_2(\mu\text{-}H)_2$ of 1.98(6) Å and 2.13(6) Å and $[Na(thf)_6][Cp_6Lu_2(\mu\text{-}H)]$ of 2.09 Å.[10]

The $Pt(C_2H_4)Yb$ interaction in (I) is obviously a weak interaction not unlike the d-transition metal olefin-alkali metal interactions found by Jonas and Klein.[11]

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Supplementary Material. Atomic positional parameters, thermal parameters, and tables of bond lengths and angles (9 pages).

References

1. (a) Yamamoto, A. "Organotransition Metal Chemistry," John Wiley, New York, 1986, Chap. 7. (b) Sinn, H.; Kaminsky, W. Adv. Organomet. Chem. **1980**, 18, 99-149. (c) Turner, H. W.; Schrock, R. R. J. Am. Chem. Soc. **1982**, 104, 2331-2333. (d) Schmidt, G. F.; Brookhart, M. Ibid. **1985**, 107, 1443-1444. (e) Clawson, L.; Soto, J.; Buchwald, S. L.; Steigerwald, M. L.; Grubbs, R. H. Ibid. **1985**, 107, 3377-3378. (f) Watson, P. L. Ibid. **1982**, 104, 337-339. (g) Watson, P. L.; Roe, D. C. Ibid. **1982**, 104, 6471-6473. (h) Watson, P. L.; Herskovitz, T. in "Initiation of Polymerization," Bailey, F. E., ed. ACS Symposium Series 212, **1983**, p. 459. (i) Watson, P. L.; Parshall, G. W. Acc. Chem. Res. **1985**, 18, 51-56. (j) Thomas, R. R.; Chebolm, V.; Sen, A. J. Am. Chem. Soc. **1986**, 108, 4096-4103.

2. (a) Green, M. L. H. "Organometallic Compounds," Vol. 2, 1968, Chapman and Hall, London, Chap. 2. (b) Herberhold, M. "Metal π-Complexes" 1974, Elsevier, Amsterdam. (c) Ittel, S. D.; Ibers, J. A. Adv. Organomet. Chem. **1976**, 14, 33-61. (d) Pasquali, M.; Floriani, C.; Gaetani-Manfredotti, A.; Chiesi-Villa, A. J. Am. Chem. Soc. **1978**, 100, 4918-4919. (e) Thompson, J. S.; Whitney, J. F. Inorg. Chem. **1984**, 23, 2813-2819.

3. (a) Cotton, F. A.; Schwotzer, W. J. Am. Chem. Soc. **1986**, 108, 4657-4658. (b) Burns, C. J.; Andersen, R. A., submitted.

4. (a) Andersen, R. A.; Boncella, J. M.; Burns, C. J.; Green, J. C.; Hohl, D.; Rosch, N. J. Chem. Soc. Chem. Comm. **1986**, 405-407. (b) Burns, C. J., unpublished results.

5. (a) Cook, C. D.; Jauhal, G. S. J. Am. Chem. Soc. **1968**, 90, 1464-1467. (b) Cheng, P. T.; Nyburg, S. C. Canad. J. Chem. **1972**, 50, 912-916.

6. $^{31}P\{^1H\}$NMR of (I): C_6D_6, 30 °C, 36.4 MHz, δ 33.0, JPtP = 3807 Hz. $(Ph_3P)_2Pt(C_2H_4)$: C_6D_6, 30 °C, 36.4 MHz, δ 3.40, JPtP = 3745 Hz. ^1H NMR of (I): C_6D_6, 30 °C, 500 MHz, δ 7.53(m), 6.94(m), phenyl H's; 2.08s Me_5C_5. $(Ph_3P)_2Pt(C_2H_4)$, C_6D_6, 30 °C, 500 MHz, δ 7.51(m), 6.94(m) phenyl H's; 2.63 JPtH = 60 Hz, C_2H_4.

7. The compound crystallizes in the triclinic space group P$\bar{1}$ with cell dimensions a = 9.577(2) Å, b = 14.797(2)Å, c = 18.429(2) Å, α = 96.90(1)°, β = 92.56(1)°, c = 102.77(1)°, V = 2522(1) Å^3, Z =2 and d(calc) = 1.57 gcm^{-3}. The data were collected on a Nonius CAD-4 automated diffractometer with MoK_α X-rays (λ = 0.71073 Å) at -80°C. The structure was solved from Patterson and electron density maps and refined by full-matrix least squares to a conventional R-factor of 0.020 (R_w=0.031, and GOF = 1.70) by using 5929 absorption corrected data, where $F_o^2 > 3\sigma(F_o^2)$, against 576 variables. The R-valve for all 6568 unique data was 0.026. The non-hydrogen atoms were refined anisotropically. The hydrogen atoms on the ethylene carbons were located in a difference Fourier map and they were refined isotropically. The hydrogen atoms on the phenylcarbons and on the Me_5C_5-ring methyls were located in a difference Fourier map, placed in calculated positions with fixed thermal parameters, included in structure factor calculations but were not refined.

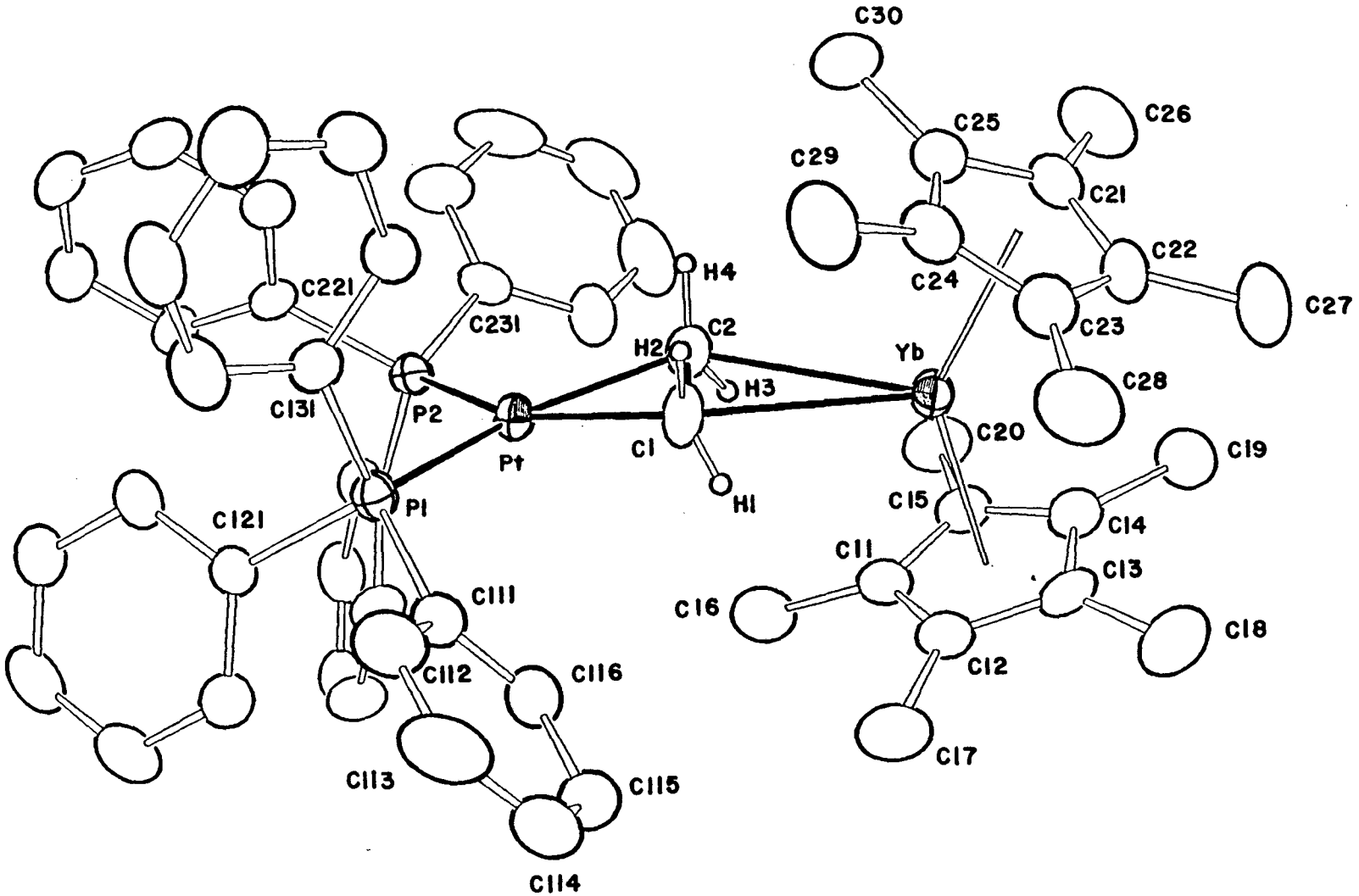
8. Tilley, T. D.; Andersen, R. A.; Spencer, B.; Ruben, H; Zalkin, A.; Templeton, D. H. Inorg. Chem. **1980**, 19, 2999-3003.

9. (a) Tilley, T. D.; Andersen, R. A.; Zalkin, A., J. Am. Chem. Soc. **1982**, 104, 3725-3727. (b) Tilley, T. D.; Andersen, R. A.; Zalkin, A. Inorg. Chem. **1984**, 23, 2271-2276. (c) Boncella, J. M.; Andersen, R. A. Organometallics **1985**, 4, 205-206. (d) Shannon, R. D. Acta Cryst. **1976**, 32A, 751-767.

10. Schumann, H.; Genthe, W.; Hahn E.; Hossain, M.B.; Van Der Helm, D. J. Organomet. Chem. **1986**, 299, 67-84.

11. (a) Jonas, K. Angew. Chem. Int. Ed. **1976**, 15, 47. (b) Brauer, D. J.; Krüger, C.; Roberts, P. J.; Tsui, Y. H. Ibid. **1976**, 15, 48. (c) Goddard, R.; Krüger, C.; Pörschke, K. R.; Wilke, G. J. Organomet. Chem. **1986**, 308, 85-103. (d) Klein, H. F.; Gross, J.; Bassett, J. M.; Schubert, U. Z. Naturforsch. **1980**, 35b, 614-619. (e) Klein, H. F.; Witty, H.; Schubert, U. J. Chem. Soc. Chem. Comm. **1983**, 231-232.

An ORTEP diagram of (I), the ellipsoids, represent 50% probability surfaces, except those of the hydrogen atoms on the ethylene unit which are arbitrary.


C30
C25
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C29
C21
C22
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C27
C23
C28
C221
C231
H4
C2
H2
H3
Yb
C131
P2
Pt
C1
C20
C19
H1
C15
C14
P1
C121
C11
C13
C111
C16
C18
C112
C116
C12
C17
C113
C115
C114

XBL 868-2846

Supplementary Material for

Preparation of the First η^2-Olefin Complex of a 4f-Transition Metal, $(Me_5C_5)_2Yb(\mu\text{-}C_2H_4)Pt(PPh_3)_2$

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Atomic Positional Parameters
Thermal Parameters
Bond Lengths and Angles

Table of Positional Parameters and Their Estimated Standard Deviations

Atom	x	y	z	B(Å^2)
PT	0.22439(1)	0.34578(1)	0.22261(1)	1.756(3)
YB	0.36512(2)	0.65579(1)	0.32113(1)	1.950(4)
P1	0.3230(1)	0.22866(7)	0.25651(5)	1.97(2)
P2	0.0619(1)	0.28012(7)	0.12718(5)	1.87(2)
C1	0.3206(5)	0.4628(3)	0.2972(2)	2.70(9)
C2	0.2050(4)	0.4824(3)	0.2539(2)	2.64(9)
C11	0.4794(4)	0.6468(3)	0.1903(2)	2.45(9)
C12	0.5963(4)	0.6745(3)	0.2430(2)	2.60(9)
C13	0.5970(4)	0.7660(3)	0.2766(2)	2.51(9)
C14	0.4782(4)	0.7939(3)	0.2445(2)	2.62(9)
C15	0.4065(4)	0.7195(3)	0.1913(2)	2.46(9)
C16	0.4504(5)	0.5601(4)	0.1341(2)	3.8(1)
C17	0.7087(5)	0.6209(4)	0.2567(3)	4.0(1)
C18	0.7148(5)	0.8261(4)	0.3293(3)	4.2(1)
C19	0.4449(5)	0.8892(3)	0.2569(2)	3.5(1)
C20	0.2768(5)	0.7210(4)	0.1423(2)	3.7(1)
C21	0.2144(5)	0.7391(3)	0.4148(2)	2.85(9)
C22	0.3609(5)	0.7739(3)	0.4407(2)	2.9(1)
C23	0.4097(5)	0.6981(3)	0.4645(2)	2.9(1)
C24	0.2957(5)	0.6194(3)	0.4556(2)	2.95(9)
C25	0.1740(5)	0.6440(3)	0.4245(2)	2.93(9)
C26	0.1162(6)	0.7933(4)	0.3840(3)	5.2(1)
C27	0.4383(6)	0.8748(4)	0.4537(3)	4.8(1)
C28	0.5577(6)	0.7005(4)	0.4965(3)	4.6(1)
C29	0.2972(6)	0.5279(3)	0.4843(3)	4.6(1)
C30	0.0250(5)	0.5831(4)	0.4101(3)	4.9(1)
C111	0.5105(4)	0.2673(3)	0.2937(2)	2.24(8)
C112	0.5692(5)	0.2235(3)	0.3466(2)	3.4(1)
C113	0.7119(5)	0.2562(4)	0.3720(2)	4.0(1)
C114	0.7965(5)	0.3301(3)	0.3451(3)	3.8(1)
C115	0.7418(5)	0.3725(3)	0.2919(3)	3.8(1)
C116	0.5974(4)	0.3405(3)	0.2669(2)	2.96(9)
C121	0.3298(4)	0.1262(3)	0.1912(2)	1.99(8)
C122	0.4548(4)	0.1220(3)	0.1560(2)	2.68(9)
C123	0.4554(5)	0.0494(3)	0.1003(2)	3.3(1)
C124	0.3325(5)	-0.0209(3)	0.0817(2)	3.3(1)
C125	0.2106(5)	-0.0179(3)	0.1166(2)	3.0(1)
C126	0.2079(5)	0.0562(3)	0.1711(2)	2.72(9)
C131	0.2259(4)	0.1706(3)	0.3311(2)	2.29(8)
C132	0.1464(4)	0.2315(3)	0.3714(2)	2.54(9)
C133	0.0709(5)	0.1990(3)	0.4297(2)	3.4(1)
C134	0.0771(6)	0.1129(4)	0.4472(3)	5.2(1)
C135	0.1591(7)	0.0508(3)	0.4009(3)	6.0(1)
C136	0.2314(6)	0.0915(3)	0.3502(3)	4.2(1)

Table of Positional Parameters and Their Estimated Standard Deviations (cont.)

Atom	x	y	z	$B(A^2)$
C211	0.1363(4)	0.2614(3)	0.0387(2)	1.96(8)
C212	0.0569(4)	0.2547(3)	-0.0279(2)	2.55(9)
C213	0.1197(5)	0.2434(3)	-0.0935(2)	3.2(1)
C214	0.2612(5)	0.2397(3)	-0.0938(2)	3.4(1)
C215	0.3412(5)	0.2454(3)	-0.0295(2)	3.5(1)
C216	0.2811(4)	0.2572(3)	0.0375(2)	2.88(9)
C221	-0.0527(4)	0.1670(3)	0.1380(2)	1.98(8)
C222	-0.0841(4)	0.0907(3)	0.0844(2)	2.39(9)
C223	-0.1660(5)	0.0060(3)	0.0986(2)	3.0(1)
C224	-0.2166(5)	-0.0028(3)	0.1665(3)	3.3(1)
C225	-0.1885(5)	0.0732(3)	0.2210(2)	3.3(1)
C226	-0.1056(4)	0.1582(3)	0.2074(2)	2.70(9)
C231	-0.0656(4)	0.3494(3)	0.1050(2)	2.34(8)
C232	-0.0111(5)	0.4421(3)	0.0952(3)	3.4(1)
C233	-0.0977(6)	0.4983(3)	0.0759(3)	4.8(1)
C234	-0.2413(5)	0.4659(4)	0.0670(3)	4.7(1)
C235	-0.3009(5)	0.3753(4)	0.0766(3)	5.3(1)
C236	-0.2140(5)	0.3165(4)	0.0955(3)	3.8(1)
H1	0.407(5)	0.489(4)	0.292(3)	6(1)*
H2	0.294(5)	0.450(3)	0.351(2)	5(1)*
H3	0.230(4)	0.521(2)	0.218(2)	2.1(8)*
H4	0.112(4)	0.479(3)	0.273(2)	2.8(9)*

* -- Atoms refined with isotropic thermal parameters.

Anisotropically refined atoms are given in the form of the isotropic equivalent thermal parameter defined as:

$$(4/3) * [a^2*B(1,1) + b^2*B(2,2) + c^2*B(3,3) + ab(\cos gamma)*B(1,2)$$

$$+ ac(\cos beta)*B(1,3) + bc(\cos alpha)*B(2,3)]$$

Table of Positional Parameters and Their Estimated Standard Deviations (cont.)

Atom	x	y	z	B(Å^2)
H112	0.5116	0.1704	0.3657	5.5**
H113	0.7530	0.2268	0.4088	5.5**
H114	0.8947	0.3527	0.3640	5.5**
H115	0.8006	0.4235	0.2711	5.5**
H116	0.5570	0.3707	0.2309	5.5**
H122	0.5409	0.1700	0.1705	5.0**
H123	0.5407	0.0476	0.0746	5.0**
H124	0.3348	-0.0727	0.0444	5.0**
H125	0.1257	-0.0682	0.1025	5.0**
H126	0.1202	0.0592	0.1952	5.0**
H132	0.1441	0.2919	0.3584	5.0**
H133	0.0137	0.2368	0.4560	5.0**
H134	0.0257	0.0903	0.4883	7.0**
H135	0.1628	-0.0020	0.4208	7.5**
H136	0.2870	0.0549	0.3224	7.0**
H161	0.5024	0.5712	0.0917	5.5**
H162	0.4796	0.5094	0.1543	5.5**
H163	0.3507	0.5405	0.1192	5.5**
H171	0.7849	0.6349	0.2256	5.5**
H172	0.7501	0.6392	0.3072	5.5**
H173	0.6698	0.5556	0.2506	5.5**
H181	0.7880	0.8602	0.3047	5.5**
H182	0.6769	0.8690	0.3624	5.5**
H183	0.7557	0.7877	0.3594	5.5**
H191	0.4972	0.9300	0.2272	5.5**
H192	0.3472	0.8864	0.2520	5.5**
H193	0.4788	0.9173	0.3089	5.5**
H201	0.3036	0.7454	0.0985	5.5**
H202	0.2172	0.6594	0.1304	5.5**
H203	0.2191	0.7596	0.1673	5.5**
H212	-0.0431	0.2567	-0.0275	5.0**
H213	0.0645	0.2383	-0.1396	5.0**
H214	0.3077	0.2344	-0.1386	5.0**
H215	0.4390	0.2383	-0.0309	5.0**
H216	0.3367	0.2641	0.0828	5.0**
H222	-0.0498	0.0965	0.0368	5.0**
H223	-0.1870	-0.0473	0.0609	5.0**
H224	-0.2693	-0.0630	0.1763	5.0**
H225	-0.2302	0.0671	0.2677	5.0**
H226	-0.0837	0.2120	0.2452	5.0**
H232	0.0903	0.4678	0.1033	5.0**
H233	-0.0540	0.5632	0.0699	7.0**
H234	-0.3043	0.5053	0.0533	7.0**
H235	-0.4042	0.3508	0.0690	7.0**

Table of Positional Parameters and Their Estimated Standard Deviations (cont.)

Atom	x	y	z	B($Å^2$)
H236	-0.2578	0.2520	0.1031	6.0**
H261	0.0700	0.8212	0.4234	7.0**
H262	0.1727	0.8443	0.3620	7.0**
H263	0.0479	0.7558	0.3494	7.0**
H271	0.4181	0.9032	0.5008	7.0**
H272	0.5387	0.8809	0.4531	7.0**
H273	0.4077	0.9091	0.4173	7.0**
H281	0.5629	0.7162	0.5480	7.0**
H282	0.5804	0.6413	0.4846	7.0**
H283	0.6258	0.7467	0.4763	7.0**
H291	0.2674	0.5298	0.5331	7.0**
H292	0.2305	0.4776	0.4540	7.0**
H293	0.3896	0.5157	0.4835	7.0**
H301	-0.0291	0.5864	0.4522	7.0**
H302	-0.0257	0.6012	0.3703	7.0**
H303	0.0288	0.5186	0.3968	7.0**
CP1	0.5115	0.7201	0.2292	
CP2	0.2909	0.6949	0.4400	

** -- Atoms included but not refined.

Table of General Temperature Factor Expressions - B's

Name	B(1,1)	B(2,2)	B(3,3)	B(1,2)	B(1,3)	B(2,3)	Beqv
PT	1.801(6)	1.496(6)	1.838(6)	0.207(5)	0.084(5)	0.028(5)	1.756(3)
YB	2.248(7)	1.575(7)	1.916(6)	0.205(6)	0.380(6)	0.141(6)	1.950(4)
P1	2.04(4)	1.87(4)	1.92(4)	0.35(3)	0.10(3)	0.07(3)	1.97(2)
P2	1.72(4)	1.86(4)	1.96(4)	0.33(3)	0.24(3)	0.09(3)	1.87(2)
C1	3.1(2)	1.6(2)	3.0(2)	0.0(1)	-0.3(2)	-0.1(1)	2.70(9)
C2	2.8(2)	1.9(2)	3.1(2)	0.3(1)	0.5(1)	0.2(1)	2.64(9)
C11	2.4(2)	2.8(2)	2.3(2)	0.7(1)	0.8(1)	0.7(1)	2.45(9)
C12	2.3(2)	2.8(2)	3.0(2)	0.8(1)	0.8(1)	1.0(1)	2.60(9)
C13	1.7(2)	2.8(2)	2.9(2)	-0.1(1)	0.3(1)	1.2(1)	2.51(9)
C14	2.7(2)	2.5(2)	2.8(2)	0.4(1)	0.8(1)	1.0(1)	2.62(9)
C15	2.5(2)	2.9(2)	2.1(1)	0.4(1)	0.7(1)	1.0(1)	2.46(9)
C16	3.8(2)	4.4(2)	3.0(2)	0.7(2)	1.2(2)	-0.1(2)	3.8(1)
C17	2.7(2)	4.6(2)	5.3(2)	1.4(2)	0.7(2)	1.6(2)	4.0(1)
C18	3.3(2)	4.5(2)	4.2(2)	-0.2(2)	-0.3(2)	0.7(2)	4.2(1)
C19	4.2(2)	2.8(2)	4.0(2)	1.1(2)	1.1(2)	1.2(2)	3.5(1)
C20	2.9(2)	4.9(2)	3.4(2)	0.8(2)	0.0(2)	1.2(2)	3.7(1)
C21	3.2(2)	2.6(2)	2.7(2)	0.9(1)	0.7(1)	-0.1(1)	2.85(9)
C22	4.0(2)	2.1(2)	2.2(2)	0.1(2)	0.7(1)	-0.4(1)	2.9(1)
C23	3.3(2)	3.3(2)	2.0(2)	0.5(2)	0.2(1)	0.1(1)	2.9(1)
C24	4.3(2)	2.8(2)	2.0(1)	1.0(2)	1.2(1)	0.5(1)	2.95(9)
C25	3.3(2)	2.8(2)	2.5(2)	0.4(2)	1.1(1)	0.1(1)	2.93(9)
C26	5.6(2)	5.0(2)	5.8(3)	3.2(2)	1.1(2)	0.7(2)	5.2(1)

Table of General Temperature Factor Expressions - B's (Continued)

Name	B(1,1)	B(2,2)	B(3,3)	B(1,2)	B(1,3)	B(2,3)	Beqv
C27	6.7(3)	2.8(2)	4.1(2)	-Ø.1(2)	1.2(2)	-Ø.7(2)	4.8(1)
C28	4.6(2)	6.3(3)	3.1(2)	1.8(2)	-Ø.1(2)	Ø.1(2)	4.6(1)
C29	7.7(3)	3.5(2)	3.Ø(2)	1.6(2)	1.4(2)	1.1(2)	4.6(1)
C3Ø	3.5(2)	4.8(3)	5.6(2)	-Ø.2(2)	1.9(2)	-Ø.4(2)	4.9(1)
C111	2.2(2)	2.5(2)	1.9(1)	Ø.6(1)	-Ø.1(1)	-Ø.2(1)	2.24(8)
C112	3.6(2)	4.3(2)	2.6(2)	1.4(2)	Ø.4(2)	Ø.6(2)	3.4(1)
C113	3.5(2)	6.6(2)	2.4(2)	2.6(2)	-Ø.4(2)	Ø.1(2)	4.Ø(1)
C114	2.3(2)	4.2(2)	4.5(2)	1.1(2)	-Ø.5(2)	-1.3(2)	3.8(1)
C115	2.1(2)	3.Ø(2)	6.3(3)	Ø.7(2)	Ø.1(2)	-Ø.Ø(2)	3.8(1)
C116	2.4(2)	2.4(2)	4.1(2)	Ø.9(1)	-Ø.2(2)	Ø.3(2)	2.96(9)
C121	2.Ø(1)	2.Ø(1)	2.Ø(1)	Ø.6(1)	-Ø.Ø(1)	Ø.1(1)	1.99(8)
C122	2.8(2)	2.7(2)	2.4(2)	Ø.4(1)	Ø.4(1)	Ø.1(1)	2.68(9)
C123	4.Ø(2)	3.9(2)	2.4(2)	2.Ø(2)	Ø.8(1)	Ø.3(2)	3.3(1)
C124	4.6(2)	3.1(2)	2.4(2)	1.8(2)	Ø.Ø(2)	-Ø.7(2)	3.3(1)
C125	3.3(2)	2.3(2)	3.2(2)	Ø.4(2)	-Ø.1(2)	-Ø.2(2)	3.Ø(1)
C126	3.1(2)	2.2(2)	2.8(2)	Ø.7(1)	Ø.1(1)	-Ø.3(1)	2.72(9)
C131	2.1(2)	2.3(2)	2.4(2)	Ø.2(1)	Ø.3(1)	Ø.3(1)	2.29(8)
C132	2.9(2)	2.6(2)	2.2(2)	Ø.7(1)	Ø.4(1)	Ø.5(1)	2.54(9)
C133	3.7(2)	3.4(2)	3.3(2)	1.Ø(2)	1.Ø(2)	Ø.4(2)	3.4(1)
C134	7.8(3)	3.7(2)	4.6(2)	1.Ø(2)	3.2(2)	1.6(2)	5.2(1)
C135	1Ø.2(3)	2.5(2)	6.Ø(2)	1.8(2)	5.2(2)	1.6(2)	6.Ø(1)
C136	6.3(2)	2.7(2)	4.3(2)	1.7(2)	2.3(2)	1.Ø(2)	4.2(1)

Table of General Temperature Factor Expressions - B's (Continued)

Name	B(1,1)	B(2,2)	B(3,3)	B(1,2)	B(1,3)	B(2,3)	Beqv
C211	2.3(2)	1.Ø(1)	2.3(1)	-Ø.Ø(1)	Ø.4(1)	-Ø.1(1)	1.96(8)
C212	2.8(2)	2.3(2)	2.5(2)	Ø.6(1)	Ø.1(1)	Ø.2(1)	2.55(9)
C213	5.Ø(2)	2.5(2)	2.1(2)	Ø.8(2)	Ø.5(2)	Ø.2(1)	3.2(1)
C214	5.Ø(2)	2.1(2)	2.9(2)	Ø.3(2)	2.Ø(2)	Ø.1(1)	3.4(1)
C215	2.8(2)	4.1(2)	3.5(2)	Ø.4(2)	1.3(2)	Ø.3(2)	3.5(1)
C216	2.4(2)	3.3(2)	2.9(2)	Ø.2(2)	Ø.6(1)	Ø.6(2)	2.88(9)
C221	1.3(1)	2.3(2)	2.1(1)	Ø.1(1)	Ø.1(1)	Ø.2(1)	1.98(8)
C222	2.4(2)	2.3(2)	2.2(2)	-Ø.Ø(1)	Ø.3(1)	Ø.2(1)	2.39(9)
C223	2.8(2)	2.5(2)	3.4(2)	Ø.1(2)	-Ø.1(2)	Ø.Ø(2)	3.Ø(1)
C224	2.8(2)	2.4(2)	4.4(2)	-Ø.2(2)	Ø.7(2)	1.Ø(2)	3.3(1)
C225	2.9(2)	3.5(2)	3.4(2)	Ø.2(2)	1.3(1)	1.Ø(2)	3.3(1)
C226	2.5(2)	2.7(2)	2.9(2)	Ø.5(1)	Ø.6(1)	Ø.3(1)	2.7Ø(9)
C231	2.1(1)	3.3(2)	1.6(1)	1.Ø(1)	Ø.Ø(1)	-Ø.3(1)	2.34(8)
C232	3.5(2)	2.4(2)	4.3(2)	1.Ø(2)	-Ø.3(2)	Ø.3(2)	3.4(1)
C233	6.6(3)	3.6(2)	4.9(2)	2.6(2)	-Ø.5(2)	Ø.4(2)	4.8(1)
C234	5.4(2)	6.1(2)	3.9(2)	4.Ø(2)	Ø.5(2)	Ø.7(2)	4.7(1)
C235	2.7(2)	9.7(3)	4.4(2)	3.4(2)	Ø.3(2)	Ø.7(2)	5.3(1)
C236	2.4(2)	5.2(2)	3.9(2)	1.Ø(2)	Ø.3(2)	Ø.6(2)	3.8(1)

The form of the anisotropic thermal parameter is:

$\exp[-Ø.25(h^2 a^{*2} B(1,1) + k^2 b^{*2} B(2,2) + l^2 c^{*2} B(3,3)' + 2hka^*b^*B(1,2) + 2hla^*c^*B(1,3)$

$+ 2klb^*c^*B(2,3))]$, where a*,b*, and c* are reciprocal lattice constants.

Intramolecular Angles

ATOM 1	ATOM 2	ATOM 3	ANGLE
YB	C1	PT	142.68(17)
YB	C2	PT	141.06(17)
YB	C1	C2	75.89(19)
YB	C2	C1	74.18(19)
PT	C1	C2	69.80(19)
PT	C2	C1	69.82(20)
C1	YB	C2	29.93(11)
C1	PT	C2	40.30(14)
H1	C1	H2	116.6(37)
H1	C1	C2	120.8(33)
H2	C1	C2	113.3(21)
H3	C2	H4	117.6(28)
H3	C2	C1	116.9(20)
H4	C2	C1	120.3(20)
H1	C1	YB	68.3(33)
H2	C1	YB	97.2(20)
H3	C2	YB	70.7(20)
H4	C2	YB	102.8(20)
H1	C1	PT	118.0(32)
H2	C1	PT	109.5(20)
H3	C2	PT	113.4(20)
H4	C2	PT	107.8(20)
CP1	YB	C1	108.7
CP1	YB	C2	105.5
CP2	YB	C1	106.8
CP2	YB	C2	111.6
CP1	YB	H1	95.4
CP1	YB	H2	127.2
CP1	YB	H3	88.0
CP1	YB	H4	118.7
CP2	YB	H1	115.9
CP2	YB	H2	86.8
CP2	YB	H3	127.3
CP2	YB	H4	96.6
CP1	YB	CP2	142.3
C12	C11	C15	108.4(3)
C11	C12	C13	107.9(3)
C12	C13	C14	107.7(3)
C13	C14	C15	107.5(3)
C11	C15	C14	108.5(3)
C12	C11	C16	125.6(3)
C15	C11	C16	125.4(3)
C11	C12	C17	126.2(3)
C13	C12	C17	125.7(3)
C12	C13	C18	124.9(3)
C14	C13	C18	126.8(3)
C13	C14	C19	125.9(3)
C15	C14	C19	126.3(3)
C11	C15	C20	126.6(3)
C14	C15	C20	124.9(3)
C22	C21	C25	108.5(3)
C21	C22	C23	106.8(3)
C22	C23	C24	108.8(3)
C23	C24	C25	108.4(3)
C21	C25	C24	107.5(3)
C22	C21	C26	126.8(4)
C25	C21	C26	124.7(4)
C21	C22	C27	125.7(4)
C23	C22	C27	126.5(4)
C22	C23	C28	126.6(3)
C24	C23	C28	124.5(4)
C23	C24	C29	125.4(4)
C25	C24	C29	125.7(4)
C21	C25	C30	125.6(4)
C24	C25	C30	126.7(3)
P1	PT	C1	104.70(10)
P1	PT	C2	144.18(10)
P2	PT	C1	148.91(10)
P2	PT	C2	108.69(10)
P1	PT	P2	106.31(3)
PT	P1	C111	114.25(11)
PT	P1	C121	121.51(10)
PT	P1	C131	109.13(11)
C111	P1	C121	101.60(14)
C111	P1	C131	104.77(14)
C121	P1	C131	103.86(14)
PT	P2	C211	115.51(10)
PT	P2	C221	115.33(10)
PT	P2	C231	115.21(10)
C211	P2	C221	104.91(13)
C211	P2	C231	100.62(14)
C221	P2	C231	103.45(15)
P1	C111	C112	122.7(3)
P1	C111	C116	118.50(24)
C112	C111	C116	118.7(3)
C111	C112	C113	119.6(4)
C112	C113	C114	120.7(4)
C113	C114	C115	120.5(3)
C114	C115	C116	118.8(4)
C111	C116	C115	121.6(3)
P1	C121	C122	120.36(24)
P1	C121	C126	120.52(24)
C122	C121	C126	118.9(3)
C121	C122	C123	120.4(3)
C122	C123	C124	120.0(3)
C123	C124	C125	119.9(3)
C124	C125	C126	120.5(3)
C121	C126	C125	120.3(3)
P1	C131	C132	118.21(25)
P1	C131	C136	123.1(3)
C132	C131	C136	118.7(3)
C131	C132	C133	121.8(3)
C132	C133	C134	118.5(3)
C133	C134	C135	121.2(4)
C134	C135	C136	119.5(4)
C131	C136	C135	120.3(4)
P2	C211	C212	122.85(25)
P2	C211	C216	118.50(24)
C212	C211	C216	118.5(3)
C211	C212	C213	120.6(3)
C212	C213	C214	120.3(3)
C213	C214	C215	120.4(3)
C214	C215	C216	120.6(3)
C211	C216	C215	119.6(3)
P2	C221	C222	124.40(23)
P2	C221	C226	116.34(23)
C222	C221	C226	119.2(3)
C221	C222	C223	120.6(3)
C222	C223	C224	120.2(3)
C223	C224	C225	120.4(3)
C224	C225	C226	119.7(3)
C221	C226	C225	119.9(3)
P2	C231	C232	117.8(3)
P2	C231	C236	125.2(3)
C232	C231	C236	117.0(3)
C231	C232	C233	122.0(4)
C232	C233	C234	120.6(4)
C233	C234	C235	119.8(4)
C234	C235	C236	120.4(4)
C231	C236	C235	120.2(4)

Intramolecular Distances

ATOM 1	ATOM 2	DISTANCE
PT	C1	2.084(3)
PT	C2	2.085(3)
YB	C1	2.770(3)
YB	C2	2.792(3)
C1	C2	1.436(5)
C1	H1	0.85(5)
C1	H2	1.07(4)
C2	H3	0.93(3)
C2	H4	0.97(4)
YB	H1	2.58(5)
YB	H2	3.09(4)
YB	H3	2.64(3)
YB	H4	3.15(3)
PT	H1	2.59(5)
PT	H2	2.64(4)
PT	H3	2.60(3)
PT	H4	2.55(4)
YB	C11	2.693(3)
YB	C12	2.675(3)
YB	C13	2.672(3)
YB	C14	2.681(3)
YB	C15	2.690(3)
YB	C21	2.660(3)
YB	C22	2.650(3)
YB	C23	2.634(3)
YB	C24	2.679(3)
YB	C25	2.694(3)
YB	CP1	2.398
YB	CP2	2.378
C11	C12	1.403(5)
C11	C15	1.405(5)
C12	C13	1.419(5)
C13	C14	1.422(5)
C14	C15	1.410(5)
C11	C16	1.515(5)
C12	C17	1.502(5)
C13	C18	1.501(5)
C14	C19	1.511(5)
C15	C20	1.508(5)
C21	C22	1.422(5)
C21	C25	1.410(5)
C22	C23	1.414(5)
C23	C24	1.396(5)
C24	C25	1.414(5)
C21	C26	1.498(5)
C22	C27	1.499(5)
C23	C28	1.503(5)
C24	C29	1.515(5)
C25	C30	1.503(5)
PT	P1	2.284(1)
PT	P2	2.266(1)
P1	C111	1.833(3)
P1	C121	1.834(3)
P1	C131	1.833(3)
P2	C211	1.823(3)
P2	C221	1.825(3)
P2	C231	1.822(3)
C111	C112	1.398(5)
C111	C116	1.369(5)
C112	C113	1.384(6)
C113	C114	1.369(6)
C114	C115	1.370(6)
C115	C116	1.396(5)
C121	C122	1.396(4)
C121	C126	1.381(5)
C122	C123	1.389(5)
C123	C124	1.387(5)
C124	C125	1.364(5)
C125	C126	1.400(5)
C131	C132	1.384(5)
C131	C136	1.388(5)
C132	C133	1.397(5)
C133	C134	1.364(6)
C134	C135	1.396(6)
C135	C136	1.389(5)
C211	C212	1.397(4)
C211	C216	1.403(5)
C212	C213	1.381(5)
C213	C214	1.368(5)
C214	C215	1.367(5)
C215	C216	1.394(5)
C221	C222	1.377(4)
C221	C226	1.406(4)
C222	C223	1.385(5)
C223	C224	1.373(5)
C224	C225	1.383(5)
C225	C226	1.389(5)
C231	C232	1.393(5)
C231	C236	1.392(5)
C232	C233	1.361(5)
C233	C234	1.348(6)
C234	C235	1.373(7)
C235	C236	1.391(6)
PT	YB	4.605

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