Reactions of Di-isopropylcarbodi-imide with Some Ruthenium and Osmium Hydrides, leading to Ready Dehydrogenation of an Isopropyl Group; Crystal and Molecular Structure of Carbonylchloro(*N*-isopropenyl-*N'*-isopropylformamidinato)bis(triphenylphosphine)ruthenium(II)[1]

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The reactions of $Pr^iN=C=NPr^i$ with the complexes $[RuH(X)(CO)(PPh_3)_3]$ (X = Cl or Br) in boiling benzene involve insertion of the carbodi-imide moiety into the Ru–H bond and concomitant dehydrogenation of an isopropyl group to yield the derivatives $[RuX\{CH_2{=}C(Me)N \overset{\cdots}{-} CH \overset{\cdots}{-} NCHMe_2\}(CO)(PPh_3)_2]$. A single-crystal *X*-ray diffraction study of the chloro-complex (X = Cl) has confirmed the presence of an *N*-isopropenyl-*N'*-isopropylformamidinate ligand in which the isopropenyl skeleton is essentially coplanar with, and conjugated to, the formamidinate group. The crystals are monoclinic, space group $P2_1/a$, with a = 17.836(2), b = 18.443(3), c = 12.607(1) Å, β = 104.59(3)°, and Z = 4. The structure has been solved *via* the heavy-atom method and refined by least squares using 4 381 observed $[I > 1.5\sigma(I)]$ diffractometer intensities to a final R of 0.086. The octahedral molecule comprises two *trans* phosphines [Ru–P 2.383(4) and 2.400(4) Å], a chelating formamidinate ligand [Ru–N 2.048(10) and 2.242(10) Å], a carbonyl group [Ru–C 1.805(12) Å], and a chlorine atom [Ru–Cl 2.434(4) Å], the latter being *trans* to the more strongly bound nitrogen atom. The complex $[OsH_2(CO)(PPh_3)_3]$ reacts with $Pr^iN=C=NPr^i$ to afford the intermediate product $[OsH(Me_2HCN \overset{\cdots}{-} CH \overset{\cdots}{-} NCHMe_2)(CO)(PPh_3)_2]$ which, on further heating, dehydrogenates to $[OsH\{CH_2{=}C(Me)N \overset{\cdots}{-} CH \overset{\cdots}{-} NCHMe_2\}(CO)(PPh_3)_2]$ in good yield.

IN previous papers within the present series we have described reactions of some heteroallenes including carbon disulphide,[2] alkyl or aryl isothiocyanates,[3] and diarylcarbodi-imides[4] with platinum-metal hydrides leading to formation of complexes containing dithioformate ($S \overset{\cdots}{-} CH \overset{\cdots}{-} S$), thioformamide ($RN \overset{\cdots}{-} CH \overset{\cdots}{-} S$), and formamidinate ($RN \overset{\cdots}{-} CH \overset{\cdots}{-} NR$) ligands respectively. We now discuss the extension of this work to include reactions of di-isopropylcarbodi-imide, and report a novel reaction in which co-ordinated di-isopropylformamidinate ligands undergo dehydrogenation at an isopropyl group. A preliminary account of this work has been published.[5] To the best of our knowledge the reactions described herein, and a similar one involving trinuclear osmium carbonyl clusters recently reported by Adams and Selegue,[6] afford the first examples of the ready dehydrogenation of an isopropyl group to an isopropenyl residue in a transition-metal complex.

EXPERIMENTAL

Platinum-metal salts were supplied by Johnson Matthey and Co. Ltd. Di-isopropylcarbodi-imide was purchased from the Aldrich Chemical Co., and was checked for purity by ^1H n.m.r. spectroscopy. All reactions were performed under a nitrogen atmosphere but products were worked up in the presence of air. Analyses were performed by the Analytical Laboratory at University College, London. Melting points were taken in sealed tubes under nitrogen on a Buchi apparatus. Proton and ^{31}P n.m.r. spectra were obtained in $CDCl_3$ solution using a Bruker HFX90 spectrometer operating in Fourier-transform mode and are referenced to internal $SiMe_4$ and external H_3PO_4 respectively. Positive ^{31}P chemical shifts are to low field of H_3PO_4. Infrared spectra were recorded on a Perkin-Elmer 457 grating spectrometer using samples mulled in Nujol.

Carbonylchloro(N-*isopropenyl*-N'-*isopropylformamidinato*)-*bis*(*triphenylphosphine*)*ruthenium*(II), (A).—Di-isopropylcarbodi-imide (0.3 cm^3) and carbonylchlorohydridotris(triphenylphosphine)ruthenium (0.4 g) in benzene (20 cm^3) were heated under reflux for 6 h. The dark yellow-brown solution was cooled to ambient temperature, filtered, concentrated under reduced pressure, and then diluted with methanol (10 cm^3) to precipitate a bright yellow solid. After washing with methanol and light petroleum (b.p. 60—80 °C), the precipitate was recrystallised from dichloromethane–methanol to give the required product as yellow *microcrystals* (0.21 g, 61%) (Found: C, 64.45; H, 5.40; N, 2.75; P, 8.05. Calc. for $C_{44}H_{43}ClN_2OP_2Ru$: C, 64.75; H, 5.55; N, 3.40; P, 7.60%).

Bromocarbonyl(N-*isopropenyl*-N'-*isopropylformamidinato*)-*bis*(*triphenylphosphine*)*ruthenium*(II) was similarly prepared from bromocarbonylhydridotris(triphenylphosphine)ruthenium as bright yellow *microcrystals* (60%) (Found: C, 61.2; H, 5.05; N, 2.90; P, 7.0. Calc. for $C_{44}H_{43}BrN_2OP_2Ru$: C, 61.4; H, 5.25; N, 3.25; P, 7.20%).

Carbonyl(N,N'-*di-isopropylformamidinato*)*hydridobis*(*triphenylphosphine*)*osmium*(II).—Di-isopropylcarbodi-imide (1.0 cm^3) and carbonyldihydridotris(triphenylphosphine)osmium (1.0 g) in dry toluene (75 cm^3) were heated under reflux for 6 h. The solution was then evaporated to dryness under reduced pressure; the residue was dissolved in the minimum volume of dichloromethane and reprecipitated by addition of methanol. The pale yellow microcrystalline *product* was filtered off, washed with methanol and n-hexane, and dried *in vacuo*. Yield 0.30 g (35%), m.p. 186 °C (Found: C, 60.5; H, 5.35; N, 3.15. Calc. for $C_{44}H_{46}N_2OOsP_2$: C, 60.65; H, 5.30; N, 3.20%).

Carbonylhydrido(N-*isopropenyl*-N'-*isopropylformamidinato*)*bis*(*triphenylphosphine*)*osmium*(II).—Di-isopropylcarbodi-imide (0.7 cm^3) and $[OsH_2(CO)(PPh_3)_3]$ (0.5 g) in dry ethylbenzene (50 cm^3) were heated under reflux for 6 h. The solution was evaporated to dryness and the product isolated as described above as yellow *microcrystals* (0.32 g, 74%), m.p. 174—178 °C (Found: C, 60.7; H, 5.10; N, 3.20. Calc. for $C_{44}H_{44}N_2OOsP_2$: C, 60.8; H, 5.10; N, 3.20%).

Conversion of Carbonyl(N,N′-*di-isopropylformamidinato*)-*hydridobis*(*triphenylphosphine*)*osmium into Carbonylhydrido*(N-*isopropenyl*-N′-*isopropylformamidinato*)*bis*(*triphenylphosphine*)*osmium*.—The starting complex (0.30 g) in dry toluene (75 cm³) was heated under reflux for 6 h. The solution was evaporated to dryness and worked up as described above to yield the required product as yellow *microcrystals* (0.285 g, 95%) identical with an authentic sample.

X-*Ray Crystallographic Study of Complex* (A).—Suitable crystals of (A) were obtained, with great difficulty, by crystallisation from methylene chloride–methanol. All *X*-ray work was carried out using a small fragment of approximate volume 6×10^{-3} mm³ from a multiple cluster. Cell dimensions and crystal orientation were obtained *via* the SEARCH and INDEX routines on an Enraf Nonius CAD4 diffractometer, and refined on the basis of setting angles for 25 high-angle ($30 < \theta < 35°$) reflections.

Crystal data. $C_{44}H_{43}ClN_2OP_2Ru$, $M = 816.28$, Monoclinic, $a = 17.836(2)$, $b = 18.443(3)$, $c = 12.607(1)$ Å, $\beta = 104.59(3)°$, $U = 4\,013.3$ Å³, $D_m = 1.33$ g cm⁻³, $Z = 4$, $D_c = 1.35$ g cm⁻³, $F(000) = 1\,620$, $\mu(\text{Cu-}K_\alpha) = 30.13$ cm⁻¹, space group $P2_1/a$.

Intensity data were recorded on the CAD4 as described previously [7] using an ω–2θ scan mode and Cu-K_α radiation ($\lambda = 1.541\,8$ Å). Scan widths ($\omega = 1.0 + 0.35 \tan\theta$) were rather large, in order to accommodate the variable peak widths indicated during the setting-up procedure. Of the 6 412 data measured, 4 381 satisfied the condition $I > 1.5\sigma(I)$ and were used for the structure analysis and refinement, following correction for Lorentz polarisation and adjustment to allow for the small amount of crystal decay. No corrections were made for absorption.

The structure was solved *via* the heavy-atom method and refined by least squares. Non-hydrogen atoms were assigned anisotropic temperature factors; hydrogen atoms were inserted in idealised positions (C–H 1.08 Å) and assigned a common overall $U_{\text{iso.}}$ value which was refined (final value 0.11 Å²). The weighting scheme $w = 1/[\sigma^2(F_o) + 0.003 F_o^2]$ gave flat agreement analyses. The final $R\ (= \Sigma|\Delta F|/\Sigma|F_o|)$ and $R'\ [= \Sigma(\Delta F w^{\frac{1}{2}})/\Sigma|F_o|w^{\frac{1}{2}}]$ values were 0.086 and 0.084 respectively. Computers, programs, and scattering-factor data sources are as given in ref. 7.

The final non-hydrogen co-ordinates are given in Table 1. Thermal parameters, $|F_o|$ and $|F_c|$ values, and a complete list of bond lengths and angles are available as Supplementary Publication No. SUP 23030 (23 pp.).*

TABLE 1

Atom co-ordinates ($\times 10^4$)

Atom	*x*	*y*	*z*
Ru(1)	2 425	2 438	1 980(1)
Cl(1)	1 816(2)	3 624(1)	1 919(2)
N(1)	2 766)4)	1 376(4)	2 018(5)
N(2)	1 554(5)	1 567(4)	2 007(6)
C(1)	3 325(5)	2 890(6)	1 990(7)
C(2)	4 098(7)	1 279(8)	1 970(9)
C(3)	3 423(6)	953(6)	2 028(7)
C(4)	3 363(8)	150(5)	2 063(11)
C(5)	754(5)	1 352(5)	2 058(8)
C(6)	358(6)	899(6)	1 047(8)
C(7)	765(7)	943(7)	3 110(9)
C(8)	2 091(6)	1 083(6)	2 047(7)
O(1)	3 906(4)	3 194(4)	2 035(5)
P(1)	2 012(1)	2 430(1)	15(2)
C(111)	2 646(6)	2 920(5)	−671(7)
C(112)	2 867(6)	3 610(5)	−310(8)
C(113)	3 338(7)	4 021(6)	−790(10)
C(114)	3 596(7)	3 730(8)	−1 642(10)
C(115)	3 383(7)	3 048(7)	−2 018(10)
C(116)	2 900(7)	2 649(6)	−1 539(9)
C(121)	1 069(5)	2 853(5)	−565(8)
C(122)	889(7)	3 161(7)	−1 581(8)
C(123)	169(7)	3 492(8)	−2 020(10)
C(124)	−387(7)	3 451(8)	−1 431(11)
C(125)	−229(7)	3 118(8)	−428(10)
C(126)	508(6)	2 830(7)	29(8)
C(131)	1 891(6)	1 541(6)	−665(8)
C(132)	1 175(6)	1 295(6)	−1 271(8)
C(133)	1 120(8)	607(6)	−1 718(9)
C(134)	1 781(9)	167(7)	−1 608(9)
C(135)	2 483(7)	414(6)	−1 008(9)
C(136)	2 543(7)	1 081(6)	−552(9)
P(2)	2 676(1)	2 518(1)	3 925(2)
C(211)	3 510(6)	3 092(6)	4 563(7)
C(212)	3 476(7)	3 819(6)	4 280(9)
C(213)	4 045(9)	4 291(8)	4 740(10)
C(214)	4 694(8)	4 022(9)	5 536(11)
C(215)	4 747(7)	3 323(8)	5 832(10)
C(216)	4 150(6)	2 842(7)	5 354(8)
C(221)	1 924(6)	2 949(5)	4 429(8)
C(222)	2 076(7)	3 329(7)	5 416(9)
C(223)	1 480(9)	3 614(7)	5 818(11)
C(224)	723(8)	3 523(7)	5 212(12)
C(225)	553(7)	3 140(8)	4 246(10)
C(226)	1 152(6)	2 865(7)	3 843(8)
C(231)	2 861(6)	1 686(5)	4 722(8)
C(232)	2 450(7)	1 497(7)	5 515(9)
C(233)	2 623(9)	852(8)	6 070(11)
C(234)	3 209(9)	407(8)	5 958(11)
C(235)	3 618(8)	574(7)	5 204(10)
C(236)	3 448(6)	1 209(6)	4 607(9)

RESULTS AND DISCUSSION

Suspensions of the ruthenium complexes $[RuH(X)(CO)(PPh_3)_3]$ (X = Cl or Br) in boiling benzene react with di-isopropylcarbodi-imide over a period of *ca.* 6 h to afford dark yellow-brown solutions from which yellow crystalline products can readily be isolated in *ca.* 60% yield. The yellow crystals dissolve in dichloromethane in the presence of air to afford an emerald green solution from which they can be recovered by addition of methanol. Development of the green colour in the solution, which is not accompanied by changes in the i.r. or n.m.r. spectra, is attributed to formation of a highly coloured, oxidised product in very small concentrations. The i.r. spectra of the new complexes show a carbonyl absorption, $\nu(CO)$ at *ca.* 1 940 cm⁻¹, and high-intensity bands at *ca.* 1 550 and 1 250 cm⁻¹ characteristic of the chelate formamidinate ligand.[4] The ¹H n.m.r. spectra are consistent with the presence of *N*-isopropenyl-*N*′-isopropylformamidinate ligands (L) and therefore indicate that the anticipated insertion of the carbodi-imide moiety into the ruthenium–hydrogen bond of the parent complex has been accompanied by dehydrogenation of one of the isopropyl groups.

For the chloro-complex the methyl groups (a) give rise to a doublet signal [τ 9.34, $^3J(HH') = 6$ Hz, relative intensity 6], one member of which is coincident with a singlet (τ 9.30, 3) attributable to methyl group (b).

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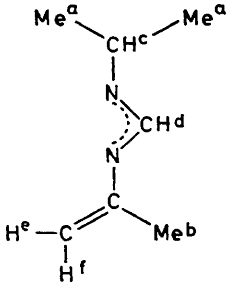
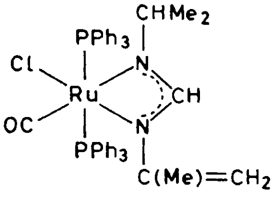
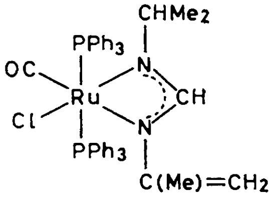
* For details see Notices to Authors No. 7, *J. Chem. Soc., Dalton Trans.*, 1980, Index issue.

Proton (c) generates a septet pattern [τ 6.18, ^{3}J(HH') = 6 Hz, 1] by coupling with the protons of methyl groups (a). The triplet resonance (τ 3.20, 1), although situated at rather higher field than those observed for co-ordinated di-*p*-tolylformamidinate ligands,[4] is attributed to the central proton (d) of the formamidinate moiety. Phosphorus-decoupling experiments confirm that the triplet-splitting arises from coupling of the proton with the ^{31}P nuclei of a pair of equivalent triphenylphosphine ligands. The size of the coupling [^{4}J(PH) = 2.6 Hz] is indicative of ^{31}P nuclei co-ordinated *cis* to the formamidinate ligand.[4] Finally, the geminal olefinic protons (e) and (f) give rise to a pair of singlets at τ 6.42 and 6.85. The proton-decoupled ^{31}P n.m.r. spectrum of the chloro-complex consists of a singlet (δ 31.4) and when taken with the ^{1}H n.m.r. and analytical data is indicative of either stereochemistry (Ia) or (Ib). An *X*-ray diffraction study (see below) has established (Ia) as the correct stereochemistry.





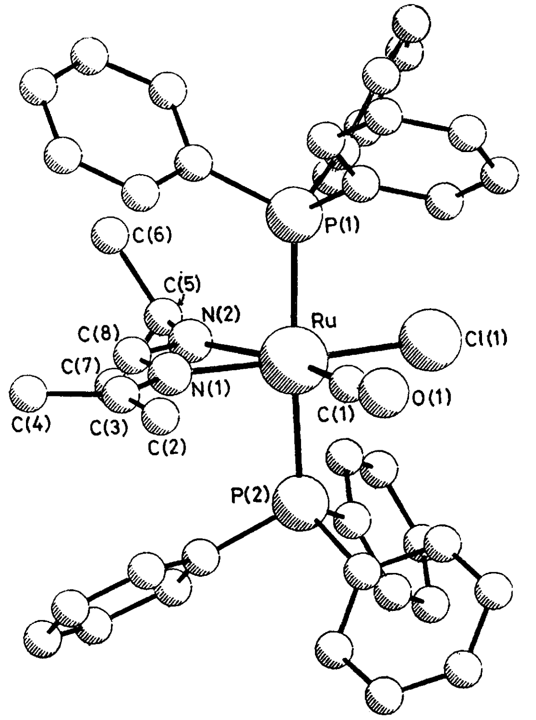
(Ia)



(Ib)

Similar spectroscopic data were recorded for the corresponding bromo-complex [Me^a, τ 9.28(d), ^{3}J(HH') = 6.5 Hz, relative intensity 6; Me^b, 9.25(s), 3; H^c, 6.11-(septet), 1; H^d, 3.17(t), ^{4}J(PH) = 2.5 Hz, 1; H^e, H^f, 6.30(s), 6.68(s); δ(PPh$_3$) = 31.5 p.p.m.]. Attempts to prepare the corresponding osmium complexes gave only unchanged starting material or, under more vigorous conditions, intractable products that we have been unable to characterise. It appears that the conditions required to induce reaction with the more inert osmium complex are sufficient to decompose the rather fragile carbodi-imide reagent.

The molecular structure of (A) is shown in the Figure; important bond lengths and angles are given in Table 2, a full list being included in the Supplementary Publication. The metal atom has a distorted-octahedral co-ordination with *trans* phosphines and with the formamidinate ligand acting as a σ-bonded chelate ligand. Bond lengths within the ligand suggest a delocalised N ⋯ C(H) ⋯ N system with contributions from both canonical forms (IIa) and (IIb). However, the bonding of the ligand to the metal atom is highly asymmetric with the nitrogen atom *trans* to chloride (and attached to the isopropenyl group) much more strongly bound [Ru–N 2.048(10) Å] than the nitrogen *trans* to carbonyl [Ru–N 2.242(10) Å]. Corresponding bond lengths for Ru–N linkages *trans* to hydride and carbonyl ligands in the closely related di-*p*-tolylformamidinato-complex [RuH(*p*-MeC$_6$H$_4$N ⋯ CH ⋯ NC$_6$H$_4$Me-*p*)(CO)(PPh$_3$)$_2$] are 2.248(8) and 2.183(5) Å respectively.[4] The differences in Ru–N bond lengths recorded for these complexes are in accord with the accepted order of *trans* influence for



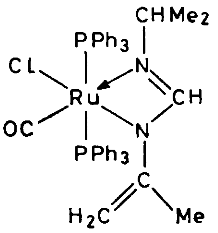
Molecular geometry and atom-labelling scheme for [RuCl{CH$_2$=C(Me)N ⋯ CH ⋯ NCHMe$_2$}(CO)(PPh$_3$)$_2$]

TABLE 2

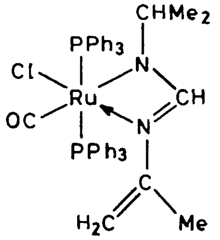
Selected bond lengths (Å) and angles (°)

(*a*) Lengths			
Ru(1)–Cl(1)	2.434(4)	Ru(1)–C(1)	1.805(12)
Ru(1)–N(1)	2.048(10)	Ru(1)–P(1)	2.400(4)
Ru(1)–N(2)	2.242(10)	Ru(1)–P(2)	2.383(4)
N(1)–C(3)	1.406(15)	N(2)–C(5)	1.498(14)
N(1)–C(8)	1.328(14)	N(2)–C(8)	1.300(14)
C(3)–C(2)	1.363(18)	C(1)–O(1)	1.167(13)
C(3)–C(4)	1.487(16)	C(5)–C(6)	1.539(16)
C(5)–C(7)	1.521(17)	P–C	1.806—1.838(13)
(*b*) Angles			
Cl(1)–Ru(1)–N(2)	109.8(3)	P(1)–Ru(1)–N(1)	91.7(3)
Cl(1)–Ru(1)–C(1)	88.5(4)	P(1)–Ru(1)–N(2)	88.7(3)
Cl(1)–Ru(1)–P(1)	91.7(3)	P(1)–Ru(1)–C(1)	92.9(4)
Cl(1)–Ru(1)–P(2)	86.8(2)	P(2)–Ru(1)–N(1)	93.3(4)
N(1)–Ru(1)–N(2)	61.1(4)	P(2)–Ru(1)–N(2)	88.8(3)
N(1)–Ru(1)–C(1)	100.6(5)	P(2)–Ru(1)–C(1)	91.6(4)
N(1)–C(8)–N(2)	112.6(10)	N(1)–C(3)–C(2)	120.1(11)
N(1)–C(3)–C(4)	119.1(11)	N(2)–C(5)–C(6)	110.5(9)
N(2)–C(5)–C(7)	114.4(8)		

octahedral platinum-metal complexes in general,[8] and ruthenium(II) species in particular,[9,10] H $\gg$ CO $\geqslant$ Cl. However, in view of the similar magnitudes of the *trans* influence exerted by carbonyl and chloride ligands it is difficult to reconcile the large difference in Ru–N bond lengths found in the present complex in terms of *trans* influence alone.



(IIa)

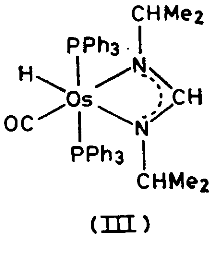


(IIb)

The isopropenyl group is not directly bonded to the metal, the closest approach being Ru $\cdots$ C(3) 3.26 Å, but is held coplanar with the chelate ring, probably through conjugation with the formamidinate π system, as indicated by the C(3)–N(1) distance of 1.41 Å.

Attempts to stop the ruthenium reaction prior to the dehydrogenation step, and thereby isolate the anticipated intermediate complexes [RuX($Me_2HCN \overset{\cdots}{-} CH \overset{\cdots}{-} NCHMe_2$)(CO)($PPh_3$)$_2$], were unsuccessful. Reactions run for shorter periods or under milder conditions gave mixtures in which the dominant species were either the precursor [RuH(X)(CO)(PPh_3)$_3$] or the dehydrogenated product described above. Attempts to dehydrogenate the second isopropyl group by use of more vigorous and/or prolonged reaction conditions were also unsuccessful.

The ruthenium dihydride [RuH_2(CO)(PPh_3)$_3$] reacted with di-isopropylcarbodi-imide to give dark solutions from which no characterisable products could be isolated. However, the corresponding osmium complex [OsH_2(CO)(PPh_3)$_3$] reacted cleanly in boiling toluene over a period of 6 h to yield the *N,N'*-di-isopropylformamidinato-complex [OsH($Me_2HCN \overset{\cdots}{-} CH \overset{\cdots}{-} NCHMe_2$)(CO)($PPh_3$)$_2$]

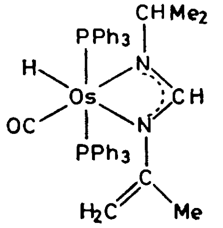


(III)

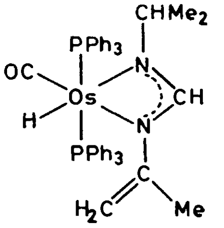
which on further heating (*ca.* 6 h, boiling toluene) dehydrogenates to afford two isomeric forms of the *N*-isopropenyl-*N'*-isopropylformamidinato-derivative [OsH{CH_2=C(Me)N $\overset{\cdots}{-}$ CH $\overset{\cdots}{-}$ $NCHMe_2$}(CO)(PPh_3)$_2$]. The latter product may also be obtained directly from [OsH_2(CO)(PPh_3)$_3$] using the same reaction time but a solvent of higher boiling point (ethylbenzene).

The air-stable yellow, crystalline *N,N'*-di-isopropylformamidinato-complex can be assigned stereochemistry (III) on the basis of its proton and ^{31}P n.m.r. spectra [OsH, τ 23.97(t); ^2J(PH) = 18.5 Hz; CH*Me*$_2$, 9.57(d) and 9.87(d), ^3J(HH') = 6.5 Hz; C*H*Me$_2$, 8.00(septet) and 7.49(septet); N $\overset{\cdots}{-}$ CH $\overset{\cdots}{-}$ N, 1.51 (peak with symmetrical shoulders); ^{31}P, δ = 17 p.p.m.(s)]. The failure of the low-field proton signal at τ 1.51 to resolve into its component parts probably reflects the presence of coupling between the formamidinato-proton (N $\overset{\cdots}{-}$ CH $\overset{\cdots}{-}$ N) and the hydride ligand [^4J(HH')] as well as the coupling to the two ^{31}P nuclei [^4J(PH)].

The dehydrogenated product [OsH{CH_2=C(Me)N $\overset{\cdots}{-}$ CH $\overset{\cdots}{-}$ $NCHMe_2$}(CO)(PPh_3)$_2$], obtained from [OsH($Me_2HCN \overset{\cdots}{-} CH \overset{\cdots}{-} NCHMe_2$)(CO)($PPh_3$)$_2$] on further heating, exists in two isomeric forms (IVa) and (IVb).



(IVa)



(IVb)

The major isomer (*ca.* 80% based on n.m.r. integrals) displays a singlet (δ = 20.3 p.p.m.) in its ^{31}P n.m.r. spectrum (proton decoupled) and a triplet [OsH, τ 25.03, ^2J(PH) = 18 Hz] in its high-field proton spectrum. Proton n.m.r. data for the *N*-isopropenyl-*N'*-isopropylformamidinate ligand [CH_2=C(Me)N $\overset{\cdots}{-}$ CH $\overset{\cdots}{-}$ $NCHMe_2$] are: *Me*$_2$CH, τ 9.39(d), ^3J(HH') = 6.5 Hz, relative intensity 6; C(*Me*)=CH_2, 9.00(s), 3; C(Me)=C*H*$_2$, 7.02(s), 1 and 6.43(s), 1; N $\overset{\cdots}{-}$ CH $\overset{\cdots}{-}$ N, 1.95 (peak with symmetrical shoulders), 1. The corresponding data for the minor isomer (*ca.* 20% of recovered product) are: ^{31}P, δ 19.7(s) p.p.m.; ^1H, OsH, τ 23.35(t), ^2J(PH) = 20 Hz; *Me*$_2$CH, 9.70(d), ^3J(HH') = 6.5 Hz, relative intensity 6; C(*Me*)=CH_2, 8.85(s), 3; the remaining signals are not adequately resolved.

We thank the S.R.C. for supporting this work. One of us (A. D. H.) thanks California State College, San Bernardino, for sabbatical leave.

[0/1670 *Received, 3rd November,* 1980]

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