# Synthesis, Reactivity, and *X*-Ray Crystal Structure of $[Rh\{C_6H_4N(O)O\text{-}2\}(\eta\text{-}C_5Me_5)Cl]$

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Reaction of $[\{Rh(\eta\text{-}C_5Me_5)Cl_2\}_2]$ with $[Hg(C_6H_4NO_2\text{-}2)_2]$ (1/1) or $[Hg(C_6H_4NO_2\text{-}2)Cl]$ (1/2) in the presence of an excess of $[NMe_4]Cl$ gives $[Rh\{C_6H_4N(O)O\text{-}2\}(\eta\text{-}C_5Me_5)Cl]$. This compound reacts with $AgClO_4$ giving solutions from which, after addition of the corresponding ligands, the cationic complexes $[Rh\{C_6H_4N(O)O\text{-}2\}(\eta\text{-}C_5Me_5)(PPh_3)]ClO_4$, $[Rh\{C_6H_4N(O)O\text{-}2\}(\eta\text{-}C_5Me_5)(py)]ClO_4$ (py = pyridine), $[Rh(C_6H_4NO_2\text{-}2)(\eta\text{-}C_5Me_5)(bipy)]ClO_4$ (bipy = 2,2′-bipyridyl) can be obtained. The crystal structure of $[Rh\{C_6H_4N(O)O\text{-}2\}(\eta\text{-}C_5Me_5)Cl]$ has been determined by an *X*-ray diffraction study. Crystals are orthorhombic, space group *Pbca*, with $a$ = 14.574 2(5), $b$ = 14.917 8(6), $c$ = 14.678 2(6) Å, and $Z$ = 8. The molecule exhibits a pseudo-octahedral geometry around the rhodium atom with the $C_6H_4(O)O\text{-}2$ group acting as a chelating ligand, bonded to the metal through a carbon atom of the phenyl ring [Rh–C(1) 2.023(3) Å] and an oxygen atom of the nitro group [Rh–O(1) 2.142(3) Å]. The co-ordination of the nitro group causes a lengthening of the N–O(Rh) bond distance [N–O(1) 1.260(4) Å] compared with the unco-ordinated N–O bond length [1.224(4) Å]. Infrared data indicate that the aryl ligand co-ordinates in the same way in the complexes $[Rh\{C_6H_4N(O)O\text{-}2\}(\eta\text{-}C_5Me_5)(PPh_3)]ClO_4$ and $[Rh\{C_6H_4N(O)O\text{-}2\}(\eta\text{-}C_5Me_5)(py)]ClO_4$ but it is unidentate in the compound $[Rh(C_6H_4NO_2\text{-}2)(\eta\text{-}C_5Me_5)(bipy)]ClO_4$.

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While a large number of rhodium(III) pentamethylcyclopentadienyl derivatives ($\eta\text{-}C_5Me_5$) have been reported,[1] relatively few examples having an aryl group are known; most have been prepared by using lithium or Grignard reagents and are of type $[Rh(aryl)(\eta\text{-}C_5Me_5)X(PR_3)]$ (aryl = Ph and several Me-, $Me_2$-, $CF_3$-, F-, MeO-, or $Me_2N$-substituted phenyl groups; X = Cl, Br, or I; R = Me, Ph, or $C_6H_4Me\text{-}p$).[2] The carbon–hydrogen bond activation of different arenes have also been shown to give $Rh(aryl)(\eta\text{-}C_5Me_5)$ complexes.[3] To our knowledge only one of these has been structurally characterized, $[RhPh(\eta\text{-}C_5Me_5)Br(PMe_3)]$.[2]

We report here the synthesis of some $Rh\{C_6H_4N(O)O\text{-}2\}$ complexes by the use of the arylmercurials $[Hg(C_6H_4NO_2\text{-}2)_2]$ or $[Hg(C_6H_4NO_2\text{-}2)Cl]$ and the *X*-ray crystal structure of $[Rh\{C_6H_4N(O)O\text{-}2\}(\eta\text{-}C_5Me_5)Cl]$. This method of synthesis has only scarcely been used in rhodium chemistry.[4] This work is one of a series of studies we are carrying out to see how useful organomercury compounds are as transmetallating agents in the synthesis of organometallic compounds.[5] We are demonstrating that this method is useful for the synthesis of organo complexes containing functionalized groups especially those whose lithium or Grignard derivatives are not known or difficult to prepare. One of these aryl groups is 2-nitrophenyl: the corresponding lithium derivative is very unstable[6] and, probably for this reason, scarcely used as a transmetallating agent.[7] However, we have been able to prepare by the 'mercurial route' several 2-nitrophenyl complexes of gold,[8] palladium,[9] platinum,[10] and rhodium.[4b,c]

## Results and Discussion

*Synthesis.*—The compound $[\{Rh(\eta\text{-}C_5Me_5)Cl_2\}_2]$ reacts with $[Hg(C_6H_4NO_2\text{-}2)_2]$ (reflux in acetone for 2 d, molar ratio 1/2) giving a mixture of the starting materials along with $[Rh\{C_6H_4N(O)O\text{-}2\}(\eta\text{-}C_5Me_5)Cl]$ (**1**) and $[Hg(R)Cl]$ (R = $C_6H_4NO_2\text{-}2$) [equation (1)] which is difficult to separate. Because $[Hg(R)Cl]$ is symmetrized by $(NMe_4)Cl$ [equation (2)] we repeated the reaction in an excess of this salt and found that it was complete and complex (**1**) obtained in high yield [equation (3)]. In this way, the synthesis of (**1**) is not only cleanly achieved but also $[Hg(R)Cl]$, instead of $[HgR_2]$, or just half the amount of $[HgR_2]$ (1/1), can be used. The by-product of these processes is always the insoluble $[NMe_4]_2[Hg_2Cl_6]$.

$$[\{Rh(\eta\text{-}C_5Me_5)Cl_2\}_2] + 2[HgR_2] \longrightarrow 2[RhR(\eta\text{-}C_5Me_5)Cl] + 2[Hg(R)Cl] \quad (1)$$

$$2[Hg(R)Cl] + Cl^- \longrightarrow [HgR_2] + \tfrac{1}{2}[Hg_2Cl_6]^{2-} \quad (2)$$

$$[\{Rh(\eta\text{-}C_5Me_5)Cl_2\}_2] + [HgR_2] + Cl^- \longrightarrow 2[RhR(\eta\text{-}C_5Me_5)Cl] + \tfrac{1}{2}[Hg_2Cl_6]^{2-} \quad (3)$$

The result of this transmetallation reaction contrasts with that observed in reactions between $[HgR_2]$ and $[MCl_4]^{n-}$ (M = Au, $n$ = 1;[8a,b] M = Pt, $n$ = 2[10a]) or $MCl_n$ (M = Pd[9] or Pt,[10a] $n$ = 2; M = Rh, $n$ = 3[4c]) which always lead first to diaryl

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† Chloro(2-nitrophenyl-$C^1O$)($\eta$-pentamethylcyclopentadienyl)rhodium(III).

*Supplementary data available:* see Instructions for Authors, *J. Chem. Soc., Dalton Trans.*, 1990, Issue 1, pp. xix—xxii.

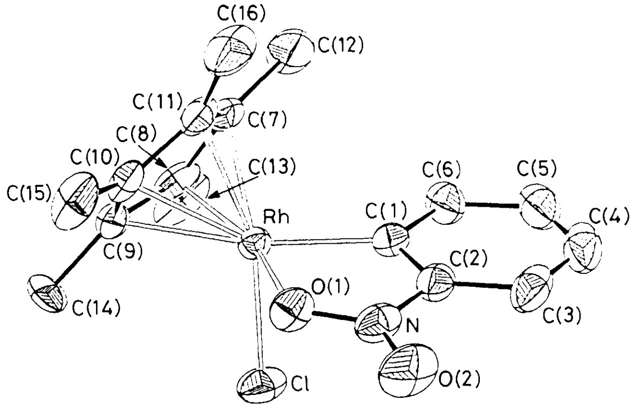


**Figure.** The molecular structure of $[Rh\{C_6H_4N(O)O\text{-}2\}(\eta\text{-}C_5Me_5)Cl]$ (**1**), showing the crystallographic numbering scheme

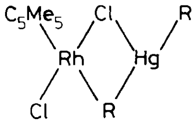
**Table 1.** Selected bond distances (Å) and angles (°) for the complex $[Rh\{C_6H_4N(O)O\text{-}2\}(\eta\text{-}C_5Me_5)Cl]$ (**1**)

| | | | |
|---|---|---|---|
| Rh–Cl | 2.382(2) | Rh–C(9) | 2.224(3) |
| Rh–O(1) | 2.142(3) | Rh–C(10) | 2.225(3) |
| Rh–C(1) | 2.023(3) | Rh–C(11) | 2.154(3) |
| Rh–C(7) | 2.158(3) | Rh–C* | 1.813(2) |
| Rh–C(8) | 2.123(4) | | |
| N–O(1) | 1.260(4) | C(2)–C(3) | 1.409(5) |
| N–O(2) | 1.224(4) | C(3)–C(4) | 1.363(8) |
| N–C(2) | 1.425(5) | C(4)–C(5) | 1.405(7) |
| C(1)–C(2) | 1.389(5) | C(5)–C(6) | 1.363(6) |
| C(1)–C(6) | 1.404(5) | | |
| C(7)–C(8) | 1.429(5) | C(7)–C(12) | 1.504(7) |
| C(7)–C(11) | 1.397(5) | C(8)–C(13) | 1.501(7) |
| C(8)–C(9) | 1.440(6) | C(9)–C(14) | 1.492(6) |
| C(9)–C(10) | 1.389(4) | C(10)–C(15) | 1.496(5) |
| C(10)–C(11) | 1.432(5) | C(11)–C(16) | 1.504(6) |
| Cl–Rh–O(1) | 86.2(1) | Cl–Rh–C* | 124.7(1) |
| Cl–Rh–C(1) | 87.7(1) | O(1)–Rh–C* | 129.9(1) |
| O(1)–Rh–C(1) | 77.6(1) | C(1)–Rh–C* | 134.0(1) |
| Rh–O(1)–N | 114.5(2) | O(1)–N–C(2) | 117.1(3) |
| O(1)–N–O(2) | 120.2(3) | O(2)–N–C(2) | 122.7(3) |
| C(2)–C(1)–C(6) | 115.2(3) | C(2)–C(3)–C(4) | 117.8(3) |
| C(1)–C(2)–N | 115.9(3) | C(3)–C(4)–C(5) | 120.0(4) |
| N–C(2)–C(3) | 119.9(3) | C(4)–C(5)–C(6) | 120.7(4) |
| C(1)–C(2)–C(3) | 124.2(4) | C(1)–C(6)–C(5) | 122.1(4) |
| C(7)–C(8)–C(9) | 108.4(3) | C(7)–C(11)–C(10) | 108.9(3) |
| C(8)–C(9)–C(10) | 107.2(3) | C(8)–C(7)–C(11) | 106.8(3) |
| C(9)–C(10)–C(11) | 108.5(3) | | |

C* represents the centroid of the $\eta^5$-$C_5Me_5$ ligand.

complexes even when a deficiency of the mercurial is used. We have postulated a simultaneous transfer of both aryl groups through a binuclear intermediate containing both aryls as bridging ligands. Probably, in the present case the steric requirements might prevent the formation of such a bridge and only a mixed halide–aryl bridge could be formed. The cleavage of Rh–Cl and Hg–R bonds of the bridges would give complex (**1**) and [Hg(R)Cl] [reaction (1)].

The $C_6H_4NO_2$-2 group (R) in (**1**) acts as a chelating ligand



through the co-ordination of an oxygen atom of the nitro group as indicated by *X*-ray data and the i.r. spectrum [$\nu_{sym}(NO_2)$ at 1 250vs cm$^{-1}$].[4b,c,8–10] This Rh–O bond is very strong because complex (**1**) does not react at room temperature with ligands such as $PPh_3$ (1/1) or pyridine (py) in excess, in contrast with the observed weakness of the Pd–O or Pt–O bonds in palladium[9] and platinum[10] 2-nitrophenyl complexes. We have previously observed this behaviour in the rhodium complex $[\{Rh[C_6H_4N(O)O\text{-}2]_2(\mu\text{-}Cl)\}_2]$ which reacts with different ligands ($PPh_3$, $AsPh_3$, or py) by cleaving the bridge but never the oxygen–rhodium bond.[4c]

The inertness of complex (**1**) can be circumvented by replacing the chloro ligand by the poorly co-ordinating anion $ClO_4^-$. Thus, (**1**) reacts with $AgClO_4$ giving an orange solution from which a brown compound can be isolated in a high yield. This solid seems to be by i.r. spectroscopy and its reactivity $[Rh\{C_6H_4N(O)O\text{-}2\}(\eta\text{-}C_5Me_5)(OClO_3)]$ (**2**) but we have been unable to get a spectroscopically pure sample (see Experimental section). It behaves as an 1:1 electrolyte in acetone probably because the co-ordinated anion is substituted by the solvent.

Solutions of complex (**2**) can be prepared *in situ* and treated with $PPh_3$ or pyridine giving the cationic complexes $[Rh\{C_6H_4N(O)O\text{-}2\}(\eta\text{-}C_5Me_5)L]ClO_4$ [L = $PPh_3$, (**3**) or py, (**4**)], $\nu_{sym}(NO_2)$ at 1 250vs cm$^{-1}$. When the reaction is carried out with 2,2′-bipyridine (bipy) the oxygen–rhodium bond is cleaved, as shown by i.r. spectroscopy [$\nu_{sym}(NO_2)$ at 1 350vs cm$^{-1}$], giving rise to $[Rh(C_6H_4NO_2\text{-}2)(\eta\text{-}C_5H_5)(bipy)]ClO_4$ (**5**). This is the first example of such a cleavage in 2-nitrophenylrhodium(III) complexes. In contrast, reactions of $[\{Rh[C_6H_4N(O)O\text{-}2]_2(\mu\text{-}Cl)\}_2]$ with L–L = 1,10-phenanthroline or bipy give complexes of the type $[Rh\{C_6H_4N(O)O\text{-}2\}_2(L\text{-}L)]$ and $[Rh\{C_6H_4N(O)O\text{-}2\}_2Cl_2]$ which contain chelating $C_6H_4NO_2$-2 groups in both the anion and the cation.[4c] Compounds (**3**)—(**5**) also behave as 1:1 electrolytes in acetone solution. Complex (**1**) shows an absorption at 285m cm$^{-1}$ which (**2**)—(**5**) do not and can be assigned to $\nu$(Rh–Cl). The $^1H$ n.m.r. resonances due to the aryl group appear as a set of complex multiplets at $\delta$ 6.9—8.2 which are partially obscured by the resonances due to the ligands in (**3**) and (**4**).

*Structure of Complex* (**1**).—The crystal structure of complex (**1**) consists of discrete molecules packed by van der Waals forces (Figure). The structure is chiral at the rhodium atom, with both enantiomers being present in the unit cell. Selected geometrical parameters are given in Table 1.

In these molecules the co-ordination geometry around the rhodium atom could be described as pseudo-octahedral with a $\eta^5$-$C_5Me_5$ group occupying three *fac* co-ordination sites. The metal co-ordination sphere is completed by a chelating 2-nitrophenyl ligand (bonded through an *ortho* carbon of the aromatic group and through an oxygen atom of the nitro substituent) and by a terminal chlorine atom.

Individual rhodium–carbon distances involving the $C_5Me_5$ ligand show significant variations [ranging from 2.123(4) to 2.225(3) Å], probably reflecting the asymmetry of the ligand arrangements on the other side of the metal. As indicated by Maitlis,[11] longer distances are observed [Rh–C(9) 2.224(3) and Rh–C(10) 2.225(3) Å] nearly opposite to the atom with lower electronegativity, that is the carbon of the phenyl group. As is usual for metal polyenyl complexes,[12] the different rhodium–carbon interactions result in slight differences in the internal C–C bond distances of the $C_5Me_5$ ring, longer Rh–C distances being associated with shorter C–C bond lengths (see Table 1). These differences are consistent, with a slight degree of localization in carbon–carbon bonding, with a weak distortion of the ring towards a diene representation.[12]

The Rh atom lies 1.810(2) Å from the least-squares plane of the $C_5Me_5$ ring, with no atom deviation from this plane bigger than 0.030 Å. This distance falls at the long end of the usual range reported for neutral $Rh^{III}(\eta\text{-}C_5Me_5)$ complexes (1.74—1.82 Å),[11] and compares well with that determined in $[Rh(\eta\text{-}C_5Me_5)(NO_3)_2(PPh_3)]$ (1.815 Å).[13]

The Rh–C(phenylic) bond distance [Rh–C(1) 2.023(3) Å] is similar to the lengths observed [2.041(3) and 1.984(4) Å] in the closely related complex $[\overline{Rh\{C_6H_4N(O)O}\text{-}2\}_2Cl(CO)]$, although the atoms situated in relative *trans* positions in the latter complex are rather different.[4b] When compared with other analogous rhodium(III) complexes, this Rh–C distance is slightly shorter than that found in $[RhPh(\eta\text{-}C_5Me_5)Br(PMe_3)]$ [2.054(9) Å],[2] or slightly longer than those reported for rhodium(III) azophenyl derivatives (1.98—2.00 Å).[14] The Rh–O length [2.142(3) Å] is identical, within experimental error, to the distance observed in $[\overline{Rh\{C_6H_4N(O)O}\text{-}2\}_2Cl(CO)]$ for the Rh–O bond *trans* to the carbonyl group, but markedly shorter than that *trans* to the phenyl group. However, it is very similar to those observed for analogous M–O bonds in related complexes of Pd and Pt.[9,10]

The N–O(Rh) length [1.260(4) Å] is longer than the other N–O distance [1.224(4) Å]. This observation and the C–N bond length [1.425(5) Å] are as in other chelating 2-nitrophenyl ligands.[4c]

The C–C bond distances in the phenyl ring show an irregular distribution. Three of them [C(2)–C(3) 1.409(5), C(4)–C(5) 1.405(7), and C(6)–C(1) 1.404(5) Å] are significantly longer and one marginally longer [C(1)–C(2) 1.389(5) Å] than the other two [C(3)–C(4) 1.363(8) and C(5)–C(6) 1.363(6) Å]. According to a previous study[4c] the C–C bonding pattern of the chelating 2-nitrophenyl ligand in complex (**1**) could be considered intermediate between that in unidentate and chelating 2-nitrophenyl complexes of Pd and Pt, where an average value of 1.391(10) Å is found independently of the type of co-ordination of the aryl ligand, and that in $[\overline{Rh\{C_6H_4N(O)O}\text{-}2\}_2Cl(CO)]$ where great C–C bond-length distortions occur [range 1.233(7)—1.568(6) Å]. According to that previous discussion, the differences between this carbonyl rhodium(III) complex and (**1**) could be due to the weaker $\pi$-acceptor ability of the $C_5Me_5$ ligand compared with that of the CO group leading to a smaller withdrawal of electron density by the nitro group from its own aromatic ring in complex (**1**) than in the carbonyl complex.

The Rh–Cl bond distance, 2.382(2) Å, can be considered as normal for rhodium(III) complexes, comparing well with that found in $[\{Rh(\eta\text{-}C_5Me_5)Cl_2\}_2]$ [2.397(1) Å][15] and being slightly shorter than in the related complex $[\overline{Rh\{C_6H_4N(O)O}\text{-}2\}_2Cl(CO)]$ [2.416(1) Å].[4b]

## Experimental

Infrared spectra were recorded in the range 4 000—200 cm$^{-1}$ on a Perkin-Elmer 1 430 spectrophotometer using Nujol mulls between polyethylene sheets, $^1H$ and $^{31}P$ n.m.r. spectra in $CDCl_3$ solutions ($\delta$ with respect to $SiMe_4$ and external $H_3PO_4$, respectively) on a Varian FT-80 spectrometer. Conductivities were measured with a Philips conductimeter for acetone solutions. Melting points were determined on a Reichert apparatus and are uncorrected. The C, H, and N analyses were carried out with a Perkin-Elmer 240C microanalyzer. Reactions were carried out with magnetic stirring and without special precautions against light or atmospheric moisture unless otherwise stated. The starting complexes $[Hg(C_6H_4NO_2\text{-}2)Cl]$[16] and $[\{Rh(\eta\text{-}C_5Me_5)Cl_2\}_2]$[17] were prepared by literature methods.

$[\overline{Rh\{C_6H_4N(O)O}\text{-}2\}(\eta\text{-}C_5Me_5)Cl]$ (**1**).—A mixture of $[\{Rh(\eta\text{-}C_5Me_5)Cl_2\}_2]$ (235 mg, 0.38 mmol), $[Hg(C_6H_4NO_2\text{-}2)Cl]$ (272 mg, 0.76 mmol), and $(NMe_4)Cl$ (201 mg, 1.83 mmol) in acetone (8 cm$^3$) was heated in a closed tube at 75 °C for 40 h. Solvent was removed *in vacuo* and the residue extracted with $CH_2Cl_2$ until the extracts were colourless. These extracts were filtered over anhydrous $MgSO_4$ and evaporated to dryness *in vacuo* giving a residue which was washed with water and recrystallized from $CH_2Cl_2$–$Et_2O$ giving complex (**1**) as a brown solid (89% yield), m.p. 220 °C (decomp.), $\Lambda_M = 0\ \Omega^{-1}$ cm$^2$ mol$^{-1}$, $\delta$(Me) 1.7 (s) (Found: C, 48.90; H, 4.95; N, 3.30. Calc. for $C_{16}H_{19}ClNO_2Rh$: C, 48.55; H, 4.85; N, 3.55%. Single crystals of (**1**) were obtained by slow diffusion of diethyl ether into a saturated solution of the complex in dichloromethane.

$[\overline{Rh\{C_6H_4N(O)O}\text{-}2\}(\eta\text{-}C_5Me_5)(OClO_3)]$ (**2**).—Complex (**1**) (62 mg, 0.16 mmol) was added to an acetone solution (10 cm$^3$) of $AgClO_4$ (32 mg, 0.16 mmol) and the light-protected mixture stirred at room temperature for 30 min, filtered, and the resulting solution evaporated *in vacuo*. The residue was recrystallized from $CH_2Cl_2$–$Et_2O$ to give (**2**) as a brown solid (82% yield), m.p. 112 °C, $\Lambda_M = 94\ \Omega^{-1}$ cm$^2$ mol$^{-1}$, $\delta$(Me) 1.68 (s); an impurity (*ca.* 10%) showed a small signal at $\delta$ 1.36. I.r.: $\nu_{sym}(NO_2)$ at 1 250vs, $\nu(OClO_3)$ 1 130br, vs, 1 080br, vs, 635s, 620m, and 880 cm$^{-1}$ (Found: C, 43.95; H, 4.80; N, 3.60. Calc. for $C_{16}H_{19}ClNO_6Rh$: C, 41.80; H, 4.15; N, 3.05%).

$[\overline{Rh\{C_6H_4N(O)O}\text{-}2\}(\eta\text{-}C_5Me_5)(PPh_3)]ClO_4$ (**3**).—Complex (**1**) (71 mg, 0.18 mmol) was added to $AgClO_4$ (37 mg, 0.18 mmol) in acetone (10 cm$^3$) and the light-protected mixture stirred at room temperature for 15 min. The precipitated AgCl was filtered off and $PPh_3$ (47 mg, 0.18 mmol) added to the solution which was stirred for 30 min. The solvent was removed *in vacuo* and the residue recrystallized from $CH_2Cl_2$–$Et_2O$ giving yellow complex (**3**) (92% yield), m.p. 228 °C (decomp.), $\Lambda_M = 108\ \Omega^{-1}$ cm$^2$ mol$^{-1}$, $\delta$(Me) 1.49 (d) ($J_{PH}$ = 3 Hz), $\delta$(P) 35.2 (d) p.p.m. ($J_{RhP}$ = 149 Hz) (Found: C, 57.20; H, 4.75; N, 1.90. Calc. for $C_{34}H_{34}ClNO_6PRh$: C, 56.55; H, 4.75; N, 1.95%).

$[\overline{Rh\{C_6H_4N(O)O}\text{-}2\}(\eta\text{-}C_5Me_5)(py)]ClO_4$ (**4**).—This compound was similarly prepared from $AgClO_4$ (46 mg, 0.22 mmol), (**1**) (88 mg, 0.22 mmol), and pyridine (three drops) as an orange solid (93% yield), m.p. 174 °C, $\Lambda_M = 109\ \Omega^{-1}$ cm$^2$ mol$^{-1}$, $\delta$(Me) 1.63 (s) (Found: C, 46.85; H, 4.50; N, 5.20. Calc. for $C_{21}H_{24}ClN_2O_6Rh$: C, 46.80; H, 4.50; N, 5.20%).

$[Rh(C_6H_4NO_2\text{-}2)(\eta\text{-}C_5Me_5)(bipy)]ClO_4$ (**5**).—Yellow complex (**5**) was prepared in a similar way from (**1**) (73 mg, 0.18 mmol), $AgClO_4$ (38 mg, 0.18 mmol), and 2,2′-bipyridine (29 mg, 0.18 mmol) in 93% yield, m.p. 198 °C (decomp.), $\Lambda_M = 111\ \Omega^{-1}$ cm$^2$ mol$^{-1}$. It is not soluble enough for n.m.r. measurements (Found: C, 50.10; H, 4.75; N, 6.45. Calc. for $C_{26}H_{27}ClN_3O_6Rh$: C, 50.70; H, 4.40; N, 6.80%).

X-*Ray Structure Determination of Complex* (**1**).—*Crystal data.* $C_{16}H_{19}ClNO_2Rh$, $M$ = 395.69, orthorhombic, space group *Pbca* (no. 61), $a$ = 14.574 2(5), $b$ = 14.917 8(6), $c$ = 14.678 2(6) Å, $U$ = 3 191.3(2) Å$^3$, $Z$ = 8, $D_c$ = 1.647 g cm$^{-3}$, Mo-$K_\alpha$ radiation ($\lambda$ = 0.710 69 Å), crystal size 0.45 × 0.40 × 0.57 mm, $\mu$ = 12.3 cm$^{-1}$, $F$(000) = 1 600.

*Data collection and processing.* Stoe-Siemens AED-2 four-circle diffractometer, profile-fitting mode,[18] $2\theta_{max.}$ 52°, 6 995 reflections measured, 3 185 unique ($R_{int}$ 0.016), 2 685 with $F > 5\sigma(F)$ used for all the calculations. Absorption correction based on empirical method (DIFABS program[19]), minimum and maximum corrections 0.892 and 1.234. No crystal decay. Cell constants refined from $2\theta$ values of 38 reflections in the range 20—35°.

*Structure solution and refinement.* Heavy-atom method. Re-

**Table 2.** Final atomic co-ordinates ( × $10^4$) for complex (**1**)

| Atom | *x* | *y* | *z* |
|---|---|---|---|
| Rh | 1 789(1) | 1 842(1) | 1 031(1) |
| Cl | 234(1) | 1 390(1) | 833(1) |
| O(1) | 1 519(2) | 2 828(2) | 5(2) |
| O(2) | 859(2) | 4 096(2) | −243(2) |
| N | 1 135(2) | 3 529(2) | 294(2) |
| C(1) | 1 316(2) | 2 888(2) | 1 772(2) |
| C(2) | 1 044(2) | 3 622(2) | 1 256(3) |
| C(3) | 693(3) | 4 423(2) | 1 626(4) |
| C(4) | 611(3) | 4 478(3) | 2 549(4) |
| C(5) | 854(3) | 3 743(3) | 3 097(3) |
| C(6) | 1 188(3) | 2 976(3) | 2 716(3) |
| C(7) | 2 867(2) | 1 373(3) | 1 919(2) |
| C(8) | 2 277(2) | 652(2) | 1 659(3) |
| C(9) | 2 377(2) | 507(2) | 694(3) |
| C(10) | 2 972(2) | 1 160(2) | 369(2) |
| C(11) | 3 258(2) | 1 707(3) | 1 119(3) |
| C(12) | 3 054(4) | 1 652(4) | 2 886(4) |
| C(13) | 1 746(3) | 49(4) | 2 285(4) |
| C(14) | 1 901(3) | −190(3) | 139(4) |
| C(15) | 3 285(3) | 1 296(4) | −592(3) |
| C(16) | 3 977(3) | 2 430(3) | 1 095(5) |

finement by full-matrix least squares on *F* using the SHELX system[20] with initial isotropic and subsequent anisotropic thermal parameters for non-H atoms. Hydrogen atoms incorporated using a riding model and common fixed thermal parameter; weighting scheme $w = 4.1132/[\sigma^2(F) + 0.0003F^2]$; 190 parameters. Final *R* and *R'* values are 0.0313 and 0.0347. Final atomic co-ordinates for the non-hydrogen atoms of complex (**1**) are given in Table 2.

Additional material available from the Cambridge Crystallographic Data centre comprises H-atom co-ordinates, thermal parameters, and remaining bond distances and angles.

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