Complexes of the Platinum Metals. Part 42.[1] Binuclear N,O-Succinimido-bridged Ruthenium(II) Derivatives: Crystal Structure of $[Ru_2(\mu\text{-}Cl)(\mu\text{-}H)(\mu\text{-}C_4H_4NO_2)_2(PPh_3)_4]\cdot 2CH_2Cl_2\cdot 0.5H_2O$†

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Succinimide reacts with $[RuHCl(PPh_3)_3]$ in refluxing toluene to afford a maroon air-stable complex **1**. A crystal of **1** obtained from CH_2Cl_2–MeOH solution has been shown by X-ray diffraction methods to possess the succinimido-bridged structure $[Ru_2(\mu\text{-}Cl)(\mu\text{-}H)(\mu\text{-}C_4H_4NO_2)_2(PPh_3)_4]\cdot 2CH_2Cl_2\cdot 0.5H_2O$. Succinimide also reacts with $[RuCl_2(PPh_3)_3]$ in refluxing toluene in the presence of an excess of NEt_3 to form a closely related monocarbonyl derivative $[Ru_2(\mu\text{-}Cl)(\mu\text{-}H)(\mu\text{-}C_4H_4NO_2)_2(CO)(PPh_3)_3]$ **2** as orange needles. Carbonylation of **2** in refluxing toluene affords the dicarbonyl $[Ru_2(\mu\text{-}Cl)(\mu\text{-}H)(\mu\text{-}C_4H_4NO_2)_2(CO)_2(PPh_3)_2]$ **3** as pale yellow crystals. The products **1**–**3** have been characterised by infrared and ^1H and $^{31}P\text{-}\{^1H\}$ NMR spectroscopies.

Complexes containing co-ordinated succinimide anions were first reported by Ley and Werner[2] in 1907 and numerous examples are now known.[3–6] The planar succinimide anion has a highly delocalised electronic structure and is theoretically capable of binding to transition-metal ions through the endocyclic nitrogen atom and/or one or both of the exocyclic oxygen atoms.[7] However, in virtually all known instances the anion functions as a monodentate N- or O-donor ligand, with N-co-ordination being by far the most commonly encountered bonding mode. Complexes in which the succinimide anion functions as a bi- or tri-dentate ligand co-ordinated in chelate or bridging mode are virtually unknown. To the best of our knowledge the only examples of succinimido-bridged transition-metal complexes are the two cyclometallated palladium(II) species $[\{Pd(o\text{-}C_6H_4CH_2NMe_2)(\mu\text{-}NCOC_2H_4CO)\}_2]$ and $[\{Pd(o\text{-}C_6H_4CH{=}NPh)(\mu\text{-}NCOC_2H_4CO)\}_2]$.[4]

Having previously observed that amides react with certain ruthenium complexes to generate products in which the amido anions $RC(O)NH^-$ function as bridging rather than chelating ligands[8] we have undertaken a parallel study of the reactions of succinimide with the same ruthenium precursors. In the course of this work we have isolated and characterised three binuclear succinimido-bridged diruthenium(II/II) complexes which form the subject of the present paper.

Experimental

Ruthenium complexes were prepared by literature methods,[9,10] succinimide was used as purchased. Reagent grade organic solvents were dried over molecular sieves. Reactions were performed under nitrogen but products were worked up in open flasks. Elemental analyses were performed by the microanalytical service at University College, London. Melting points were taken in sealed tubes under nitrogen.

$[Ru_2(\mu\text{-}Cl)(\mu\text{-}H)(\mu\text{-}C_4H_4NO_2)_2(PPh_3)_4]\cdot 2CH_2Cl_2\cdot 0.5H_2O$ **1**.—Chlorohydridotris(triphenylphosphine)ruthenium (0.8 g, 0.86 mmol) and succinimide (0.39 g, 3.93 mmol) were added to degassed toluene (40 cm^3) and the mixture heated under reflux for *ca.* 6 h. The dark reddish brown solution was cooled to ambient temperature, filtered and then concentrated under reduced pressure to leave an oil. Crystallisation from dichloromethane–methanol afforded the product as maroon crystals (0.45 g, 63%), m.p. 241–243 °C (Found: C, 59.25; H, 4.05; N, 1.65. Calc. for $C_{80}H_{69}ClN_2O_4P_4Ru_2\cdot 2CH_2Cl_2\cdot 0.5H_2O$: C, 58.3; H, 4.45; N, 1.70%). NMR: δ_H(360 MHz, solvent $CDCl_3$, standard $SiMe_4$), −11.43 [t of t, $^2J(HP)_{trans}$ 49, $^2J(HP)_{cis}$ 17 Hz]; δ_P(101.26 MHz, solvent $CDCl_3$, standard external 85% H_3PO_4) 57.33 and 28.36 (unresolved multiplets, relative intensities 1:1).

$[Ru_2(\mu\text{-}Cl)(\mu\text{-}H)(\mu\text{-}C_4H_4NO_2)_2(CO)(PPh_3)_3]$ **2**.—Dichlorotris(triphenylphosphine)ruthenium (1 g, 1.04 mmol), succinimide (0.46 g, 4.64 mmol) and triethylamine (4 cm^3) were heated together under reflux in toluene (50 cm^3) for *ca.* 10 h. During the course of the reaction further portions (4 cm^3) of triethylamine were added at 2.5 h intervals. The dark reddish brown solution was cooled to ambient temperature and then concentrated to leave an oil. This was dissolved in dichloromethane (40 cm^3) and on addition of methanol (3 cm^3) a small amount (0.04 g) of unidentified yellow powder formed. This was filtered off and the filtrate was kept for a few days whereupon the major product slowly deposited as orange microcrystals. Recrystallisation from dichloromethane–methanol afforded the product as orange needles (0.43 g, 66%), m.p. 258–260 °C (decomp.) (Found: C, 59.85; H, 3.85; N, 2.20. Calc. for $C_{63}H_{54}ClN_2O_5P_3Ru_2$: C, 60.55; H, 4.3; N, 2.25%). IR(Nujol): ν(CO) 1944 cm^{-1}. NMR: δ_H(360 MHz, solvent $C_6D_5CD_3$, standard $SiMe_4$), −10.22 [d of d of d, $^2J(HP)_{trans}$ *ca.* 48, $^2J(HP')_{trans}$ *ca.* 49, $^2J(HP)_{cis}$ *ca.* 24 Hz]; δ_P(101.26 MHz, solvent $C_6D_5CD_3$, standard external 85% H_3PO_4), 60.86 and 31.90 (d of d and m, relative intensities 1:2).

$[Ru_2(\mu\text{-}Cl)(\mu\text{-}H)(\mu\text{-}C_4H_4NO_2)_2(CO)_2(PPh_3)_2]$ **3**.—Compound **2** (0.1 g, 0.08 mmol) was dissolved in toluene (30 cm^3) and the orange solution was heated under reflux in an atmosphere of carbon monoxide for 20 min. The pale yellow solution was cooled, filtered and evaporated to dryness to give a

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† *Supplementary data available:* see Instructions for Authors, *J. Chem. Soc., Dalton Trans.*, 1993, Issue 1, pp. xxiii–xxviii.

Table 1 Fractional atomic coordinates (× 10^4) for compound **1**

Atom	*x*	*y*	*z*
Ru(1)	2 235.4(4)	8 299.0(3)	2 293.6(2)
Ru(2)	3 353.1(4)	7 535.6(3)	3 067.6(2)
Cl(1)	2 403(1)	8 463(1)	3 185(1)
P(1)	2 237(1)	8 102(1)	1 466(1)
P(2)	1 194(1)	9 218(1)	2 255(1)
P(3)	4 111(1)	6 612(1)	2 890(1)
P(4)	3 751(1)	7 564(1)	3 970(1)
O(1)	2 094(4)	6 976(3)	2 983(2)
C(1)	1 357(6)	7 115(4)	2 676(3)
C(2)	477(6)	6 697(5)	2 653(4)
C(3)	−256(6)	7 020(5)	2 250(4)
C(4)	266(5)	7 590(4)	2 068(3)
O(2)	−81(4)	7 979(3)	1 744(2)
N(1)	1 204(4)	7 614(3)	2 342(2)
O(3)	3 491(3)	8 867(2)	2 389(2)
C(5)	4 230(5)	8 735(4)	2 710(3)
C(6)	5 065(6)	9 191(4)	2 774(3)
C(7)	5 818(6)	8 839(4)	3 161(3)
C(8)	5 345(5)	8 212(4)	3 312(3)
O(4)	5 691(4)	7 818(3)	3 604(2)
N(2)	4 400(4)	8 213(2)	3 017(2)
C(111)	4 141(4)	8 091(2)	1 483(2)
C(112)	4 948(4)	7 915(2)	1 319(2)
C(113)	4 922(4)	7 391(2)	984(2)
C(114)	4 090(4)	7 044(2)	812(2)
C(115)	3 283(4)	7 220(2)	976(2)
C(116)	3 308(4)	7 743(2)	1 312(2)
C(121)	2 048(4)	8 692(2)	503(2)
C(122)	2 115(4)	9 215(2)	180(2)
C(123)	2 365(4)	9 841(2)	377(2)
C(124)	2 547(4)	9 944(2)	896(2)
C(125)	2 480(4)	9 421(2)	1 219(2)
C(126)	2 230(4)	8 795(2)	1 023(2)
C(131)	548(4)	7 747(2)	771(2)
C(132)	−166(4)	7 303(2)	573(2)
C(133)	−116(4)	6 654(2)	747(2)
C(134)	648(4)	6 450(2)	1 118(2)
C(135)	1 362(4)	6 894(2)	1 316(2)
C(136)	1 312(4)	7 543(2)	1 142(2)
C(211)	275(4)	9 379(3)	1 256(2)
C(212)	−71(4)	9 727(3)	812(2)
C(213)	45(4)	10 409(3)	799(2)
C(214)	506(4)	10 743(3)	1 231(2)
C(215)	853(4)	10 395(3)	1 675(2)
C(216)	737(4)	9 713(2)	1 688(2)
C(221)	−753(4)	9 357(3)	2 217(2)
C(222)	−1 581(4)	9 229(3)	2 376(2)
C(223)	−1 568(4)	8 819(3)	2 789(2)
C(224)	−727(4)	8 536(3)	3 043(2)
C(225)	102(4)	8 664(3)	2 884(2)
C(226)	88(4)	9 074(3)	2 471(2)
C(231)	2 620(4)	10 107(3)	2 648(2)
C(232)	3 089(4)	10 576(3)	2 986(2)
C(233)	2 691(4)	10 802(3)	3 377(2)
C(234)	1 825(4)	10 559(3)	3 429(2)
C(235)	1 355(4)	10 090(3)	3 091(2)
C(236)	1 753(4)	9 864(3)	2 701(2)
C(311)	6 093(4)	6 434(3)	3 182(2)
C(312)	7 007(4)	6 591(3)	3 149(2)
C(313)	7 161(4)	7 033(3)	2 783(2)
C(314)	6 402(4)	7 319(3)	2 450(2)
C(315)	5 488(4)	7 162(3)	2 484(2)
C(316)	5 333(4)	6 719(3)	2 850(2)
C(321)	4 064(4)	6 083(3)	1 903(2)
C(322)	3 680(4)	5 684(3)	1 492(2)
C(323)	2 825(4)	5 367(3)	1 471(2)
C(324)	2 354(4)	5 448(3)	1 861(2)
C(325)	2 738(4)	5 847(3)	2 273(2)
C(326)	3 593(4)	6 165(3)	2 294(2)
C(331)	3 409(4)	5 824(2)	3 569(2)
C(332)	3 322(4)	5 246(2)	3 833(2)
C(333)	3 907(4)	4 710(2)	3 804(2)
C(334)	4 578(4)	4 753(2)	3 511(2)
C(335)	4 665(4)	5 331(2)	3 247(2)
C(336)	4 080(4)	5 866(2)	3 276(2)
C(411)	5 191(4)	8 199(3)	4 675(2)
C(412)	5 727(4)	8 738(3)	4 887(2)
C(413)	5 524(4)	9 366(3)	4 683(2)
C(414)	4 784(4)	9 454(3)	4 267(2)
C(415)	4 247(4)	8 915(3)	4 055(2)
C(416)	4 450(4)	8 287(3)	4 260(2)
C(421)	2 599(4)	8 187(3)	4 549(2)
C(422)	1 771(4)	8 262(3)	4 719(2)
C(423)	1 031(4)	7 822(3)	4 563(2)
C(424)	1 120(4)	7 306(3)	4 237(2)
C(425)	1 948(4)	7 231(3)	4 068(2)
C(426)	2 688(4)	7 671(3)	4 224(2)
C(431)	4 022(3)	6 617(3)	4 760(2)
C(432)	4 507(3)	6 099(3)	5 039(2)
C(433)	5 328(3)	5 859(3)	4 922(2)
C(434)	5 665(3)	6 137(3)	4 527(2)
C(435)	5 180(3)	6 655(3)	4 248(2)
C(436)	4 358(3)	6 895(3)	4 365(2)

pale yellow solid. This was washed several times with methanol and then dried *in vacuo* (0.07 g, 87%), m.p. 278–282 °C (Found: C, 53.65; H, 3.9; N, 2.55. Calc. for $C_{46}H_{39}ClN_2O_6P_2Ru$: C, 54.4; H, 3.85; N, 2.75%). IR(Nujol): ν(CO) 1980 and 1955 cm^{-1}. NMR: δ_H(360 MHz, solvent $C_6D_5CD_3$, standard $SiMe_4$), −7.79 [d of d, $^2J(HP)_{trans}$ 54, $^2J(PH)_{cis}$ 18 Hz]; δ_P 60.22 and 32.29 [both doublets, $^4J(PP')$ 6 Hz].

Crystallography.—Crystal data. $C_{80}H_{69}ClN_2O_4P_4Ru_2\cdot 2CH_2Cl_2\cdot 0.5H_2O$, $M = 1662.81$, monoclinic, space group $P2_1/c$, $a = 14.611(4)$, $b = 20.282(4)$, $c = 27.282(4)$ Å, $\beta = 102.62(1)°$, $U = 7865.7$ Å^3, $Z = 4$, $D_c = 1.404$ g cm^{-3}, $F(000) = 3396$, μ(Mo-Kα) = 6.765 cm^{-1}.

Data collection. Unit-cell dimensions and intensity data were obtained at 295 K using an Enraf-Nonius area detector diffractometer and graphite monochromated Mo-Kα radiation, following previously described procedures [detector to crystal distance = 40 mm, $2\theta = 18°$].[11] A total of 30 580 reflections were measured, of which 13 212 were unique and 7953 satisfied the condition $F_o > 3[F_o]$.

Structure solution. The structure was solved *via* the heavy-atom method using SHELX-S.[12] Absorption corrections were applied at the isotropic refinement stage using the DIFABS procedure,[13a] adapted for FAST geometry.[13b] The phenyl carbons were refined assuming regular hexagons with C–C 1.395 Å and C–C–C 120.0°. All hydrogen atoms were allowed to ride on their parent carbon atoms in the calculated positions (C–H 0.96 Å). The position of the bridging hydride ligand could not be detected crystallographically.

The solvent molecules occupy isolated positions in the lattice, and do not participate in hydrogen-bonding interactions. The final *R* and *R'* values are 0.057 and 0.049 respectively. Final fractional atomic coordinates are given in Table 1. Selected bond lengths and angles are given in Table 2.

All calculations were made on a T800 transputer hosted by an IBM/AT personal computer.

Additional material available from the Cambridge Crystallographic Data Centre comprises H-atom coordinates, thermal parameters and remaining bond lengths and angles.

Results and Discussion

The reaction of $[RuHCl(PPh_3)_3]$ with an excess of succinimide in boiling degassed toluene over a period of 6 h generates a dark

Table 2 Selected bond lengths (Å) and angles (°) for compound **1**

Ru(2)–Ru(1)	2.827(4)	C(416)–P(4)	1.862(7)	Cl(1)–Ru(1)	2.406(4)	C(336)–P(3)	1.846(7)
P(1)–Ru(1)	2.286(4)	C(436)–P(4)	1.835(7)	P(2)–Ru(1)	2.394(4)	C(426)–P(4)	1.845(9)
N(1)–Ru(1)	2.076(8)	C(2)–C(1)	1.531(15)	O(3)–Ru(1)	2.133(7)	C(1)–O(1)	1.242(10)
Cl(1)–Ru(2)	2.400(4)	C(3)–C(2)	1.504(15)	P(3)–Ru(2)	2.281(4)	N(1)–C(1)	1.345(11)
P(4)–Ru(2)	2.395(4)	O(2)–C(4)	1.209(11)	O(1)–Ru(2)	2.131(7)	C(4)–C(3)	1.526(15)
N(2)–Ru(2)	2.082(8)	C(5)–O(3)	1.259(9)	C(116)–P(1)	1.856(8)	N(1)–C(4)	1.410(10)
C(126)–P(1)	1.850(7)	N(2)–C(5)	1.339(10)	C(136)–P(1)	1.836(7)	C(6)–C(5)	1.509(13)
C(216)–P(2)	1.839(8)	C(8)–C(7)	1.546(14)	C(226)–P(2)	1.859(9)	C(7)–C(6)	1.523(13)
C(236)–P(2)	1.850(8)	N(2)–C(8)	1.439(9)	C(316)–P(3)	1.827(9)	O(4)–C(8)	1.161(10)
C(326)–P(3)	1.869(8)						
Cl(1)–Ru(1)–Ru(2)	53.9(1)	P(1)–Ru(1)–Ru(2)	120.9(2)	C(236)–P(2)–Ru(1)	109.7(3)	C(236)–P(2)–C(216)	101.0(4)
P(1)–Ru(1)–Cl(1)	173.9(1)	P(1)–Ru(1)–Ru(2)	135.9(1)	C(236)–P(2)–C(226)	101.1(4)	C(316)–P(3)–Ru(2)	116.2(3)
P(2)–Ru(1)–Cl(1)	82.0(2)	P(2)–Ru(1)–P(1)	103.3(2)	C(326)–P(3)–Ru(2)	117.4(3)	C(326)–P(3)–C(316)	102.7(4)
N(1)–Ru(1)–Ru(2)	84.0(2)	N(1)–Ru(1)–Cl(1)	86.8(3)	C(336)–P(3)–Ru(2)	118.7(3)	C(336)–P(3)–C(316)	106.1(3)
N(1)–Ru(1)–P(1)	95.9(3)	N(1)–Ru(1)–P(2)	93.5(3)	C(336)–P(3)–C(326)	92.3(4)	C(416)–P(4)–Ru(2)	116.2(3)
O(3)–Ru(1)–Ru(2)	82.2(2)	O(3)–Ru(1)–Cl(1)	84.7(2)	C(426)–P(4)–Ru(2)	110.5(3)	C(426)–P(4)–C(416)	100.2(4)
O(3)–Ru(1)–P(1)	91.6(2)	O(3)–Ru(1)–P(2)	96.1(2)	C(436)–P(4)–Ru(2)	124.1(3)	C(436)–P(4)–C(416)	100.6(3)
O(3)–Ru(1)–N(1)	166.2(2)	Cl(1)–Ru(2)–Ru(1)	54.1(2)	C(436)–P(4)–C(426)	101.9(4)	C(1)–O(1)–Ru(2)	123.6(6)
P(3)–Ru(2)–Ru(1)	121.4(2)	P(3)–Ru(2)–Cl(1)	173.4(1)	C(2)–C(1)–O(1)	119.9(9)	N(1)–C(1)–O(1)	128.1(8)
P(4)–Ru(2)–Ru(1)	136.1(1)	P(4)–Ru(2)–Cl(1)	82.1(2)	N(1)–C(1)–C(2)	112.0(8)	C(3)–C(2)–C(1)	104.2(9)
P(4)–Ru(2)–P(3)	102.5(2)	O(1)–Ru(2)–Ru(1)	82.3(2)	C(4)–C(3)–C(2)	104.0(8)	O(2)–C(4)–C(3)	124.9(8)
O(1)–Ru(2)–Cl(1)	85.5(2)	O(1)–Ru(2)–P(3)	89.2(3)	N(1)–C(4)–C(3)	110.5(8)	N(1)–C(4)–O(2)	124.6(9)
O(1)–Ru(2)–P(4)	97.7(3)	N(2)–Ru(2)–Ru(1)	84.4(2)	C(1)–N(1)–Ru(1)	122.0(6)	C(4)–N(1)–Ru(1)	128.6(6)
N(2)–Ru(2)–Cl(1)	86.9(3)	N(2)–Ru(2)–P(3)	97.5(3)	C(4)–N(1)–C(1)	109.3(7)	C(5)–O(3)–Ru(1)	123.7(6)
N(2)–Ru(2)–P(4)	92.0(3)	N(2)–Ru(2)–O(1)	166.7(2)	C(6)–C(5)–O(3)	120.0(7)	N(2)–C(5)–O(3)	127.9(7)
Ru(2)–Cl(1)–Ru(1)	72.1(2)	C(116)–P(1)–Ru(1)	118.3(3)	N(2)–C(5)–C(6)	112.1(7)	C(7)–C(6)–C(5)	103.8(8)
C(126)–P(1)–Ru(1)	120.6(3)	C(126)–P(1)–C(116)	92.0(3)	C(8)–C(7)–C(6)	105.7(7)	O(4)–C(8)–C(7)	126.6(8)
C(136)–P(1)–Ru(1)	114.9(3)	C(136)–P(1)–C(116)	102.6(3)	N(2)–C(8)–C(7)	106.3(7)	N(2)–C(8)–O(4)	127.1(8)
C(136)–P(1)–C(126)	104.9(3)	C(216)–P(2)–Ru(1)	125.0(3)	C(5)–N(2)–Ru(2)	121.7(5)	C(8)–N(2)–Ru(2)	126.3(6)
C(226)–P(2)–Ru(1)	116.9(3)	C(226)–P(2)–C(216)	99.8(4)	C(8)–N(2)–C(5)	111.9(7)		

Table 3 Comparison of salient bond lengths (Å) for [Ru_2(μ-Cl)(μ-H){μ-NHC(O)CF_3}$_2$(PPh_3)$_4$] **4** and [Ru_2(μ-Cl)(μ-H)(μ-$C_4H_4NO_2$)$_2$(PPh_3)$_4$] **1**

Complex	**4**	**1**
Ru–Ru	2.811(4)	2.827(4)
Ru–N	2.061(8)	2.082(8)
	2.041(8)	2.076(8)
Ru–O	2.153(7)	2.133(7)
	2.162(7)	2.131(7)
Ru–Cl	2.427(4)	2.406(4)
	2.429(4)	2.400(4)
Ru–P(*trans* to H)	2.339(4)	2.395(4)
	2.336(4)	2.394(4)
Ru–P(*trans* to Cl)	2.281(4)	2.281(4)
	2.298(4)	2.286(4)

reddish brown solution which, on evaporation, followed by crystallisation (CH_2Cl_2–MeOH) of the residual oil, affords air-stable maroon crystals. The high-field proton NMR spectrum reveals the presence of a single hydride (δ_H −11.43) coupled to two pairs of equivalent phosphorus nuclei (triplet of triplets). The ^{31}P-{^{1}H} NMR spectrum displays two sharp partially resolved multiplets at δ 57.33 and 28.36 with relative intensities of 1:1. These data are not consistent with the presence of a mononuclear octahedral complex but can be interpreted in terms of a binuclear hydride-bridged species **1**. However, the elemental analysis required the presence of dichloromethane within the crystal lattice in order to fit the proposed stoichiometry. Therefore to confirm the succinimido-bridged binuclear structure and to quantify the amount of CH_2Cl_2 present in the crystals an X-ray diffraction study was undertaken. The molecular structure of **1** is given in Fig. 1, selected bond lengths and angles are listed in Table 2. The complex possesses the anticipated binuclear ruthenium(II) structure with four bridging groups: chloride, hydride and two N,O-bonded succinimide anions arranged in head-to-tail configuration. The remaining four co-ordination sites, two per ruthenium, are occupied by four triphenylphosphine ligands. The X-ray crystal structure determination also reveals that each dimer is associated with two molecules of CH_2Cl_2 and half a molecule of water, and this allows the calculated elemental (C,H,N) analysis to be reconciled with the experimental figures.

The structure of **1** closely parallels that recently reported from our laboratories[8] for the related trifluoroacetamido derivative [Ru_2(μ-H)(μ-Cl){μ-NHC(O)CF_3}$_2$(PPh_3)$_4$] **4** and displays bond length and angle data very similar to those found for the latter complex (Table 3). The most significant difference is found in the Ru–P bond lengths, those *trans* to chloride are virtually identical in both complexes, and are unusually short (mean 2.286 Å), whereas those *trans* to hydride increase from 2.336(4)/2.339(4) Å in the trifluoroacetamide complex to 2.394(4)/2.395(4) Å in the succinimide. Inspection of bond lengths for the bridging succinimide ligands (Fig. 2) reveals that direct participation in the bridge leads to a marked lengthening of the C=O bonds and a concomitant shortening of the C–N distances, thus indicating a significant degree of charge delocalisation within the co-ordinated O┈C┈N groups.

Finally, it is interesting to note that the mean C=O and C–N bond lengths for free succinimide (1.227 and 1.385 Å respectively)[7] fall almost midway between those found for the corresponding co-ordinated and non-co-ordinated bonds in complex **1**.

The sequence of reactions leading to the formation of **1** has not been determined but may involve steps similar to those previously proposed[8] for the formation of the related trifluoroacetamido complex [Ru_2(μ-Cl)(μ-H){μ-NHC(O)CF_3}$_2$(PPh_3)$_4$].

Succinimide also reacts with [$RuCl_2(PPh_3)_3$] in boiling degassed toluene over a period of *ca.* 10 h in the presence of excess triethylamine to generate a red-brown solution which on evaporation and crystallisation from CH_2Cl_2–MeOH afforded as the major product an air-stable orange crystalline material **2**. This was identified on the basis of analytical and spectroscopic data as a monocarbonylated derivative of **1**. The hydride and

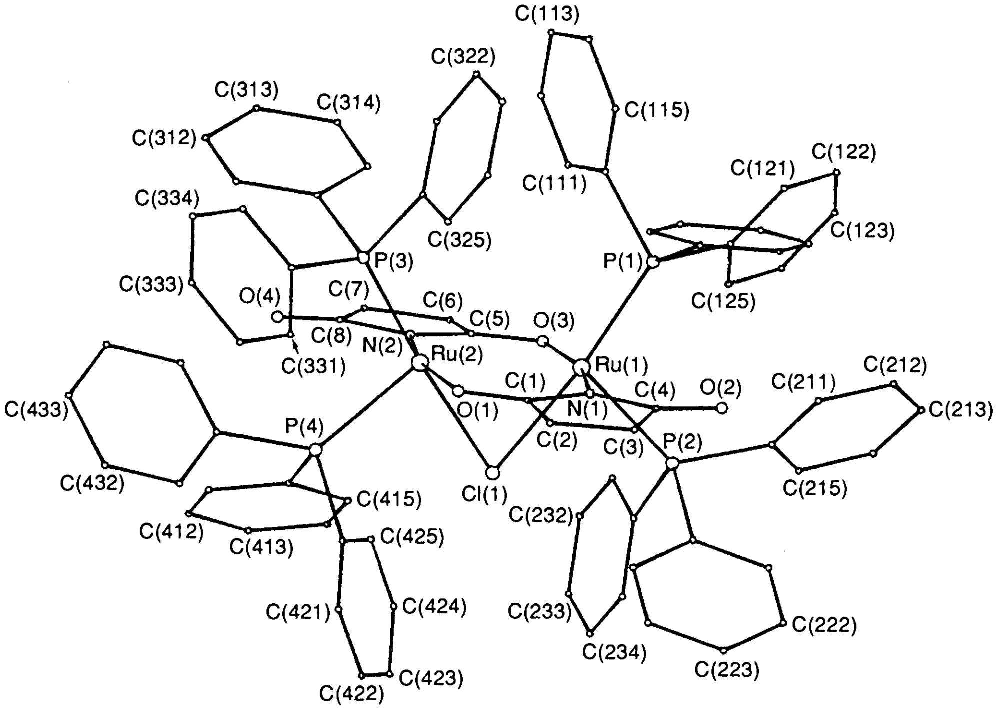

Fig. 1 Molecular structure of complex **1**

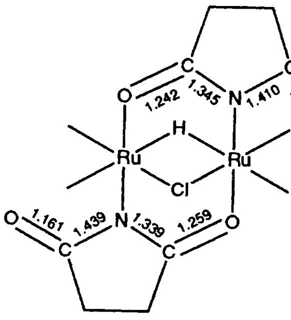

Fig. 2 Selected molecular dimensions (Å) of complex **1**

carbonyl ligands, which are present in the crude product prior to the recrystallisation step, are thought to arise from traces of alcohol in the solvent. The high-field proton NMR spectrum consists of a doublet of doublets in which the two central doublets coincide almost exactly thereby indicating that two of the three $^2J(\mathrm{PH})$ couplings involved are virtually identical. The $^{31}\mathrm{P}$-$\{^1\mathrm{H}\}$ NMR spectrum consists of two narrow partially resolved multiplets, of relative intensities 1:2 at δ 60.86 and 31.90 respectively. The signal of relative intensity two centred at δ 31.90 is attributed to the two phosphorus nuclei *trans* to the bridging hydride ligand, which must therefore possess almost identical chemical shifts as well as virtually identical $^2J(\mathrm{PH})$ coupling constants. The signal at δ 60.86 with a relative intensity of one is, by a process of elimination, assigned to the unique phosphorus *cis* to the bridging hydride.

Confirmation of these NMR assignments was provided by the spectrum of the product obtained on carbonylation of **2** in boiling toluene. The yellow carbonylated solution gave a yellow solid which was shown by elemental analysis and spectroscopic methods to be the dicarbonyl $[\mathrm{Ru_2(\mu\text{-}Cl)(\mu\text{-}H)(\mu\text{-}C_4H_4NO_2)_2(CO)_2(PPh_3)_2}]$ **3**. The high-field proton NMR spectrum consists of a doublet of doublets with couplings $^2J(\mathrm{PH})$ of 54 and 18 Hz indicating phosphorus nuclei *trans* and *cis* respectively to the hydride ligand. The $^{31}\mathrm{P}$ NMR spectrum shows doublets at δ 60.22 and 32.29 with $^4J(\mathrm{PP'})$ equal to ≈6 Hz, consistent with triphenylphosphine *trans* to chloride and hydride respectively.

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