Dinitrogen Complexes of Osmium(II)

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A series of 12 dinitrogen complexes of osmium(II), mer- $[OsX_2(N_2)(QR_3)_3]$ (X = Cl or Br; QR₃ = tertiary phosphine or arsine) has been prepared by the zinc reduction under dinitrogen of solutions of mer-[OsX₃(QR₃)₃] in anhydrous tetrahydrofuran (THF). The compounds are assigned the meridional configuration with dinitrogen trans to halogen on the basis of their n.m.r. and i.r. spectra. The co-ordinated dinitrogen is displaced quantitatively by diphosphine Ph₂P·CH₂·CH₂·PPh₂ or by halide ion in the presence of air. Attempts to prepare an analogous series of complexes of ruthenium(II) were unsuccessful.

ALTHOUGH a large number of dinitrogen complexes of the transition metals has been prepared during the past five years 1 there has been only one extensive series of compounds reported, those of the general formula $[CoH(N_2)(PR_3)_3]$, and many members of this series are unstable oils, difficult to characterise.^{2,3} Attempts to prepare analogous series of other dinitrogen complexes have proved unsuccessful.^{3,4} Recently, however, we have been able to prepare two series of stable dinitrogen complexes, one of osmium(II),⁵ which we report here in detail, and another of rhenium(1).6



Q = tertiary phosphine or arsine $\tilde{X} = halogen$

Twelve stable crystalline compounds [OsX2(N2)- $(QR_3)_3$] (X = Cl or Br; Q = P or As) (I) have been

¹ R. Murray and D. C. Smith, Co-ordination Chem. Rev., 1968, 3, 429; Yu. G. Borodko and A. E. Shilov, Uspekhi Khim., 1969, 35, 761.

² A. Misono, Y. Uchida, T. Saito, M. Hidai, and M. Araki, Inorg. Chem., 1969, **8**, 168.

J. Chatt, D. P. Melville, and R. L. Richards, J. Chem. Soc. (A), 1969, 2841. ⁴ J. P. Collman, M. Kubota, F. D. Vastine, J. Y. Sun, and

J. W. Kang, J. Amer. Chem. Soc., 1968, 90, 5430.

prepared directly from dinitrogen, and here we describe the characterisation and physical properties of the series, together with some simple reactions.

Preparation of Complexes.—Following the discovery by Shilov et al., that ruthenium trichloride and osmium compounds when reduced in THF solution took up molecular nitrogen to give dinitrogen complexes,^{7,8} we reduced mer- $[OsX_3(QR_3)_3]$ (X = Cl or Br; $QR_3 =$ tertiary phosphine or tertiary arsine) in THF solution with amalgamated zinc. Under 1 atm. of dinitrogen the red colour of the osmium(III) compound changed rapidly to green (except for $QR_3 = PMe_2Ph$), and then slowly to a pale yellow. Dinitrogen was usually passed through the solution overnight until the solvent was removed, then treatment of the residual yellow oil with methanol gave pale pink solids which had analyses close to that calculated for $[OsX_2(N_2)(QR_3)_3]$. Later experiments with about 100 atm pressure of dinitrogen gave compounds which were white and crystalline, as might be expected of spin-paired compounds of osmium(II). The green intermediate colour probably

⁵ J. Chatt, G. J. Leigh, and R. L. Richards, Chem. Comm., 1969, 515; J. Chatt, G. J. Leigh, and D. M. P. Mingos, Chem. and Ind., 1969, 109.

⁶ J. Chatt, J. R. Dilworth, and G. J. Leigh, Chem. Comm., 1969, 687.

Yu. G. Borodko, A. K. Shilova, and A. E. Shilov, Doklady

Akad. Nauk S.S.S.R., 1967, **176**, 1297. ⁸ Yu. G. Borodko, V. S. Bukreev, G. J. Kozub, M. L. Khidekel, and A. E. Shilov, Zur. strukt. Khim., 1967, **8**, 542.

J. Chem. Soc. (A), 1970

corresponds to the presence of a compound $[OsX_2-(THF)(QR_3)_3]$; the co-ordinated THF is very labile towards dinitrogen and other ligands, but this THF compound is extremely reactive to dioxygen and has not been characterised. If argon is passed through the reduced solution instead of dinitrogen, the green intermediate changes slowly into yellow chloro-bridged osmium(II) species.⁹ No green colour is formed by $\nu(N\equiv N)$ moves to lower frequency, as is also found in other series of dinitrogen complexes.^{2,3} Increasing σ -electron donation caused by the alkyl substituents on the ligands presumably increases the electron density on the metal and causes greater electron drift from the filled *d*-orbitals of the osmium into the anti-bonding orbitals of the dinitrogen and reduces the N=N stretching frequency; $\nu(N=N)$ is rather lower for arsine complexes

TABLE	1
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Dinitrogen complexes	of	osmium(11)
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	Wold			Analyses "			Мп
Compound		Colour	С	́Н	N	Mol. wt.	(decomp.)
[OsCl.(N.)(PMe,Ph),]	65	White	41.0(41.0)	4.8(4.7)	$3 \cdot 9(4 \cdot 0)$	$692(703)^{b}$	150 - 152
OsBr.(N.)(PMe.Ph)]	50	White	36·4(36·4)	$4 \cdot 3(4 \cdot 2)$	3.3(3.5)	· · ·	132 - 134
OsCl, (N,) (PEt, Ph)	70	White	45·9(45·7)	5.9(5.8)	3 ·5(3·6)	755(787) ^b	ء 1 59—16 0 د
OsCl, (15N,) (PEt, Ph),]	60	Pink ^e	、	. ,	3.5(3.6)		ء 15 9—16 0 د
OsBr. (N.) (PEt. Ph)	70	White	$41 \cdot 1(41 \cdot 1)$	$5 \cdot 2(5 \cdot 1)$	$3 \cdot 1(3 \cdot 2)$	$905(875)^{d}$	148 - 150
OsCl, (N,) (PPr ⁿ , Ph),	65	White	49·8(49·6)	6.8(6.6)	3.0(3.2)	786(871) »	137 - 139
OsCl, (N,) (PBu ⁿ , Ph),	70	White	52.7(52.8)	$7 \cdot 4(7 \cdot 3)$	$2 \cdot 8 (2 \cdot 9)$	$912(955)^{b}$	125 - 126
OsCl, (N,) (PMePh,)	65	White	$52 \cdot 6(52 \cdot 8)$	4·3(4·4)	$2 \cdot 9(3 \cdot 2)$	• •	137 - 140
OsCl, N, (PEtPh,)	65	White	$54 \cdot 3(54 \cdot 1)$	$5 \cdot 1(4 \cdot 8)$	$3 \cdot 0(3 \cdot 1)$	$943(931)^{d}$	130 - 131
$\left[OsCl_{2}(N_{2})(PEt_{3})_{3}\right]$	35	V. pale pink ^e	33.5(33.6)	6.8(7.0)	$4 \cdot 2(4 \cdot 4)$. ,	101—104 °
[OsCl, (N,) (AsMe, Ph)]	80	Cream	34·6(34·9)	$4 \cdot 1 (4 \cdot 0)$	$3 \cdot 4 (3 \cdot 4)$	853(824) d	150 - 151
$\left[\operatorname{OsCl}_{2}(N_{2})(\operatorname{AsEt}_{2}\operatorname{Ph})_{3}\right]$	50	Fawn ^e	39.6(39.5)	5.3(5.3)	3.0(3.1)	$973(912)^{d}$	154 - 156
^a Calculated values in p	parentheses.	^b In chloroform.	^e True m.p. (see text).	^d In benzene	e. ^e Slight e	contamination by	r [OsCl ₃ (QR ₃)]

 $[OsCl_3(PMe_2Ph)_3]$, probably because the reaction with dinitrogen is too fast, *e.g.* $[OsCl_2(N_2)(PMe_2Ph)_3]$ can be formed in 1—2 h at 100 atm of dinitrogen, whereas other compounds of the series require up to 18 h for complete reaction. A ¹⁵N labelled derivative was prepared from dinitrogen (¹⁵N₂) by the method described previously.¹⁰

TABLE 2

 $v(N\equiv N)$ of dinitrogen complexes

$\nu(N \equiv N)^{a}$	(cm-1)
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Compound	Nujol	Benzene
[OsCl ₂ (N ₂)(PMe ₂ Ph) ₃]	2078, 2060sh	2082
$OsBr_{2}(N_{2})(PMe_{2}Ph)_{3}$	2100, 2070sh	2090
$\left[OsCl_2(N_2)(PEt_2Ph)_3\right]$	2080, 2050sh	2063
$[OsCl_2(15N_2)(PEt_2Ph)_3]$	2010 6	1998 ^b
$[OsBr_2(N_2)(PEt_2Ph)_3]$	2087, 2053sh	2070
$[OsCl_2(N_2)(PPr^n_2Ph)_3]$	2061, 2071	2062
$[OsCl_2(N_2)(PBun_2Ph)_3]$	2080w, 2064, 2050w	2060
$[OsCl_2(N_2)(PMePh_2)_3]$	2092	2092
OsCl ₂ (N ₂)(PEtPh ₂) ₃]	2090, 2050sh	2090
$\left[OsCl_{2}(N_{2})(PEt_{3})_{3}\right]$	2064	2069
[OsCl(N,) (AsMe, Ph)]	2063sh, 2050	2070
$[OsCl_2(N_2)(AsEt_2Ph)_3]$	2070, 2049sh	2059
		(1537-1537)

All bands v. strong unless otherwise stated. $b \nu(^{15}N \equiv ^{15}N)$

If dinitrogen oxide is passed through the green solution dinitrogen complexes are formed, but in a lower state of purity than when dinitrogen is used. The complexes and analytical data are shown in Table 1.

I.r. Spectra.—All compounds show a very intense band in the range 2059—2092 cm⁻¹, assigned to $\nu(N\equiv N)$, shifted by 65 cm⁻¹ on ¹⁵N substitution (Table 2). As the number of alkyl groups or length of alkyl chain of substituents of the phosphine or arsine ligands increases

 $^{\rm 9}$ J. Chatt, D. P. Melville, and R. L. Richards, unpublished results.

than for corresponding phosphine complexes, and suggests a lower d_{π} -acceptor power of arsine ligands. Bromo-complexes have slightly higher frequencies than chloro-complexes consistent with a greater *trans*-influence of bromide in these compounds.

The far i.r. spectra (Table 3) show two clearly identifiable osmium-chlorine stretching frequencies which are

	TABLE 3	
Far i.r. spectra (550	40 cm ¹) of	dinitrogen complexes a
	$\nu(Os-X)$	Other bands
$[\mathrm{OsCl}_2(\mathrm{N_2})(\mathrm{PMe_2Ph})_3]$	304vs, 263vs	486s, 468sh, 433s, 416s, 356m, 342w
$[\mathrm{OsBr_2(N_2)(PMe_2Ph)_3}]$	170s	506m, 494s, 464s, 422s, 419vs, 412s, 356m, 320w, 124w, 116w
$[OsCl_2(N_2)(PEt_2Ph)_3]$	302vs, 252vs	512s, 495s, 446s, 406w
$[\mathrm{OsBr}_2(\mathrm{N}_2)(\mathrm{PEt}_2\mathrm{Ph})_3]$	164m, 150m	512s, 498s, 468s, 450s, 430w, 408w, 328vw, 255w
$[\mathrm{OsCl}_2(\mathrm{N}_2)(\mathrm{PPr}^n_2\mathrm{Ph})_3]$	294vs, 262vs	497s, 453s, 409s, 394sh, 237m
$[OsCl_2(N_2)(PBun_2Ph)_3]$	296vs, 263vs	497s, 456s, 426m, 385m
$[OsCl_2(N_2)(PEtPh_2)_3]$	307vs, 264vs	453s, 443sh, 425m, 387m
$[OsCl_2(N_2)(AsMe_2Ph)_3]$	306vs, 272vs	468s, 253m
	" Nujol mull	s.

not present in the bromo-compounds. Osmiumbromine stretching bands are broader and less easily assigned. Osmium-nitrogen and osmium-phosphorus stretching frequencies are not assignable because the $500-400 \text{ cm}^{-1}$ region of the spectrum, in which these bands probably occur, is obscured by the bands of the phosphine or arsine ligands.

¹⁰ J. Chatt, J. E. Fergusson, J. L. Love, A. B. Nikolsky, R. L. Richards, and J. R. Sanders, *J. Chem. Soc.* (*A*), 1970, 1479.

Inorg. Phys. Theor.

N.m.r. Spectra.—The spectra of complexes with ligands which have methyl groups are particularly useful in assigning configurations 11 and are shown in Table 4. These spectra show that there are two equivalent phosphine or arsine ligands which give rise to a signal of twice the intensity of that at higher field from the unique phosphine or arsine ligand. The low-field signal of the phosphine complexes consists of a triplet for the PPh₂Me complex and two overlapping triplets for the PMe₂Ph complexes. Thus, these phosphines are trans and 'virtually coupled', i.e., the three phosphine ligands are meridional.¹¹ Splitting of the triplet of the PMe₂Ph complexes and splitting of the corresponding

neutral ligands by Ph2P·CH2·CH2·PPh2 in vacuo 5 requires several days in refluxing toluene [equation (3)].

$$\begin{array}{l} [\mathrm{OsCl}_2(\mathrm{N}_2)(\mathrm{QR}_3)_3] + 2\mathrm{Ph}_2\mathrm{P}\cdot\mathrm{CH}_2\cdot\mathrm{CH}_2\cdot\mathrm{PPh}_2 \xrightarrow{\mathrm{Toluene\ 120^\circ}} \\ [\mathrm{OsCl}_2(\mathrm{Ph}_2\mathrm{P}\cdot\mathrm{CH}_2\cdot\mathrm{CH}_2\cdot\mathrm{PPh}_2)_2] + \mathrm{N}_2 + 3\mathrm{QR}_3 \quad (3) \end{array}$$

Their inert character, except the sensitivity to dioxygen, recalls the stability of the ammine-dinitrogen complexes $[Os(NH_3)_5(N_2)]^{2+,12}$ but strong oxidising agents are necessary to displace the dinitrogen ligand from the ammine complex.

Attempts to reduce the co-ordinated dinitrogen to ammonia with TiCl3-KOH, SnCl2, NaBH4, H2, and

		TABLE 4		
	¹ H N.m.r. spe	ectra of dinitrog	gen complexes ^a	
Compound	$\begin{array}{c} \text{Chemical} \\ \text{Shift} \\ (\tau) \end{array}$	$ {}^{2}J(\text{PCH}) $ (Hz)	$ ^{2}J(\text{PCH}) + $ $ ^{4}J(POsPCH) $ (Hz)	Assignment
$[\mathrm{OsCl}_2(\mathrm{N_2})(\mathrm{PMe_2Ph})_3]$	$2 \cdot 3 - 3$ 8 \cdot 17 8 \cdot 68	9.0	8.0	Phenyl multiplet trans-methyl sextet cis-methyl doublet
$[\mathrm{OsBr_2(N_2)(PMe_2Ph)_3}]$	$2 \cdot 3 - 3 \\ 8 \cdot 01 \\ 8 \cdot 59$	9.1	7.8	Phenyl multiplet trans-methyl sextet cis-methyl doublet
$[\mathrm{OsCl}_2(\mathrm{N}_2)(\mathrm{PPh}_2\mathrm{Me})_3]$	$2-3\cdot 3$ 7 \cdot 88 8 \cdot 13	9.4	8.0	Phenyl multiplet trans-methyl triplet cis-methyl doublet
$[\mathrm{OsCl}_2(\mathrm{N}_2)(\mathrm{AsMe}_2\mathrm{Ph})_3]$	$2 \cdot 3 - 3 \cdot 2$ 8 \cdot 28, 8 \cdot 40 8 \cdot 80			Phenyl multiplet trans-methyl doublet cis-methyl singlet

 $a \tau$ Values (measured from multiplet centres with respect to tetramethylsilane in CDCl₃ solution) generally ± 0.01 ; coupling constants generally ± 0.05 Hz.

signal of the AsMe₂Ph complexes into a doublet is consistent with the configuration shown in structure (I), the asymmetry of the complex in the plane perpendicular to that of the dinitrogen and *trans*-phosphorus or arsine ligands causes the methyl groups of the trans-ligands to be in inequivalent environments. The compound [IrHCl₂(PMe₂Ph₃] which has the same configuration has a similar spectrum.¹¹

Reactions of the Complexes.-The complexes are of moderate thermal stability shown by their decomposition points, but in solution in the presence of halide ion they are very sensitive to oxidation. Thus they revert to the parent $[OsX_3(QR_3)_3]$ compounds during their preparation if there is adventitious contact of the yellow reduced osmium solutions with air [equation (1)].

$$[OsCl_2(N_2)(QR_3)_3] + Cl^{-} \xrightarrow[THF]{O_2}$$
$$[OsCl_3(QR_3)] + N_2 \quad (1)$$

In the absence of dioxygen the complexes are comparatively inert; chlorine oxidation in chloroform solution *in vacuo* requires several hours to reach completion $\left[equation (2) \right].$

$$[\operatorname{OsCl}_2(N_2)(QR_3)_3] + \frac{1}{2}\operatorname{Cl}_2 \xrightarrow{25^{\circ}}_{\operatorname{CHCl}_3, 24 \text{ h}} N_2 + \\ [\operatorname{OsCl}_3(QR_3)_3](+\text{a little } [\operatorname{OsCl}_4(QR_3)_2]) \quad (2)$$

Complete displacement of dinitrogen and other

HCl have proved unsuccessful. Further reactions of the dinitrogen complexes and of the reactive green solution, which afford convenient routes to complexes of carbon monoxide and other dinitrogen analogues 5,9 will be reported subsequently.

Attempts to Prepare Analogous Complexes of Ruthenium(II).—The compounds $[RuCl_3(PR_2Ph)_3]$ (R = Me or Et) were reduced with zinc or sodium amalgam under the same conditions as were used for the osmium reactions, but dinitrogen complexes could not be isolated. For R = Et a band at 2120–2140 cm⁻¹ was observed in the i.r. spectrum of the crude product and nitrogen analysis gave values of 0.3-0.5%, but the dinitrogen complex was too unstable to be purified.

EXPERIMENTAL

All manipulations were carried out under dinitrogen using Schlenk-tube techniques unless otherwise stated. Reactions under pressure of dinitrogen were performed in 100-ml glass flasks placed inside a Baskerville rocking autoclave.

The i.r. spectra were measured on the R.I.I.C. Fourier spectrophotometer FS-620, and Grubb-Parsons D.M.4 and Unicam SP 1200 instruments. The n.m.r. spectra were measured using a Varian A60 instrument, and an

¹¹ J. M. Jenkins and B. L. Shaw, J. Chem. Soc. (A), 1966, 1407 and references therein.
¹² A. D. Allen and J. R. Stevens, Chem. Comm., 1967, 1147.

AEI MS 10 instrument was used for mass spectral measurements.

M.p.s were measured on a Kofler hot stage and molecular weights were determined with an Hitachi-Perkin-Elmer 115 osmometer. A Faraday balance was used for magnetic measurements. Microanalyses were by Mr. A. G. Olney of Sussex University.

Tetrahydrofuran was distilled from sodium diphenylketyl into reaction vessels under dinitrogen. Other solvents were degassed before use. The compounds $[MX_3(QR_3)_3]$ $(M = Os \text{ or } Ru; X = Cl \text{ or } Br; QR_3 = tertiary phosphine$ or tertiary arsine) were prepared by established methods.¹³ All the dinitrogen complexes were diamagnetic in the solid state and nonconducting in nitrobenzene solution.

Preparation of Dinitrogen Complexes.-Since the method is general, a typical preparation is described and analytical data, etc. are presented in Table 1.

mer-Dichlorotris(diethylphenylphosphine)(dinitrogen)os-

mium(II). (a) To trichlorotris(diethylphenylphosphine)osmium(III) (2.8 g) dissolved in tetrahydrofuran (30 ml) was added amalgamated zinc (5 g); the mixture was then shaken overnight under 100 atm pressure of dinitrogen in an autoclave.

The autoclave was vented and the flask was quickly stoppered. The zinc was filtered off and the pale yellow filtrate was concentrated under reduced pressure to small volume which upon addition of degassed methanol gave offwhite crystals. These were filtered off and dried; they crystallised from benzene-methanol as white needles (2 g, 70%).

The other compounds listed in the Table were obtained similarly, but some were faintly pink, owing to contamination with < 1% of starting material, as shown by their u.v. spectra.

mer-Dichloro(dinitrogen)tris(triethylphosphine)osmium-

(II) was the most difficult complex to purify because it was very soluble and was only recrystallised in low yield from aqueous ethanol.

(b) As an alternative procedure dinitrogen at 1 atm was vigorously bubbled overnight via a sinter through the reduced osmium solution, so that the initial green colour of the solution was discharged. The resulting yellow oil was worked up as before, but probably because of greater likelihood of contact with air the compounds obtained were always pink or orange in colour.

Preparation of mer-dichlorotris(diethylphenylphosphine)-{dinitrogen(15N2)}osmium(11). A solution of trichlorotris(diethylphenylphosphine)osmium(III) (0.2 g) in tetrahydrofuran (5 ml) was reduced with zinc under argon until the solution was green. The solution was then quickly transferred under argon to one limb of an H-tube,¹⁰ degassed under vacuum, and cooled to -196° . Sufficient dinitrogen-(¹⁵N₂) was condensed onto activated charcoal in the other limb of the H-tube to give a pressure of dinitrogen(15N2) of ca. 3 atm after the H-tube had been sealed and allowed to warm up. After 50 h the tube was opened via a break-seal, and the excess of dinitrogen was removed into storage bulbs by means of an automatic Topler pump; the yellow solution was filtered under argon and worked up as above under argon to give pink needles of dichlorotris(diethylphenylphosphine){dinitrogen(15N2)}osmium(II) (0.12 g, 60%).

Reaction of the Reduced Solution with Dinitrogen Oxide.-Procedure (b) was followed with dinitrogen oxide and after removal of solvent from the reduced solution overnight in a stream of dinitrogen oxide, the resulting orange oil was worked up to give orange crystals, shown by their i.r. and u.v. spectra to be mainly $[OsCl_2(N_2)(PR_3)_3]$ (PR₃ = PEt₂Ph or PPh₂Et) contaminated with [OsCl₃(PR₃)₃].

Reactions of Dinitrogen Complexes .-- Reactions were carried out in sealed glass bulbs (ca. 50 ml.), with a sidearm which could be sealed and a second side-arm carrying a break-seal. In general, a weighed quantity of complex was added to the bulb then the reagent was either weighed or distilled in vacuo. Solvent was then distilled into the bulb at -196° in vacuo and the bulb was sealed. The bulb was warmed, if necessary, to complete the reaction after which it was cooled to -196° , attached to the vacuum line, and opened via the break-seal. The evolved gas was measured by means of the Topler pump and analysed by mass spectrometry.

Reactions with Bis(diphenylphosphino)ethane.-(a) mer-Dichlorotris(diethylphenylphosphine)(dinitrogen)osmium(II). A solution of the osmium compound (0.0385 g) and bis(diphenylphosphino)ethane(dpe) (0.2 g) in toluene was heated at 120° for 48 h. Dinitrogen (1.083 ml, 99.1%) was evolved and a bright yellow solid precipitated. This was filtered off, washed, dried, and was shown to be trans- $[OsCl_2(dpe)_2]^{14}$ by its i.r. spectrum and m.p. (yield 0.050 g, 90%).

(b) mer-Dichloro(dinitrogen)tris(phenyldi-n-propylphosphine)osmium(II). This compound (0.0392 g) reacted similarly with dpe (0.2 g) in toluene (20 ml) to give dinitrogen (1.05 ml, 93%) and trans- $[OsCl_2(dpe)_2]$ (0.043 g, 1.05 ml, 1.05 ml)90%).

Reaction of mer-Dichlorotris(diethylphenylphosphine)(dinitrogen)osmium(II) with Chlorine.—A solution of the osmium compound (0.0385 g) and chlorine (0.075 g) in chloroform (20 ml) was warmed at 30° for 70 h; the solution became orange-red, and dinitrogen (0.984 ml, 98%) was evolved. After removal of solvent [OsCl₃(PEt₂Ph)₃] (0.032 g, 80%) and [OsCl₄(PEt₂Ph)₂] (0.006 g, 16%) were obtained, with identical m.p. and u.v. and i.r. spectra to authentic samples.

Reduction of mer-trichlorotris-(diethylphenylphosphine)ruthenium(III) under dinitrogen.-The ruthenium compound (0.26 g) in THF solution (20 ml) was reduced with sodium amalgam (30 g of 1% amalgam) under 150 atm of dinitrogen for 64 h. Solvent was removed from the brownish solution after decantation from residual amalgam; a sticky brown oil resulted. The i.r. spectrum of this oil showed a strong band at 2120 cm⁻¹. Addition of hexane gave a khaki solid which had a nitrogen content of 0.5%.

In a similar experiment with amalgamated zinc as the reducing agent, a light brown solid was obtained which showed a band at 2130 cm⁻¹ and had a nitrogen content of 0.32%. These products decomposed on attempted purification.

[0/346 Received, March 3rd, 1970]

J. Chatt, G. J. Leigh, D. M. P. Mingos, and R. J. Paske, J. Chem. Soc. (A), 1968, 2636.
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