Crystal structure of the new palladium complexes tetrakis(1,3-dimethylimidazolium-2-ylidene)-palladium(II) hexadecacarbonyltetra-rhenium diethyl ether disolvate and octa-μ-carbonyl-di-carbonyltetrakis(triphenylphosphane)palladium-dirhenium (unknown solvate)

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The investigation of the coordination chemistry of heterometallic transition-metal complexes of palladium (Pd) and rhenium (Re) led to the isolation and crystallographic characterization of tetrakis(1,3-dimethylimidazolium-2-ylidene)palladium(II) hexadecacarbonyltetra-rhenium diethyl ether disolvate, [Pd(C5H8N2)4][Re6(CO)16]·2C2H5O or [Pd(IMe)4][Re6(CO)16]·2C2H5O, (1), and octa-μ-carbonyl-di-carbonyltetrahedral(triphenylphosphane)palladium-dirhenium, [Pd4Re2(C18H15P)4(C10)=CO]·Pd4Re2(PPh3)4(μ-CO)8(CO)2, (2), from the reaction of Pd(PPh3)4 with 1,3-dimethylimidazolium-2-carboxylate and Re2(CO)10 in a toluene–acetonitrile mixture. In complex 1 the Re–Re bond lengths [2.9767 (3)–3.0133 (2) Å] are close to double the covalent Re radii (1.51 Å). The palladium–rhenium carbonyl cluster 2 has not been structurally characterized previously; the Pd–Re bond lengths [2.7582 (2)–2.7796 (2) Å] are about 0.1 Å shorter than the sum of the covalent Pd and Re radii (1.39 + 1.51 = 2.90 Å). One carbene ligand and a diethyl ether molecule are disordered over two positions with occupancy ratios of 0.5:0.5 and 0.625 (15):0.375 (15) in 1. An unidentified solvent is present in compound 2. The given chemical formula and other crystal data do not take into account the unknown solvent molecule(s).

1. Chemical context

Bimetallic catalysts comprising palladium (Pd) and rhenium (Re) have important applications in alkane reforming, industrial chemical production, hydrodechlorination and biomass conversion (Thompson & Lamb, 2016; Bonarowska et al., 1999; Malinowski et al., 1998; Juszczyk & Karpinski, 2001). Heterometallic Pd–Re clusters are suitable precursors for such a catalytic system. We found that the reaction of Pd(PPh3)4 with 1,3-dimethylimidazolium-2-carboxylate and Re2(CO)10 in a toluene–acetonitrile mixture produces a mixture of two compounds: [Pd(IMe)4][Re6(CO)16]·2C2H5O (1) and Pd4Re2(PPh3)4(μ-CO)8(CO)2 (2) where IMe is 1,3-dimethylimidazolium-2-ylidene. Two other products, triphenylphosphine oxide and the known complex Re2(CO)8(PPh3)2 (Adams et al., 2013) were isolated from the reaction mixture.
2. Structural commentary

The displacement ellipsoid plot of 1 is depicted in Fig. 1. The molecular unit of 1 comprises a palladium(II) cation with four coordinated N-heterocyclic carbenes (NHC) lying on a twofold rotoinversion axis, and one [Re₄(CO)₁₆] anion. The geometry around the Pd atom is square-planar with one carbene unit being disordered. The C—Pd—C angles range from 86.9 (4) to 97.7 (4)°. The cluster anion lying on the inversion center has a perfectly flat rhombus geometry with the shortest Re—Re bond [2.9767 (3) Å] corresponding to the short diagonal. The other four Re—Re bond lengths [3.001 (2)–3.0132 (2) Å] are also close to double the covalent Re radii (1.51 Å; Cordero et al., 2008). The Re—Re—Re angles are 59.330 (6)–60.542 (6)°.

The displacement ellipsoid plot of 2 is depicted in Fig. 2. The geometry of the Re₂Pd₄ core is found to be slightly distorted from that of a D₄h-symmetric tetragonal–bipyramidal prism. In complex 2, the Pd—Re bond lengths [2.7582 (2)–2.7796 (2) Å] are close to the sum of the covalent Pd and Re radii (1.39 + 1.51 = 2.90 Å). In comparison, the Pd—Re bond lengths in the PdRe₄(CO)₁₀(μ-SbPh₂)₂(μ-H)₂ clus (Adams et al., 2015) are in the range 2.9348 (18)–2.9823 (19) Å. The Pd₄ fragment has an almost square geometry [the Pd—Pd—Pd angles are 89.865 (6)–90.135 (6)° and the Pd—Pd bond lengths are 2.9678 (2)–2.99 (2) Å).

3. Supramolecular features

In the ionic crystal of 1, each cation is surrounded by six anions and vice versa (Fig. 3). No classical hydrogen-bonding interactions are observed between cations and anions, but...
and many carbonyl-O···H$_2$C and carbonyl-O···HC intermolecular contacts (Table 1) are present. The diethyl ether molecule resides in voids between four adjacent cations and anions featuring an O···HC contact (2.32 Å) with one of the carbenes at the palladium atom. No π-π stacking is observed in structure 2, but several weak C···H···π and C···H···OC contacts (Fig. 4 and Table 2) are present. The axial CO groups of the Re(CO)$_5$ fragments point towards voids filled with an unidentified solvent (Fig. 5).

4. Database survey

A search for related structures of palladium cations in the Cambridge Structural Database (CSD Version 5.42, update of November 2020; Groom et al., 2016) resulted in 27 hits. Of the structures found, the closest structures considering the connectivity of the atoms are tetrakis(N-methylimidazol-2-ylidene)palladium(II) diiodide (JOKCIV; Fehlhammer et al., 1992) and bis[methylenebis(3-methylimidazol-2-ylidene)]palladium(II) diiodide dimethylsulfoxide solvate (REFQID; Heckenroth et al., 2006). The cation in 1 is the first structurally characterized palladium complex ion containing four NHC ligands with substituents at the 1,3 positions of the imidazole ring. There are a number of compounds containing the tetranuclear [Re$_4$(CO)$_{16}$]$_2^{2-}$ anion, which is also found in the compound reported here. A search of the CSD found two closely related cluster compounds, viz. bis(tetraethylammonium) hexadecacarbonyl-tetrarhenium (EAMCRE; Ciani et al., 1978) and bis(tetra-n-butylammonium)hexadecacarbonyl-tetrarhenium (BATCRE10; Churchill & Bau, 1968). The palladium–rhenium carbonyl cluster in 2 has not been structurally characterized previously.

5. Synthesis and crystallization

Under a nitrogen atmosphere, Pd(PPh$_3$)$_4$ (241 mg, 0.185 mmol) was added to a toluene–acetonitrile mixture (8 and 6 mL, respectively) and 1,3-dimethylimidazolium-2-carboxylate (104 mg, 0.704 mmol). The reaction mixture was refluxed for 1.5 h, then Re$_4$(CO)$_{16}$ (242 mg, 0.141 mmol) was added, the solution turned dark red and the solvents were removed in vacuo. The solid was washed with benzene (3 × 5 ml) and recrystallized from an acetonitrile–diethyl ether mixture. X-ray quality crystals of Pd(IMe)$_4$Re$_4$(CO)$_{16}$–2C$_2$H$_6$O (37 mg, 13%) were grown from a dichloromethane–diethyl ether mixture at 277 K. 1H NMR (300.13 MHz, DMSO-$d_6$, ppm): 3.41 (s, 24H, 8Me), 7.37 (s, 24H, 8CH). 13C{H} NMR

![Figure 4](image4.png)

A view of the packing of compound 2.

![Figure 5](image5.png)

The axial CO groups of the Re(CO)$_5$ fragments in 2 point towards voids filled with an unidentified solvent.

Table 1

| D−H ·· A | D−H | H ·· A | D ·· A | D−H ·· A |
|----------|-----|-------|-------|--------|
| C11−H11A···O4 | 0.98 | 2.49 | 3.436 (6) | 161 |
| C13−H13···O9$^{ii}$ | 0.95 | 2.44 | 3.36 (3) | 165 |
| C13−H13···O9A$^{ii}$ | 0.95 | 2.32 | 3.25 (5) | 163 |
| C15−H15A···O6$^{iii}$ | 0.98 | 2.44 | 3.326 (5) | 150 |
| C16−H16B···O9 | 0.98 | 2.57 | 3.49 (3) | 158 |
| C18−H18···O7$^{iii}$ | 0.95 | 2.43 | 3.230 (9) | 141 |
| C19−H19···O7$^{iii}$ | 0.95 | 2.56 | 3.483 (16) | 163 |
| C20−H20C···O5$^{ii}$ | 0.98 | 2.35 | 3.203 (12) | 145 |
| C20−H20C···O5$^{ii}$ | 0.98 | 2.54 | 3.491 (11) | 149 |
| C21−H21C···O5 | 0.98 | 2.59 | 3.494 (12) | 153 |
| C24−H24B···O8$^{iv}$ | 0.99 | 2.58 | 3.473 (12) | 150 |

Symmetry codes: (i) −x+1, −y+1, −z+1; (ii) −x, −y+1, −z+1; (iii) x, −y+2, z+1$\frac{1}{2}$; (iv) x−1, y, −z+1$\frac{1}{2}$; (v) −x, −y+2, −z; (vi) −x+y+1, y+1, −z+1$\frac{1}{2}$; (vii) x+1, y+1, z+1$\frac{1}{2}$.

Table 2

| D−H ·· A | D−H | H ·· A | D ·· A | D−H ·· A |
|----------|-----|-------|-------|--------|
| C9−H9···O5$^{iii}$ | 0.95 | 2.49 | 3.188 (3) | 130 |
| C39−H39···O2$^{iv}$ | 0.95 | 2.60 | 3.491 (4) | 157 |
| C20−H20C···Cg1 | 0.95 | 2.84 | 3.635 (3) | 142 |
| C34−H34···Cg3$^{iv}$ | 0.95 | 2.90 | 3.683 (3) | 140 |

Symmetry codes: (i) −x+1, −y+1, −z+1; (ii) −x, −y+1, −z+2; (iii) −x+1, −y+2, −z+1; (iv) x, y, z.

![Image](image.png)
Table 3
Experimental details.

| 1 | 2 |
|---|---|
| Crystal data | [Pd(C,H,N),][Re(CO),]2C,H,O | [Pd-Re(C,H,P),]4(CO),] |
| Chemical formula | 1832.13 | 2127.18 |
| M | Monoclinic, C2/c | Triclinic, P |
| Crystal system, space group | 100 | 100 |
| Temperature (K) | 21.1079 (9), 14.0026 (6), 19.4346 (8) | 12.9278 (4), 13.5132 (5), 14.1184 (5) |
| a, b, c (Å) | 90, 10.342 (1), 90 | 100 |
| α, β, γ (°) | 5420.0 (4) | 2060.09 (12) |
| V (Å³) | 218.6 (CO) | 201.1 (CO) |
| Z | IR (ATR, benzene solution, after several days, by slow ether diffusion | 197.7 (CO) |
| Radiation type | Mo Kα | Mo Kα |
| μ (mm⁻¹) | 9.30 | 3.91 |
| Crystal size (mm) | 0.17 × 0.11 × 0.03 | 0.23 × 0.18 × 0.18 |

Data collection

| | Bruker APEXII CCD | Bruker APEXII CCD |
|---|---|---|
| Diffractometer | Multi-scan (SADABS; Krause et al., 2015) | Multi-scan (SADABS; Krause et al., 2015) |
| Absorption correction | Tmin, Tmax | 0.11, 0.23 |
| No. of measured, independent and observed [F > 2σ(F)] reflections | 128368, 9046, 7392 | 151194, 11588, 10906 |
| Rint | 0.087 | 0.042 |
| (sin θ/λ)max (Å⁻¹) | 0.736 | 0.696 |

Refinement

| | | |
|---|---|---|
| R[F² > 2σ(F²)], wR(F²), S | 0.028, 0.065, 1.06 | 0.018, 0.042, 1.10 |
| No. of reflections | 9046 | 11588 |
| No. of parameters | 427 | 461 |
| No. of restraints | 45 | 0 |
| H-atom treatment | H-atom parameters constrained | H-atom parameters constrained |
| Δρmax, Δρmin (e Å⁻³) | 1.43, −1.83 | 0.95, −0.70 |

Computer programs: APEX2 and SAINT (Bruker, 2015), SHELXT2014/5 (Sheldrick, 2015a), SHELXL2014/7 (Sheldrick, 2015b) and OLEX2 (Dolomanov et al., 2009).

A few crystals of Pd-Re2(PPh3)4(m-CO)8(CO)2 suitable for X-ray diffraction analysis were obtained from a yellow benzene solution, after several days, by slow ether diffusion into a concentrated solution of benzene at 277 K. IR (ATR, ν, cm⁻¹): 3850 (vw), 3054 (vw, br), 2955 (vw, br), 2986 (s), 1821 (vs, br), 1585 (vw), 1571 (vw), 1515 (vw), 1477 (w), 1434 (m), 1307 (vw), 1263 (vw), 1236 (vw, br), 1182 (vw), 1159 (vw), 1119 (vw), 1092 (m), 1071 (vw), 1026 (vw), 997 (w), 907 (vw), 846 (vw), 741 (m), 690 (vs), 618 (vw), 565 (w), 541 (vw), 496 (m), 436 (vw), 412 (vw).

Triphenylphosphine oxide (14 mg, 28%) and Re(CO)₄(PPh₃)₂ (29 mg, 14%) were also isolated from this crystallization.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. All H atoms were positioned geometrically and refined using a riding model, with C—H = 0.95 Å (sp²), 0.98 Å (methyl) and 0.99 Å (methylene), with common isotropic temperature factors for all hydrogen atoms of the aromatic rings and methyl groups. SADI restraints on bond lengths and DELU restraints on anisotropic thermal parameters were used to model the disordered carbene ligand and diethyl ether molecule over two positions. For the refinement of 2, four reflections (100, 010, 200, 021) were omitted because they showed a significantly lower intensity than calculated, most probably caused by obstruction from the beam stop. The residual electron density in 2 was difficult to model and therefore, the SQUEEZE routine (Spek, 2015) in PLATON (Spek, 2020) was used to remove the contribution of the electron density in the solvent region from the intensity data and the solvent-free model was employed for the final refinement. The cavity with a volume of ca 311 Å³ contains approximately 98 electrons.

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Crystal structure of the new palladium complexes tetrakis(1,3-dimethylimidazolium-2-ylidene)palladium(II) hexadecacarbonyltetrarhenium diethyl ether disolvate and octa-µ-carbonyl-dicarbonyltetrakis(triphenylphosphane)palladiumdirhenium (unknown solvate)

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Computing details

For both structures, data collection: APEX2 (Bruker, 2015); cell refinement: SAINT (Bruker, 2015); data reduction: SAINT (Bruker, 2015); program(s) used to solve structure: SHELXT2014/5 (Sheldrick, 2015a); program(s) used to refine structure: SHELXL2014/7 (Sheldrick, 2015b); molecular graphics: OLEX2 (Dolomanov et al., 2009); software used to prepare material for publication: OLEX2 (Dolomanov et al., 2009).

Tetrakis(1,3-dimethylimidazolium-2-ylidene)palladium(II) hexadecacarbonyltetrarhenium diethyl ether disolvate (1)

Crystal data

\[\text{[Pd(C}_5\text{H}_8\text{N}_2\text{)}_4][\text{Re}_4\text{(CO)}_{16}]\cdot2\text{C}_4\text{H}_10\text{O}\]

\[M_p = 1832.13\]

Monoclinic, \(\text{C}_2/\text{c}\)

\(a = 21.1079 (9) \text{ Å}\)

\(b = 14.0026 (6) \text{ Å}\)

\(c = 19.4346 (8) \text{ Å}\)

\(\beta = 109.342 (1)^\circ\)

\(V = 5420.0 (4) \text{ Å}^3\)

\(Z = 4\)

\(F(000) = 3448\)

\(D_x = 2.245 \text{ Mg m}^{-3}\)

\(\lambda = 0.71073 \text{ Å}\)

Cell parameters from 9678 reflections

\(\theta = 2.9−31.5^\circ\)

\(\mu = 9.30 \text{ mm}^{-1}\)

\(T = 100 \text{ K}\)

Plate, brownish yellow

0.17 × 0.11 × 0.03 mm

Data collection

Bruker APEXII CCD

\(\varphi\) and \(\omega\) scans

Absorption correction: multi-scan

(SADABS; Krause et al., 2015)

\(T\_\text{min} = 0.285, \ T\_\text{max} = 0.746\)

128368 measured reflections

9046 independent reflections

7392 reflections with \(I > 2\sigma(I)\)

\(R_{\text{int}} = 0.087\)

\(\theta_{\text{max}} = 31.5^\circ, \ \theta_{\text{min}} = 1.8^\circ\)

\(h = -31\rightarrow 31\)

\(k = -20\rightarrow 20\)

\(l = -28\rightarrow 28\)

Refinement

Refinement on \(F^2\)

Least-squares matrix: full

\(R(F^2 > 2\sigma(F^2)) = 0.028\)

\(wR(F^2) = 0.065\)

\(S = 1.06\)

9046 reflections

427 parameters

45 restraints

Primary atom site location: dual
Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

\[ w = \frac{1}{\sigma^2(F_o^2) + (0.0239P)^2 + 18.7547P} \]
where \( P = (F_o^2 + 2F_c^2)/3 \)

\((\Delta \sigma)_{\text{max}} = 0.002\)

\(\Delta \rho_{\text{max}} = 1.43 \text{ e Å}^{-3}\)

\(\Delta \rho_{\text{min}} = -1.83 \text{ e Å}^{-3}\)

Extinction correction: SHELXL-2014/7

(Sheldrick, 2015b),

\[ F_c^e = kF_c[1 + 0.001xF_c^2\lambda^3/\sin(2\theta)]^{1/4} \]

Extinction coefficient: 0.000167 (12)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

|     | x     | y     | z     | U(eq) |
|-----|-------|-------|-------|-------|
| Re1 | 0.24123 (2) | 0.64550 (2) | 0.00757 (2) | 0.01933 (4) |
| Re2 | 0.12119 (2) | 0.77460 (2) | -0.02794 (2) | 0.01862 (4) |
| O1  | 0.26345 (18) | 0.6591 (2) | 0.17414 (18) | 0.0402 (8) |
| O2  | 0.33004 (14) | 0.4665 (2) | 0.04457 (16) | 0.0282 (6) |
| O3  | 0.21798 (18) | 0.6155 (2) | -0.15832 (16) | 0.0395 (8) |
| O4  | 0.12446 (17) | 0.5083 (3) | -0.0062 (3) | 0.0723 (15) |
| O5  | 0.14525 (17) | 0.7926 (3) | 0.13868 (17) | 0.0467 (9) |
| O6  | 0.02877 (15) | 0.9489 (2) | -0.05934 (19) | 0.0360 (7) |
| O7  | 0.09872 (16) | 0.7518 (2) | -0.19407 (16) | 0.0298 (6) |
| O8  | 0.00308 (15) | 0.6418 (2) | -0.03953 (18) | 0.0344 (7) |
| C1  | 0.2553 (2) | 0.6594 (3) | 0.1126 (2) | 0.0259 (8) |
| C2  | 0.30013 (19) | 0.5380 (3) | 0.0300 (2) | 0.0222 (7) |
| C3  | 0.2273 (2) | 0.6311 (3) | -0.0977 (2) | 0.0265 (8) |
| C4  | 0.1656 (2) | 0.5633 (3) | -0.0026 (3) | 0.0436 (13) |
| C5  | 0.1390 (2) | 0.7867 (3) | 0.0783 (2) | 0.0284 (8) |
| C6  | 0.06388 (19) | 0.8834 (3) | -0.0472 (2) | 0.0265 (8) |
| C7  | 0.10950 (19) | 0.7595 (3) | -0.1328 (2) | 0.0203 (7) |
| C8  | 0.0477 (2) | 0.6912 (3) | -0.0359 (2) | 0.0240 (7) |
| Pd1 | 0.0000 | 0.73266 (3) | 0.2500 | 0.01708 (8) |
| N1  | 0.01104 (15) | 0.5294 (2) | 0.20006 (16) | 0.0208 (6) |
| N2  | 0.08671 (16) | 0.7599 (2) | 0.40944 (16) | 0.0207 (6) |
| N3  | 0.14126 (16) | 0.6837 (3) | 0.35200 (18) | 0.0264 (7) |
| C9  | 0.0000 | 0.5885 (3) | 0.2500 | 0.0163 (9) |
| C10 | 0.0072 (2) | 0.4355 (3) | 0.2188 (2) | 0.0291 (8) |
| H10 | 0.0135 | 0.3810 | 0.1927 | 0.035* |
| C11 | 0.0244 (2) | 0.5602 (3) | 0.1342 (2) | 0.0280 (8) |
| H11A | -0.0169 | 0.5553 | 0.0921 | 0.042* |
| H11B | 0.0591 | 0.5194 | 0.1264 | 0.042* |
| H11C | 0.0399 | 0.6266 | 0.1400 | 0.042* |
| C12 | 0.08194 (18) | 0.7269 (3) | 0.34247 (19) | 0.0197 (7) |
| C13 | 0.1480 (2) | 0.7374 (3) | 0.4598 (2) | 0.0272 (8) |
| H13 | 0.1630 | 0.7529 | 0.5102 | 0.033* |
| C14 | 0.1825 (2) | 0.6895 (3) | 0.4242 (2) | 0.0306 (9) |
| Atom | x     | y     | z     | Ueq  |
|------|-------|-------|-------|------|
| H14  | 0.2265| 0.6643| 0.4445| 0.037*|
| C15  | 0.0357 (2) | 0.8150 (3) | 0.4261 (2) | 0.0263 (8) |
| H15A | 0.0515 | 0.8807 | 0.4381 | 0.040*|
| H15B | 0.0265 | 0.7864 | 0.4678 | 0.040*|
| H15C | −0.0055 | 0.8154 | 0.3837 | 0.040*|
| C16  | 0.1617 (2) | 0.6371 (4) | 0.2951 (3) | 0.0372 (11) |
| H16A | 0.1578 | 0.5677 | 0.2988 | 0.056*|
| H16B | 0.2084 | 0.6538 | 0.3015 | 0.056*|
| H16C | 0.1327 | 0.6587 | 0.2471 | 0.056*|
| N4   | −0.0689 (5) | 0.9322 (6) | 0.2333 (5) | 0.0253 (17) 0.5 |
| N5   | 0.0364 (4) | 0.9444 (6) | 0.2548 (5) | 0.0250 (16) 0.5 |
| C17  | −0.0127 (4) | 0.8789 (5) | 0.2461 (10) | 0.0184 (18) 0.5 |
| C18  | −0.0558 (5) | 1.0282 (6) | 0.2331 (5) | 0.038 (2) 0.5 |
| H18  | −0.0875 | 1.0788 | 0.2239 | 0.045* 0.5 |
| C19  | 0.0112 (8) | 1.0359 (6) | 0.2485 (19) | 0.037 (4) 0.5 |
| H19  | 0.0361 | 1.0935 | 0.2541 | 0.044* 0.5 |
| C20  | −0.1373 (5) | 0.8957 (8) | 0.2173 (6) | 0.034 (2) 0.5 |
| H20A | −0.1550 | 0.8755 | 0.1662 | 0.051* 0.5 |
| H20B | −0.1659 | 0.9462 | 0.2261 | 0.051* 0.5 |
| H20C | −0.1367 | 0.8411 | 0.2491 | 0.051* 0.5 |
| C21  | 0.1067 (6) | 0.9243 (7) | 0.2723 (6) | 0.030 (2) 0.5 |
| H21A | 0.1261 | 0.9091 | 0.3243 | 0.045* 0.5 |
| H21B | 0.1295 | 0.9803 | 0.2612 | 0.045* 0.5 |
| H21C | 0.1126 | 0.8698 | 0.2433 | 0.045* 0.5 |
| O9   | 0.3252 (14) | 0.7269 (9) | 0.3616 (17) | 0.034 (3) 0.625 (15) |
| C23  | 0.3548 (9) | 0.5629 (10) | 0.3719 (8) | 0.038 (3) 0.625 (15) |
| H23A | 0.3132 | 0.5457 | 0.3330 | 0.057* 0.625 (15) |
| H23B | 0.3473 | 0.5604 | 0.4191 | 0.057* 0.625 (15) |
| H23C | 0.3903 | 0.5177 | 0.3721 | 0.057* 0.625 (15) |
| C22  | 0.3752 (7) | 0.6610 (9) | 0.3592 (8) | 0.027 (3) 0.625 (15) |
| H22A | 0.4184 | 0.6774 | 0.3970 | 0.033* 0.625 (15) |
| H22B | 0.3815 | 0.6642 | 0.3110 | 0.033* 0.625 (15) |
| C24  | 0.3390 (5) | 0.8233 (8) | 0.346 (6) | 0.032 (2) 0.625 (15) |
| H24A | 0.3523 | 0.8250 | 0.3021 | 0.038* 0.625 (15) |
| H24B | 0.3766 | 0.8490 | 0.3877 | 0.038* 0.625 (15) |
| C25  | 0.2780 (5) | 0.8827 (7) | 0.3352 (5) | 0.045 (2) 0.625 (15) |
| H25A | 0.2418 | 0.8594 | 0.2924 | 0.068* 0.625 (15) |
| H25B | 0.2881 | 0.9493 | 0.3274 | 0.068* 0.625 (15) |
| H25C | 0.2639 | 0.8784 | 0.3783 | 0.068* 0.625 (15) |
| O9A  | 0.333 (3) | 0.7156 (16) | 0.371 (3) | 0.033 (6) 0.375 (15) |
| C22A | 0.3655 (13) | 0.6404 (13) | 0.3477 (13) | 0.029 (5) 0.375 (15) |
| H22C | 0.4123 | 0.6592 | 0.3540 | 0.035* 0.375 (15) |
| H22D | 0.3422 | 0.6280 | 0.2952 | 0.035* 0.375 (15) |
| C24A | 0.3289 (11) | 0.8017 (10) | 0.3296 (9) | 0.027 (4) 0.375 (15) |
| H24C | 0.2916 | 0.7966 | 0.2827 | 0.032* 0.375 (15) |
| H24D | 0.3712 | 0.8108 | 0.3188 | 0.032* 0.375 (15) |
| C23A | 0.3655 (13) | 0.5520 (12) | 0.3901 (11) | 0.020 (3) 0.375 (15) |
| H23D | 0.3939 | 0.5037 | 0.3783 | 0.030* 0.375 (15) |
### Atomic displacement parameters (Å²)

|   | $U^{11}$  | $U^{22}$  | $U^{33}$  | $U^{12}$  | $U^{13}$  | $U^{23}$  |
|---|-----------|-----------|-----------|-----------|-----------|-----------|
| Re1 | 0.01832 (7) | 0.01206 (7) | 0.02740 (8) | 0.00109 (5) | 0.00727 (5) | -0.00045 (5) |
| Re2 | 0.01763 (7) | 0.01595 (7) | 0.02921 (7) | 0.00130 (5) | 0.00757 (5) | -0.00270 (5) |
| O1  | 0.0373 (18) | 0.075 (3)  | 0.0284 (16) | 0.0133 (18) | 0.0116 (14) | -0.0083 (17) |
| O2  | 0.0276 (14) | 0.0205 (15) | 0.059 (2)   | 0.0059 (12) | 0.0128 (14) | -0.0041 (14) |
| O3  | 0.0316 (16) | 0.0266 (16) | 0.0279 (15) | 0.0001 (12) | 0.0122 (12) | 0.0020 (12)  |
| O4  | 0.0324 (15) | 0.0296 (17) | 0.0465 (18) | -0.0084 (13) | 0.0202 (14) | -0.0073 (14) |
| C1  | 0.0295 (19) | 0.0196 (19) | 0.035 (2)   | 0.0015 (15) | 0.0191 (17) | 0.0027 (16)  |
| C2  | 0.0258 (17) | 0.0211 (19) | 0.0217 (16) | -0.0004 (14) | 0.0107 (14) | 0.0010 (14)  |
| C3  | 0.0299 (19) | 0.0134 (18) | 0.032 (2)   | 0.0042 (15) | 0.0052 (16) | -0.0015 (15) |
| C4  | 0.0262 (16) | 0.027 (2)   | 0.088 (4)   | 0.0035 (17) | 0.018 (2)  | 0.001 (2)    |
| C5  | 0.0239 (18) | 0.030 (2)   | 0.032 (2)   | 0.0046 (16) | 0.0101 (16) | -0.0061 (17) |
| N1  | 0.0257 (15) | 0.0122 (14) | 0.0230 (14) | -0.0005 (12) | 0.0062 (12) | -0.0024 (12) |
| N2  | 0.0265 (15) | 0.0175 (16) | 0.0190 (14) | -0.0068 (12) | 0.0085 (12) | -0.0013 (11) |
| N3  | 0.0223 (15) | 0.0286 (19) | 0.0294 (17) | -0.0082 (13) | 0.0101 (13) | -0.0123 (14) |
| C9  | 0.0152 (12) | 0.020 (2)   | 0.020 (2)   | 0.00 (2)    | 0.0026 (17) | 0.000 (2)    |
| C10 | 0.036 (2)   | 0.022 (2)   | 0.0274 (19) | 0.0000 (17) | 0.0123 (17) | -0.0041 (16) |
| C11 | 0.0253 (17) | 0.0152 (17) | 0.0201 (16) | -0.0068 (14) | 0.0096 (14) | -0.0023 (13) |
| C12 | 0.0213 (2)  | 0.026 (2)   | 0.0219 (17) | -0.0112 (16) | 0.0042 (15) | -0.0039 (15) |
| C13 | 0.0220 (18) | 0.031 (2)   | 0.034 (2)   | -0.0065 (16) | 0.0023 (16) | -0.0061 (18) |
| C14 | 0.033 (2)   | 0.026 (2)   | 0.0236 (18) | -0.0035 (17) | 0.0148 (16) | -0.0063 (16) |
| C15 | 0.025 (2)   | 0.0047 (3)  | 0.041 (2)   | -0.0039 (19) | 0.0137 (18) | -0.0019 (2)  |
| N4  | 0.035 (4)   | 0.023 (4)   | 0.021 (4)   | 0.014 (4)   | 0.012 (5)   | 0.004 (3)    |
| N5  | 0.041 (4)   | 0.020 (4)   | 0.019 (4)   | -0.005 (3)  | 0.017 (4)   | -0.004 (3)   |
| C17 | 0.030 (6)   | 0.015 (3)   | 0.013 (4)   | 0.004 (3)   | 0.011 (7)   | 0.000 (3)    |
| C18 | 0.074 (6)   | 0.019 (4)   | 0.026 (5)   | 0.018 (4)   | 0.024 (5)   | 0.010 (3)    |
| C19 | 0.068 (9)   | 0.016 (3)   | 0.029 (4)   | -0.001 (5)  | 0.020 (12)  | -0.001 (5)   |
| C20 | 0.039 (5)   | 0.041 (7)   | 0.027 (5)   | 0.018 (4)   | 0.017 (5)   | 0.009 (5)    |
| C21 | 0.043 (5)   | 0.020 (5)   | 0.032 (5)   | -0.017 (5)  | 0.018 (6)   | -0.003 (4)   |
| O9  | 0.038 (5)   | 0.033 (5)   | 0.037 (9)   | -0.011 (5)  | 0.021 (6)   | -0.010 (5)   |
### Geometric parameters (Å, °)

|   |   |   |   |   |   |   |   |   |   |   |   |   |   |   |   |   |   |   |   |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
|   |   |   |   |   |   |   |   |   |   |   |   |   |   |   |   |   |   |   |   |
| C23 | 0.043 (8) | 0.046 (5) | 0.022 (7) | 0.000 (5) | 0.008 (6) | 0.001 (4) |
| C22 | 0.021 (4) | 0.036 (5) | 0.026 (5) | -0.005 (4) | 0.010 (4) | -0.005 (4) |
| C24 | 0.035 (4) | 0.036 (5) | 0.023 (5) | -0.008 (4) | 0.006 (4) | -0.006 (4) |
| C25 | 0.050 (6) | 0.043 (5) | 0.038 (5) | 0.002 (4) | 0.008 (4) | -0.006 (4) |
| O9A | 0.055 (15) | 0.022 (5) | 0.028 (9) | 0.000 (6) | 0.022 (10) | 0.002 (5) |
| C22A | 0.037 (11) | 0.028 (8) | 0.019 (8) | 0.001 (7) | 0.005 (7) | 0.003 (7) |
| C24A | 0.044 (9) | 0.016 (6) | 0.015 (7) | -0.005 (6) | 0.002 (6) | -0.006 (4) |
| C23A | 0.023 (7) | 0.023 (6) | 0.009 (8) | -0.007 (5) | -0.001 (6) | -0.005 (5) |
| C25A | 0.044 (8) | 0.024 (6) | 0.046 (8) | 0.005 (5) | 0.016 (7) | -0.006 (5) |

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| Bond          | Distance (Å) | Bond          | Distance (Å) |
|--------------|--------------|--------------|--------------|
| C11—H11A     | 0.9800       | C24A—H24C    | 0.9900       |
| C11—H11B     | 0.9800       | C24A—H24D    | 0.9900       |
| C11—H11C     | 0.9800       | C24A—C25A    | 1.479 (14)   |
| C13—H13      | 0.9500       | C23A—H23D    | 0.9800       |
| C13—C14      | 1.338 (6)    | C23A—H23E    | 0.9800       |
| C14—H14      | 0.9500       | C23A—H23F    | 0.9800       |
| C15—H15A     | 0.9800       | C25A—H25D    | 0.9800       |
| C15—H15B     | 0.9800       | C25A—H25E    | 0.9800       |
| C15—H15C     | 0.9800       | C25A—H25F    | 0.9800       |
| C16—H16A     | 0.9800       |               |              |
| Re1°—Re1—Re2 | 60.542 (6)   | H15B—C15—H15C| 109.5       |
| Re1°—Re1—Re2i| 60.127 (5)   | N3—C16—H16A | 109.5       |
| Re2—Re1—Re2i | 120.669 (6)  | N3—C16—H16B | 109.5       |
| C1—Re1—Re1i | 91.41 (12)   | N3—C16—H16C | 109.5       |
| C1—Re1—Re2i | 91.11 (12)   | H16A—C16—H16B| 109.5     |
| C1—Re1—Re2  | 90.28 (12)   | H16A—C16—H16C| 109.5     |
| C1—Re1—C3   | 179.77 (18)  | H16B—C16—H16C| 109.5     |
| C2—Re1—Re1i | 134.85 (11)  | C17—N4—C18  | 112.1 (9)   |
| C2—Re1—Re2  | 164.59 (11)  | C17—N4—C20  | 126.2 (8)   |
| C2—Re1—Re2i | 74.73 (11)   | C18—N4—C20  | 121.7 (9)   |
| C2—Re1—C1   | 88.73 (16)   | C17—N5—C19  | 111.2 (8)   |
| C2—Re1—C3   | 91.05 (15)   | C17—N5—C21  | 125.8 (8)   |
| C2—Re1—C4   | 90.39 (17)   | C19—N5—C21  | 122.9 (9)   |
| C3—Re1—Re1i | 88.79 (11)   | N4—C17—Pd1  | 130.5 (6)   |
| C3—Re1—Re2  | 89.91 (11)   | N5—C17—Pd1  | 125.7 (6)   |
| C3—Re1—Re2i | 88.89 (12)   | N5—C17—N4   | 103.8 (8)   |
| C4—Re1—Re1i | 134.75 (13)  | N4—C18—H18  | 127.0       |
| C4—Re1—Re2  | 165.04 (13)  | C19—C18—N4  | 105.9 (9)   |
| C4—Re1—Re2i | 74.23 (13)   | C19—C18—H18 | 127.0       |
| C4—Re1—C1   | 90.0 (2)     | N5—C19—H19  | 126.5       |
| C4—Re1—C3   | 89.9 (2)     | C18—C19—N5  | 107.0 (9)   |
| Re1—Re2—Re1i| 59.330 (6)   | C18—C19—H19 | 126.5       |
| C5—Re2—Re1i | 89.40 (12)   | N4—C20—H20A | 109.5       |
| C5—Re2—Re1  | 87.57 (12)   | N4—C20—H20B | 109.5       |
| C5—Re2—C7   | 176.24 (15)  | N4—C20—H20C | 109.5       |
| C6—Re2—Re1i | 163.81 (12)  | H20A—C20—H20B| 109.5   |
| C6—Re2—C5   | 104.51 (12)  | H20A—C20—H20C| 109.5   |
| C6—Re2—C7   | 91.43 (17)   | H20B—C20—H20C| 109.5   |
| C6—Re2—C8   | 91.67 (16)   | N5—C21—H21A | 109.5       |
| C7—Re2—Re1  | 88.86 (10)   | N5—C21—H21B | 109.5       |
| C7—Re2—Re1i | 87.76 (11)   | H21A—C21—H21B| 109.5   |
| C8—Re2—Re1i | 163.93 (12)  | H21A—C21—H21C| 109.5   |
| C8—Re2—C5   | 104.61 (12)  | H21B—C21—H21C| 109.5   |
| C8—Re2—C7   | 90.28 (17)   | C22—O9—C24  | 113.7 (13)  |
| C8—Re2—C7   | 91.76 (15)   | H23A—C23—H23B| 109.5    |
| C1—C1—Re1   | 174.2 (4)    | H23A—C23—H23C| 109.5    |
| Bond                  | Value (Å) | Bond                  | Value (Å) |
|----------------------|-----------|----------------------|-----------|
| O2—C2—Re1           | 172.3 (3) | C23—C22—H22A        | 109.7     |
| O2—C2—Re1           | 174.7 (4) | C22—C23—H22B        | 109.7     |
| O4—C4—Re1           | 174.2 (4) | C22—C23—H22B        | 109.5     |
| O5—C5—Re2           | 175.9 (4) | C22—C23—H22C        | 109.5     |
| O6—C6—Re2           | 179.4 (4) | C22—C22—H22A        | 109.7     |
| O7—C7—Re2           | 175.8 (3) | O9—C22—H22B         | 109.7     |
| O8—C8—Re2           | 178.7 (4) | O9—C22—H22B         | 109.7     |
| C9—Pd1—C12          | 87.72 (10) | O9—C22—H22B         | 109.7     |
| C9—Pd1—C12          | 87.72 (10) | O9—C22—H22B         | 109.7     |
| C12a—Pd1—C12        | 173.0 (2)  | O9—C24—H24A          | 109.8     |
| C12a—Pd1—C12        | 175.4 (2)  | O9—C24—H24B          | 109.8     |
| C12—N2—C13          | 111.0 (3)  | C24—C25—H25C         | 109.5     |
| C12—N2—C15          | 125.0 (3)  | C24—C25—H25B         | 109.5     |
| C12—N3—C14          | 110.9 (3)  | C25—C25—H25C         | 109.5     |
| C12—N3—C16          | 126.1 (3)  | C25—C25—H25B         | 109.5     |
| N1—C9—Pd1           | 127.7 (2)  | C22A—C22A—H22C       | 109.5     |
| N1—C9—Pd1           | 127.7 (2)  | C22A—C22A—H22D       | 109.5     |
| N1—C10—C11          | 125.2 (3)  | O9A—C24A—C25A        | 109.5     |
| N1—C10—C11          | 124.0 (3)  | O9A—C24A—C25A        | 109.5     |
| N1—C10—C11          | 111.0 (3)  | O9A—C24A—C25A        | 109.5     |
| N1—C10—C11          | 125.0 (3)  | O9A—C24A—C25A        | 109.5     |
| H11A—C11—H11B       | 109.5      | O9A—C24A—C25A        | 109.5     |
| H11A—C11—H11C       | 109.5      | O9A—C24A—C25A        | 109.5     |
| H11B—C11—H11C       | 109.5      | O9A—C24A—C25A        | 109.5     |
| N2—C12—Pd1          | 127.2 (3)  | C22A—C22A—H22B       | 109.5     |
| N2—C12—Pd1          | 128.1 (3)  | C22A—C22A—H22B       | 109.5     |
| N2—C12—Pd1          | 104.6 (3)  | C22A—C22A—H22B       | 109.5     |
| N3—C12—N2           | 126.5      | C22A—C22A—H22B       | 109.5     |
| N3—C12—N2           | 126.7      | C22A—C22A—H22B       | 109.5     |
| N3—C12—N2           | 106.6 (4)  | C22A—C22A—H22B       | 109.5     |
| N3—C12—N2           | 126.5      | C22A—C22A—H22B       | 109.5     |
| C14—C13—N2          | 126.5      | C22A—C22A—H22B       | 109.5     |
| C14—C13—N2          | 107.0 (3)  | C22A—C22A—H22B       | 109.5     |
| C14—C13—N2          | 126.5      | C22A—C22A—H22B       | 109.5     |
| C14—C13—N2          | 106.6 (4)  | C22A—C22A—H22B       | 109.5     |
| C14—C13—N2          | 126.7      | C22A—C22A—H22B       | 109.5     |
| C14—C13—N2          | 107.0 (3)  | C22A—C22A—H22B       | 109.5     |
| C14—C13—N2          | 126.7      | C22A—C22A—H22B       | 109.5     |
| C14—C13—N2          | 106.6 (4)  | C22A—C22A—H22B       | 109.5     |
| C14—C13—N2          | 126.7      | C22A—C22A—H22B       | 109.5     |
| C14—C13—N2          | 107.0 (3)  | C22A—C22A—H22B       | 109.5     |
| C14—C13—N2          | 126.7      | C22A—C22A—H22B       | 109.5     |
| C14—C13—N2          | 106.6 (4)  | C22A—C22A—H22B       | 109.5     |
| H15A—C15—H15B       | 109.5      | C22A—C22A—H22B       | 109.5     |
| H15A—C15—H15C       | 109.5      | C22A—C22A—H22B       | 109.5     |
Hydrogen-bond geometry (Å, °)

| D—H···A       | D—H | H···A | D···A | D—H···A |
|---------------|------|-------|-------|---------|
| C11—H11A···O4iii | 0.98 | 2.49  | 3.436 (6) | 161     |
| C13—H13···O9vi | 0.95 | 2.44  | 3.36 (3)  | 165     |
| C13—H13···O9Av | 0.95 | 2.32  | 3.25 (5)  | 163     |
| C15—H15A···O6v | 0.98 | 2.44  | 3.326 (5) | 150     |
| C16—H16B···O9  | 0.98 | 2.57  | 3.49 (3)  | 158     |
| C18—H18···O7vi | 0.95 | 2.43  | 3.230 (9) | 141     |
| C19—H19···O7v | 0.95 | 2.56  | 3.483 (16) | 163     |
| C20—H20C···O5vi | 0.98 | 2.35  | 3.203 (12) | 145     |
| C21—H21A···O2vii | 0.98 | 2.54 | 3.413 (11) | 149     |
| C21—H21C···O5  | 0.98 | 2.59  | 3.494 (12) | 153     |
| C24—H24B···O8viii | 0.99 | 2.58 | 3.473 (12) | 150     |

Octa-\(\mu\)-carbonyl-dicarbonyltetrakis(triphenylphosphane)palladiumdirhenium (2)

Crystal data

\[\text{[Pd}_4\text{Re}_2\text{(C}_18\text{H}_{15}\text{P})_4\text{(CO)}_{10}]\]

\(M_r = 2127.18\)  
Triclinic, \(\overline{P}\)  
\(a = 12.9278 \text{ (4) Å}\)  
\(b = 13.5132 \text{ (5) Å}\)  
\(c = 14.1184 \text{ (5) Å}\)  
\(\alpha = 120.983 (1)°\)  
\(\beta = 108.510 (1)°\)

\(\gamma = 106.051 (1)°\)

\(y = 106.129 (1)^\circ\)

\(V = 2060.09 \text{ (12) Å}^3\)

\(Z = 1\)

\(F(000) = 1026\)

\(D_o = 1.715 \text{ Mg m}^3\)

\(\text{Mo Kα radiation, } \lambda = 0.71073 \text{ Å}\)

\(\theta = 2.8–29.6°\)

Cell parameters from 9325 reflections
Supporting information

\( \mu = 3.91 \text{ mm}^{-1} \)
\( T = 100 \text{ K} \)
Block, red
\( 0.23 \times 0.18 \times 0.18 \text{ mm} \)

Data collection

Bruker APEXII CCD
diffractometer
\( \varphi \) and \( \omega \) scans
Absorption correction: multi-scan
(SADABS; Krause et al., 2015)
\( T_{\text{min}} = 0.515, T_{\text{max}} = 0.746 \)
151194 measured reflections

Refinement

Refinement on \( F^2 \)
Least-squares matrix: full
\( R[F^2 > 2\sigma(F^2)] = 0.018 \)
\( wR(F^2) = 0.042 \)
\( S = 1.10 \)
11588 reflections
461 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

\[ w = 1/[\sigma^2(F_o^2) + (0.0148P)^2 + 2.1161P] \]
where \( P = (F_o^2 + 2F_c^2)/3 \)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

|    | x     | y     | z     | \( U_{	ext{iso}}/U_{	ext{eq}} \) |
|----|-------|-------|-------|-------------------------------|
| Re1| −0.03789 (2) | 0.58284 (2) | 0.59592 (2) | 0.01288 (3) |
| Pd1| 0.11939 (2)  | 0.64837 (2)  | 0.50923 (2)  | 0.01377 (3)  |
| Pd2| 0.13798 (2)  | 0.50124 (2)  | 0.63029 (2)  | 0.01360 (3)  |
| P1 | 0.27753 (4)  | 0.79978 (4)  | 0.52970 (4)  | 0.01537 (9)  |
| P2 | 0.27280 (5)  | 0.48255 (4)  | 0.77255 (4)  | 0.01634 (10) |
| O1 | −0.11990 (18)| 0.71938 (18)| 0.74578 (16)| 0.0433 (5)   |
| O2 | −0.12746 (15)| 0.42760 (14)| 0.70708 (13)| 0.0266 (3)   |
| O3 | 0.18975 (14) | 0.70585 (13)| 0.81880 (12)| 0.0236 (3)   |
| O4 | 0.03880 (14) | 0.82425 (13)| 0.59258 (13)| 0.0240 (3)   |
| O5 | 0.30356 (13) | 0.45945 (14)| 0.53745 (13)| 0.0236 (3)   |
| C1 | −0.0874 (2)  | 0.66930 (19)| 0.69145 (18)| 0.0246 (4)   |
| C2 | −0.10429 (18)| 0.45891 (18)| 0.64488 (17)| 0.0198 (4)   |
| C3 | 0.12010 (18) | 0.64157 (17)| 0.73175 (16)| 0.0178 (4)   |
| C4 | 0.03254 (17) | 0.73328 (17)| 0.57964 (16)| 0.0175 (4)   |
| C5 | 0.20750 (18) | 0.45438 (17)| 0.51815 (16)| 0.0174 (4)   |
| C6 | 0.37893 (17) | 0.75557 (17)| 0.48099 (17)| 0.0188 (4)   |
| C7 | 0.4316 (2)   | 0.69504 (19)| 0.53169 (19)| 0.0242 (4)   |
| H7 | 0.4156       | 0.6822      | 0.5894      | 0.029*       |

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| Atom | x    | y    | z    | Ueq  |
|------|------|------|------|------|
| C8   | 0.5067 (2) | 0.65387 (19) | 0.4984 (2) | 0.0270 (5) |
| H8   | 0.5409  | 0.6118  | 0.5322  | 0.032*   |
| C9   | 0.5315 (2) | 0.6744 (2) | 0.41544 (19) | 0.0283 (5) |
| H9   | 0.5830  | 0.6464  | 0.3924  | 0.034*   |
| C10  | 0.4819 (2) | 0.7353 (2) | 0.36630 (19) | 0.0278 (5) |
| H10  | 0.5008  | 0.7504  | 0.3106  | 0.033*   |
| C11  | 0.40389 (18) | 0.77520 (18) | 0.39750 (17) | 0.0215 (4) |
| H11  | 0.3683  | 0.8154  | 0.3619  | 0.026*   |
| C12  | 0.38172 (18) | 0.90224 (17) | 0.66794 (16) | 0.0180 (4) |
| C13  | 0.34659 (19) | 0.91169 (18) | 0.75257 (17) | 0.0216 (4) |
| H13  | 0.2686  | 0.8649  | 0.7381  | 0.026*   |
| C14  | 0.4253 (2) | 0.9896 (2) | 0.85853 (18) | 0.0264 (5) |
| H14  | 0.4004  | 0.9956  | 0.9156  | 0.032*   |
| C15  | 0.5392 (2) | 1.0578 (2) | 0.88081 (19) | 0.0299 (5) |
| H15  | 0.5922  | 1.1113  | 0.9527  | 0.036*   |
| C16  | 0.5758 (2) | 1.0476 (2) | 0.7971 (2) | 0.0292 (5) |
| H16  | 0.6544  | 1.0935  | 0.8122  | 0.035*   |
| C17  | 0.49795 (19) | 0.97081 (18) | 0.69209 (18) | 0.0230 (4) |
| H17  | 0.5237  | 0.9645  | 0.6356  | 0.028*   |
| C18  | 0.22924 (18) | 0.88071 (17) | 0.45395 (16) | 0.0184 (4) |
| C19  | 0.3060 (2) | 0.98359 (18) | 0.46489 (18) | 0.0229 (4) |
| H19  | 0.3875  | 1.0152  | 0.5148  | 0.027*   |
| C20  | 0.2634 (2) | 1.0399 (2) | 0.4029 (2) | 0.0281 (5) |
| H20  | 0.3162  | 1.1093  | 0.4102  | 0.034*   |
| C21  | 0.1449 (2) | 0.9955 (2) | 0.3312 (2) | 0.0347 (6) |
| H21  | 0.1161  | 1.0346  | 0.2897  | 0.042*   |
| C22  | 0.0676 (2) | 0.8933 (3) | 0.3198 (2) | 0.0387 (6) |
| H22  | 0.0676 (2) | 0.8933 (3) | 0.3198 (2) | 0.0387 (6) |
| C23  | 0.1098 (2) | 0.8368 (2) | 0.3815 (2) | 0.0282 (5) |
| H23  | 0.0566  | 0.7676  | 0.3742  | 0.034*   |
| C24  | 0.42622 (18) | 0.57138 (19) | 0.80973 (17) | 0.0208 (4) |
| C25  | 0.5207 (2) | 0.5376 (2) | 0.8357 (2) | 0.0294 (5) |
| H25  | 0.5070  | 0.4651  | 0.8366  | 0.035*   |
| C26  | 0.6346 (2) | 0.6093 (2) | 0.8602 (2) | 0.0401 (6) |
| H26  | 0.6982  | 0.5855  | 0.8774  | 0.048*   |
| C27  | 0.6561 (2) | 0.7155 (3) | 0.8598 (2) | 0.0409 (6) |
| H27  | 0.7342  | 0.7642  | 0.8765  | 0.049*   |
| C28  | 0.5630 (2) | 0.7502 (3) | 0.8347 (3) | 0.0415 (7) |
| H28  | 0.5776  | 0.8233  | 0.8353  | 0.050*   |
| C29  | 0.4486 (2) | 0.6779 (2) | 0.8087 (2) | 0.0326 (5) |
| H29  | 0.3850  | 0.7015  | 0.7901  | 0.039*   |
| C30  | 0.26705 (19) | 0.34177 (18) | 0.75156 (17) | 0.0199 (4) |
| C31  | 0.3181 (2) | 0.3138 (2) | 0.83814 (19) | 0.0296 (5) |
| H31  | 0.3604  | 0.3699  | 0.9103  | 0.036*   |
| C32  | 0.3073 (3) | 0.2043 (2) | 0.8192 (2) | 0.0365 (6) |
| H32  | 0.3425  | 0.1860  | 0.8784  | 0.044*   |
| C33  | 0.2453 (2) | 0.1214 (2) | 0.7143 (2) | 0.0335 (5) |
| H33  | 0.2382  | 0.0465  | 0.7018  | 0.040*   |
### Atomic displacement parameters (Å²)

|       | U₁₁   | U₂₂   | U₃₃   | U₁₂   | U₁₃   | U₂₃   |
|-------|-------|-------|-------|-------|-------|-------|
| Re1   | 0.01245 (4) | 0.01357 (4) | 0.01647 (4) | 0.00659 (3) | 0.00863 (3) | 0.00710 (3) |
| Pd1   | 0.01249 (7) | 0.01311 (7) | 0.01861 (7) | 0.00468 (5) | 0.00858 (5) | 0.00854 (5) |
| Pd2   | 0.01236 (7) | 0.01464 (7) | 0.01648 (7) | 0.00662 (5) | 0.00670 (5) | 0.00807 (5) |
| P1    | 0.0134 (2) | 0.0141 (2) | 0.0215 (2) | 0.00506 (18) | 0.00935 (18) | 0.00943 (19) |
| P2    | 0.0163 (2) | 0.0175 (2) | 0.0174 (2) | 0.00879 (19) | 0.00679 (18) | 0.00836 (19) |
| O1    | 0.0443 (11) | 0.0454 (11) | 0.0439 (11) | 0.0246 (9) | 0.0282 (9) | 0.0054 (9) |
| O2    | 0.0308 (8) | 0.0263 (8) | 0.0236 (7) | 0.0053 (7) | 0.0156 (7) | 0.0124 (6) |
| O3    | 0.0248 (8) | 0.0196 (7) | 0.0221 (7) | 0.0100 (6) | 0.0058 (6) | 0.0062 (6) |
| O4    | 0.0241 (8) | 0.0162 (7) | 0.0341 (8) | 0.0097 (6) | 0.0128 (7) | 0.0111 (6) |
| O5    | 0.0180 (7) | 0.0314 (8) | 0.0300 (8) | 0.0145 (6) | 0.0135 (6) | 0.0156 (7) |
| C1    | 0.0216 (10) | 0.0260 (11) | 0.0263 (10) | 0.0104 (9) | 0.0120 (9) | 0.0075 (9) |
| C2    | 0.0168 (9) | 0.0221 (10) | 0.0220 (9) | 0.0069 (8) | 0.0104 (8) | 0.0090 (8) |
| C3    | 0.0190 (9) | 0.0182 (9) | 0.0213 (9) | 0.0089 (8) | 0.0109 (8) | 0.0109 (8) |
| C4    | 0.0138 (8) | 0.0190 (9) | 0.0199 (9) | 0.0070 (7) | 0.0074 (7) | 0.0073 (7) |
| C5    | 0.0190 (9) | 0.0161 (9) | 0.0218 (9) | 0.0082 (7) | 0.0109 (8) | 0.0101 (7) |
| C6    | 0.0142 (9) | 0.0151 (9) | 0.0270 (10) | 0.0041 (7) | 0.0108 (8) | 0.0079 (8) |
| C7    | 0.0234 (10) | 0.0231 (10) | 0.0361 (12) | 0.0112 (9) | 0.0180 (9) | 0.0177 (9) |
| C8    | 0.0252 (11) | 0.0224 (11) | 0.0411 (13) | 0.0132 (9) | 0.0172 (10) | 0.0163 (10) |
| C9    | 0.0222 (11) | 0.0284 (12) | 0.0331 (12) | 0.0127 (9) | 0.0131 (9) | 0.0063 (9) |
| C10   | 0.0257 (11) | 0.0367 (13) | 0.0256 (10) | 0.0144 (10) | 0.0157 (9) | 0.0113 (9) |
| C11   | 0.0174 (9) | 0.0242 (10) | 0.0233 (10) | 0.0077 (8) | 0.0092 (8) | 0.0104 (8) |
| C12   | 0.0175 (9) | 0.0154 (9) | 0.0226 (9) | 0.0069 (7) | 0.0079 (8) | 0.0100 (7) |
| C13   | 0.0216 (10) | 0.0218 (10) | 0.0255 (10) | 0.0108 (8) | 0.0098 (8) | 0.0132 (8) |
| C14   | 0.0305 (12) | 0.0291 (11) | 0.0236 (10) | 0.0146 (10) | 0.0112 (9) | 0.0139 (9) |
| C15   | 0.0316 (12) | 0.0213 (11) | 0.0251 (11) | 0.0071 (9) | 0.0031 (9) | 0.0071 (9) |
| C16   | 0.0219 (11) | 0.0220 (11) | 0.0326 (12) | 0.0024 (9) | 0.0058 (9) | 0.0089 (9) |
| C17   | 0.0201 (10) | 0.0196 (10) | 0.0287 (10) | 0.060 (8) | 0.0104 (8) | 0.0110 (8) |
| C18   | 0.0200 (9) | 0.0191 (9) | 0.0228 (9) | 0.0097 (8) | 0.0122 (8) | 0.0120 (8) |
| C19   | 0.0243 (10) | 0.0201 (10) | 0.0284 (10) | 0.0082 (8) | 0.0143 (9) | 0.0126 (8) |
| C20 | 0.0370 (13) | 0.0227 (11) | 0.0358 (12) | 0.0133 (10) | 0.0215 (10) | 0.0190 (10) |
| C21 | 0.0403 (14) | 0.0394 (14) | 0.0441 (14) | 0.0236 (12) | 0.0217 (12) | 0.0318 (12) |
| C22 | 0.0243 (12) | 0.0525 (17) | 0.0478 (15) | 0.0158 (12) | 0.0116 (11) | 0.0357 (14) |
| C23 | 0.0199 (10) | 0.0316 (12) | 0.0356 (12) | 0.0069 (9)  | 0.0106 (9)  | 0.0219 (10) |
| C24 | 0.0159 (9)  | 0.0252 (10) | 0.0209 (9)  | 0.0081 (8)  | 0.0070 (8)  | 0.0097 (8)  |
| C25 | 0.0229 (11) | 0.0282 (12) | 0.0347 (12) | 0.0144 (9)  | 0.0076 (9)  | 0.0103 (10) |
| C26 | 0.0217 (12) | 0.0435 (15) | 0.0497 (16) | 0.0166 (11) | 0.0105 (11) | 0.0130 (13) |
| C27 | 0.0200 (11) | 0.0465 (16) | 0.0527 (16) | 0.0072 (11) | 0.0150 (11) | 0.0218 (14) |
| C28 | 0.0262 (13) | 0.0395 (15) | 0.0630 (18) | 0.0097 (11) | 0.0167 (12) | 0.0322 (14) |
| C29 | 0.0216 (11) | 0.0350 (13) | 0.0496 (15) | 0.0127 (10) | 0.0150 (11) | 0.0278 (12) |
| C30 | 0.0208 (10) | 0.0204 (10) | 0.0236 (10) | 0.0118 (8)  | 0.0100 (8)  | 0.0115 (8)  |
| C31 | 0.0352 (13) | 0.0260 (11) | 0.0273 (11) | 0.0163 (10) | 0.0071 (10) | 0.0134 (9)  |
| C32 | 0.0438 (15) | 0.0329 (13) | 0.0403 (14) | 0.0248 (12) | 0.0120 (12) | 0.0223 (11) |
| C33 | 0.0376 (13) | 0.0249 (12) | 0.0465 (14) | 0.0204 (11) | 0.0179 (12) | 0.0183 (11) |
| C34 | 0.0304 (12) | 0.0219 (11) | 0.0335 (12) | 0.0132 (9)  | 0.0152 (10) | 0.0087 (9)  |
| C35 | 0.0213 (10) | 0.0202 (10) | 0.0238 (10) | 0.0097 (8)  | 0.0098 (8)  | 0.0093 (8)  |
| C36 | 0.0226 (10) | 0.0250 (10) | 0.0195 (9)  | 0.0154 (8)  | 0.0091 (8)  | 0.0120 (8)  |
| C37 | 0.0246 (11) | 0.0393 (14) | 0.0319 (12) | 0.0155 (10) | 0.0143 (10) | 0.0157 (10) |
| C38 | 0.0430 (16) | 0.0624 (19) | 0.0491 (16) | 0.0326 (15) | 0.0350 (14) | 0.0348 (15) |
| C39 | 0.076 (2)   | 0.0585 (19) | 0.0360 (14) | 0.0490 (18) | 0.0393 (15) | 0.0307 (14) |
| C40 | 0.068 (2)   | 0.0340 (14) | 0.0219 (11) | 0.0274 (14) | 0.0168 (12) | 0.0101 (10) |
| C41 | 0.0364 (13) | 0.0240 (11) | 0.0215 (10) | 0.0124 (10) | 0.0105 (9)  | 0.0094 (9)  |

**Geometric parameters (Å, °)**

| Re1—Pd1 | 2.7748 (2) | C15—H15 | 0.9500 |
| Re1—Pd1i| 2.7555 (2) | C15—C16 | 1.394 (4) |
| Re1—Pd2 | 2.7796 (2) | C16—H16 | 0.9500 |
| Re1—Pd2i| 2.7582 (2) | C16—C17 | 1.383 (3) |
| Re1—C1  | 1.921 (2)  | C17—H17 | 0.9500 |
| Re1—C2  | 2.058 (2)  | C17—C18 | 1.400 (3) |
| Re1—C3  | 2.062 (2)  | C18—C19 | 1.391 (3) |
| Re1—C4  | 2.092 (2)  | C19—H19 | 0.9500 |
| Re1—C5i | 2.087 (2)  | C19—C20 | 1.393 (3) |
| Pd1—Re1 | 2.7747 (2) | C20—H20 | 0.9500 |
| Pd1—Pd2 | 2.9678 (2) | C20—C21 | 1.379 (4) |
| Pd1—Pd2i| 2.9909 (2) | C21—H21 | 0.9500 |
| Pd1—P1  | 2.3291 (5) | C21—C22 | 1.393 (4) |
| Pd1—C2i | 2.170 (2)  | C22—H22 | 0.9500 |
| Pd1—C4  | 2.088 (2)  | C22—C23 | 1.392 (3) |
| Pd2—Re1 | 2.7796 (2) | C23—H23 | 0.9500 |
| Pd2—Pd1 | 2.9910 (2) | C24—C25 | 1.398 (3) |
| Pd2—P2  | 2.3317 (5) | C24—C29 | 1.393 (3) |
| Pd2—C3  | 2.158 (2)  | C25—H25 | 0.9500 |
| Pd2—C5  | 2.094 (2)  | C25—C26 | 1.387 (4) |
| P1—C6   | 1.825 (2)  | C26—H26 | 0.9500 |
| P1—C12  | 1.830 (2)  | C26—C27 | 1.385 (4) |
| P1—C18  | 1.825 (2)  | C27—H27 | 0.9500 |
| Bond                  | Length (Å) | Bond                  | Length (Å) | Bond                  | Length (Å) |
|----------------------|------------|----------------------|------------|----------------------|------------|
| P2—C24               | 1.822 (2)  | C27—C28              | 1.389 (4)  | P2—C30               | 1.819 (2)  |
| P2—C30               | 1.821 (2)  | C28—H28              | 0.9500     | O1—C1                | 1.140 (3)  |
| P2—C36               | 1.158 (3)  | C29—H29              | 0.9500     | O2—C2                | 1.162 (2)  |
| O1—C1                | 1.158 (3)  | C30—C31              | 1.398 (3)  | O3—C3                | 1.167 (3)  |
| O2—C2                | 1.140 (3)  | C30—C35              | 1.396 (3)  | O4—C4                | 1.161 (2)  |
| O3—C3                | 1.162 (2)  | C31—H31              | 0.9500     | O5—C5                | 1.161 (2)  |
| O4—C4                | 1.167 (3)  | C31—C32              | 1.386 (3)  | C2—Pd1<sup>i</sup>   | 2.170 (2)  |
| O5—C5                | 1.161 (2)  | C32—H32              | 0.9500     | C5—Re<sup>1</sup>    | 2.087 (2)  |
| C2—Pd1<sup>i</sup>   | 2.170 (2)  | C32—C33              | 1.387 (4)  | C6—C7                | 1.403 (3)  |
| C5—Re<sup>1</sup>    | 2.087 (2)  | C33—H33              | 0.9500     | C6—C11               | 1.389 (3)  |
| C6—C7                | 1.403 (3)  | C33—C34              | 1.383 (4)  | C7—H7                | 0.9500     |
| C6—C11               | 1.389 (3)  | C34—H34              | 0.9500     | C7—C8                | 1.385 (3)  |
| C7—H7                | 0.9500     | C34—C35              | 1.390 (3)  | C8—H8                | 0.9500     |
| C7—C8                | 1.385 (3)  | C35—H35              | 0.9500     | C8—C9                | 1.385 (3)  |
| C8—H8                | 0.9500     | C36—C37              | 1.398 (3)  | C9—H9                | 0.9500     |
| C8—C9                | 1.385 (3)  | C36—C41              | 1.386 (3)  | C9—C10               | 1.376 (3)  |
| C9—H9                | 0.9500     | C37—H37              | 0.9500     | C10—H10              | 0.9500     |
| C9—C10               | 1.376 (3)  | C37—C38              | 1.386 (4)  | C10—C11              | 1.398 (3)  |
| C10—H10              | 0.9500     | C38—H38              | 0.9500     | C11—H11              | 0.9500     |
| C11—H11              | 0.9500     | C38—C39              | 1.379 (5)  | C12—C13              | 1.394 (3)  |
| C12—C13              | 1.394 (3)  | C39—H39              | 0.9500     | C12—C17              | 1.400 (3)  |
| C12—C17              | 1.400 (3)  | C39—C40              | 1.374 (5)  | C13—H13              | 0.9500     |
| C13—H13              | 0.9500     | C40—H40              | 0.9500     | C13—C14              | 1.397 (3)  |
| C13—C14              | 1.397 (3)  | C40—C41              | 1.386 (3)  | C14—H14              | 0.9500     |
| C14—H14              | 0.9500     | C41—H41              | 0.9500     | C14—C15              | 1.381 (3)  |

**Bond Angles:**

| Angle                  |   | Angle                  |   | Angle                  |   |
|-----------------------|---|-----------------------|---|-----------------------|---|
| Pd1—Re1—Pd1<sup>i</sup> | 99.105 (5) | C6—C7—H7              | 119.7    |                            |   |
| Pd1—Re1—Pd1<sup>2</sup> | 65.413 (5) | C6—C7—C6              | 120.6 (2) |                            |   |
| Pd1—Re1—Pd2<sup>i</sup> | 64.596 (5) | C8—C7—H7              | 119.7    |                            |   |
| Pd1—Re1—Pd2<sup>2</sup> | 65.131 (5) | C7—C8—H8              | 120.2    |                            |   |
| Pd2—Re1—Pd1<sup>i</sup> | 65.445 (5) | C7—C8—C9              | 119.6 (2) |                            |   |
| Pd2—Re1—Pd2<sup>2</sup> | 99.238 (5) | C9—C8—H8              | 120.2    |                            |   |
| C1—Re1—Pd1            | 131.04 (7) | C8—C9—H9              | 119.9    |                            |   |
| C1—Re1—Pd1<sup>i</sup> | 129.69 (7) | C10—C9—C8             | 120.3 (2) |                            |   |
| C1—Re1—Pd2            | 133.48 (7) | C10—C9—H9             | 119.9    |                            |   |
| C1—Re1—Pd2<sup>i</sup> | 127.28 (7) | C9—C10—H10            | 119.7    |                            |   |
| C1—Re1—C2             | 83.99 (9)  | C9—C10—C11            | 120.7 (2) |                            |   |
| C1—Re1—C3             | 85.84 (9)  | C11—C10—H10           | 119.7    |                            |   |
| C1—Re1—C4             | 83.85 (9)  | C6—C11—C10            | 119.5 (2) |                            |   |
| C1—Re1—C5<sup>i</sup> | 81.63 (9)  | C6—C11—H11            | 120.3    |                            |   |
| C2—Re1—Pd1            | 141.37 (6) | C10—C11—H11           | 120.3    |                            |   |
| C2—Re1—Pd1<sup>i</sup> | 50.76 (6)  | C13—C12—P1            | 119.88 (16) |                            |   |
| C2—Re1—Pd2            | 78.91 (6)  | C13—C12—C17           | 118.49 (19) |                            |   |
| C2—Re1—Pd2<sup>i</sup> | 109.51 (6) | C17—C12—P1            | 121.62 (16) |                            |   |
| C2—Re1—C3             | 87.43 (8)  | C12—C13—H13           | 119.8    |                            |   |
| C2—Re1—C4             | 166.84 (8) | C12—C13—C14           | 120.4 (2) |                            |   |
| Bond                  | Distance (Å) | Angle (°)   |
|-----------------------|-------------|------------|
| C2—Re1—C5i           | 91.78 (8)   |            |
| C3—Re1—Pd1^1         | 109.34 (6)  | C14—C13—H13 | 119.8 |
| C3—Re1—Pd1           | 105.38 (6)  | C13—C14—H14 | 119.8 |
| C3—Re1—Pd2           | 50.73 (6)   | C15—C14—H14 | 120.4 (2) |
| C3—Re1—Pd2^1         | 143.08 (6)  | C14—C15—H15 | 119.8 |
| C3—Re1—C4            | 86.69 (8)   |            |
| C3—Re1—C5i           | 167.46 (8)  | C16—C15—H15 | 120.2 |
| C4—Re1—Pd1           | 48.71 (5)   | C15—C16—H16 | 119.9 |
| C4—Re1—Pd1^1         | 142.40 (5)  | C17—C16—C15 | 120.2 (2) |
| C4—Re1—Pd2           | 82.00 (5)   | C17—C16—H16 | 119.9 |
| C4—Re1—Pd2^1         | 106.14 (5)  | C12—C17—H17 | 119.5 |
| C5^—Re1—Pd1          | 107.18 (6)  | C16—C17—C12 | 120.9 (2) |
| C5^—Re1—Pd1^1        | 79.46 (5)   |            |
| C5^—Re1—Pd2          | 141.24 (5)  | C19—C18—P1  | 123.40 (16) |
| C5^—Re1—Pd2^1        | 48.43 (5)   | C23—C18—P1  | 117.73 (16) |
| C5^—Re1—C4           | 91.42 (8)   | C23—C18—C19 | 118.9 (2) |
| Re1—Pd1—Re1^1        | 80.894 (5)  | C18—C19—H19 | 119.8 |
| Re1—P1—Pd1—Pd2       | 57.781 (5)  | C20—C19—C18 | 120.3 (2) |
| Re1—P1—Pd1—Pd2^1     | 57.011 (4)  |            |
| Re1—Pd1—Pd2^2        | 57.477 (4)  |            |
| Re1—Pd1—Pd2^2^1      | 57.681 (5)  |            |
| Pd2—Pd1—Pd2^1        | 90.135 (6)  |            |
| P1—Pd1—Re1^1         | 133.608 (14)|            |
| P1—Pd1—Re1^2         | 143.861 (14)|            |
| P1—Pd1—Pd2^1         | 143.699 (14)|            |
| P1—Pd1—Pd2^2         | 125.624 (14)|            |
| C2—Pd1—Re1^1         | 47.26 (6)   | C23—C22—C21 | 119.9 (2) |
| C2—Pd1—Re1^2         | 122.58 (6)  | C23—C22—H22 | 120.0 |
| C2—Pd1—Pd2           | 72.07 (5)   | C18—C23—C22 | 120.6 (2) |
| C2—Pd1—Pd2^2         | 100.12 (6)  | C18—C23—H23 | 119.7 |
| C2—Pd1—P1            | 93.27 (6)   | C22—C23—H23 | 119.7 |
| C4—Pd1—Re1^1         | 48.82 (6)   | C25—C24—P2  | 123.95 (18) |
| C4—Pd1—Re1^2         | 125.79 (6)  | C29—C24—P2  | 117.27 (17) |
| C4—Pd1—Pd2           | 99.39 (6)   | C29—C24—C25 | 118.8 (2) |
| C4—Pd1—Pd2^2         | 76.94 (5)   | C24—C25—H25 | 119.8 |
| C4—Pd1—P1            | 100.27 (6)  | C26—C25—C24 | 120.3 (2) |
| C4—Pd1—C2^1          | 143.15 (8)  | C26—C25—H25 | 119.8 |
| Re1—Pd2—Re1^1        | 80.762 (5)  | C25—C26—H26 | 119.8 |
| Re1—Pd2—Pd1^1        | 57.545 (5)  | C27—C26—C25 | 120.5 (2) |
| Re1—Pd2—Pd1^2        | 57.623 (5)  | C27—C26—H26 | 119.8 |
| Re1—Pd2—Pd1^2^1      | 57.392 (5)  | C26—C27—H27 | 120.2 |
| Re1—Pd2—Pd1^2^2      | 56.904 (4)  | C26—C27—C28 | 119.7 (2) |
| Pd1—Pd2—Pd1^2^1      | 89.865 (6)  | C28—C27—H27 | 120.2 |
| P2—Pd2—Re1^1         | 139.454 (14)| C27—C28—H28 | 120.0 |
| P2—Pd2—Re1^2         | 138.742 (14)| C27—C28—C29 | 119.9 (3) |
| P2—Pd2—Pd1^1         | 128.138 (15)| C29—C28—H28 | 120.0 |
| P2—Pd2—Pd1^2         | 141.995 (15)| C24—C29—H29 | 119.6 |
| C3—Pd2—Re1           | 47.70 (5)   | C28—C29—C24 | 120.8 (2) |
| Bond                  | Angle (°) (E) | Bond                  | Angle (°) (E) |
|----------------------|--------------|----------------------|--------------|
| C3—Pd2—Re1          | 123.96 (5)   | C28—C29—H29         | 119.6        |
| C3—Pd2—Pd1          | 99.53 (5)    | C31—C30—P2          | 122.07 (17)  |
| C3—Pd2—Pd1i         | 74.39 (5)    | C35—C30—P2          | 119.19 (16)  |
| C3—Pd2—P2           | 96.08 (6)    | C35—C30—C31         | 118.7 (2)    |
| C5—Pd2—Re1          | 124.34 (6)   | C30—C31—H31         | 119.9        |
| C5—Pd2—Re1i         | 48.21 (6)    | C32—C31—C30         | 120.3 (2)    |
| C5—Pd2—Pd1i         | 99.26 (6)    | C32—C31—H31         | 119.9        |
| C5—Pd2—Pd1          | 74.81 (6)    | C31—C32—H32         | 119.8        |
| C5—Pd2—P2           | 96.36 (6)    | C31—C32—C33         | 120.5 (2)    |
| C6—P1—Pd1           | 143.64 (8)   | C33—C32—H32         | 119.8        |
| C6—P1—C12           | 112.32 (7)   | C32—C33—H33         | 120.0        |
| C12—P1—Pd1          | 100.69 (9)   | C34—C33—C32         | 119.9 (2)    |
| C12—P1—Pd1          | 117.97 (7)   | C34—C33—H33         | 120.0        |
| C18—P1—Pd1          | 112.89 (7)   | C33—C34—H34         | 120.1        |
| C18—P1—C6           | 105.65 (10)  | C33—C34—C35         | 119.8 (2)    |
| C18—P1—C12          | 105.98 (9)   | C35—C34—H34         | 120.1        |
| C24—P2—Pd2          | 111.89 (7)   | C30—C35—C35         | 119.6        |
| C30—P2—Pd2          | 116.83 (7)   | C34—C35—C30         | 120.9 (2)    |
| C30—P2—C24          | 106.51 (10)  | C34—C35—H35         | 119.6        |
| C30—P2—C36          | 101.65 (10)  | C37—C36—P2          | 117.43 (17)  |
| C36—P2—Pd2          | 114.10 (7)   | C41—C36—P2          | 123.41 (18)  |
| C36—P2—C24          | 104.65 (10)  | C41—C36—C37         | 119.1 (2)    |
| O1—C1—Re1           | 178.1 (2)    | C36—C37—H37         | 119.9        |
| Re1—C2—Pd1i         | 81.98 (7)    | C38—C37—C36         | 120.1 (2)    |
| O2—C2—Re1           | 152.98 (18)  | C38—C37—H37         | 119.9        |
| O2—C2—Pd1i          | 124.90 (17)  | C37—C38—H38         | 119.8        |
| Re1—C3—Pd2          | 81.57 (7)    | C39—C38—C37         | 120.4 (3)    |
| O3—C3—Re1           | 152.27 (17)  | C39—C38—H38         | 119.8        |
| O3—C3—Pd2           | 126.08 (16)  | C38—C39—H39         | 120.2        |
| Pd1—C4—Re1          | 124.90 (17)  | C40—C39—C38         | 119.6 (2)    |
| O4—C4—Re1           | 149.43 (17)  | C40—C39—H39         | 120.2        |
| O4—C4—Pd1           | 128.10 (16)  | C39—C40—H40         | 119.6        |
| Re1i—C5—Pd2         | 83.36 (7)    | C39—C40—C41         | 120.8 (3)    |
| O5—C5—Re1i          | 149.52 (17)  | C41—C40—H40         | 119.6        |
| O5—C5—Pd2           | 127.10 (16)  | C36—C41—C40         | 120.0 (3)    |
| C7—C6—P1            | 115.78 (16)  | C36—C41—H41         | 120.0        |
| C11—C6—P1           | 124.87 (17)  | C40—C41—H41         | 120.0        |
| C11—C6—C7           | 119.32 (19)  | C15—C16—C17—C12    | 0.0 (4)      |
| Pd1—P1—C6—C7        | −58.98 (17)  | C17—C12—C13—C14    | 1.2 (3)      |
| Pd1—P1—C6—C11       | 119.34 (17)  | C18—P1—C6—C7       | 177.54 (16)  |
| Pd1—P1—C12—C13      | −21.58 (19)  | C18—P1—C6—C11      | −4.1 (2)     |
| Pd1—P1—C12—C17      | 157.12 (15)  | C18—P1—C12—C13     | 106.03 (18)  |
| Pd1—P1—C18—C19      | 171.11 (16)  | C18—P1—C12—C17     | −75.28 (19)  |
| Pd1—P1—C18—C23      | −9.1 (2)     | C18—C19—C20—C21   | 0.8 (4)      |
| Pd2—P2—C24—C25      | −139.46 (18) | C19—C18—C23—C22   | 0.9 (4)      |
| Pd2—P2—C24—C29      | 38.6 (2)     | C19—C20—C21—C22   | −0.6 (4)     |
| Pd2—P2—C30—C31      | −162.18 (17) |                     |              |
Pd2—P2—C30—C35 14.8 (2) C20—C21—C22—C23 0.6 (4)
Pd2—P2—C36—C37 66.91 (19) C21—C22—C23—C18 −0.8 (4)
Pd2—P2—C36—C41 −109.26 (18) C23—C18—C19—C20 −0.9 (3)
P1—C6—C7—C8 177.66 (18) C24—P2—C30—C31 72.0 (2)
P1—C6—C11—C10 −178.91 (17) C24—P2—C30—C35 −111.09 (18)
P1—C12—C13—C14 179.96 (17) C24—P2—C36—C37 −170.48 (18)
P1—C12—C17—C16 −179.77 (18) C24—P2—C36—C41 13.4 (2)
P1—C18—C19—C20 188.85 (17) C24—C25—C26—C27 0.3 (4)
P1—C18—C23—C22 −178.9 (2) C25—C26—C27—C28 −0.9 (5)
P2—C24—C25—C26 178.2 (2) C25—C26—C27—C28 0.1 (5)
P2—C24—C29—C28 −179.2 (2) C26—C27—C28—C29 1.4 (5)
P2—C30—C31—C32 177.2 (2) C27—C28—C29—C24 0.1 (4)
P2—C30—C35—C34 −176.80 (17) C30—P2—C24—C25 −10.7 (2)
P2—C30—C35—C34 −176.80 (17) C30—P2—C36—C37 −59.76 (19)
P2—C36—C37—C38 175.83 (19) C30—P2—C36—C41 124.07 (19)
P2—C36—C37—C38 175.83 (19) C31—C30—C35—C34 0.2 (3)
P1—C6—C7—C8 −144.12 (17) C31—C32—C33—C34 −0.1 (4)
P1—C6—C11—C10 34.58 (19) C32—C33—C34—C35 0.5 (4)
P1—C12—C13—C14 179.96 (17) C33—C34—C35—C30 −0.6 (4)
P1—C12—C17—C16 67.42 (18) C34—C35—C30—C31 1.1 (4)
P1—C18—C19—C20 −1.1 (4) C35—C30—C31—C32 0.2 (4)
P1—C18—C23—C22 67.42 (18) C36—P2—C24—C25 96.5 (2)
P1—C18—C23—C22 67.42 (18) C36—P2—C24—C29 −85.4 (2)
P1—C18—C23—C22 67.42 (18) C36—P2—C30—C31 −37.3 (2)
P1—C18—C23—C22 67.42 (18) C36—P2—C30—C35 139.62 (18)
P1—C18—C23—C22 67.42 (18) C36—P2—C30—C35 139.62 (18)
P1—C18—C23—C22 67.42 (18) C36—P2—C30—C35 139.62 (18)
P1—C18—C23—C22 67.42 (18) C36—P2—C30—C35 139.62 (18)
P1—C18—C23—C22 67.42 (18) C36—P2—C30—C35 139.62 (18)
P1—C18—C23—C22 67.42 (18) C36—P2—C30—C35 139.62 (18)

Hydrogen-bond geometry (Å, °)

| D—H···A | D—H | H···A | D···A | D—H···A |
|---------|-----|------|-------|---------|
| C9—H9···O5ii | 0.95 | 2.49 | 3.188 (3) | 130 |
| C39—H39···O2ii | 0.95 | 2.60 | 3.491 (4) | 157 |
| C20—H20···Cg1iv | 0.95 | 2.84 | 3.635 (3) | 142 |
| C34—H34···Cg3v | 0.95 | 2.90 | 3.683 (3) | 140 |

Symmetry codes: (ii) −x, −y+1, −z+1; (iii) −x, −y+1, −z+2; (iv) −x+1, −y+2, −z+1; (v) x, y−1, z.

Supplementary tables are available in the supporting information.