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# Reactions of $Ru(CO)Cl(RC{=}CHR')(PPh_3)_2$ with carboxylates. The crystal structure of $Ru(O_2CMe)(CO)(HC{=}CHPh)(PPh_3)_2$

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## Abstract

The 16-electron complexes $Ru(CO)Cl(RC{=}CHR')(PPh_3)_2$ ($R = H$, $R' = CMe_3$, $SiMe_3$, Ph, $CO_2Me$ and $CO_2Et$; $R = R' = H$, Me, Ph and $CO_2Me$ react readily with carboxylates to give the saturated complexes $Ru(O_2CR'')(CO)(RC{=}CHR')(PPh_3)_2$ ($R'' = Me$ and Et). The crystal structure of the complex $Ru(O_2CMe)(CO)(HC{=}CHPh)(PPh_3)_2$ has been determined, and reveals an octahedral geometry for the ruthenium atom bearing two phosphines in a *trans*-disposition and $\eta^2$-acetate, CO and $\eta^1$-phenylethenyl ligands.

## Introduction

Recently the reactivity of unsaturated complexes of ruthenium-containing carbonyl and phosphine ligands has been investigated by several groups [1,2,3]. The results indicate that ready addition of small molecules to the 16-electron complexes leads to saturated species. In some cases, the ligands react with small molecules. This is the case for the complexes $Ru(CO)Cl(HC{=}CHR)(PPh_3)_2$, which add $CS_2$ to give an alkenedithiocarboxylate ligand [4]. Such compounds normally react with 3,5-dimethylpyrazole to give the saturated complexes $Ru(CO)Cl(HC{=}CHR)(PPh_3)_2(Me_2HPz)$, but in $Et_2O/EtOH$ solution the alkenyl ligands are converted into alkynyl ligands [5].

The carboxylate complexes of ruthenium have been known for several years, and have been investigated because of their extensive chemistry and their catalytic activity in some important reactions [6,7]. In particular, use of acetate complexes in catalytic hydrogenation and hydroformylation of alkenes has been reported recently [8].

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In this paper we describe the synthesis and characterization of ruthenium complexes containing alkenyl, carbonyl, carboxylato and triphenylphosphine ligands formed from unsaturated alkenyl complexes of ruthenium by treatment with methanolic solutions of sodium acetate or propionate.

## Results and discussion

Treatment of $CH_2Cl_2$ solutions of the 16-electron complexes Ru(CO)Cl(RC=CHR′)($PPh_3$) where R = H, R′ = $CMe_3$, $SiMe_3$, Ph, $CO_2Me$ and $CO_2Et$; R = R′ = H, Me, Ph and $CO_2Me$ with methanolic solutions of sodium acetate or sodium propionate to give the crystalline compounds Ru($O_2CR''$)(CO)(RC=CHR′)$(PPh_3)_2$ with R″ = Me or Et. The carboxylate products are quite stable to air and soluble in $CH_2Cl_2$ but insoluble in alcohols, allowing slow crystallization from $CH_2Cl_2$/MeOH mixtures. Single crystals of the complex Ru($O_2CMe$)(CO)(HC=CHPh)$(PPh_3)_2$ suitable for X-ray diffraction studies were obtained in this way. The IR spectra of the new complexes show typical absorptions of dihapto-carboxylate ligands [9,10]; $\nu(OCO)_{asym}$: 1516–1553 $cm^{-1}$ and $\nu(OCO)_{sym}$: 1452–1470 $cm^{-1}$. The $\nu(C{\equiv}O)$ band appears at 1910–1925 $cm^{-1}$, the exact position depending on the substituents on the alkenyl ligands as well as those in the initial unsaturated complex [5,11,12]. The $\nu(C{=}C)$ band for all the complexes with medium intensity appears near 1555 $cm^{-1}$.

The $^1H$ NMR spectra of all the complexes show the signals of the carboxylate and alkenyl ligands. In comparison with the position of the signals of the alkenyl protons of the Ru(CO)Cl(RC=CHR′)$(PPh_3)_2$ complexes there is a general shift upfield for the =CHR′ protons and downfield for the CH= protons observed. This effect is attributed to the increase in electronic density at the ruthenium centre.

The positions of the $\nu(C{\equiv}O)$ are also affected, and lower frequencies are observed for the saturated carboxylato complexes. The geometry of the alkenyl ligands is maintained after replacement of Cl by carboxylato groups; thus, two doublets are observed in the complexes containing monosubstituted alkenyl groups, with $J$(HH) ≈ 14–18 Hz, characteristic of *trans*-alkenyl protons.

From the spectroscopic data we conclude that the replacement of Cl by carboxylato ligands results in saturation of the complex without modification of the alkenyl groups. There is only an electronic effect on the $\nu(C{\equiv}O)$ band positions and on the $\delta$ values for the alkenyl protons. The new structures involve a six-coordinate ruthenium atom with $\eta^2$-$O_2CR''$, C≡O and alkenyl ligands with two phosphines probably in a *trans* disposition. In order to establish their exact nature, the structure of Ru($O_2CMe$)(CO)(HCCHPh)$(PPh_3)_2$ was determined.

## The structure of Ru($O_2CMe$)(CO)(HCCHPh)$(PPh_2)_3$ (4)

The crystal of complex **4** consists of individual molecules of Ru($O_2CMe$)(CO)(HCCHPh)$(PPh_3)_2$ linked by Van der Waals forces. Table 1 lists selected bond

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results in a small O2–Ru–O3 angle of 55.9(4)°. This compression causes a modification of the remaining equatorial angles around the ruthenium atom (C1–Ru–O2: 116.4(7)°, C1–Ru–C4: 121(7)° and C4–Ru–O3: 66.2(4)°). The O2–C2 and O3–C2 bonds have lengths of 1.322(10) and 1.271(9) Å, showing that there is strong

Table 1

Relevant bond lengths (Å) and angles (°) for $Ru(O_2CMe)(CO)(HCCPh)(PPh_3)_2$, with e.s.d.'s in parentheses.

| | | | |
|---|---|---|---|
| Ru-P1 | 2.344(9) | P2-C201 | 1.834(8) |
| Ru-P2 | 2.400(9) | P2-C211 | 1.783(10) |
| Ru-O2 | 2.381(11) | P2-C221 | 1.852(9) |
| Ru-O3 | 2.259(10) | O1-C1 | 1.144(23) |
| Ru-C1 | 1.683(16) | O2-C2 | 1.322(11) |
| Ru-C4 | 2.030(15) | O3-C2 | 1.281(9) |
| P1-C101 | 1.848(9) | C2-C3 | 1.493(1) |
| P1-C111 | 1.877(10) | C4-C5 | 1.294(14) |
| P1-C121 | 1.842(9) | C5-C6 | 1.493(1) |
| C1-Ru-C4 | 120.9(7) | P1-Ru-C1 | 86.0(6) |
| O3-Ru-C4 | 66.2(4) | P1-Ru-O3 | 88.9(4) |
| O3-Ru-C1 | 171.4(6) | P1-Ru-O2 | 83.2(3) |
| O2-Ru-C4 | 122.0(4) | P1-Ru-P2 | 174.5(3) |
| O2-Ru-C1 | 116.4(7) | Ru-O2-C2 | 91.7(5) |
| O2-Ru-O3 | 55.9(4) | Ru-O3-C2 | 98.5(5) |
| P2-Ru-C4 | 92.5(4) | Ru-C1-O1 | 175.3(14) |
| P2-Ru-C1 | 91.9(5) | O2-C2-O3 | 113.7(7) |
| P2-Ru-O3 | 92.5(4) | O3-C2-C3 | 123.6(5) |
| P2-Ru-O2 | 93.2(3) | O2-C2-C3 | 120.5(5) |
| P1-Ru-C4 | 92.9(5) | Ru-C4-C5 | 125.6(8) |
| | | C4-C5-C6 | 126.8(6) |

$\eta^2$-bonded to the metal, with Ru-O2 and Ru-O3 distances of 2.381(11) and 2.259(10) Å. The shorter distance is probably the result of the *trans* relationship of O3 to CO. The geometry of the acetate ligand (O2-C2-O3 angle of 113.7(6)°)

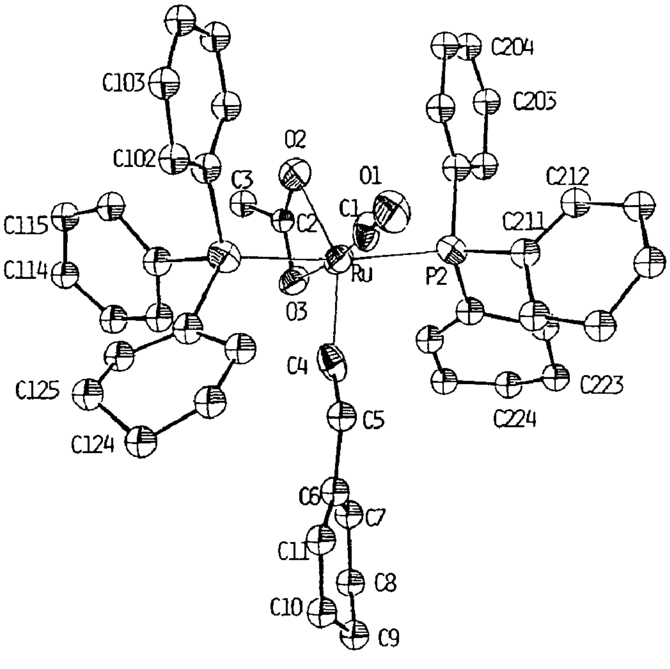

Fig. 1. The molecular structure of $Ru(O_2CMe)(CO)(HC{=}CHPh)(PPh_3)_2$.

results in a small O2–Ru–O3 angle of 55.9(4)°. This compression causes a modification of the remaining equatorial angles around the ruthenium atom (C1–Ru–O2: 116.4(7)°, C1–Ru–C4: 121(7)° and C4–Ru–O3: 66.2(4)°). The O2–C2 and O3–C2 bonds have lengths of 1.322(10) and 1.271(9) Å, showing that there is strong electron delocalization. The bond length Ru–C1 of 1.68(2) Å is short but is comparable with those in related compounds [6,13]. Other distances C1–O1 (1.14(2) Å), Ru–P1 (2.344(9) Å) and Ru–P2 (2.400(9) Å) are as expected [5]. The phenylethenyl ligand lies in a plane and the double C4–C5 bond has a length of 1.29(1) Å. The ruthenium–alkenyl bond Ru–C4 has a length of 2.03(1) Å, indicative of a σ bond.

## Experimental

The IR spectra were recorded on a Perkin–Elmer 1710-FT apparatus with KBr pellets. The $^1$H NMR spectra were recorded on a Bruker AM 400 spectrometer with $CDCl_3$ solutions; values containing $SiMe_4$ as reference. The C and H elemental analysis were performed with a Perkin Elmer 240B analyzer.

All reactions were performed under nitrogen by Schlenk techniques. Crystallization of the new products was made by slow evaporation of solutions in the air. The $Ru(CO)Cl(RCCHR')(PPh_3)_2$ complexes were made by published methods [3,11,12].

### *Preparation of complexes*

The same general method was used for all the new $Ru(O_2CR'')(CO)(RCCHR')(PPh_3)_2$ complexes. A slight excess of sodium acetate or sodium propionate dissolved in 5 ml of methanol was added with stirring to a solution of $Ru(CO)Cl(RCCHR')(PPh_3)_2$ (0.2 g) in $CH_2Cl_2$. The solution turned green-yellow and the stirring was maintained until it became yellow (30 min). The solution was then concentrated until a yellow precipitate appeared and, to bring about complete precipitation hexane was added slowly. The precipitate was filtered off and dissolved in $CH_2Cl_2$. The solution was filtered than concentrated, and hexane was added to precipitate the pure product. The complexes could be recrystallized from $CH_2Cl_2$/MeOH mixtures.

$Ru(O_2CMe)(CO)(HCCH_2(PPh_3)_2$ (**1**). Found: C, 66.6; H, 4.9. $C_{41}H_{38}O_3P_2Ru$ calc.: C, 66.59; H, 4.87%. IR: ν(CO) 1920, ν(C=C) 1562, ν(OCO) 1523, 1457 cm$^{-1}$. $^1$H NMR (ppm): δ 0.64(s,3H), 4.73(d,16Hz,1H), 4.96(d,7Hz,1H), 7.26–7.42(m,31H).

$Ru(O_2CMe)(CO)(HCCHCMe_3)(PPh_3)_2$ (**2**). Found: C, 67.8; H, 5.6. $C_{45}H_{44}O_3P_2Ru$ calc.: C, 67.93; H, 5.53%. IR: ν(CO) 1910, ν(OCO) 1527, 1452 cm$^{-1}$. $^1$H NMR (ppm): δ 0.35(s,9H), 0.59(s,3H), 5.00(d,15Hz,1H), 6.29(d,15Hz,1H), 7.25–7.44(m,30H).

$Ru(O_2CMe)(CO)(HCCHSiMe_3)(PPh_3)_2$ (**3**). Found: C, 67.8,; H, 5.6. $C_{44}H_{44}O_3P_2RuSi$ calc.: C, 65.11; H, 5.42%. IR: ν(CO) 1910, ν(C=C) 1507, ν(OCO) 1531, 1455 cm$^{-1}$. $^1$H NMR (ppm): δ −0.59(s,9H), 0.54(s,3H), 5.52(d,18Hz,1H), 7.24–7.40(m,30H), 7.65(d,18Hz,1H).

$Ru(O_2CMe)(CO)(HCCHPh)(PPh_3)_2$ (**4**). Found: C, 68.7; H, 4.9. $C_{47}H_{40}O_3P_2Ru$ calc.: C, 69.21; H, 4.90%. IR: ν(CO) 1924, ν(C=C) 1550, ν(OCO) 1532, 1458 cm$^{-1}$. $^1$H NMR (ppm): δ 0.60(s,3H), 5.79(d,16Hz,1H), 6.32–8.40(m,35H), 7.81(d,16Hz, 1H).

$Ru(O_2CMe)(CO)(MeCCHMe)(PPh_3)_2$ (**5**). Found: C, 64.6; H, 4.7. $C_{43}H_{40}O_3P_2Ru$

calc.: C, 67.29; H, 5.21%. IR: $\nu$(CO) 1916, $\nu$(OCO) 1530, 1458 cm$^{-1}$. $^1$H NMR (ppm): $\delta$ 0.56(s,3H), 1.07(s,3H), 1.35(s,3H), 4.62(br,1H), 7.35–7.45(m,30H).

$Ru(O_2CMe)(CO)(PhCCHPh)(PPh_3)_2$ (**6**). Found: C, 71.6; H, 4.7. $C_{43}H_{44}O_3P_2Ru$ calc.: C, 71.39; H, 4.93%. IR: $\nu$(CO) 1919, $\nu$(OCO) 1534, 1460 cm$^{-1}$. $^1$H NMR (ppm): $\delta$ 0.38(s,3H), 6.10–7.39(m,41H).

$Ru(O_2CMe)(CO)(HCCHCO_2Me)(PPh_3)_2$ (**7**). Found: C, 64.7; H, 5.0. $C_{43}H_{38}O_5P_2Ru$ calc.: C, 64.76; H, 4.77. IR: $\nu$(CO) 1919, 1683, $\nu$(C=C) 1556, $\nu$(OCO) 1526, 1468 cm$^{-1}$. $^1$H NMR (ppm): $\delta$ 0.55(s,3H), 3.35(s,3H), 5.56(d, 15Hz, 1H), 7.25–7.70(m,30H), 9.50(d,15Hz, 1H).

$Ru(O_2CMe)(CO)(HCCHCO_2Et)(PPh_3)_2$ (**8**). Found: C, 65.2; H, 5.0. $C_{44}H_{40}O_5P_2Ru$ calc.: C, 65.12; H, 4.93%. IR: $\nu$(CO) 1925, 1692, $\nu$(OCO) 1553, 1460 cm$^{-1}$. $^1$H NMR (ppm): $\delta$ 0.54(s,3H), 1.04(t,7Hz,3H), 3.80(q,7Hz,2H), 5.62(d,16Hz, 1H), 7.25–7.70(m,30H), 9.45(d,16Hz,1H).

$Ru(O_2CMe)(CO)(MeO_2CCCHCO_2Me)(PPh_3)_2$ (**9**). Found: c, 63.1; H, 4.8. $C_{45}H_{40}O_7P_2Ru$ calc.: C, 63.17; H, 4.68%. IR: $\nu$(CO) 1938, 1708, $\nu$(C=C 1521, $\nu$(OCO) 1564, 1463 cm$^{-1}$. $^1$H NMR (ppm): $\delta$ 0.45(s,3H), 3.38(s,3H), 3.51(s,3H), 5.08(s,1H), 7.4–7.7(m,30H).

$Ru(O_2CEt)(CO)(HCCH_2)(PPh_3)_3$ (**10**). Found: C, 66.7; H, 5.0. $C_{42}H_{38}P_2O_3Ru$ calc.: C, 66.95; H, 5.04%. IR: $\nu$(CO) 1914, $\nu$(C=C) 1567, $\nu$(OCO) 1522, 1469 cm$^{-1}$. $^1$H NMR (ppm): $\delta$ 0.16(t,7Hz,3H), 0.92(q,7Hz,2H), 4.79(d,16Hz,1H), 5.04(d,8Hz, 1H), 7.2–7.7(m,31H).

$Ru(O_2CEt)(CO)(HCCHCMe_3)(PPh_3)_2$ (**11**). Found: C, 68.3; H, 5.5. $C_{46}H_{46}P_2O_3Ru$ calc.: C, 68.25; H, 5.68%. IR: $\nu$(CO) 1910, $\nu$(C=C) 1558, $\nu$(OCO) 1516, 1469 cm$^{-1}$. $^1$H NMR (ppm): $\delta$ 0.34(s,9H), 0.84(br,3H), 1.62(br,2H), 5.00(d,15Hz,1H), 6.23(d,15Hz,1H), 7.2–7.7(m,30H).

$Ru(O_2CEt)(CO)(HCCHSiMe_3)(PPh_3)_2$ (**12**). Found: C, 65.9; H, 5.7. $C_{45}H_{46}P_2O_3SiRu$ calc.: C, 65.45; H, 5.57%. IR: $\nu$(CO) 1912, $\nu$(OCO) 1519, 1469 cm$^{-1}$. $^1$H NMR (ppm): $\delta$ −0.39(s,9H), 0.18(br,3H), 0.92(br,2H), 5.62(d,16Hz,1H), 7.0–7.7(m,31H).

$Ru(O_2CEt)(CO)(HCCHPh)(PPh_3)_2$ (**13**). Found: C, 69.5; H, 5.1. $C_{48}H_{42}P_2O_3Ru$ calc.: C, 69.49; H, 5.06%. IR $\nu$(CO) 1924, $\nu$(C=C) 1551, $\nu$(OCO) 1519, 1468 cm$^{-1}$. $^1$H NMR (ppm): $\delta$ 0.93(t,7Hz,3H), 1.79(q,7Hz,2H), 5.82(d,15Hz,1H), 7.36–7.54(m, 45H), 7.81(d,15Hz,1H).

$Ru(O_2CEt)(CO)(MeCCHMe)(PPh_3)_2$ (**14**). Found: C, 67.6; H, 5.4. $C_{44}H_{42}P_2O_3Ru$ calc.: C, 67.26; H, 5.37%. IR: $\nu$(CO) 1908, $\nu$(OCO) 1523, 1470 cm$^{-1}$. $^1$H NMR (ppm): $\delta$0.85(t7Hz,3H), 1.07(br,3H), 1.34(s,3H), 1.76(q,7Hz,2H), 4.59(br,1H), 7.35–7.7(m,30H).

$Ru(O_2CEt)(CO)(PhCCHPh)(PPh_3)_2$ (**15**). Found: C, 71.7; H, 5.1. $C_{54}H_{46}P_2O_3Ru$ calc.: C, 71.61; H, 5.08%. IR: $\nu$(CO) 1918, $\nu$(C=C) 1559, $\nu$(OCO) 1518, 1470 cm$^{-1}$. $^1$H NMR (ppm): $\delta$ 0.6(t,7Hz,3H), 1.52(q,7Hz,2H), 6.25(s,1H),6.12–7.7(m,40H).

$Ru(O_2CEt)(CO)(HCCHCO_2Me)(PPh_3)_2$ (**16**). Found: C, 65.6; H, 4.7. $C_{44}H_{40}P_2O_5Ru$ calc.: C, 65.12; H, 4.92%. IR: $\nu$(CO) 1922, $\nu$(C=C) 1551, $\nu$(OCO) 1520, 1469 cm$^{-1}$. $^1$H NMR (ppm): $\delta$ 0.80(br,3H), 1.75(br,2H), 3.34(s,3H), 5.51(d,14Hz,1H), 7.0–7.7(m,30H), 9.52(d,14Hz,1H).

$Ru(O_2CEt)(CO)(HCCHCO_2Et)(PPh_3)_2$ (**17**). Found: C, 65.5; H, 5.0. $C_{45}H_{42}P_2O_5Ru$ calc.: C, 65.47; H, 5.09%. IR: $\nu$(CO) 1922, $\nu$(C=C) 1556, $\nu$(OCO) 1519, 1468 cm$^{-1}$ $^1$H NMR (ppm): $\delta$ 0.84(br,3H), 1.06(br,3H), 1.62(br,2H), 3.79(br,2H), 5.49(d,14Hz,1H), 7.0–7.7(m,30H), 9.46(d,14Hz,1H).

*X-ray structure determination*

Crystal data and experimental details for the structure determination, with the methods of solution and refinement, are summarized in Table 2. The final atomic coordinates are given in Table 3. The crystal was mounted on a Nonius CAD-4 diffractometer, and graphite-monochromatized Mo-$K_\alpha$ radiation ($\lambda$ 0.7107 Å) was used. A total of 5742 reflections were measured up to $\theta$ 30°, with bisecting geometry and $w/2\theta$ scan mode; 1628 were considered as observed on the criterion $I > 2\sigma(I)$. The data were corrected for Lorentz polarization and absorption factors.

The structure was solved by MULTAN80 [16] and DIRDIF [17]. The refinement was carried out with anisotropic temperature factors for Ru, P, O and Cl atoms and isotropic factors for the others atoms. H atoms were fixed in calculated positions. An empirical weighting scheme [15] was used so as to give no trends in $\langle \Delta^2 F \rangle$ vs. $\langle |F_o| \rangle$ and $\langle \sin\theta/\lambda \rangle$. The final mixed refinement gave $R = 0.045$ ($R_w = 0.050$), and a maximal residual electron density of 3.2 eÅ$^{-3}$ in the neighbourhood of the ruthenium atom. Scattering factors were taken from International Tables for X-ray Crystallography [19] and anomalous dispersion was taken into account only for Ru, O and P atoms. Calculations were performed with XRAY76 system [18] on a VAX 11/750 computer.

Table 2

Crystal data, data collection and structure refinement

| | |
|---|---|
| *Crystal data* | |
| Formula | $C_{47}H_{40}O_3P_2Ru$ |
| Crystal habit | Yellow, prismatic |
| Crystal size (mm) | 0.37×0.14×0.12 |
| Symmetry | Monoclinic, *Cc* |
| Unit cell dimensions | 14.160(5), 14.250(3), 19.749(9) Å; 90.0, 96.33, 90.0° |
| Packing: $V$ (Å$^3$), $Z$ | 3961(2), 4 |
| $D_c$ (g cm$^{-3}$), $M$, $F(000)$ | 1.30, 775.5, 1520 |
| $\mu$(cm$^{-1}$)(Mo-$K_\alpha$) | 5.03 |
| *Experimental data* | |
| Technique | Four circle diffractometer: Nonius CAD-4 bisecting geometry |
| Scanning range for $\theta$ | $1 < \theta < 30$° |
| Number of reflexions: | |
| Measured | 5742 |
| Observed | 1628 ($2\sigma(I)$ criterion) |
| Absorption | Correction applied [14] |
| *Solution and refinement* | |
| Solution | Direct methods |
| Refinement | Anisotropic for Ru, P, O and Cl |
| H atoms | Geometrical calculation |
| $w$-scheme | Empirical as to give no trends in $\langle w\,\Delta^2 F \rangle$ vs. $\langle |F_o| \rangle$ and $\langle \sin\theta/\lambda \rangle$ [15] |
| Final $F$ peaks | 3.2e Å$^{-3}$ in the neighbouring of the Ru |
| Final $R$ and $R_w$ | 4.5 and 5.0 |
| Computer and programs | VAX 11/750, MULTAN 80 [16], DIRDIF [17], XRAY76 [18] |
| Scattering factors | Int. Tables for X-ray Crystallography [19] |
| Anomalous dispersion | Applied for Ru, O and P. Tables for X-ray Crystallography [19] |

Table 3

Atomic coordinates for $C_{47}H_{40}O_3P_2Ru$

| Atom | *x* | *y* | *z* |
|---|---|---|---|
| Ru | 0.46577(0) | 0.19836(8) | 0.73467(0) |
| P1 | 0.56590(64) | 0.20547(64) | 0.83732(51) |
| P2 | 0.36419(71) | 0.20731(59) | 0.62922(49) |
| O1 | 0.62095(107) | 0.26008(127) | 0.66789(74) |
| O2 | 0.37916(72) | 0.31549(78) | 0.78770(53) |
| O3 | 0.34261(70) | 0.16753(64) | 0.79487(52) |
| C1 | 0.56032(109) | 0.23561(140) | 0.69741(70) |
| C2 | 0.32119(0) | 0.25202(0) | 0.80914(0) |
| C3 | 0.25645(0) | 0.27702(0) | 0.86116(0) |
| C4 | 0.43982(76) | 0.05933(107) | 0.74533(69) |
| C5 | 0.49523(0) | −0.00824(0) | 0.73083(0) |
| C6 | 0.47977(0) | −0.11088(0) | 0.74057(0) |
| C7 | 0.38973(0) | −0.15318(0) | 0.73611(0) |
| C8 | 0.37905(0) | −0.24918(0) | 0.73600(0) |
| C9 | 0.46095(0) | −0.30487(0) | 0.72452(0) |
| C10 | 0.54628(0) | −0.26045(0) | 0.74765(0) |
| C11 | 0.55929(0) | −0.1636(0) | 0.74619(0) |
| C101 | 0.62730(0) | 0.32010(0) | 0.84420(0) |
| C102 | 0.72275(0) | 0.33020(0) | 0.86064(0) |
| C103 | 0.76714(0) | 0.42185(0) | 0.86225(0) |
| C104 | 0.71462(0) | 0.50296(0) | 0.85459(0) |
| C105 | 0.61740(0) | 0.48900(0) | 0.83540(0) |
| C106 | 0.57480(0) | 0.40160(0) | 0.83160(0) |
| C111 | 0.50250(0) | 0.19950(0) | 0.91580(0) |
| C112 | 0.44920(0) | 0.12180(0) | 0.92420(0) |
| C113 | 0.39950(0) | 0.11710(0) | 0.98250(0) |
| C114 | 0.41201(0) | 0.18097(0) | 1.03201(0) |
| C115 | 0.46290(0) | 0.26170(0) | 1.02090(0) |
| C116 | 0.50900(0) | 0.27390(0) | 0.96330(0) |
| C121 | 0.65929(0) | 0.11746(0) | 0.86021(0) |
| C122 | 0.71567(0) | 0.09502(0) | 0.80142(0) |
| C123 | 0.79040(0) | 0.03540(0) | 0.81920(0) |
| C124 | 0.80150(0) | −0.01310(0) | 0.88270(0) |
| C125 | 0.77090(0) | 0.04230(0) | 0.93750(0) |
| C126 | 0.68700(0) | 0.08370(0) | 0.92280(0) |
| C201 | 0.30253(0) | 0.32062(0) | 0.62744(0) |
| C202 | 0.20300(0) | 0.32490(0) | 0.60450(0) |
| C203 | 0.16610(0) | 0.41890(0) | 0.60350(0) |
| C204 | 0.21980(0) | 0.49550(0) | 0.61930(0) |
| C205 | 0.32010(0) | 0.49020(0) | 0.63650(0) |
| C206 | 0.35770(0) | 0.40140(0) | 0.63920(0) |
| C211 | 0.42407(0) | 0.20414(0) | 0.55442(0) |
| C212 | 0.41660(0) | 0.26900(0) | 0.50600(0) |
| C213 | 0.46500(0) | 0.26280(0) | 0.44640(0) |
| C214 | 0.52000(0) | 0.19150(0) | 0.43720(0) |
| C215 | 0.52549(0) | 0.11300(0) | 0.48238(0) |
| C216 | 0.47986(0) | 0.12381(0) | 0.54459(0) |
| C221 | 0.26650(0) | 0.12040(0) | 0.61800(0) |
| C222 | 0.23936(0) | 0.09607(0) | 0.54345(0) |
| C223 | 0.17634(0) | 0.02084(0) | 0.53432(0) |
| C224 | 0.11426(0) | 0.00555(0) | 0.58647(0) |
| C225 | 0.15535(0) | 0.01277(0) | 0.65279(0) |
| C226 | 0.22400(0) | 0.07872(0) | 0.66308(0) |

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