Crystal Structure of *trans*-Bis(acetone hydrazone)tetrakis(trimethyl phosphite)ruthenium(II) Bis(tetraphenylborate): Stabilisation of Metal–Hydrazone Complexes through Hydrogen Bonding

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The crystal structure of the title compound has been determined by *X*-ray diffraction methods from counter data. Crystals are triclinic, space group $P\bar{1}$, with $Z = 1$, $a = 15{\cdot}30$, $b = 11{\cdot}54$, $c = 13{\cdot}74$ ($\pm 0{\cdot}01$) Å, $\alpha = 92{\cdot}55°$, $\beta = 59{\cdot}02°$, $\gamma = 64{\cdot}49°$ ($\pm 0{\cdot}01$). The structure was solved by heavy-atom methods and refined by least-squares to *R* 0·069 for 5696 observed reflections. The ruthenium atom has almost ideal octahedral symmetry and the two hydrazone ligands are *trans*-oriented. Hydrogen bonding between the *N*-hydrogen of the hydrazone and the oxygen of the phosphite was inferred from observed N–O distances of 2·82 (1) and 2·85 (1) Å. Mean Ru–P, Ru–N, and N–C distances are 2·35, 2·17, and 1·28 Å.

RECENT studies[1] on $[(C_8H_{12})Ru(N_2H_4)_4][BPh_4]_2$ and $[(C_8H_{12})RuH(NH_2NMe_2)_3]PF_6$ have shown that these salts react readily with a variety of substrates of varying electronic and steric properties to give a range of ruthenium(II) salts in which, in general, total replacement of the hydrazine ligands occurs. With small phosphites and phosphonites, however, the reaction with $[(C_8H_{12})Ru(N_2H_4)_4][BPh_4]_2$ when heated under reflux in acetone solution produced compounds of formulae $[Ru(NH_2{\cdot}N{:}CMe_2)_2L_4][BPh_4]_2$ [$L = P(OMe)_3$, $P(OEt)_3$, $P(OMe)_2Ph$, and $P(OEt)_2Ph$] which, from i.r. spectroscopic evidence, appeared to contain bonded hydrazone

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[1] J. J. Hough and E. Singleton, *J.C.S. Chem. Comm.*, 1972, 371.

ligands.[2] These salts were exceedingly stable in solution and practically inert to further reactions under reasonable conditions, which is unusual considering the instability of the few known transition-metal–hydrazone complexes.[3,4] We therefore decided to determine the structure of one of these complexes, $[Ru(NH_2{\cdot}N{:}CMe_2)\{P(OMe_3)\}_4][BPh_4]_2$, in order to confirm the presence of bonded hydrazone ligands, thus gaining some insight into the factors controlling the unusual stability of the cations in solution, and also finding the nitrogen to which bonding to the metal atom occurs.

EXPERIMENTAL

The crystals were white prisms, and one with dimensions 0·24 × 0·30 × 0·50 mm was chosen for data collection. Intensity data were collected with a Hilger and Watts four-circle single-crystal diffractometer for the diffraction range θ 1—25°, by use of graphite-monochromated Mo-K_α radiation. By scanning in the ω—2θ mode, the intensities of 6250 reflections were measured of which 554, having $I < 3\sigma(I)$, were rejected. The background was measured between two lattice rows as a function of θ and subtracted from the intensities. Intensities were measured in 30 steps of 0·02, counting for 1·5 s at each step. Reflections 011 and 331 were monitored every 100 reflections. No corrections were made for extinction or absorption.

Crystal Data.—$C_{66}H_{92}B_2N_4O_{12}P_4Ru$, $M = 1380{\cdot}13$, Triclinic, $a = 15{\cdot}30(1)$, $b = 11{\cdot}54(1)$, $c = 13{\cdot}74(1)$ Å, $\alpha = 92{\cdot}55(1)$, $\beta = 59{\cdot}02(1)$, $\gamma = 64{\cdot}49(1)°$, $U = 1762{\cdot}4$ Å^3, D_m (by flotation) = 1·30, $Z = 1$, $D_c = 1{\cdot}30$, $F(000) = 726$. Mo-$K_{\alpha 1}$ radiation, $\lambda = 0{\cdot}7093$ Å; μ(Mo-K_α) = 3·667 cm^{-1}. Space group $P\bar{1}$.

Structure Solution and Refinement.—Because the exact composition of the compound was uncertain, initial calculations (on an IBM 360/65 computer) were carried out in space group $P1$. The positions of the ruthenium and

TABLE 1

Final atomic co-ordinates (× 10^4) and anisotropic temperature factors * (10^4 × Å^2), with their standard deviations in parentheses

	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Ru	0	0	0	46(1)	82(1)	74(1)	−28(1)	−35(1)	24(1)
P(1)	−2023(1)	795(2)	867(2)	46(1)	95(2)	98(2)	−29(1)	−40(1)	32(2)
P(2)	−63(1)	−855(2)	1538(2)	59(1)	83(2)	80(2)	−33(1)	−42(1)	31(1)
N(1)	−611(4)	1936(5)	1136(5)	65(4)	76(6)	99(6)	−34(4)	−40(4)	19(5)
N(2)	−18(5)	2676(5)	591(5)	91(5)	91(6)	115(6)	−49(5)	−64(5)	39(5)
C(1)	378(6)	3019(7)	1102(7)	83(6)	102(8)	125(9)	−47(6)	−54(6)	23(7)
C(2)	948(8)	3833(9)	546(9)	150(10)	156(12)	189(13)	−115(10)	−93(10)	69(10)
C(3)	318(10)	2748(11)	2195(9)	203(13)	247(16)	150(11)	−158(13)	−133(11)	97(11)
O(11)	−2139(4)	408(5)	−162(5)	80(4)	184(8)	147(6)	−66(5)	−86(5)	67(6)
O(12)	−2884(4)	510(5)	1976(5)	67(4)	133(6)	125(6)	−47(4)	−45(4)	50(5)
O(13)	−2746(4)	2409(5)	1461(5)	54(4)	93(5)	171(7)	−23(4)	−39(4)	22(5)
O(21)	1111(4)	−2091(5)	1323(5)	83(4)	117(6)	132(6)	−35(4)	−75(4)	50(5)
O(22)	−1012(4)	1285(5)	2166(5)	101(5)	163(7)	126(6)	−88(5)	−78(5)	85(5)
O(23)	−394(4)	301(5)	2560(4)	109(5)	111(6)	93(5)	−53(4)	−63(4)	33(4)
C(11)	−3253(9)	985(12)	−69(11)	135(11)	330(21)	279(18)	−122(13)	−176(13)	148(16)
C(12)	−2928(7)	−726(8)	1889(9)	103(8)	157(11)	178(11)	−92(8)	−84(8)	83(9)
C(13)	−4039(7)	3335(9)	2430(11)	49(6)	116(11)	254(17)	1(7)	−1(8)	2(11)
C(21)	1607(8)	−3500(7)	674(9)	132(9)	77(8)	179(12)	−25(7)	−93(9)	34(8)
C(22)	−1285(9)	−1775(11)	3179(9)	174(12)	282(18)	143(11)	−166(13)	−107(10)	140(12)
C(23)	−264(9)	141(9)	3526(8)	189(12)	183(13)	124(10)	−103(11)	−121(10)	69(9)
B	4178(6)	−1831(7)	2838(7)	58(6)	87(8)	80(7)	−35(6)	−40(5)	20(6)
C(A1)	5165(5)	−2267(6)	3164(6)	63(5)	85(7)	85(6)	−39(5)	−43(5)	31(5)
C(A2)	6341(6)	−3403(7)	2339(7)	70(6)	102(8)	108(7)	−38(6)	−53(6)	38(6)
C(A3)	7139(6)	−3870(8)	2672(8)	81(7)	127(9)	136(9)	−43(7)	−69(7)	53(8)
C(A4)	6802(7)	−3240(8)	3771(8)	105(8)	136(10)	139(10)	−68(7)	−86(7)	67(8)
C(A5)	5653(7)	−2096(8)	4599(8)	128(9)	146(10)	132(9)	−73(8)	−97(8)	57(8)
C(A6)	4854(6)	−1639(7)	4270(7)	94(7)	122(9)	99(7)	−43(6)	−64(6)	28(7)
C(B1)	4922(5)	−2334(6)	1360(6)	50(5)	94(7)	84(6)	−28(5)	−42(5)	23(5)
C(B2)	5252(6)	−3595(6)	694(6)	69(5)	94(7)	90(7)	−28(5)	−49(5)	14(6)
C(B3)	5957(6)	−4074(7)	−576(7)	72(6)	117(9)	94(7)	−29(6)	−48(6)	15(6)
C(B4)	6368(6)	−3316(8)	−1222(7)	78(6)	129(9)	92(7)	−31(6)	−52(6)	27(7)
C(B5)	6051(6)	−2060(7)	−584(7)	81(6)	131(9)	96(7)	−47(6)	−55(6)	41(7)
C(B6)	5337(5)	−1586(7)	638(6)	64(5)	115(8)	93(7)	−42(6)	−47(5)	37(6)
C(C1)	3263(5)	−153(6)	3390(5)	62(5)	103(7)	65(5)	−37(5)	−36(5)	22(5)
C(C2)	2063(6)	476(7)	3758(6)	64(5)	117(8)	86(7)	−36(6)	−44(5)	32(6)
C(C3)	1314(6)	1899(7)	4160(7)	80(6)	114(8)	105(8)	−30(6)	−56(6)	32(7)
C(C4)	1718(7)	2746(7)	4192(7)	101(7)	112(9)	101(8)	−38(7)	−62(6)	32(7)
C(C5)	2900(7)	2166(7)	3833(7)	103(7)	108(8)	103(8)	−50(6)	−64(6)	33(6)
C(C6)	3634(6)	746(7)	3440(6)	81(6)	98(8)	93(7)	−46(6)	−51(5)	25(6)
C(D1)	3435(5)	−2596(6)	3434(6)	57(5)	80(7)	86(6)	−28(5)	−28(5)	6(5)
C(D2)	3472(6)	−3204(7)	4292(7)	83(7)	113(9)	120(8)	−46(6)	−44(6)	50(7)
C(D3)	2845(7)	−3886(8)	4772(9)	97(8)	131(10)	161(11)	−56(8)	−51(8)	63(9)
C(D4)	2161(7)	−3940(8)	4395(9)	87(7)	108(9)	148(11)	−52(7)	−27(7)	10(8)
C(D5)	2100(6)	−3345(8)	3567(7)	77(7)	119(9)	112(9)	−49(7)	−22(6)	−8(7)
C(D6)	2729(6)	−2673(7)	3090(7)	66(6)	109(8)	101(7)	−43(6)	−31(5)	−3(6)

* In the form $T = \exp[-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + 2\beta_{12}hk + 2\beta_{13}hl + 2\beta_{23}kl)]$.

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[2] T. Ashworth and E. Singleton, to be published.

[3] C. H. Stapfer, R. W. D'Andrea, and R. H. Herber, *Inorg. Chem.*, 1972, **11**, 204.

[4] C. H. Stapfer and R. W. D'Andrea, *Inorg. Chem.*, 1971, **10**, 1224.

phosphorus atoms were deduced from a three-dimensional Patterson map aided by knowledge of the approximate interatomic distances. Consecutive cycles of difference Fourier syntheses and full-matrix least-squares refinements with the ORFLS program [5] led to the identification of the remaining non-hydrogen atoms in the structure. It was evident that the molecule was centrosymmetric about the ruthenium atom and that the space group was actually $P\bar{1}$. Refinement of positional parameters and individual isotropic temperature factors reduced R to 0·114. By introducing anisotropic thermal parameters and continuing the refinement, R was reduced to 0·069 and the weighted R' $\{= [\Sigma w(\Delta F)^2/\Sigma wF_o^2]^{\frac{1}{2}}\}$ to 0·083 using unit weights. Although the number of parameters was approximately halved by introducing the inversion centre, the 402 parameters were still in excess of the maximum that could be handled by the full-matrix refinement program. Refinement was therefore carried out in two stages during which the cation and anion were refined separately. Observed and calculated structure factors are listed in Supplementary Publication No. SUP 21081 (26 pp., 1 microfiche).* Table 1 gives the final positional and thermal parameters with their standard deviations. Bond distances and angles calculated with the ORFFE program [6] are summarised in Tables 2 and 3. Interatomic distances and angles, some of which are involved in hydrogen bonding, are listed in Table 4.

TABLE 2

Bond lengths (Å), with standard deviations

Ru–P(1)	2·35(1)	C(A2)–C(A3)	1·42(1)
Ru–P(2)	2·35(1)	C(A3)–C(A4)	1·36(1)
Ru–N(1)	2·17(1)	C(A4)–C(A5)	1·39(1)
N(1)–N(2)	1·46(1)	C(A5)–C(A6)	1·42(1)
N(2)–C(1)	1·28(1)	C(A6)–C(A1)	1·39(1)
C(1)–C(2)	1·52(1)	C(B1)–C(B2)	1·41(1)
C(1)–C(3)	1·51(1)	C(B2)–C(B3)	1·41(1)
P(1)–O(11)	1·58(1)	C(B3)–C(B4)	1·38(1)
P(1)–O(12)	1·59(1)	C(B4)–C(B5)	1·40(1)
P(1)–O(13)	1·59(1)	C(B5)–C(B6)	1·40(1)
P(2)–O(21)	1·60(1)	C(B6)–C(B1)	1·40(1)
P(2)–O(22)	1·57(1)	C(C1)–C(C2)	1·42(1)
P(2)–O(23)	1·59(1)	C(C2)–C(C3)	1·40(1)
O(11)–C(11)	1·47(1)	C(C3)–C(C4)	1·37(1)
O(12)–C(12)	1·46(1)	C(C4)–C(C5)	1·40(1)
O(13)–C(13)	1·47(1)	C(C5)–C(C6)	1·40(1)
O(21)–C(21)	1·46(1)	C(C6)–C(C1)	1·39(1)
O(22)–C(22)	1·45(1)	C(D1)–C(D2)	1·41(1)
O(23)–C(23)	1·45(1)	C(D2)–C(D3)	1·42(1)
B–C(A1)	1·67(1)	C(D3)–C(D4)	1·41(1)
B–C(B1)	1·65(1)	C(D4)–C(D5)	1·38(1)
B–C(C1)	1·65(1)	C(D5)–C(D6)	1·41(1)
B–C(D1)	1·65(1)	C(D6)–C(D1)	1·42(1)
C(A1)–C(A2)	1·41(1)		

TABLE 3

Bond angles (°), with standard deviations

P(1)–Ru–P(2)	92·8(1)	B–C(A1)–C(A2)	119·6(6)
P(1)–Ru–N(1)	87·6(1)	B–C(A1)–C(A6)	123·0(5)
P(2)–Ru–N(2)	87·1(2)	C(A6)–C(A1)–C(A2)	117·2(6)
Ru–N(1)–N(2)	118·8(4)	C(A1)–C(A2)–C(A3)	119·6(7)
N(1)–N(2)–C(1)	116·1(6)	C(A2)–C(A3)–C(A4)	121·8(7)
N(2)–C(1)–C(2)	114·9(8)	C(A3)–C(A4)–C(A5)	120·0(7)
N(2)–C(1)–C(3)	128·2(7)	C(A4)–C(A5)–C(A6)	118·1(7)
C(2)–C(1)–C(3)	116·9(7)	C(A5)–C(A6)–C(A1)	123·3(7)
Ru–P(1)–O(11)	108·9(2)	B–C(B1)–C(B2)	121·8(6)
Ru–P(1)–O(12)	126·8(2)	B–C(B1)–C(B6)	122·1(6)
Ru–P(1)–O(13)	106·8(2)	C(B6)–C(B1)–C(B2)	116·0(6)
Ru–P(2)–O(21)	122·1(2)	C(B1)–C(B2)–C(B3)	122·1(7)
Ru–P(2)–O(22)	114·9(2)	C(B2)–C(B3)–C(B4)	120·5(7)
Ru–P(2)–O(23)	108·4(2)	C(B3)–C(B4)–C(B5)	118·5(7)
P(1)–O(11)–C(11)	126·3(6)	C(B4)–C(B5)–C(B6)	120·4(7)
P(1)–O(12)–C(12)	124·7(5)	C(B5)–C(B6)–C(B1)	122·4(6)
P(1)–O(13)–C(13)	127·7(5)	B–C(C1)–C(C2)	121·9(6)
P(2)–O(21)–C(21)	121·0(5)	B–C(C1)–C(C6)	122·8(5)
P(2)–O(22)–C(22)	126·5(5)	C(C6)–C(C1)–C(C2)	115·1(6)
P(2)–O(23)–C(23)	127·6(5)	C(C1)–C(C2)–C(C3)	121·6(6)
O(11)–P(1)–O(12)	105·4(3)	C(C2)–C(C3)–C(C4)	121·3(6)
O(11)–P(1)–O(13)	110·0(3)	C(C3)–C(C4)–C(C5)	118·9(7)
O(12)–P(1)–O(13)	97·9(3)	C(C4)–C(C5)–C(C6)	119·1(7)
O(21)–P(2)–O(22)	103·4(3)	C(C5)–C(C6)–C(C1)	124·0(6)
O(21)–P(2)–O(23)	99·1(3)	B–C(D1)–C(D2)	122·4(6)
O(22)–P(2)–O(23)	107·1(3)	B–C(D1)–C(D6)	120·4(6)
C(A1)–B–C(B1)	108·5(5)	C(D6)–C(D1)–C(D2)	117·2(6)
C(A1)–B–C(C1)	109·9(5)	C(D1)–C(D2)–C(D3)	121·4(8)
C(A1)–B–C(D1)	107·4(5)	C(D2)–C(D3)–C(D4)	119·2(9)
C(B1)–B–C(C1)	108·4(5)	C(D3)–C(D4)–C(D5)	120·7(7)
C(B1)–B–C(D1)	111·3(5)	C(D4)–C(D5)–C(D6)	119·9(8)
C(C1)–B–C(D1)	111·3(5)	C(D5)–C(D6)–C(D1)	121·7(8)

TABLE 4

Some interatomic distances (Å) and angles (°)

N(1) ··· O(11)	4·40(1)	Ru–N(1) ··· O(11)	44·4(1)
N(1) ··· O(12)	4·14(1)	Ru–N(1) ··· O(12)	58·7(1)
N(1) ··· O(13)	2·85(1)	Ru–N(1) ··· O(13)	77·7(2)
N(1) ··· O(21)	4·34(1)	Ru–N(1) ··· O(21)	52·3(1)
N(1) ··· O(22)	4·21(1)	Ru–N(1) ··· O(22)	51·6(1)
N(1) ··· O(23)	2·82(1)	Ru–N(1) ··· O(23)	79·4(2)
N(1) ··· O(31)	3·30(1)	Ru–N(1) ··· O(31)	68·8(2)
N(1) ··· O(32)	4·15(1)	Ru–N(1) ··· O(32)	58·3(1)
N(1) ··· O(33)	4·65(1)	Ru–N(1) ··· O(33)	36·7(1)
N(1) ··· O(41)	3·83(1)	Ru–N(1) ··· O(41)	64·0(2)
N(1) ··· O(42)	3·72(1)	Ru–N(1) ··· O(42)	62·5(2)
N(1) ··· O(43)	4·71(1)	Ru–N(1) ··· O(43)	36·0(1)

DISCUSSION

The asymmetric unit comprises half the molecule with the ruthenium atom situated on a centre of symmetry in almost ideal octahedral co-ordination (Figure 1). The two hydrazone ligands are mutually *trans* and bonding to the metal atom is by the protonated nitrogen and not, as would be expected from electronegativity arguments,[3] by the –N=C nitrogen. However, the situation observed could arise because this configuration gives minimal steric interaction between

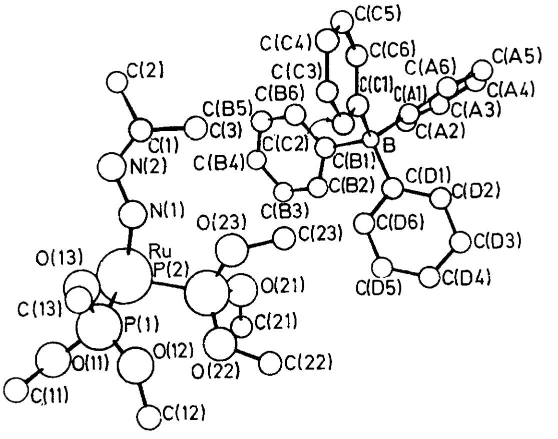

FIGURE 1 Atomic numbering of the asymmetric unit of a molecule

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* Notice to Authors No. 7, *J.C.S. Dalton*, 1973, Index issue.

[5] W. R. Busing, K. O. Martin, and H. A. Levy, Report ORNL TM 305, Oak Ridge National Laboratories, Oak Ridge, Tennessee, 1962.

[6] W. R. Busing, K. O. Martin, and H. A. Levy, Report ORNL TM 306, Oak Ridge National Laboratories, Oak Ridge, Tennessee, 1964.

the hydrazones and the phosphite ligands. Figure 2 is a stereogram of the molecule obtained from an ORTEP program.[7] Bond distances found in this structure correspond reasonably closely with related values from other structure analyses. The Ru–N bond length [2·17(1) Å] is longer than expected from covalent radii summations (2·03 Å) and suggests a mutual *trans*-influence. The values of the N–N, N–C, and C–C parameters [1·46(1), 1·28(1), and 1·52(1) Å], are close to those found in metal–hydrazine complexes,[8] bisacetyldicarbonylbis(acetone hydrazone)nickel(0),[9] and *trans*-dimethylglyoxime.[10] The mean value [2·35(1) Å] for the Ru–P bonds is somewhat larger than those [2·21(1) and 2·23(4) Å] found for $[(C_4H_7)_2Ru\{P(OMe)_3\}_2]$ [11] containing *cis*-phosphite ligands, and identical with that for $[Ru(H)(OAc)(PPh_3)_3]$ [12] with *trans*-triphenylphosphine groups. Mean P–O and O–C distances are 1·59(1) and 1·46(1) Å. There are, however, large deviations from ideal tetrahedral symmetry around the phosphorus atoms, and this indicates the following as a possible stabilising factor for the cation in solution. The Ru–P–O and P–O–C angles are summarised in Table 3. The inner co-ordination of N, P, and O atoms around the ruthenium is compared in Figure 3 for two different pairs of phosphite ligands (each pair being mutually *trans*) and a hydrazone group. As can be seen in Figure 3(a), two oxygen atoms O(13) and O(23) from two symmetrically unrelated phosphite ligands are 2·85(1) and 2·82(1) Å from nitrogen N(1) of the $-NH_2$ moiety. This close approach of N(1) to O(13) and O(23) is well within the value of 2·91 Å suggested by Tenzer *et al.*[13] as indicative of hydrogen bonding. Further evidence for N–H · · · O interactions comes from the bending of O(13) and O(23) towards N(1) with corresponding contractions of the N–Ru–P (87·1 and 87·6°) and Ru–P–O (106·8 and 108·4°) angles (the mean Ru–P–O angle in $[(C_4H_7)_2Ru\{P(OMe)_3\}_2]$ is 118·6° [11]). The Ru–N–O angles (77·7 and 79·4°) imply, however, a marked deviation from linearity of the N–H · · · O bond, but nevertheless represent the best possible choice from among the available angles as determined by electrostatic forces and steric influences. For the ligands depicted in Figure 3(b), the N–Ru–P (92·4 and 92·9°) and Ru–P–O (114·9 and 108·9°) angles are slightly increased and the shortest N · · · O distances are 3·30(1) and 3·72(1) Å. Ru–N–O angles of 62·5 and 68·8° would lead to larger deviations from linearity of the N–H · · · O bond and from these parameters it is unlikely that even weak hydrogen–oxygen interaction occurs with this set of ligands. Thus it seems that the unreactivity of the cations $[Ru(NH_2{\cdot}N{:}CMe_2)_2L_4]^{2+}$ in solution is due entirely to strong hydrogen bonding between the hydrazone hydrogen atoms and the phosphite or phosphonite oxygens. No stable hydrazone products were obtained with phosphinites, and it is possible that steric interactions of these larger ligands with the hydrazone groups may become a dominant factor which destabilises the molecule in solution.

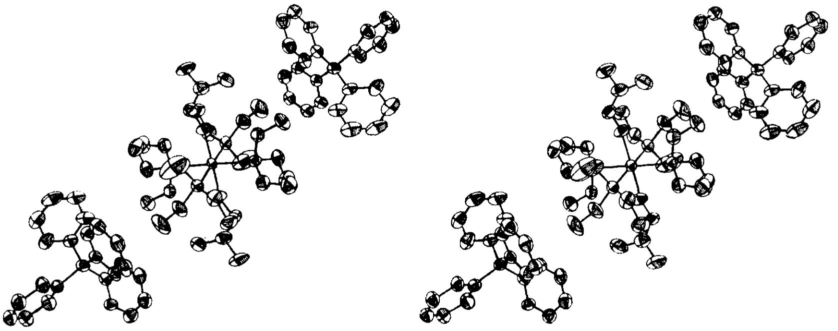

FIGURE 2 A stereoscopic drawing of a molecule

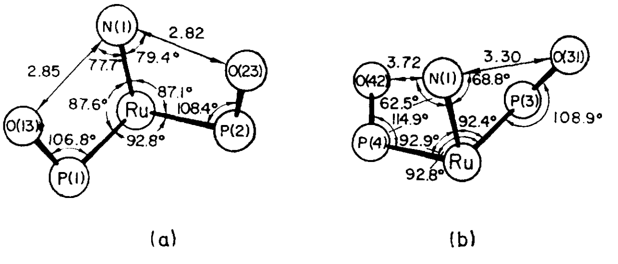

FIGURE 3 The inner co-ordination around the Ru atom contrasted for two different pairs of phosphite ligands and a hydrazone group

Tetraphenylborate Ions.—The geometry of these ions is in agreement with that found in other structures.[14,15] Crowding around the boron atom is manifested by some close non-bonded approaches [2·95(1)—3·17(1) Å] be-

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[7] C. K. Johnson, Report ORNL TM 3794, Oak Ridge National Laboratories, Oak Ridge, Tennessee, 1965.

[8] F. Bottomley, *Quart. Rev.*, 1970, **24**, 617, and refs. therein.

[9] H. D. Hausen and K. Krogmann, *Z. anorg. Chem.*, 1972, **389**, 247.

[10] L. L. Merritt, jun., and E. Lanterman, *Acta Cryst.*, 1952, **5**, 811.

[11] R. A. Marsh, J. Howard, and P. Woodward, *J.C.S. Dalton*, 1972, 778.

[12] A. C. Skapski and F. A. Stephens, *Chem. Comm.*, 1969, 1008.

[13] L. Tenzer, B. C. Frazer, and R. Pepinsky, *Acta Cryst.*, 1958, **11**, 505.

[14] T. L. Blundell and H. M. Powell, *Acta Cryst.*, 1972, **B27**, 2304.

[15] M. Di Vaira and A. B. Orlandini, *J.C.S. Dalton*, 1972, 1704.

tween α and β carbon atoms of different phenyl rings. Each anion can be considered to be a discrete entity, no interionic distance involving non-hydrogen atoms being $<3{\cdot}47(1)$ Å.

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