[5166 with $I > 3\sigma(I)$] (I) and 7919 [3007 with $I > 3\sigma(I)$] (II) diffracted intensities were collected at room temperature with variable scan speed (from 2 to 20° $\min^{-1}$) and a variable scan range of ($\alpha + 0.35 \tan \theta$), with $\alpha = 0.8°$ (I) and $\alpha = 0.9°$ (II); the maximum scanning time for reflections was 60 (I) and 70 s (II). The crystal stability under diffraction condition was checked by monitoring three standard reflections every 60 min, and no crystal decay was observed. The measured intensities were corrected for Lorentz, polarization, and background effects and reduced to F_o. An empirical absorption correction was applied as described[17] using ψ-scans of three suitable reflections having χ values close to 90°.

(b) Solution and Structure Refinement. Crystal data are summarized in Table V. For compound I the metal atoms positions were taken from that of the isomorphous (PPN)[$RuRh_5(CO)_{16}$][4] in the centrosymmetric space group $C2/c$ (No. 15). For compound II from systematic absences ($0k0$, $k = 2n + 1$; $h0l$, $l = 2n + 1$), the centrosymmetric space group $P2_1/c$ (No. 14) was uniquely determined. The positions of the four metal atoms were obtained from a Patterson map. In both structures the remaining lighter P, O, N, and C atoms were located from subsequent difference Fourier syntheses; the hydrogen atoms of the PPN cations were placed in idealized positions (C–H = 0.95 Å) and refined riding on their parent atoms with fixed isotropic thermal parameters (B = 5.0 Å^2). Anisotropic thermal parameters were assigned to all the atoms of the anions and to the P atoms of the cations, while to the N and C atoms of the cations were given isotropic B's. Full-matrix least-squares refinement of 515 (I) and 419 (II) variables converged to R, R_w, and GOF factors of 0.030, 0.031, and 1.166 (I) and 0.037, 0.037, and 1.138 (II), the minimized function being $\sum w(|F_o| - k|F_c|)^2$. Individual weights were assigned as $w = 1/\sigma^2(F_o)$, where $\sigma(F_o) = \sigma(F_o^2)/2F_o$ and $\sigma(F_o^2) = [\sigma^2(I) + (pI)^2]^{1/2}/LP$, where $\sigma(F_o^2)$ is the standard deviation for each reflection as derived from counting statistics, p (0.030 for I and 0.035 for II is a coefficient for improving the goodness of fit, and LP is the Lorentz–polarization factor. In compound I all the metal atoms were found having variable mixed Os/Rh occupancies; practically, we refined all the metals as Rh atoms with free multiplicities. This lead to the final values of 1.04, 1.04, 1.12, 1.05, 1.31, and 1.13 for the M1–M6 multiplicities, respectively, in perfect agreement with the presence of just one Os atom. In the last residual Fourier map, peaks not exceeding 0.43 (I) and 0.56 (II) e/Å^3 were found. Scattering factors for neutral atoms and anomalous dispersion corrections for scattering factors were taken from refs 18 and 19, respectively. Final selected atomic coordinates with their estimated standard deviations are presented in Tables VI (I) and VII (II), respectively. All the calculations were performed on a PDP 11/73 computer using the SDP-Plus structure determination package.[20]

Supplementary Material Available: Detailed lists of crystallographic parameters (Tables S1 (I) and S7 (II)), full lists of atomic coordinates (Tables S2 (I) and S8 (II)), lists of anisotropic thermal factors (Tables S3 (I) and S9 (II)), and full lists of bond distances and angles (Tables S4 and S5 (I) and Tables S10 and S11 (II)) (18 pages); lists of observed and calculated structure factor moduli (Tables S6 (I) and S12 (II)) (56 pages). Ordering information is given on any current masthead page.

---

(17) North, A. C. T.; Phillips, D. C.; Mathews, F. S. *Acta Crystallogr., Sect. A* **1968**, *24*, 351.

(18) Cromer, D. T. *International Tables for X-Ray Crystallography*; The Kynoch Press: Birmingham, England, 1974 (present distributor Kluwer Academic Publishers, Dordrecht, The Netherlands); Vol. 4, Table 2.3.1.

(19) Cromer, D. T.; Waber, J. T. *International Tables for X-Ray Crystallography*; The Kynoch Press: Birmingham, England, 1974 (present distributor Kluwer Academic Publishers, Dordrecht, The Netherlands); Vol. 4, Table 2.2.b.

(20) Frenz, B. A.; et al. *SDP Structure Determination Package Plus*, Version 1.0; Enraf-Nonius: Delft, The Netherlands, 1980.

Contribution from the Departments of Chemistry, University of Warwick, Coventry, U.K., and Imperial College of Science, Technology and Medicine, London, U.K.

Ruthenatetraboranes: Synthesis of [$Ru(B_3H_8)(PPh_3)\{HB(pz)_3\}$] and Crystal Structure of [$RuCl(PPh_3)_2\{HB(pz)_3\}$] (pz = Pyrazol-1-yl)

Nathaniel W. Alcock,[†] Ian. D. Burns,[‡] Kanwaljit S. Claire,[†] and Anthony F. Hill*[,‡]

Received December 20, 1991

The reaction of [$RuCl_2(PPh_3)_3$] with K[$HB(pz)_3$] (pz = pyrazol-1-yl) affords [$RuCl(PPh_3)_2\{\kappa^3\text{-}HB(pz)_3\}$] [crystallographically characterized with monoclinic crystals of space group $P2_1/a$, $Z = 8$, in a unit cell of dimensions a = 19.75 (1) Å, b = 22.48 (1) Å, c = 20.283 (7) Å, and β = 112.94 (3)°], which upon treatment with $NR_4[B_3H_8]$ (R = Me, Bu) provides the ruthenatetraborane [$Ru(B_3H_8)(PPh_3)\{\kappa^3\text{-}HB(pz)_3\}$].

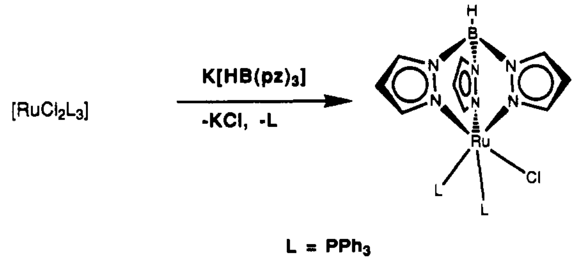
Introduction

Metallaboranes of low nuclearity, in particular the metallatetraboranes, are of interest as possible precursors to clusters of higher nuclearity.[1] Nevertheless very little is known about the reactivity of these compounds. The established reactions of metal octahydrotriborates are essentially limited to (i) displacement of the B_3H_8 moiety,[2] (ii) halogenation of the unique wingtip BH_2 group,[3] (iii) *agostic* coordination of the wingtip BH_2 group to an otherwise coordinatively unsaturated metal center,[3] and (iv) dehydrohalogenative formation of boraallyl complexes[4,5] (by implication). We describe here the convenient synthesis of an air-stable ruthenatetraborane from the new complex [$RuCl(PPh_3)_2\{\kappa^3\text{-}HB(pz)_3\}$] (pz = pyrazol-1-yl).

Results and Discussion

Synthesis of [$RuCl(PPh_3)_2\{\kappa^3\text{-}HB(pz)_3\}$]. The complex [$RuCl_2(PPh_3)_3$][6] has been shown to react with TlC_5H_5 to provide the half-sandwich compound [$RuCl(PPh_3)_2(\eta^5\text{-}C_5H_5)$],[7] the chemistry of which is sufficiently rich to deserve review in its own right.[8] The firmly established analogy between the ligands $\eta\text{-}C_5H_5$ and $\kappa^3\text{-}HB(pz)_3$ suggests that a similarly rich chemistry should be anticipated for the complex [$RuCl(PPh_3)_2\{\kappa^3\text{-}HB(pz)_3\}$]. We have obtained this compound by the simple reaction of Wilkinson's

Scheme I



[†] University of Warwick.

[‡] Imperial College.

(1) Kennedy, J. D. *Prog. Inorg. Chem.* **1984**, *32*, 519.

(2) Klanberg, F.; Muetterties, E. L. *J. Am. Chem. Soc.* **1968**, *90*, 3296.

(3) Gaines, D. F.; Hildebrandt, S. J. *Inorg. Chem.* **1978**, *17*, 794.

(4) Guggenberger, L. J.; Kane, A. R.; Meutterties, E. L. *J. Am. Chem. Soc.* **1972**, *94*, 5665.

(5) Greenwood, N. N.; Kennedy, J. D.; Reed, D. *J. Chem. Soc., Dalton Trans.* **1980**, 196. Bould, J.; Greenwood, N. N.; Kennedy, J. D.; McDonald, W. S. *J. Chem. Soc., Dalton Trans.* **1985**, 1843.

(6) Hallman, P. S.; Stephenson, T. A.; Wilkinson, G. *Inorg. Synth.* **1970**, *12*, 237.

(7) Blackmore, T.; Bruce, M. I.; Stone, F. G. A. *J. Chem. Soc. A* **1971**, 2376.

0020-1669/92/1331-2906$03.00/0

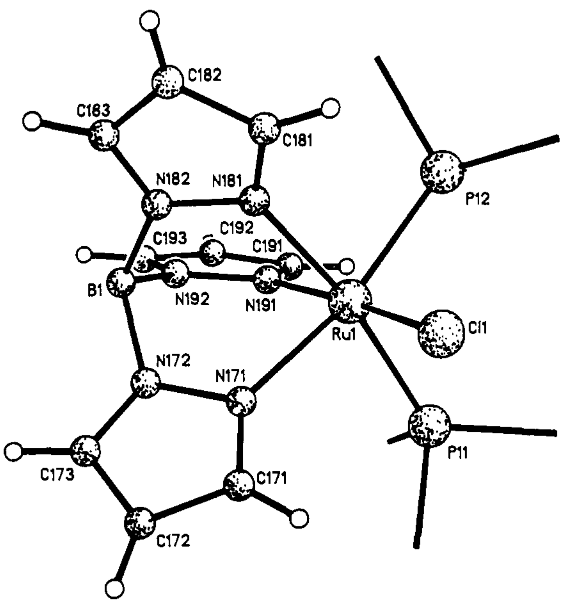
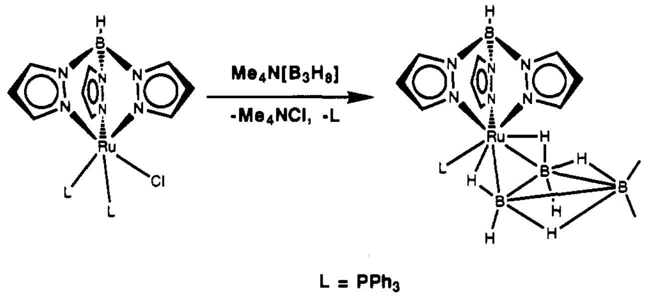


Figure 1. Molecular structure of the $[RuCl(PPh_3)_2\{HB(pz)_3\}]$ molecule based on Ru(I). Phenyl groups were omitted for clarity.

Scheme II



complex $[RuCl_2(PPh_3)_3]$ with potassium hydrotris(pyrazol-1-yl)borate in dichloromethane (Scheme I). The reaction proceeds cleanly, quickly, and in high yield to provide a yellow complex characterized by FAB-MS and ^1H, ^{11}B, and ^{31}P NMR as $[RuCl(PPh_3)_2\{\kappa^3\text{-}HB(pz)_3\}]$: The ^1H NMR spectrum clearly shows that there are two chemical environments adopted by the pyrazolyl rings, while FAB-MS data indicate a molecular ion in addition to peaks due to loss of Cl, pzH, PPh_3, and $HB(pz)_3$ ligands. The molecular geometry was finally confirmed by a single-crystal X-ray diffraction analysis (Figure 1).

Perhaps the most important factor dictating the chemistry of $[RuCl(PPh_3)_2(\eta\text{-}C_5H_5)]$ is the sterically induced lability of one triphenylphosphine.[8] The hydrotris(pyrazol-1-yl)borato ligand has a larger cone angle than that of the cyclopentadienyl group in related complexes. We might therefore expect that the phosphines in $[RuCl(PPh_3)_2\{\kappa^3\text{-}HB(pz)_3\}]$ are at least as labile.

Synthesis of $[Ru(B_3H_8)(PPh_3)\{\kappa^3\text{-}HB(pz)_3\}]$. Treating a solution of $[RuCl(PPh_3)_2\{\kappa^3\text{-}HB(pz)_3\}]$ with $NMe_4[B_3H_8]$ or $^nBu_4N[B_3H_8]$ leads to slow replacement of the chloride and one phosphine ligand with formation of the pale yellow ruthenatetraborane $[Ru(B_3H_8)(PPh_3)\{\kappa^3\text{-}HB(pz)_3\}]$ (Scheme II). This complex is indefinitely air stable as a solid and only slowly decomposed by air in halocarbon solution. The ^1H NMR spectrum is comparatively simple due to the plane of symmetry which the molecule possesses. A signal at −14.34 ppm may be attributed to the protons bridging the Ru–B vectors, while the $B(\mu\text{-}H)B$ resonance appears at −0.52 ppm. The terminal hydrogen resonances occur at 0.9, 1.2, and 2.2 ppm (signals very broad). The ^{11}B NMR spectrum is surprising in that only two peaks are observed with equal integrals. Ruthenatetraboranes typically show resonances in the region −28 to −32 ppm for the boron atoms bound to the metal and a resonance around 0 ppm for the unique "wingtip" boron. It would appear that, in the present case, the ^{11}B resonance for the hydrotris(pyrazolyl)borate ligand is not resolved from that of the wingtip boron. The ^{31}P NMR spectrum shows an unremarkable singlet for the phosphine phosphorus nucleus.

Chart I. Possible Conformations for $[Ru(B_3H_8)(PPh_3)(Tp)]$ [Tp = $HB(pz)_3$]

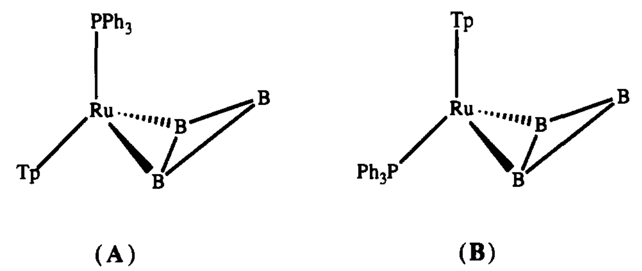


Table I. Comparison of Geometries (Å, deg) of the "$RuCl(PPh_3)_2$" Fragments of the Complexes $[RuCl(PPh_3)_2\{HB(pz)_3\}]$ and $[RuCl(PPh_3)_2(\eta\text{-}C_5H_5)]$

$[RuCl(PPh_3)_2\{HB(pz)_3\}]$		$[RuCl(PPh_3)_2(\eta\text{-}C_5H_5)]$[11]	
Ru(1)–Cl(1)	2.409 (3)	Ru–Cl	2.453 (2)
Ru(1)–P(11)	2.332 (3)	Ru–P(1)	2.337 (1)
Ru(1)–P(12)	2.349 (3)	Ru–P(2)	2.335 (1)
P(11)–Ru(1)–P(12)	101.9 (1)	P(1)–Ru–P(2)	103.99 (4)
P(11)–Ru(1)–Cl(1)	89.0 (1)	P(1)–Ru–Cl	89.05 (3)
P(12)–Ru(1)–Cl(1)	96.0 (1)	P(2)–Ru–Cl	90.41 (4)

Two isomeric arrangements are possible for the MB_3 unit such that the phosphine is endo (A, Chart I) or exo (B, Chart I) with respect to the butterfly metallaborane moiety. We have been unable to obtain single crystals of $[Ru(B_3H_8)(PPh_3)\{\kappa^3\text{-}HB(pz)_3\}]$ suitable for diffractometry; however, it would appear most reasonable that the more bulky $HB(pz)_3$ ligand is bound to the ruthenatetraborane in the exo position of the "butterfly" and that the phosphine ligand coordinates in the endo position. However, it must be borne in mind that barriers to inversion and rotation in metallatetraboranes are often low.[1]

It is worthwhile in passing to note the relationship between $[Ru(B_3H_8)(PPh_3)\{\kappa^3\text{-}HB(pz)_3\}]$ and the known ruthenaboranes $[Ru(B_3H_8)Cl(\eta\text{-}C_6Me_6)]$[9] and $[Ru(B_3H_8)(CO)(\eta\text{-}C_5H_5)]$[10] bearing in mind the isolobal mappings between "$Ru(PPh_3)\{\kappa^3\text{-}HB(pz)_3\}$", "$RuCl(\eta\text{-}C_6Me_6)$", and "$Ru(CO)(\eta\text{-}C_5H_5)$" (isolobal with CH).

Description of the Molecular Structure of $[RuCl(PPh_3)_2\{\kappa^3\text{-}HB(pz)_3\}]$. The compound forms air-stable yellow prisms of the ethanol hemisolvate from dichloromethane and ethanol mixtures. The asymmetric unit contains one molecule of ethanol and two crystallographically independent molecules of the complex. The molecular geometry of one molecule is depicted in Figure 1. Bond lengths and angles for the inner coordination sphere are given in Table I along with relevant data for the related complex $[RuCl(PPh_3)_2(\eta\text{-}C_5H_5)]$.[11]

Despite the well-established increase in steric requirements for the $HB(pz)_3$ chelating ligand compared with the cyclopentadienyl ligand, the geometry of the *fac*-$RuCl(PPh_3)_2$ unit of the $[RuCl(PPh_3)_2\{HB(pz)_3\}]$ molecule is not particularly distorted from octahedral geometry. The recently reported complex $[Ru\{C(C{\equiv}CPh){=}CHPh\}(CO)(PPh_3)\{HB(pz)_3\}]$[12] also shows an essentially octahedral coordination geometry at ruthenium. The

---

(8) Davies, S. G.; McNally, J. P.; Smallridge, A. J. *Adv. Organomet. Chem.* **1990**, *30*, 1.

(9) Bown, M.; Fontaine, X. L. R.; Greenwood, N. N.; MacKinnon, P.; Kennedy, J. D.; Thornton-Pett, M. *J. Chem. Soc., Dalton Trans.* **1987**, 2781.

(10) Gaines, D. F.; Hildebrandt, S. J. Unpublished results referred to in: *Metal Interactions with Boron Clusterss*; Grimes, R. N., Ed.; Plenum: New York, 1982.

(11) Bruce, M. I.; Wong, F. S.; Skelton, B. W.; White, A. H. *J. Chem. Soc., Dalton Trans.* **1981**, 1398.

(12) Hill, A. F. *J. Organomet. Chem.* **1990**, *395*, C35. Alcock, N. W.; Hill, A. F.; Melling, R. P. *Organometallics* **1991**, *10*, 3898.

pseudooctahedral [hydrotris(pyrazolyl)borato]ruthenium complexes $[Ru(OH_2)_3\{HB(pz)_3\}]^+$[13] and $[Ru_2(CO)_4\{HB(pz)_3\}_2]$[14] have also been the subjects of recent crystallographic studies.

Concluding Remarks

The chemistry of the hydrotris(pyrazolyl)borate chelating ligand bound to ruthenium has received surprisingly little attention until the very recent isolation of $[Ru(OH_2)_3\{\kappa^3\text{-}HB(pz)_3\}]^+$,[13] $[Ru_2(CO)_4\{\kappa^3\text{-}HB(pz)_3\}_2]$,[14] and the organometallic derivatives $[RuR(CO)(PPh_3)\{\kappa^3\text{-}HB(pz)_3\}]$.[12] Given the wealth of chemistry originating from the complex $[RuCl(PPh_3)_2(\eta\text{-}C_5H_5)]$, the synthesis of the $HB(pz)_3$ analogue should considerably open up the area to further development. Beyond the simple analogy, however, differences in reactivity may be expected; e.g., in our hands $[RuCl(PPh_3)_2(\eta\text{-}C_5H_5)]$ does not react with sources of $B_3H_8^-$ to provide ruthenatetraboranes.

Experimental Section

General Procedures. All manipulations were carried out under an atmosphere of prepurified dinitrogen using conventional Schlenk-tube techniques. Dichloromethane was distilled from calcium hydride and degassed prior to use.

^1H, ^{11}B, and $^{31}P\{^1H\}$ NMR spectra were recorded on a Bruker WH-400 or Perkin-Elmer R34 NMR spectrometer and calibrated against internal Me_4Si (^1H), external BF_3OEt_2 (^{11}B), or H_3PO_4 (^{31}P). The assistance of O. W. Howarth and the Warwick University NMR service is gratefully acknowledged. Infrared spectra were recorded using a Perkin-Elmer 1720-X FT-IR spectrometer. FAB mass spectrometry was carried out using a Kratos MS80 mass spectrometer using nitrobenzyl alcohol as matrix. Elemental microanalyses were carried out by Medac Ltd., Reading, U.K. The complex $[RuCl_2(PPh_3)_3]$ was prepared according to a published procedure.[6] $NMe_4[B_3H_8]$ was obtained commercially (Strem).

Preparation of $[RuCl(PPh_3)_2\{\kappa^3\text{-}HB(pz)_3\}]$. A suspension of $[RuCl_2(PPh_3)_3]$ (3.00 g, 3.13 mmol) in dichloromethane (150 cm^3) was treated with $K[HB(pz)_3]$ (0.79 g, 3.13 mmol) and the mixture stirred for 30 min. Hexane (100 cm^3) was added and the suspension filtered through Kieselguhr resin to remove KCl. The filtrate was concentrated to ca. 50 cm^3 and the crude complex isolated by filtration and recrystallized from dichloromethane/ethanol. Yield: 2.45 g (90%). IR (CH_2Cl_2; cm^{-1}): 2476, ν(BH). IR (Nujol; cm^{-1}): 2467, ν(BH). ^1H NMR ($CDCl_3$, 25 °C): δ 5.15 [t, 1 H, H^4(pz)], 5.65 [t, 2 H, H^4(pz)], 6.86 [d, 2 H, H^5(pz)], ca. 7.0 [d, 1 H, H^5(pz)], 7.0–7.3 [m, 30 H, PC_6H_5], 7.51 [d, 2 H, H^3(pz)], 7.55 [d, 1 H, H^3(pz)]. ^{31}P NMR ($CDCl_3$, 25 °C): δ 42.9 ppm. ^{11}B NMR ($CDCl_3$, 25 °C): δ −3.67 ppm. FAB-MS (nba matrix) (*m/z*): 874, $[M]^+$; 839, $[M - Cl]^+$; 806, $[M - Hpz]^+$; 612, $[M - PPh_3]^+$; 576, $[M - PPh_3 - HCl]^+$; 363, $[RuPPh_3]^+$.

Preparation of $[Ru(B_3H_8)(PPh_3)\{\kappa^3\text{-}HB(pz)_3\}]$. A solution of $[RuCl_2(PPh_3)_2\{\kappa^3\text{-}HB(pz)_3\}]$ (1.00 g, 1.14 mmol) in dichloromethane (50 cm^3) was treated with $NBu_4[B_3H_8]$ (0.32 g, 0.35 mmol) and stirred for 5 days (monitored by solution IR and TLC) until the reaction was complete. Degassed ethanol (40 cm^3) was added and the suspension filtered through Kieselguhr resin to remove $[NBu_4]Cl$. The filtrate was concentrated to ca. 25 cm^3 under reduced pressure to precipitate the crude product, which was isolated by filtration and recrystallized from a mixture of dichloromethane and ethanol. Yield: 0.56 g (80%). Anal. Found: C, 52.0; H, 5.4; N, 13.0. Calcd for $C_{27}H_{33}B_4N_6PRu$: C, 52.5; H, 5.4; N, 13.6. IR (CH_2Cl_2; cm^{-1}): 2510, 2490, 2115, 1976, ν(BH). IR (Nujol; cm^{-1}): 2507, 2486, 2455, 2115, 1977, ν(BH) [$\{HB(pz)_3\}$ and phosphine bands omitted]. ^1H NMR ($CDCl_3$, 20 °C): δ −14.34 [s (br), 2 H, Ru–*H*–B], −0.52 [s (br), 2 H, B–*H*–B], 0.9, 1.2, 2.2 [s (vbr) × 3, 4 H, terminal BH], 3.7 [s (br), 1 H, $HB(pz)_3$], 5.81, 6.12, 6.67, 7.58, 8.34 [m × 5, 9 H, pz], 7.0–7.5 [m, 30 H, PC_6H_5]. ^{31}P NMR ($CDCl_3$, 21 °C): δ 57.5 ppm. ^{11}B NMR: δ −33.9 [s (br), 2 B, Ru–B], −4.01 [s (br), 2 B, RuB_2B and $HB(pz)_3$]. FAB-MS (nba matrix) (*m/z*): 618, $[M]^+$; 576, $[M - B_3H_8]^+$; 362, $[RuPPh_3]^+$.

Crystal Data, X-ray Data Collection, and Structural Determination. Yellow prisms of the complex $[RuCl(PPh_3)_2\{HB(pz)_3\}]\cdot 0.5EtOH$ were grown from a mixture of dichloromethane and ethanol by solvent evaporation. That chosen for data collection (ca. 0.13 × 0.23 × 0.51 mm) was mounted on a glass fiber. Diffracted intensities were collected (θ–2θ scans) at 298 K in the range $3° < 2\theta < 50°$, on a Nicolet P3m four-circle diffractometer. Of 8395 unique reflections, 6470 had $F \geq 4\sigma(F)$, and only these data were used in the solution and refinement of the structure. Corrections were applied for Lorentz, polarization, and X-ray absorption effects, the latter by an analytical method based on complete definition of crystal morphology.

Crystal data for $[\{RuCl(PPh_3)_2\{HB(pz)_3\}]\cdot 0.5EtOH$: $C_{45}H_{40}BClN_6P_2Ru\cdot 0.5EtOH$, $M = 889.2$, monoclinic, $a = 19.75$ (1) Å, $b = 22.48$ (1) Å, $c = 20.283$ (7) Å, $\beta = 112.94$ (3)°, $U = 8295$ (5) Å^3, $Z = 8$, $D_c = 1.42$ g cm^{-3}, $F(000) = 3688$, space group = $P2_1/a$, Mo Kα X-radiation ($\lambda = 0.710\,73$ Å, graphite monochromator), μ(Mo Kα) = 0.56 mm^{-1}.

The structure was solved, and all non-hydrogen atoms were located, by conventional heavy-atom and difference Fourier methods which indicated that there were two molecules in the asymmetric unit. Phenyl and pyrazolyl hydrogen atoms were included in calculated positions (C–H = 0.96 Å) with fixed isotropic thermal parameters. The difference Fourier map showed the presence of one molecule of ethanol. All non-hydrogen atoms were refined anisotropically (blocked least squares; 1021 parameters), the refinement converging at $R = 0.056$ ($R_w = 0.067$) with a weighting scheme of the form $w^{-1} = [\sigma^2(F) + 0.0206|F|^2]$. Two peaks were apparent in the final different Fourier map at unreasonable distances (ca. 1.6 Å) from B(1) and B(2) for these to be boron hydrides. Apart from these two peaks, electron density residuals were between 1.12 and −0.68 e Å^{-3}. Scattering factors and corrections for anomalous dispersion were taken from ref 15. All calculations were carried out using a DEC micro-VAX II computer with the SHELXTL-PLUS suite of programs.[16] Tables of atomic positional and thermal parameters, calculated hydrogen positions, bond lengths and angles, and procedural details have been deposited as supplementary material.

Acknowledgment. We are grateful to Johnson Matthey Ltd. for a generous loan of ruthenium salts.

Supplementary Material Available: Tables of crystallographic details, atomic and thermal parameters, hydrogen positions, and bond lengths and angles (13 pages); a table of structure factors (32 pages). Ordering information is given on any current masthead page.

---

(13) Llobet, A.; Hodgson, D. J.; Meyer, T. J. *Inorg. Chem.* **1990**, *29*, 3760.

(14) Steyn, M. M. deV.; Singleton, E.; Hietkamp, S.; Liles, D. C. *J. Chem. Soc., Dalton Trans.* **1990**, 2991.

(15) *International Tables for X-ray Crystallography*; Kynoch Press: Birmingham, U.K., 1974; Vol. 4.

(16) Sheldrick, G. M. SHELXTL-PLUS programs for use with the Nicolet X-ray system. Revision 4.0, 1990.