

Undecacarbonyl-1 κ^3 C,2 κ^4 C,3 κ^4 C-[tris(2-chloroethyl) phosphite-1 κ P]-triangulo-triruthenium(0)

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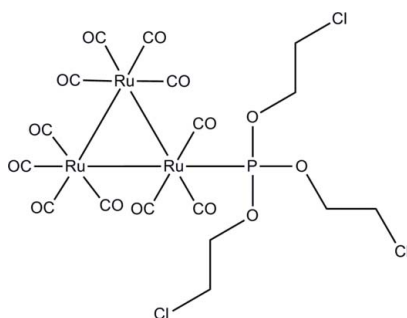
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Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.030; wR factor = 0.120; data-to-parameter ratio = 34.9.

In the title *triangulo*-triruthenium compound, $[\text{Ru}_3(\text{C}_6\text{H}_{12}\text{Cl}_3\text{O}_3\text{P})(\text{CO})_{11}]$, one equatorial carbonyl ligand is substituted by a monodentate phosphite ligand, leaving one equatorial and two axial carbonyl ligands on one Ru atom. The remaining two Ru atoms each carry two equatorial and two axial terminal carbonyl ligands. In the crystal structure, the molecules are linked into a one-dimensional column along $[100]$ by intermolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds.

Related literature

For general background to *triangulo*-triruthenium derivatives, see: Bruce *et al.* (1985, 1988*a,b*). For the synthesis, see: Bruce *et al.* (1987). For related structures, see: Shawkataly *et al.* (1991, 2010). For the stability of the temperature controller used in the data collection, see: Cosier & Glazer (1986).



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Experimental

Crystal data

$[\text{Ru}_3(\text{C}_6\text{H}_{12}\text{Cl}_3\text{O}_3\text{P})(\text{CO})_{11}]$
 $M_r = 880.80$
 Triclinic, $P\bar{1}$
 $a = 7.8592$ (9) Å
 $b = 12.5979$ (14) Å
 $c = 14.8393$ (17) Å
 $\alpha = 109.442$ (3)°
 $\beta = 93.791$ (3)°
 $\gamma = 90.763$ (3)°
 $V = 1381.4$ (3) Å³
 $Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 2.03$ mm⁻¹
 $T = 100$ K
 $0.20 \times 0.19 \times 0.03$ mm

Data collection

Bruker APEXII DUO CCD diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2001)
 $T_{\min} = 0.691$, $T_{\max} = 0.936$
 32458 measured reflections
 11981 independent reflections
 9935 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.037$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.030$
 $wR(F^2) = 0.120$
 $S = 1.07$
 11981 reflections
 343 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 1.36$ e Å⁻³
 $\Delta\rho_{\text{min}} = -1.36$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

| $D-\text{H}\cdots A$ | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|--|--------------|--------------------|-------------|----------------------|
| $\text{C}17-\text{H}17\text{B}\cdots\text{O}4^{\text{i}}$ | 0.97 | 2.58 | 3.297 (4) | 131 |
| $\text{C}17-\text{H}17\text{B}\cdots\text{O}5^{\text{ii}}$ | 0.97 | 2.54 | 3.307 (4) | 136 |

Symmetry codes: (i) $-x + 2, -y + 1, -z + 1$; (ii) $-x + 1, -y + 1, -z + 1$.

Data collection: APEX2 (Bruker, 2007); cell refinement: SAINT (Bruker, 2007); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Sheldrick, 2008); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL and PLATON (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HY2343).

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supporting information

Acta Cryst. (2010). E66, m1191–m1192 [doi:10.1107/S1600536810033891]

Undecacarbonyl- $1\kappa^3C,2\kappa^4C,3\kappa^4C$ -[tris(2-chloroethyl) phosphite- $1\kappa P$]-triangulo-triruthenium(0)

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S1. Comment

Syntheses and structures of substituted *triangulo*-triruthenium clusters have been of interest to researchers due to observed structural variations and their potential catalytic activity. A large number of substituted derivatives, $Ru_3(CO)_{12-n}L_n$ (L = group 15 ligands), have been reported (Bruce *et al.*, 1985, 1988a,b). As part of our ongoing studies on phosphite substituted *triangulo*-triruthenium clusters (Shawkataly *et al.*, 1991, 2010), herein we report the structure of the title compound.

In the title compound (Fig. 1), a monodentate phosphite ligand has replaced a single carbonyl ligand of the Ru_3 triangle. The monodentate phosphite ligand is bonded equatorially to the Ru1 atoms of the *triangulo*-triruthenium unit. Thus, the Ru2 and Ru3 atoms each carry two equatorial and two axial terminal carbonyl ligands, while the phosphite-bonded Ru1 atom binds one equatorial and two axial terminal carbonyl ligands.

In the crystal structure, the molecules are linked into a one-dimensional column along [1 0 0] by intermolecular C—H \cdots O hydrogen bonds (Fig. 2, Table 1).

S2. Experimental

All the manipulations were performed under a dry oxygen-free nitrogen atmosphere using standard Schlenk techniques. THF was dried over sodium wire and freshly distilled from sodium benzophenone ketyl solution. The title compound was prepared by mixing $Ru_3(CO)_{12}$ (Aldrich) and $P(OCH_2CH_2Cl)_3$ (Maybridge) in a 1:1 molar ratio in THF at 40°C. Diphenylketyl radical anion initiator of about 0.2 ml (synthesized as per the method of Bruce *et al.*, 1987) was introduced into the reaction mixture under a current of nitrogen. After stirring of 15 min, the solvent was removed under vacuum. Separation of the product in a pure form was done by column chromatography (Florisil, 100–200 mesh; eluant, dichloromethane:hexane). Crystals suitable for X-ray diffraction were grown by slow diffusion of CH_3OH into the CH_2Cl_2 solution.

S3. Refinement

H atoms were positioned geometrically and refined using a riding model, with C—H = 0.97 Å and $U_{iso}(H) = 1.2U_{eq}(C)$. The maximum and minimum residual electron density peaks of 1.36 and $-1.36 e\text{Å}^{-3}$ were located 1.32 and 0.81 Å from the C8 and Ru3 atoms, respectively.

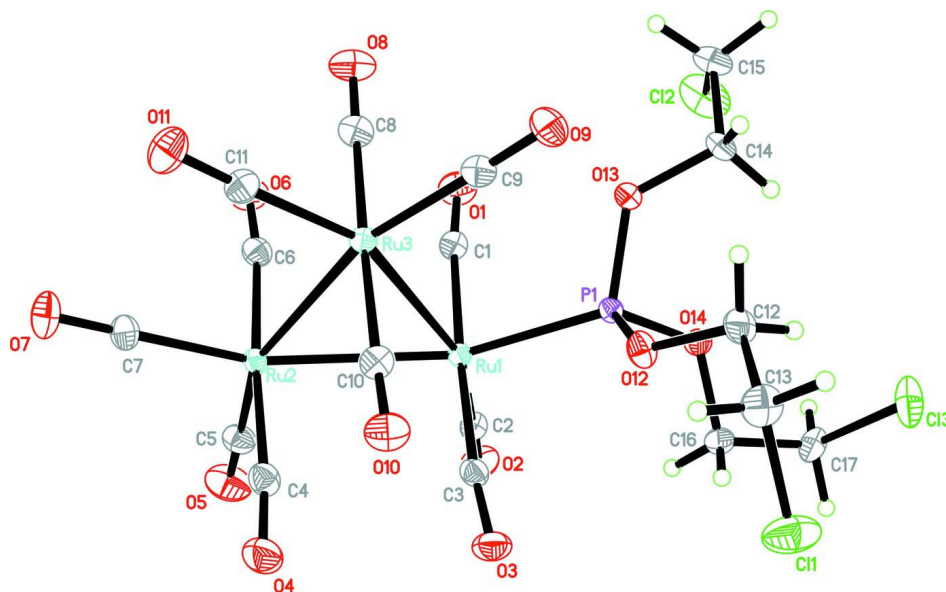


Figure 1

The molecular structure of the title compound with 50% probability ellipsoids for non-H atoms.

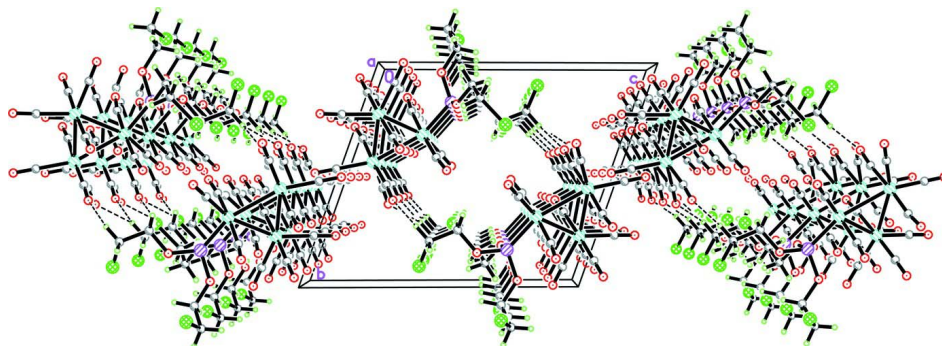


Figure 2

The crystal packing of the title compound, viewed down the *a* axis, showing the molecules linked into one-dimensional columns along the *a* axis.

Undecacarbonyl-1 κ^3 C,2 κ^4 C,3 κ^4 C- [tris(2-chloroethyl) phosphite-1 κ P]-triangulo-triruthenium(0)

Crystal data

[Ru₃(C₆H₁₂Cl₃O₃P)(CO)₁₁]

M_r = 880.80

Triclinic, *P* $\bar{1}$

Hall symbol: -P 1

a = 7.8592 (9) Å

b = 12.5979 (14) Å

c = 14.8393 (17) Å

α = 109.442 (3)°

β = 93.791 (3)°

γ = 90.763 (3)°

V = 1381.4 (3) Å³

Z = 2

F(000) = 848

D_x = 2.117 Mg m⁻³

Mo *K* α radiation, λ = 0.71073 Å

Cell parameters from 9961 reflections

θ = 2.6–34.9°

μ = 2.03 mm⁻¹

T = 100 K

Plate, orange

0.20 × 0.19 × 0.03 mm

Data collection

Bruker APEXII DUO CCD
diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
 φ and ω scans
Absorption correction: multi-scan
(*SADABS*; Bruker, 2001)
 $T_{\min} = 0.691$, $T_{\max} = 0.936$

32458 measured reflections
11981 independent reflections
9935 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.037$
 $\theta_{\max} = 35.0^\circ$, $\theta_{\min} = 1.7^\circ$
 $h = -12 \rightarrow 11$
 $k = -20 \rightarrow 20$
 $l = -23 \rightarrow 23$

Refinement

Refinement on F^2
Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.030$
 $wR(F^2) = 0.120$
 $S = 1.07$
11981 reflections
343 parameters
0 restraints
Primary atom site location: structure-invariant
direct methods

Secondary atom site location: difference Fourier
map
Hydrogen site location: inferred from
neighbouring sites
H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0712P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 1.36 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -1.36 \text{ e } \text{\AA}^{-3}$

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1) K.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|---------------|---------------|----------------------------------|
| Ru1 | 0.71803 (3) | 0.328125 (16) | 0.260791 (13) | 0.01152 (5) |
| Ru2 | 0.70797 (3) | 0.443891 (16) | 0.125659 (14) | 0.01282 (5) |
| Ru3 | 0.90548 (3) | 0.247218 (16) | 0.093890 (14) | 0.01285 (5) |
| Cl1 | 1.27297 (12) | 0.29528 (8) | 0.52857 (7) | 0.0393 (2) |
| Cl2 | 0.44453 (13) | -0.08078 (8) | 0.26311 (8) | 0.0408 (2) |
| Cl3 | 0.87744 (12) | 0.12562 (8) | 0.59050 (6) | 0.03316 (18) |
| P1 | 0.81185 (9) | 0.19414 (5) | 0.32030 (5) | 0.01243 (11) |
| O1 | 0.4173 (3) | 0.1665 (2) | 0.15829 (18) | 0.0261 (5) |
| O2 | 0.4589 (3) | 0.4615 (2) | 0.39455 (16) | 0.0272 (5) |
| O3 | 1.0116 (3) | 0.48098 (19) | 0.38775 (16) | 0.0243 (4) |
| O4 | 1.0024 (4) | 0.6040 (2) | 0.24393 (19) | 0.0312 (5) |
| O5 | 0.4624 (4) | 0.6254 (2) | 0.2229 (2) | 0.0353 (6) |
| O6 | 0.4026 (3) | 0.2931 (2) | 0.01285 (18) | 0.0264 (5) |
| O7 | 0.8072 (3) | 0.4917 (2) | -0.05275 (17) | 0.0285 (5) |
| O8 | 0.6085 (3) | 0.0883 (2) | -0.02193 (18) | 0.0286 (5) |
| O9 | 1.0676 (3) | 0.03923 (18) | 0.11984 (17) | 0.0237 (4) |
| O10 | 1.2121 (3) | 0.3946 (2) | 0.21192 (18) | 0.0246 (4) |
| O11 | 1.0379 (3) | 0.2483 (2) | -0.09539 (17) | 0.0296 (5) |
| O12 | 1.0146 (3) | 0.20752 (17) | 0.34233 (15) | 0.0185 (4) |
| O13 | 0.7651 (3) | 0.06914 (16) | 0.25139 (14) | 0.0167 (3) |
| O14 | 0.7425 (3) | 0.18383 (16) | 0.41609 (13) | 0.0156 (3) |

| | | | | |
|------|------------|-------------|--------------|------------|
| C1 | 0.5297 (4) | 0.2259 (2) | 0.1906 (2) | 0.0170 (5) |
| C2 | 0.5606 (4) | 0.4129 (2) | 0.34655 (19) | 0.0176 (5) |
| C3 | 0.9063 (4) | 0.4263 (2) | 0.33632 (19) | 0.0162 (4) |
| C4 | 0.8986 (4) | 0.5401 (2) | 0.2013 (2) | 0.0197 (5) |
| C5 | 0.5525 (4) | 0.5573 (2) | 0.1872 (2) | 0.0213 (5) |
| C6 | 0.5187 (4) | 0.3433 (2) | 0.0568 (2) | 0.0186 (5) |
| C7 | 0.7704 (4) | 0.4744 (2) | 0.0129 (2) | 0.0190 (5) |
| C8 | 0.7114 (4) | 0.1509 (2) | 0.0235 (2) | 0.0195 (5) |
| C9 | 1.0079 (4) | 0.1172 (2) | 0.1112 (2) | 0.0176 (5) |
| C10 | 1.0934 (4) | 0.3452 (2) | 0.1710 (2) | 0.0178 (5) |
| C11 | 0.9900 (4) | 0.2496 (2) | -0.0249 (2) | 0.0191 (5) |
| C12 | 1.1191 (4) | 0.1307 (2) | 0.3739 (2) | 0.0201 (5) |
| H12A | 1.1351 | 0.0632 | 0.3201 | 0.024* |
| H12B | 1.0627 | 0.1090 | 0.4217 | 0.024* |
| C13 | 1.2881 (4) | 0.1874 (3) | 0.4157 (2) | 0.0251 (6) |
| H13A | 1.3346 | 0.2200 | 0.3715 | 0.030* |
| H13B | 1.3661 | 0.1316 | 0.4238 | 0.030* |
| C14 | 0.7869 (4) | -0.0323 (2) | 0.2750 (2) | 0.0206 (5) |
| H14A | 0.7794 | -0.0160 | 0.3433 | 0.025* |
| H14B | 0.8982 | -0.0619 | 0.2585 | 0.025* |
| C15 | 0.6486 (4) | -0.1176 (2) | 0.2195 (2) | 0.0241 (6) |
| H15A | 0.6760 | -0.1907 | 0.2237 | 0.029* |
| H15B | 0.6449 | -0.1235 | 0.1525 | 0.029* |
| C16 | 0.7696 (4) | 0.2759 (2) | 0.50584 (19) | 0.0183 (5) |
| H16A | 0.6951 | 0.3368 | 0.5060 | 0.022* |
| H16B | 0.8869 | 0.3046 | 0.5150 | 0.022* |
| C17 | 0.7303 (4) | 0.2308 (3) | 0.5846 (2) | 0.0211 (5) |
| H17A | 0.6149 | 0.1984 | 0.5727 | 0.025* |
| H17B | 0.7374 | 0.2919 | 0.6455 | 0.025* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|--------------|--------------|-------------|
| Ru1 | 0.01204 (9) | 0.01129 (8) | 0.01162 (8) | 0.00113 (6) | 0.00190 (6) | 0.00414 (6) |
| Ru2 | 0.01309 (9) | 0.01271 (8) | 0.01366 (9) | 0.00183 (6) | 0.00155 (6) | 0.00559 (6) |
| Ru3 | 0.01169 (9) | 0.01307 (9) | 0.01344 (9) | 0.00218 (6) | 0.00272 (6) | 0.00355 (6) |
| Cl1 | 0.0261 (4) | 0.0352 (4) | 0.0420 (5) | 0.0005 (3) | -0.0078 (4) | -0.0045 (4) |
| Cl2 | 0.0277 (4) | 0.0341 (4) | 0.0615 (6) | 0.0012 (3) | 0.0209 (4) | 0.0140 (4) |
| Cl3 | 0.0348 (4) | 0.0420 (4) | 0.0343 (4) | 0.0164 (4) | 0.0094 (3) | 0.0264 (4) |
| P1 | 0.0123 (3) | 0.0118 (2) | 0.0135 (3) | 0.0009 (2) | 0.0010 (2) | 0.0046 (2) |
| O1 | 0.0173 (10) | 0.0285 (11) | 0.0295 (11) | -0.0045 (8) | -0.0009 (8) | 0.0065 (9) |
| O2 | 0.0225 (11) | 0.0326 (12) | 0.0220 (10) | 0.0072 (9) | 0.0069 (8) | 0.0018 (9) |
| O3 | 0.0253 (11) | 0.0209 (9) | 0.0222 (10) | -0.0057 (8) | -0.0020 (8) | 0.0023 (8) |
| O4 | 0.0335 (14) | 0.0245 (11) | 0.0345 (13) | -0.0079 (10) | -0.0086 (11) | 0.0108 (10) |
| O5 | 0.0309 (14) | 0.0253 (11) | 0.0446 (15) | 0.0083 (10) | 0.0135 (11) | 0.0026 (10) |
| O6 | 0.0194 (11) | 0.0268 (11) | 0.0283 (11) | -0.0004 (9) | -0.0045 (9) | 0.0044 (9) |
| O7 | 0.0285 (12) | 0.0391 (13) | 0.0258 (11) | 0.0016 (10) | 0.0059 (9) | 0.0205 (10) |
| O8 | 0.0215 (11) | 0.0243 (10) | 0.0319 (12) | 0.0010 (9) | 0.0000 (9) | -0.0013 (9) |

| | | | | | | |
|-----|-------------|-------------|-------------|-------------|--------------|-------------|
| O9 | 0.0224 (11) | 0.0202 (9) | 0.0298 (11) | 0.0062 (8) | 0.0044 (8) | 0.0093 (8) |
| O10 | 0.0159 (10) | 0.0268 (11) | 0.0281 (11) | -0.0014 (8) | 0.0003 (8) | 0.0054 (9) |
| O11 | 0.0287 (12) | 0.0397 (13) | 0.0222 (10) | 0.0015 (10) | 0.0085 (9) | 0.0117 (9) |
| O12 | 0.0127 (8) | 0.0203 (9) | 0.0260 (10) | 0.0015 (7) | 0.0007 (7) | 0.0125 (8) |
| O13 | 0.0218 (10) | 0.0126 (7) | 0.0158 (8) | -0.0002 (7) | 0.0008 (7) | 0.0050 (6) |
| O14 | 0.0175 (9) | 0.0153 (8) | 0.0138 (8) | -0.0015 (7) | 0.0026 (7) | 0.0046 (6) |
| C1 | 0.0152 (11) | 0.0186 (11) | 0.0176 (11) | 0.0021 (9) | 0.0017 (9) | 0.0063 (9) |
| C2 | 0.0178 (12) | 0.0191 (11) | 0.0164 (10) | 0.0013 (9) | 0.0015 (9) | 0.0064 (9) |
| C3 | 0.0179 (12) | 0.0143 (10) | 0.0170 (10) | -0.0007 (9) | 0.0031 (9) | 0.0056 (8) |
| C4 | 0.0179 (12) | 0.0191 (11) | 0.0235 (12) | 0.0007 (9) | -0.0005 (10) | 0.0093 (10) |
| C5 | 0.0212 (13) | 0.0180 (11) | 0.0234 (12) | 0.0006 (10) | 0.0048 (10) | 0.0047 (10) |
| C6 | 0.0180 (12) | 0.0169 (11) | 0.0212 (12) | 0.0027 (9) | 0.0031 (9) | 0.0065 (9) |
| C7 | 0.0171 (12) | 0.0210 (11) | 0.0215 (12) | 0.0029 (9) | 0.0018 (9) | 0.0103 (10) |
| C8 | 0.0145 (11) | 0.0198 (11) | 0.0235 (12) | 0.0027 (9) | 0.0046 (9) | 0.0056 (10) |
| C9 | 0.0148 (11) | 0.0180 (11) | 0.0188 (11) | 0.0007 (9) | 0.0043 (9) | 0.0042 (9) |
| C10 | 0.0150 (11) | 0.0198 (11) | 0.0184 (11) | 0.0023 (9) | 0.0019 (9) | 0.0059 (9) |
| C11 | 0.0174 (12) | 0.0208 (11) | 0.0183 (11) | 0.0024 (9) | 0.0034 (9) | 0.0053 (9) |
| C12 | 0.0168 (12) | 0.0188 (11) | 0.0245 (12) | 0.0054 (9) | -0.0009 (10) | 0.0074 (10) |
| C13 | 0.0143 (12) | 0.0297 (14) | 0.0306 (15) | 0.0042 (11) | 0.0002 (11) | 0.0092 (12) |
| C14 | 0.0266 (14) | 0.0119 (10) | 0.0230 (12) | 0.0020 (9) | -0.0004 (11) | 0.0057 (9) |
| C15 | 0.0252 (14) | 0.0147 (11) | 0.0292 (14) | 0.0003 (10) | 0.0065 (11) | 0.0023 (10) |
| C16 | 0.0223 (13) | 0.0160 (10) | 0.0147 (10) | 0.0022 (9) | 0.0022 (9) | 0.0022 (8) |
| C17 | 0.0204 (13) | 0.0283 (13) | 0.0156 (11) | 0.0073 (11) | 0.0020 (9) | 0.0084 (10) |

Geometric parameters (Å, °)

| | | | |
|---------|------------|----------|-----------|
| Ru1—C2 | 1.905 (3) | O5—C5 | 1.131 (4) |
| Ru1—C3 | 1.945 (3) | O6—C6 | 1.135 (4) |
| Ru1—C1 | 1.946 (3) | O7—C7 | 1.120 (4) |
| Ru1—P1 | 2.2609 (7) | O8—C8 | 1.135 (4) |
| Ru1—Ru2 | 2.8431 (4) | O9—C9 | 1.136 (4) |
| Ru1—Ru3 | 2.8610 (4) | O10—C10 | 1.133 (4) |
| Ru2—C5 | 1.922 (3) | O11—C11 | 1.130 (4) |
| Ru2—C7 | 1.929 (3) | O12—C12 | 1.450 (3) |
| Ru2—C6 | 1.936 (3) | O13—C14 | 1.443 (3) |
| Ru2—C4 | 1.949 (3) | O14—C16 | 1.446 (3) |
| Ru2—Ru3 | 2.8622 (4) | C12—C13 | 1.493 (4) |
| Ru3—C9 | 1.919 (3) | C12—H12A | 0.9700 |
| Ru3—C11 | 1.934 (3) | C12—H12B | 0.9700 |
| Ru3—C8 | 1.945 (3) | C13—H13A | 0.9700 |
| Ru3—C10 | 1.950 (3) | C13—H13B | 0.9700 |
| Cl1—C13 | 1.784 (3) | C14—C15 | 1.509 (4) |
| Cl2—C15 | 1.779 (3) | C14—H14A | 0.9700 |
| Cl3—C17 | 1.790 (3) | C14—H14B | 0.9700 |
| P1—O13 | 1.589 (2) | C15—H15A | 0.9700 |
| P1—O14 | 1.600 (2) | C15—H15B | 0.9700 |
| P1—O12 | 1.601 (2) | C16—C17 | 1.507 (4) |
| O1—C1 | 1.122 (4) | C16—H16A | 0.9700 |

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|-------------|--------------|---------------|-------------|
| O2—C2 | 1.148 (4) | C16—H16B | 0.9700 |
| O3—C3 | 1.137 (3) | C17—H17A | 0.9700 |
| O4—C4 | 1.135 (4) | C17—H17B | 0.9700 |
| C2—Ru1—C3 | 90.61 (12) | C12—O12—P1 | 124.95 (18) |
| C2—Ru1—C1 | 88.67 (12) | C14—O13—P1 | 126.29 (18) |
| C3—Ru1—C1 | 176.82 (11) | C16—O14—P1 | 120.43 (17) |
| C2—Ru1—P1 | 106.42 (9) | O1—C1—Ru1 | 173.4 (3) |
| C3—Ru1—P1 | 88.17 (8) | O2—C2—Ru1 | 176.4 (3) |
| C1—Ru1—P1 | 89.06 (8) | O3—C3—Ru1 | 173.4 (2) |
| C2—Ru1—Ru2 | 99.93 (8) | O4—C4—Ru2 | 173.8 (3) |
| C3—Ru1—Ru2 | 91.10 (8) | O5—C5—Ru2 | 178.8 (3) |
| C1—Ru1—Ru2 | 92.08 (8) | O6—C6—Ru2 | 172.8 (3) |
| P1—Ru1—Ru2 | 153.641 (19) | O7—C7—Ru2 | 179.6 (3) |
| C2—Ru1—Ru3 | 159.81 (8) | O8—C8—Ru3 | 173.9 (3) |
| C3—Ru1—Ru3 | 93.38 (8) | O9—C9—Ru3 | 178.8 (3) |
| C1—Ru1—Ru3 | 88.35 (8) | O10—C10—Ru3 | 173.8 (3) |
| P1—Ru1—Ru3 | 93.50 (2) | O11—C11—Ru3 | 178.3 (3) |
| Ru2—Ru1—Ru3 | 60.236 (9) | O12—C12—C13 | 109.3 (2) |
| C5—Ru2—C7 | 106.72 (13) | O12—C12—H12A | 109.8 |
| C5—Ru2—C6 | 90.60 (12) | C13—C12—H12A | 109.8 |
| C7—Ru2—C6 | 93.05 (12) | O12—C12—H12B | 109.8 |
| C5—Ru2—C4 | 89.78 (13) | C13—C12—H12B | 109.8 |
| C7—Ru2—C4 | 90.49 (12) | H12A—C12—H12B | 108.3 |
| C6—Ru2—C4 | 176.17 (12) | C12—C13—C11 | 112.2 (2) |
| C5—Ru2—Ru1 | 97.66 (9) | C12—C13—H13A | 109.2 |
| C7—Ru2—Ru1 | 155.59 (9) | C11—C13—H13A | 109.2 |
| C6—Ru2—Ru1 | 87.85 (9) | C12—C13—H13B | 109.2 |
| C4—Ru2—Ru1 | 88.32 (9) | C11—C13—H13B | 109.2 |
| C5—Ru2—Ru3 | 157.76 (9) | H13A—C13—H13B | 107.9 |
| C7—Ru2—Ru3 | 95.47 (9) | O13—C14—C15 | 108.5 (2) |
| C6—Ru2—Ru3 | 86.84 (8) | O13—C14—H14A | 110.0 |
| C4—Ru2—Ru3 | 91.37 (9) | C15—C14—H14A | 110.0 |
| Ru1—Ru2—Ru3 | 60.191 (8) | O13—C14—H14B | 110.0 |
| C9—Ru3—C11 | 103.01 (12) | C15—C14—H14B | 110.0 |
| C9—Ru3—C8 | 88.67 (12) | H14A—C14—H14B | 108.4 |
| C11—Ru3—C8 | 90.45 (12) | C14—C15—C12 | 112.2 (2) |
| C9—Ru3—C10 | 91.35 (12) | C14—C15—H15A | 109.2 |
| C11—Ru3—C10 | 92.68 (12) | C12—C15—H15A | 109.2 |
| C8—Ru3—C10 | 176.78 (12) | C14—C15—H15B | 109.2 |
| C9—Ru3—Ru1 | 101.60 (8) | C12—C15—H15B | 109.2 |
| C11—Ru3—Ru1 | 155.38 (9) | H15A—C15—H15B | 107.9 |
| C8—Ru3—Ru1 | 90.80 (9) | O14—C16—C17 | 107.3 (2) |
| C10—Ru3—Ru1 | 86.04 (8) | O14—C16—H16A | 110.2 |
| C9—Ru3—Ru2 | 161.17 (8) | C17—C16—H16A | 110.2 |
| C11—Ru3—Ru2 | 95.82 (9) | O14—C16—H16B | 110.2 |
| C8—Ru3—Ru2 | 91.17 (9) | C17—C16—H16B | 110.2 |
| C10—Ru3—Ru2 | 87.77 (9) | H16A—C16—H16B | 108.5 |

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|-----------------|--------------|-----------------|--------------|
| Ru1—Ru3—Ru2 | 59.573 (8) | C16—C17—C13 | 110.5 (2) |
| O13—P1—O14 | 98.19 (10) | C16—C17—H17A | 109.5 |
| O13—P1—O12 | 108.50 (12) | C13—C17—H17A | 109.5 |
| O14—P1—O12 | 104.15 (11) | C16—C17—H17B | 109.5 |
| O13—P1—Ru1 | 113.83 (8) | C13—C17—H17B | 109.5 |
| O14—P1—Ru1 | 120.97 (8) | H17A—C17—H17B | 108.1 |
| O12—P1—Ru1 | 110.01 (8) | | |
| | | | |
| C2—Ru1—Ru2—C5 | 1.98 (12) | C4—Ru2—Ru3—C9 | 88.2 (3) |
| C3—Ru1—Ru2—C5 | -88.83 (12) | Ru1—Ru2—Ru3—C9 | 0.9 (3) |
| C1—Ru1—Ru2—C5 | 91.00 (12) | C5—Ru2—Ru3—C11 | 175.3 (3) |
| P1—Ru1—Ru2—C5 | -176.92 (10) | C7—Ru2—Ru3—C11 | -1.28 (12) |
| Ru3—Ru1—Ru2—C5 | 177.91 (9) | C6—Ru2—Ru3—C11 | 91.49 (12) |
| C2—Ru1—Ru2—C7 | 179.0 (2) | C4—Ru2—Ru3—C11 | -91.90 (12) |
| C3—Ru1—Ru2—C7 | 88.2 (2) | Ru1—Ru2—Ru3—C11 | -179.18 (9) |
| C1—Ru1—Ru2—C7 | -92.0 (2) | C5—Ru2—Ru3—C8 | 84.8 (3) |
| P1—Ru1—Ru2—C7 | 0.1 (2) | C7—Ru2—Ru3—C8 | -91.85 (13) |
| Ru3—Ru1—Ru2—C7 | -5.1 (2) | C6—Ru2—Ru3—C8 | 0.91 (12) |
| C2—Ru1—Ru2—C6 | -88.33 (12) | C4—Ru2—Ru3—C8 | 177.52 (12) |
| C3—Ru1—Ru2—C6 | -179.15 (11) | Ru1—Ru2—Ru3—C8 | 90.24 (9) |
| C1—Ru1—Ru2—C6 | 0.68 (11) | C5—Ru2—Ru3—C10 | -92.2 (3) |
| P1—Ru1—Ru2—C6 | 92.76 (9) | C7—Ru2—Ru3—C10 | 91.19 (12) |
| Ru3—Ru1—Ru2—C6 | 87.59 (9) | C6—Ru2—Ru3—C10 | -176.05 (11) |
| C2—Ru1—Ru2—C4 | 91.53 (12) | C4—Ru2—Ru3—C10 | 0.56 (12) |
| C3—Ru1—Ru2—C4 | 0.72 (11) | Ru1—Ru2—Ru3—C10 | -86.72 (8) |
| C1—Ru1—Ru2—C4 | -179.46 (12) | C5—Ru2—Ru3—Ru1 | -5.5 (2) |
| P1—Ru1—Ru2—C4 | -87.38 (10) | C7—Ru2—Ru3—Ru1 | 177.90 (9) |
| Ru3—Ru1—Ru2—C4 | -92.55 (9) | C6—Ru2—Ru3—Ru1 | -89.33 (9) |
| C2—Ru1—Ru2—Ru3 | -175.93 (9) | C4—Ru2—Ru3—Ru1 | 87.28 (9) |
| C3—Ru1—Ru2—Ru3 | 93.26 (8) | C2—Ru1—P1—O13 | 117.48 (13) |
| C1—Ru1—Ru2—Ru3 | -86.91 (8) | C3—Ru1—P1—O13 | -152.42 (12) |
| P1—Ru1—Ru2—Ru3 | 5.17 (4) | C1—Ru1—P1—O13 | 29.15 (12) |
| C2—Ru1—Ru3—C9 | -168.0 (3) | Ru2—Ru1—P1—O13 | -63.64 (10) |
| C3—Ru1—Ru3—C9 | 90.97 (12) | Ru3—Ru1—P1—O13 | -59.14 (9) |
| C1—Ru1—Ru3—C9 | -86.36 (12) | C2—Ru1—P1—O14 | 0.96 (13) |
| P1—Ru1—Ru3—C9 | 2.60 (9) | C3—Ru1—P1—O14 | 91.05 (12) |
| Ru2—Ru1—Ru3—C9 | -179.70 (9) | C1—Ru1—P1—O14 | -87.38 (12) |
| C2—Ru1—Ru3—C11 | 13.7 (3) | Ru2—Ru1—P1—O14 | 179.83 (8) |
| C3—Ru1—Ru3—C11 | -87.4 (2) | Ru3—Ru1—P1—O14 | -175.67 (9) |
| C1—Ru1—Ru3—C11 | 95.3 (2) | C2—Ru1—P1—O12 | -120.51 (12) |
| P1—Ru1—Ru3—C11 | -175.7 (2) | C3—Ru1—P1—O12 | -30.41 (12) |
| Ru2—Ru1—Ru3—C11 | 2.0 (2) | C1—Ru1—P1—O12 | 151.16 (12) |
| C2—Ru1—Ru3—C8 | -79.2 (3) | Ru2—Ru1—P1—O12 | 58.37 (10) |
| C3—Ru1—Ru3—C8 | 179.78 (11) | Ru3—Ru1—P1—O12 | 62.86 (9) |
| C1—Ru1—Ru3—C8 | 2.45 (12) | O13—P1—O12—C12 | -49.8 (2) |
| P1—Ru1—Ru3—C8 | 91.41 (9) | O14—P1—O12—C12 | 54.1 (2) |
| Ru2—Ru1—Ru3—C8 | -90.89 (9) | Ru1—P1—O12—C12 | -174.9 (2) |
| C2—Ru1—Ru3—C10 | 101.4 (3) | O14—P1—O13—C14 | -41.8 (3) |

| | | | |
|-----------------|---------------|-----------------|--------------|
| C3—Ru1—Ru3—C10 | 0.41 (11) | O12—P1—O13—C14 | 66.2 (3) |
| C1—Ru1—Ru3—C10 | -176.92 (11) | Ru1—P1—O13—C14 | -171.0 (2) |
| P1—Ru1—Ru3—C10 | -87.96 (9) | O13—P1—O14—C16 | 174.3 (2) |
| Ru2—Ru1—Ru3—C10 | 89.74 (9) | O12—P1—O14—C16 | 62.8 (2) |
| C2—Ru1—Ru3—Ru2 | 11.7 (2) | Ru1—P1—O14—C16 | -61.5 (2) |
| C3—Ru1—Ru3—Ru2 | -89.33 (8) | P1—O12—C12—C13 | -162.5 (2) |
| C1—Ru1—Ru3—Ru2 | 93.34 (8) | O12—C12—C13—C11 | 71.2 (3) |
| P1—Ru1—Ru3—Ru2 | -177.703 (19) | P1—O13—C14—C15 | 148.7 (2) |
| C5—Ru2—Ru3—C9 | -4.6 (4) | O13—C14—C15—C12 | -71.2 (3) |
| C7—Ru2—Ru3—C9 | 178.8 (3) | P1—O14—C16—C17 | -166.17 (19) |
| C6—Ru2—Ru3—C9 | -88.4 (3) | O14—C16—C17—C13 | 64.2 (3) |

Hydrogen-bond geometry (Å, °)

| <i>D</i> —H... <i>A</i> | <i>D</i> —H | H... <i>A</i> | <i>D</i> ... <i>A</i> | <i>D</i> —H... <i>A</i> |
|-----------------------------|-------------|---------------|-----------------------|-------------------------|
| C17—H17B...O4 ⁱ | 0.97 | 2.58 | 3.297 (4) | 131 |
| C17—H17B...O5 ⁱⁱ | 0.97 | 2.54 | 3.307 (4) | 136 |

Symmetry codes: (i) $-x+2, -y+1, -z+1$; (ii) $-x+1, -y+1, -z+1$.