Notes

Synthesis and *X*-Ray Crystal Structure of $[Rh(NO)(PPh_3)_2(p\text{-}MeC_6H_4NNNC_6H_4Me\text{-}p)][BF_4]\cdot CH_2Cl_2$ *

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The reaction of $[Rh(NO)(NCMe)_2(PPh_3)_2][BF_4]_2$ with Na[RNNNR] (R = *p*-tolyl) in tetrahydrofuran yields $[Rh(NO)(PPh_3)_2(RNNNR)][BF_4]$, *X*-ray diffraction studies on which have revealed a trigonal-bipyramidal structure with a linear nitrosyl [Rh–N–O 177.5(6)°] and chelating triazenide ligand in the equatorial positions.

We have recently described[1] the synthesis, electrochemistry, and structural and spectroscopic characterisation of a range of triazenido-bridged binuclear complexes, derived from $[\{Rh(CO)_2(\mu\text{-}RNNNR)\}_2]$ (**1**; R = *p*-tolyl) and containing $[Rh_2]^{Z+}$ (*Z* = 2, 3, or 4) cores. In order to extend and diversify this range, we have attempted to substitute CO in (**1**) and its derivatives by the isoelectronic $[NO]^+$ group, thereby altering the overall charge of the complex without changing the oxidation state of the central Rh_2 core. The direct reaction of the binuclear carbonyl complexes, for example, $[Rh_2(CO)_2(PPh_3)_2(\mu\text{-}RNNNR)_2]$, with nitrosonium salts invariably results in one-electron oxidation. We have therefore sought alternative synthetic routes, and now report on the reaction of $[Rh(NO)(NCMe)_2(PPh_3)_2][BF_4]_2$ (**2**) with the triazenide anion $[RNNNR]^-$ (R = *p*-tolyl), the product of which has been characterised by *X*-ray crystallography as the mononuclear complex $[Rh(NO)(PPh_3)_2(RNNNR)][BF_4]$ (**3**). Structural comparisons are made between (**3**) and related nitrosyl and triazenide complexes, and the redox properties of the cation are briefly described.

Results and Discussion

The reaction of $[Rh(NO)(NCMe)_2(PPh_3)_2][BF_4]_2$ (**2**) with $[RNNNR]^-$ (R = *p*-tolyl) in tetrahydrofuran (thf) rapidly gave a good yield of a brown crystalline complex (**3**) whose elemental analysis (C, H, and N) was consistent with either a mononuclear monocationic complex, *i.e.* $[Rh(NO)(PPh_3)_2(RNNNR)][BF_4]$, or a dimeric triazenido-bridged, diaxial adduct derived from $[\{Rh(CO)_2(\mu\text{-}RNNNR)\}_2]$. The ^1H [$(CD_3)_2CO$; δ 7.59 (30 H, m, PPh_3), 6.88 (4 H, d, *J* 8.4, C_6H_4), 6.60 (4 H, d, *J* 8.4 Hz, C_6H_4), and 2.20 (6 H, s, Me)] and ^{31}P {CD_2Cl_2; δ 35.51 p.p.m. [d, $J(^{103}Rh^{31}P)$ 95.2 Hz]} n.m.r. spectra of (**3**) showed that both the *p*-tolyl groups and the phosphine ligands were equivalent, but did not distinguish between mono- and bi-nuclear structures. However, a substantial change from the square-pyramidal geometry of complex (**2**) (with a bent nitrosyl ligand)[2] was apparent from the i.r. nitrosyl absorption at 1 813 cm^{-1} (corrected to 1 703 cm^{-1} according to Ibers' rules for the prediction of NO bonding modes[3]) which suggested a linear Rh–N–O linkage.

The cyclic voltammogram (c.v.) of complex (**3**), in CH_2Cl_2 at a platinum bead electrode, showed a diffusion-controlled $[(i_p)_{ox}/v^{\frac{1}{2}}$ = constant for scan rates $50 \leqslant v \leqslant 200$ mV s^{-1}] oxidation wave, reversible at scan rates faster than 200 mV s^{-1} (with E° = 1.10 V *vs.* the saturated calomel electrode †), and a similarly diffusion-controlled but only partially chemically reversible [*e.g.* v = 50 mV s^{-1}, $(i_p)_{ox}/(i_p)_{red}$ = 0.55, v = 200 mV s^{-1}, $(i_p)_{ox}/(i_p)_{red}$ = 0.7] reduction wave with a peak potential, $(E_p)_{red}$, of −0.46 V at 200 mV s^{-1}. Although the redox properties of (**3**) are not as extensive as those of the derivatives of (**1**), they are more so than those of the mononuclear carbonyl $[Rh(CO)(PPh_3)_2(RNNNR)]$ (R = phenyl) the c.v. of which showed only ill defined oxidation waves in the potential range 0.0 to 1.7 V.

As neither the spectroscopic nor electrochemical data distinguished between the mono- and bi-nuclear structures possible for (**3**), a single-crystal *X*-ray diffraction study was carried out. The structure of (**3**) is illustrated in the Figure; Table 1 lists the derived bond lengths and inter-bond angles, and Table 2 the atomic co-ordinates.

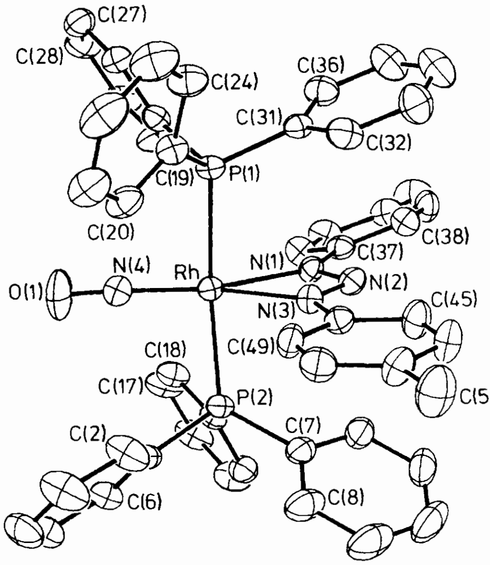

Figure. Molecular structure of the cation of (**3**) showing the atom labelling scheme. All hydrogen atoms have been omitted for clarity

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* (Di-*p*-tolyltriazenido)nitrosylbis(triphenylphosphine)rhodium tetrafluoroborate dichloromethane (1/1).

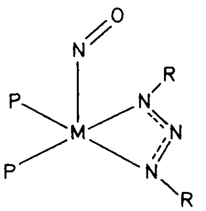
Supplementary data available: see Instructions for Authors, *J. Chem. Soc., Dalton Trans.*, 1989, Issue 1, pp. xvii—xx.

† Under the conditions of the experiment, E° for the one-electron oxidation of $[Fe(\eta^5\text{-}C_5H_5)_2]$ is + 0.47 V.

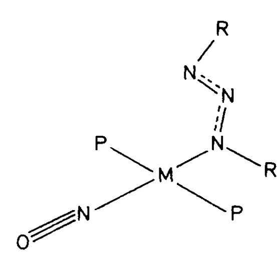
Table 1. Bond lengths (Å) and angles (°) for complex (**3**)$\cdot CH_2Cl_2$

Rh–N(1)	2.171(5)	Rh–N(3)	2.149(4)
Rh–N(4)	1.769(6)	Rh–P(1)	2.377(1)
Rh–P(2)	2.378(2)	C(37)–C(38)	1.395(9)
C(37)–C(42)	1.416(10)	C(37)–N(1)	1.393(8)
C(38)–C(39)	1.377(9)	C(39)–C(40)	1.398(10)
C(40)–C(41)	1.393(11)	C(40)–C(43)	1.508(10)
C(41)–C(42)	1.378(10)	C(44)–C(45)	1.394(9)
C(44)–C(49)	1.374(9)	C(44)–N(3)	1.406(7)
C(45)–C(46)	1.369(10)	C(46)–C(47)	1.421(11)
C(47)–C(48)	1.371(10)	C(47)–C(50)	1.513(11)
C(48)–C(49)	1.365(10)	C(51)–Cl(1)	1.673(14)
C(51)–Cl(2)	1.427(11)	N(1)–N(2)	1.302(7)
N(2)–N(3)	1.293(7)	N(4)–O(1)	1.117(8)
P(1)–C(19)	1.814(5)	P(1)–C(25)	1.812(4)
P(1)–C(31)	1.809(5)	P(2)–C(1)	1.817(5)
P(2)–C(7)	1.808(6)	P(2)–C(13)	1.805(5)
B(1)–F(1)	1.351(12)	B(1)–F(2)	1.358(11)
B(1)–F(3)	1.367(10)	B(1)–F(4)	1.385(13)
N(1)–Rh–N(3)	57.0(2)	N(1)–Rh–N(4)	150.2(2)
N(3)–Rh–N(4)	152.8(2)	N(1)–Rh–P(1)	87.5(1)
N(3)–Rh–P(1)	85.9(1)	N(4)–Rh–P(1)	92.9(2)
N(1)–Rh–P(2)	89.3(1)	N(3)–Rh–P(2)	89.2(1)
N(4)–Rh–P(2)	91.8(2)	P(1)–Rh–P(2)	175.0(1)
C(2)–C(1)–P(2)	119.2(1)	C(6)–C(1)–P(2)	120.7(2)
C(8)–C(7)–P(2)	121.4(2)	C(12)–C(7)–P(2)	118.6(2)
C(14)–C(13)–P(2)	121.5(1)	C(18)–C(13)–P(2)	118.4(1)
C(20)–C(19)–P(1)	120.8(1)	C(24)–C(19)–P(1)	119.2(1)
C(26)–C(25)–P(1)	120.7(1)	C(30)–C(25)–P(1)	119.3(1)
C(32)–C(31)–P(1)	118.3(2)	C(36)–C(31)–P(1)	121.7(2)
C(38)–C(37)–C(42)	118.3(6)	C(38)–C(37)–N(1)	124.8(6)
C(42)–C(37)–N(1)	116.9(5)	C(37)–C(38)–C(39)	119.6(6)
C(38)–C(39)–C(40)	122.1(6)	C(39)–C(40)–C(41)	118.6(6)
C(39)–C(40)–C(43)	118.8(7)	C(41)–C(40)–C(43)	122.5(7)
C(40)–C(41)–C(42)	119.8(7)	C(37)–C(42)–C(41)	121.5(7)
C(45)–C(44)–C(49)	118.6(6)	C(45)–C(44)–N(3)	122.0(5)
C(49)–C(44)–N(3)	119.4(5)	C(44)–C(45)–C(46)	120.2(6)
C(45)–C(46)–C(47)	121.3(6)	C(46)–C(47)–C(48)	116.3(6)
C(46)–C(47)–C(50)	120.5(6)	C(48)–C(47)–C(50)	123.1(7)
C(47)–C(48)–C(49)	122.7(7)	C(44)–C(49)–C(48)	120.8(6)
Cl(1)–C(51)–Cl(2)	123.4(10)	Rh–N(1)–C(37)	143.4(4)
Rh–N(1)–N(2)	98.2(4)	C(37)–N(1)–N(2)	118.2(5)
N(1)–N(2)–N(3)	105.1(5)	Rh–N(3)–C(44)	140.5(4)
Rh–N(3)–N(2)	99.6(3)	C(44)–N(3)–N(2)	119.9(5)
Rh–N(4)–O(1)	177.5(6)	Rh–P(1)–C(19)	113.3(2)
Rh–P(1)–C(25)	110.4(1)	C(19)–P(1)–C(25)	105.4(2)
Rh–P(1)–C(31)	115.0(2)	C(19)–P(1)–C(31)	104.7(2)
C(25)–P(1)–C(31)	107.3(2)	Rh–P(2)–C(1)	109.0(2)
Rh–P(2)–C(7)	115.5(2)	C(1)–P(2)–C(7)	107.9(2)
Rh–P(2)–C(13)	115.3(2)	C(1)–P(1)–C(13)	104.1(2)
C(7)–P(2)–C(13)	104.3(2)	F(1)–B(1)–F(2)	109.8(8)
F(1)–B(1)–F(3)	110.0(7)	F(2)–B(1)–F(3)	110.8(7)
F(1)–B(1)–F(4)	105.7(7)	F(2)–B(1)–F(4)	110.3(8)
F(3)–B(1)–F(4)	110.2(8)		

The cation $[Rh(NO)(PPh_3)_2(RNNNR)]^+$ (R = *p*-tolyl) is mono- rather than bi-nuclear, and its structure differs significantly from that of (**2**). The rhodium atom is trigonal bipyramidal with the linear nitrosyl [Rh–N(4)–O(1) 177.5(6)°] and the chelating triazenido ligand in the equatorial plane. The two PPh_3 ligands therefore occupy *trans* axial sites [P(1)–Rh–P(2) 175.0(1)°]. The trigonal-bipyramidal geometry at rhodium is severely distorted by the small bite angle of the chelating triazenido ligand [N(1)–Rh–N(3) 57.0(2)°; *cf.* ideal inter-equatorial ligand angle, 120°]. Nevertheless, the N(4)N(1)N(3)Rh unit is very close to planar (maximum deviation from least-squares plane = 0.008 Å) and the Rh–P vectors are essentially orthogonal to this plane (at angles of 85.6 and 85.0°).



(**A**)



(**B**)

There are two other isomeric forms that were *a priori* possible for the cation of (**3**), *viz.* (**A**) and (**B**), having square-pyramidal (with bent, apical NO) and square-planar (with linear NO) co-ordination geometries at the metal respectively. Examples of species of type (**A**) include $[Rh(NO)(PPh_3)_2(SO_4)]$ (**4**)[4] and $[Rh(NO)(PPh_3)_2\{\sigma\text{-}OC(O)CF_3\}_2]$ (**5**),[5] while *trans*-$[Ir(CO)(PPh_3)_2(\sigma\text{-}RNNNR)]$ (R = *p*-tolyl) (**6**)[6] adopts the geometry of type (**B**), with CO replacing $[NO]^+$. Finally there exist several structurally characterised species which have trigonal-bipyramidal metal geometries (with linear, equatorial NO), *e.g.* $[Ir(NO)(PPh_3)_2\{\sigma\text{-}OC(O)CF_3\}_2]$ (**7**)[5] and $[Ir(NO)(phen)(PPh_3)_2][PF_6]_2$ (**8**)[7] (phen = 1,10-phenanthroline). Clearly the balance between these forms is very delicate and dictated by factors more subtle than electron counts, *viz.* the different structures adopted by the formally isoelectronic species (**5**) and (**7**), and (**3**) and (**6**).

Experimental

The preparation and purification of the complex described were carried out under an atmosphere of dry nitrogen. The complex $[Rh(NO)(NCMe)_2(PPh_3)_2][BF_4]_2$ was prepared[2] by a published method.

Infrared spectra were recorded on a Perkin-Elmer 1710 IFTS instrument, ^1H n.m.r. spectra on a JEOL JNM GX-270 spectrometer and calibrated against tetramethylsilane as internal reference, and ^{31}P n.m.r. spectra on a JEOL FX-90Q spectrometer and calibrated against H_3PO_4 as external reference. Cyclic voltammetry was carried out as described previously.[8] Microanalyses were carried out by the staff of the Microanalytical Service of the School of Chemistry, University of Bristol.

*(Di-*p*-tolyltriazenido)nitrosylbis(triphenylphosphine)rhodium Tetrafluoroborate–Dichloromethane* (1/1), $[Rh(NO)(PPh_3)_2(RNNNR)][BF_4]\cdot CH_2Cl_2$ (R = *p-tolyl*).—To $[Rh(NO)(NCMe)_2(PPh_3)_2][BF_4]_2$ (0.58 g, 0.64 mmol) in thf (40 cm^3) was added a solution of Na[RNNNR] (R = *p*-tolyl) in thf (20 cm^3), prepared from RNNNHR (0.142 g, 0.63 mmol) and an excess of NaH. After 15 min the brown solution was evaporated to dryness, and the residue was extracted with CH_2Cl_2 (70 cm^3). Addition of n-hexane (50 cm^3), partial removal of the solvent *in vacuo*, and cooling to 0 °C gave the product as brown crystals, yield 0.47 g (70%) (Found: C, 57.3; H, 4.4; N, 5.2. $C_{50}H_{44}BF_4N_4OP_2Rh\cdot CH_2Cl_2$ requires C, 58.1; H, 4.4; N, 5.3%).

The complex is soluble in polar solvents such as CH_2Cl_2, thf, and acetone to give brown solutions which slowly decompose in air.

Structure Analysis of Complex (**3**)$\cdot CH_2Cl_2$.—*Crystal data.* $C_{50}H_{44}BF_4N_4OP_2Rh\cdot CH_2Cl_2$, M = 1 053.5, orthorhombic, space group $Pca2_1$ (no. 29), a = 19.679(3), b = 11.578(2), c = 21.706(4) Å, U = 4 945(2) Å^3, Z = 4, D_c = 1.42 g cm^{-3}, λ = 0.710 69 Å, $\mu(Mo\text{-}K_\alpha)$ = 5.66 cm^{-1}, $F(000)$ = 2 152, T = 295 K.

Table 2. Atomic co-ordinates (× 10^4) for the complex (**3**)$\cdot CH_2Cl_2$

Atom	*x*	*y*	*z*	Atom	*x*	*y*	*z*
Rh	8 419(1)	8 282(1)	10 000	C(34)	10 387	11 874	9 176
C(2)	8 088(2)	6 135(4)	11 216(2)	C(35)	9 804	11 786	8 813
C(3)	7 684	5 753	11 703	C(36)	9 207	11 338	9 063
C(4)	7 202	4 890	11 605	C(31)	9 192	10 978	9 676
C(5)	7 123	4 409	11 020	C(37)	9 263(3)	8 435(5)	8 640(3)
C(6)	7 527	4 791	10 533	C(38)	9 848(3)	8 839(6)	8 348(3)
C(1)	8 010	5 655	10 631	C(39)	9 883(3)	8 832(7)	7 714(3)
C(8)	9 586(3)	5 136(5)	10 589(2)	C(40)	9 339(4)	8 471(6)	7 347(3)
C(9)	10 239	4 667	10 612	C(41)	8 748(4)	8 088(6)	7 635(3)
C(10)	10 659	4 720	10 095	C(42)	8 707(4)	8 089(6)	8 269(3)
C(11)	10 427	5 242	9 555	C(43)	9 431(5)	8 436(8)	6 658(4)
C(12)	9 774	5 711	9 533	C(44)	9 950(3)	8 377(5)	10 677(3)
C(7)	9 354	5 658	10 050	C(45)	10 650(3)	8 463(6)	10 590(3)
C(14)	8 389(2)	4 466(3)	9 119(2)	C(46)	11 080(4)	8 469(7)	11 085(4)
C(15)	8 071	3 909	8 627	C(47)	10 829(3)	8 395(6)	11 698(3)
C(16)	7 512	4 417	8 340	C(48)	10 136(4)	8 335(6)	11 762(3)
C(17)	7 271	5 483	8 544	C(49)	9 704(3)	8 341(6)	11 270(3)
C(18)	7 589	6 041	9 035	C(50)	11 311(4)	8 443(7)	12 240(4)
C(13)	8 148	5 533	9 323	C(51)	5 113(5)	5 252(11)	7 556(10)
C(20)	8 144(2)	10 288(3)	11 272(2)	N(1)	9 178(2)	8 356(4)	9 275(2)
C(21)	8 096	10 776	11 858	N(2)	9 717(3)	8 417(4)	9 622(3)
C(22)	8 297	11 917	11 953	N(3)	9 491(2)	8 378(4)	10 181(2)
C(23)	8 546	12 570	11 463	N(4)	7 558(3)	8 227(4)	10 233(3)
C(24)	8 594	12 082	10 877	O(1)	7 020(3)	8 157(6)	10 392(4)
C(19)	8 393	10 941	10 781	P(1)	8 441(1)	10 334(1)	10 012(1)
C(26)	7 404(2)	11 933(3)	9 798(2)	P(2)	8 502(1)	6 233(1)	9 993(1)
C(27)	6 861	12 392	9 464	B(1)	6 507(4)	8 130(8)	7 768(5)
C(28)	6 629	11 830	8 936	F(1)	6 945(3)	8 146(5)	8 247(3)
C(29)	6 938	10 808	8 742	F(2)	6 140(3)	7 136(5)	7 777(4)
C(30)	7 480	10 349	9 076	F(3)	6 088(2)	9 072(4)	7 794(2)
C(25)	7 713	10 912	9 604	F(4)	6 905(4)	8 176(5)	7 242(4)
C(32)	9 775(2)	11 066(4)	10 039(2)	Cl(1)	5 625(2)	4 169(4)	7 769(3)
C(33)	10 372	11 514	9 789	Cl(2)	4 390(3)	5 183(6)	7 574(5)

A single crystal of (**3**)$\cdot CH_2Cl_2$, grown from CH_2Cl_2–hexane solution, with approximate dimensions 0.68 × 0.32 × 0.18 mm was mounted in a thin-walled glass capillary under N_2. All diffraction measurements were made on a Nicolet P3m diffractometer at room temperature, using graphite-monochromated Mo-K_α X-radiation. Unit-cell dimensions were determined from 15 centred reflections in the range $27.0 < 2\theta < 30.0°$. A total of 4 758 diffracted intensities were measured in a unique octant of reciprocal space for $4.0 < 2\theta < 50.0°$ by ω–2θ scans. For $40 < 2\theta < 50°$, reflections with intensity < 14 counts per second were not recorded. Three check reflections remeasured after every 100 ordinary data showed no significant decay, but *ca.* 2% variation over the period of data collection; an appropriate correction was therefore applied. Of the intensity data collected, 4 081 unique observations remained after averaging of duplicate and equivalent measurements and deletion of systematic absences. Of these, 3 832 with $I > 0.5\sigma(I)$ were retained for use in structure solution and refinement. An absorption correction was applied on the basis of 289 azimuthal scan data; maximum and minimum transmission coefficients were 0.641 and 0.559 respectively. Lorentz and polarisation corrections were applied.

The structure was solved by conventional heavy-atom (Patterson and Fourier) methods. All non-hydrogen atoms were assigned anisotropic displacement parameters and all hydrogen atom isotropic displacement parameters constrained such that methyl, phenyl, and ring tolyl hydrogens had the same U_{iso}. The solvent C–Cl distances were constrained to be close to 1.7 Å. Phenyl rings were forced to have C–C 1.395 Å and C–C–C angles of 120.0°. Other non-hydrogen atoms were refined without positional constraints. All hydrogen atoms were constrained to idealised geometries (C–H 0.96 Å). The absolute structure was assigned by η-refinement[9] [$\eta = 0.96(7)$ for the structure as reported]. Full-matrix least-squares refinement of this model (526 parameters) converged to final residual indices $R = 0.045$, $R' = 0.049$, and $S = 1.37$.* Weights, *w*, were set equal to $[\sigma_c^2(F_o) + gF_o^2]^{-1}$. Here $\sigma_c^2(F_o)$ is the variance in F_o due to counting statistics and $g = 0.0005$ was chosen to minimise the variation in *S* as a function of F_o. Final difference electron-density maps showed no features outside the range +0.5 to −0.5 e Å^{-3}, the largest of these being close to the solvent molecule which showed signs of partial disorder, having high and anisotropic U_{ii}. No resolution of the solvent atomic sites into partially occupied positions was possible. Table 2 lists the final atomic positional parameters for the non-hydrogen atoms.

All calculations were carried out on a Nicolet R3m/E structure-determination system using programs of the SHELXTL package.[10] Complex neutral-atom scattering factors were taken from ref. 11.

Additional material available from the Cambridge Crystallographic Data Centre comprises H-atom co-ordinates and thermal parameters.

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* $R = \Sigma|\Delta|/\Sigma|F_o|$; $R' = (\Sigma w\Delta^2/\Sigma wF_o^2)^{\frac{1}{2}}$; and $S = [\Sigma w\Delta^2/(N_o - N_v)]^{\frac{1}{2}}$, where $\Delta = |F_o| - |F_c|$ and N_o, N_v = numbers of observations and variables.

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