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Synthesis and structural characterization of rhenium(I) tricarbonyl complexes with the bidentate ligands *o*-(diphenylphosphino)benzaldehyde (P $\cap$ O) and *o*-[(diphenylphosphino)benzylidene]aniline (P $\cap$ N)

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Abstract

A series of rhenium(I) tricarbonyl complexes with bidentate P,O donor ligand *o*-(diphenylphosphino)benzaldehyde (P $\cap$ O) and its Schiff base P,N donor ligand *o*-[(diphenylphosphino)benzylidene]aniline (P $\cap$ N) have been synthesized and structurally characterized. All the complexes of the type [ReX(CO)₃(LL)] (where LL = P $\cap$ O, P $\cap$ N) reveal a distorted octahedral structure with the three carbonyl ligands arranged in the facial fashion. Crystal data for **1**, C₂₂H₁₅ClO₄Pre·1/2C₆H₁₄: triclinic, *P*1, *a*=8.7430(4), *b*=9.5767(4), *c*=13.9449(6) Å, α =93.651(1), β =101.265(1), γ =93.048(1); *V*=1140.20(9) Å³, *Z*=2.2, C₂₂H₁₅BrO₄Pre: monoclinic, *C*2/*c*, *a*=31.6800(16), *b*=8.8880(4), *c*=18.4517(9) Å, β =124.990(1); *V*=4256.4(4) Å³, *Z*=8.3, C₂₈H₂₀ClNO₃Pre: monoclinic, *C*2/*c*, *a*=22.675(4), *b*=8.803(2), *c*=28.218(5) Å, β =100.192(3); *V*=5543.5(16) Å³, *Z*=8.4, C₂₈H₂₀BrNO₃Pre: monoclinic, *C*2/*c*, *a*=23.035(1), *b*=8.7561(4), *c*=28.269(1) Å, β =100.811(1); *V*=5600.4(5) Å³, *Z*=8.

Keywords

Rhenium(I) complexes; Tricarbonyl complexes; X-ray crystal structure; Bidentate P, O donor ligand; P, N donor ligand

1. Introduction

The gamma-emitting radionuclide ^{99m}Tc and its Group VII congener ^{186/188}Re have found wide-ranging applications in radiodiagnosis [1–6] and radiotherapy [7,8], respectively. The optimal nuclear properties of ^{99m}Tc (γ -emitter, *E*_{max} = 140 keV, *t*_{1/2} = 6 h), as well as its convenient availability from ⁹⁹Mo/^{99m}Tc generator system [9], have made ^{99m}Tc radiopharmaceuticals virtually ideal for imaging low capacity high density receptors by deploying SPECT (single photon emission computerized tomography), while both isotopes ¹⁸⁶Re (*t*_{1/2} = 90.64 h, *E*_{max} = 1.1 MeV) and ¹⁸⁸Re (*t*_{1/2} = 17 h, *E*_{max} = 2.1 MeV) are suitable for therapeutic use by means of β -irradiation.

Almost all ^{99m}Tc and ^{186/188}Re radiopharmaceuticals research and development are focused on the [M=O]³⁺ (M=Tc and Re) core which forms very stable square pyramidal complexes with tetradentate N_xS_(4-x) chelators. The inevitable formation of multiple isomers differing

in their pharmacokinetics [10] aroused interest in developing an alternative '3+1' approach, in which a neutral oxometal complex in a distorted square pyramidal or trigonal bipyramidal molecular geometry was prepared by employing the mixed tridentate (SNS, ONS, ONO or SSS) ligand and monodentate thiol ligand with the appropriate MO^{3+} precursors [11–14]. These '3+1' integrated complexes eliminated the possibility of *syn* and *anti* stereoisomers that are often produced in the tetradentate complexes, but they were found to be relatively unstable in vitro and in vivo due to metabolism and replacement of the monothiol ligand through transchelation by physiological thiols such as cysteine and glutathione [15]. By replacing the monothiolate ligand with a bidentate [NO] or [PO] donor set, '3+2' six-coordinate oxometal complexes of the general formulation $[\text{ReO}(\eta^3\text{-L}^{\text{A}})(\eta^2\text{-L}^{\text{B}})]$, possessing a maximum electron count (MEC) of 18 rather than 16 which were always encountered in the case of '3+1' complexes, improved the kinetic stability substantially [16–18].

Despite the rich and diverse redox chemistry of technetium and rhenium, organometallic Tc/Re complexes in their low M(I) oxidation state received little attention until Davison and co-workers [19] discovered a very stable water-soluble hexakis (isonitrile) complex $[\text{}^{99\text{m}}\text{Tc}(\text{MIBI})_6]^+$, $^{99\text{m}}\text{Tc}$ -sestamibi, used as a myocardial perfusion agent. In an effort to develop new organometallic precursors for radiopharmaceutical applications, Alberto and co-workers developed a facile route to the important intermediate $[\text{M}(\text{H}_2\text{O})_3(\text{CO})_3]^+$ from the permethylate salt MO_4^- under atmospheric pressure of CO and exploiting a reducing agent such as NaBH_4 [20]. These complexes are water-soluble and readily undergo ligand exchange [21–23]. We have recently investigated biodistribution and gamma camera imaging studies of focal sites of *E. coli* infection in rabbits with the *fac* - $\text{M}(\text{CO})_3^+$ complex of a bipyridine derived chemotactic peptide (f-MLFK-bpy). The bidentate diimine and chloride ligands make the resultant molecule neutral in charge. It showed good in vitro and in vivo stability and high target-to-background (T/B) ratio, and was efficiently cleared from the blood pool through the urinary and hepatobiliary pathways [24]. A series of σ - and π -donors such as isonitrile, cyclopentadiene, aromatic nitrogen heterocycles and thioethers have been shown to easily react with $[\text{M}(\text{H}_2\text{O})_3(\text{CO})_3]^+$ to yield stable complexes. To further explore the coordination behavior of the $\{\text{MX}(\text{CO})_3\}^+$ core with bidentate ligands, we report herein the synthesis and x-ray crystal structure characterization of the tricarbonylrhenium(I) halide complexes of the bidentate P,O donor ligand *o*-(diphenylphosphino)benzaldehyde (PNO) and its Schiff base P,N donor ligand *o*-(diphenylphosphino)benzylidene]aniline (PNN).

2. Experimental

2.1. General considerations

NMR spectra were recorded on a Bruker DPX 300 (^1H 300.10 MHz) spectrometer in CDCl_3 (δ 7.27 ppm). IR spectra were recorded as KBr pellets with a Perkin–Elmer Paragon 1000 FTIR. Elemental analysis for carbon, hydrogen and nitrogen were carried out by Oneida Research Services, Whitesboro, NY. Reagent grade solvents were used for all preparations, $[\text{ReCl}(\text{CO})_5]$, $[\text{ReBr}(\text{CO})_5]$ and *o*-(diphenylphosphino)benzaldehyde were obtained from the Aldrich Chemical Company.

2.2. Synthesis

2.2.1. Preparation of Schiff base ligand *o*-(diphenylphosphino)benzylidene]aniline (PNN)

***o*-(diphenylphosphino)benzylidene]aniline (PNN)**—The bidentate P,N donor ligand *o*-(diphenylphosphino)benzylidene]aniline (PNN) was prepared via a Schiff base reaction [25] carried out under a nitrogen atmosphere. *o*-(diphenylphosphino)benzaldehyde (1.74 g, 6 mmol) was added to aniline (1 cm³) in methanol (30 cm³). The resulting mixture was refluxed for 3 h. The solution was cooled to room temperature (r.t.) and the crude product

was obtained as powder by removing the unreacted analine. The pure ligand was obtained upon recrystallization of this yellow powder from degassed hot ethanol. Yield: 1.42 g, 65%. ^1H NMR (CDCl_3 , δ ppm): 10.51 (d, 1H, $\text{CH}=\text{N}$, $J=5.1$ Hz), 8.47 (1H, m, ArH), 7.43 (2H, t, ArH), 7.35–6.85 (m, 16H, ArH). *Anal.* Calc. For $\text{C}_{25}\text{H}_{20}\text{NP}$: C, 82.19; H, 5.48; N, 3.84. Found C, 82.03; H, 5.39; N, 3.90%.

2.2.2. General synthesis procedure for the complexes $[\text{ReX}(\text{CO})_3(\text{P} \cap \text{O})]$ —The bidentate P,O donor ligand *o*-(diphenylphosphino)benzaldehyde ($\text{P} \cap \text{O}$) (29 mg, 0.1 mmol) dissolved in methanol (10 cm^3) was added to a stirred solution of $[\text{ReX}(\text{CO})_5]$ (where $\text{X}=\text{Cl}$ or Br) in methanol (30 cm^3). After stirring the reaction mixture at refluxing temperature for 4 h, the solvent was removed to dryness in a vacuum. The remaining residue was taken up in dry chloroform and *n*-hexane was added to precipitate the reddish product. X-ray quality crystals were grown by slow diffusion of *n*-hexane into a solution of the compound dissolved in minimum amount of CH_2Cl_2 .

2.2.2.1. $[\text{ReCl}(\text{CO})_3(\text{P} \cap \text{O})] \cdot 1/2 \text{C}_6\text{H}_{14}$ (1): Yield: 48 mg, 75%. FTIR (cm^{-1} , KBr pellet): 2039, 1938, 1935 ($\text{C}\equiv\text{O}$), 1643 ($\text{C}=\text{O}$), 1483, 1435 ($\text{C}-\text{P}$). ^1H NMR (CDCl_3 , ppm): 9.78 (d, 1H, $\text{CH}=\text{O}$, $J=5.1$ Hz), 8.10–8.00 (m, 1H, ArH), 7.9–7.7 (m, 3H, ArH), 7.55–7.40 (m, 7 H, ArH), 7.3–7.2 (m, 1 H, ArH), 7.15–7.05 (m, 2H, ArH). *Anal.* Calc. for $\text{C}_{25}\text{H}_{22}\text{ClO}_4\text{Pre}$: C, 46.99; H, 3.45. Found: C, 46.81; H, 3.53%.

2.2.2.2. $[\text{ReBr}(\text{CO})_3(\text{P} \cap \text{O})]$ (2): Yield 40 mg, 62%. FTIR (cm^{-1} , KBr pellet): 2035, 1941, 1938 ($\text{C}\equiv\text{O}$), 1647 ($\text{C}=\text{O}$), 1483, 1437 ($\text{C}-\text{P}$). ^1H NMR (CDCl_3 , ppm): 9.80 (d, 1H, $\text{CH}=\text{O}$, $J=5.4$ Hz), 8.10–7.10 (m, 14H, ArH). *Anal.* Calc. for $\text{C}_{22}\text{H}_{15}\text{BrO}_4\text{Pre}$: C, 41.25; H, 2.34. Found: C, 41.40; H, 2.39%.

2.2.3. General synthesis procedure for the complexes $[\text{ReX}(\text{CO})_3(\text{P} \cap \text{N})]$ —In a typical experiment, the appropriate pentacarbonylrhenium(I) halide (0.1 mmol) was refluxed for ca. 6 hours with a slight excess of the Schiff base ligand *o*-[(diphenylphosphino)benzylidene]analine ($\text{P} \cap \text{N}$) in 30 cm^3 of methanol. The solvent was then removed in vacuo. The solid residue was crystallized from CH_2Cl_2 –pentane solution at -20°C to yield pure, yellow crystalline $[\text{ReX}(\text{CO})_3(\text{P} \cap \text{N})]$.

2.2.3.1. $[\text{ReCl}(\text{CO})_3(\text{P} \cap \text{N})]$ (3): Yield: 40 mg, 60%. FTIR (cm^{-1} , KBr pellet): 2021, 1930, 1926 ($\text{C}\equiv\text{O}$), 1589 ($\text{C}=\text{N}$), 1483, 1434 ($\text{C}-\text{P}$). ^1H NMR (CDCl_3 , ppm): 8.36 (s, 1H, $\text{CH}=\text{N}$), 7.96–7.85 (m, 1H, ArH), 7.65–7.45 (m, 11H, ArH), 7.40–7.15 (m, 5H, ArH), 7.19 (t, 1H, ArH), 6.95 (dd, 1 H, ArH). *Anal.* Calc. for $\text{C}_{28}\text{H}_{20}\text{ClNO}_3\text{Pre}$: C, 50.11; H, 2.98; N, 2.09. Found: C, 50.28; H, 3.04; N, 2.01%.

2.2.3.2. $[\text{ReBr}(\text{CO})_3(\text{P} \cap \text{N})]$ (4): Yield: 35 mg, 49%. FTIR (cm^{-1} , KBr pellet): 2024, 1925, 1921 ($\text{C}\equiv\text{O}$), 1594 ($\text{C}=\text{N}$), 1484, 1437 ($\text{C}-\text{P}$). ^1H NMR (CDCl_3 , ppm): 8.39 (s, 1H, $\text{CH}=\text{N}$), 7.90–7.00 (m, 19H, ArH). *Anal.* Calc. for $\text{C}_{28}\text{H}_{20}\text{BrNO}_3\text{Pre}$: C, 46.99; H 2.80; N, 1.96. Found: C, 47.13; H, 2.75; N, 1.91%.

2.3. X-ray crystallography

The selected crystals of **1–4** were studied on a Bruker diffractometer equipped with the SMART CCD system [26] and using graphite-monochromated Mo- $\text{K}\alpha$ radiation ($\lambda=0.71073 \text{ \AA}$). All the data collections were carried out at $295(5) \text{ K}$. The data were corrected for Lorentz and polarization effects, and absorption corrections were made using SADABS [27]. Neutral atom scattering factors were taken from Cromer and Waber [28]. And anomalous dispersion corrections were taken from those of Creagh and McAuley [29]. All calculations were performed using SHELXL [30]. The structures were solved by direct methods [31] and all of

the non-hydrogen atoms were located from the initial solution. After locating all the initial nonhydrogen atoms in each structure, the models were refined against F^2 , initially using isotropic and later anisotropic thermal displacement parameters until the final value of $\Delta/\sigma_{\max}$ was less than 0.001. At this point the hydrogen atoms were located from the electron density difference map and a final cycle of refinements was performed, until the final value of $\Delta/\sigma_{\max}$ was again less than 0.001. No anomalies were encountered in the refinement of any of the structures. The relevant parameters for crystal data, data collection, structure solution and refinement are summarized in Table 1, and important bond lengths and angles in Table 2 and Table 3. A complete description of the details of the crystallographic methods is given in supplementary material.

3. Results and discussion

The rhenium(I) tricarbonyl complexes **1–4** were prepared by reaction of $[\text{ReX}(\text{CO})_5]$ with the bidentate, P,O donor ligand *o*-(diphenylphosphino)benzaldehyde (PNO) or its Schiff base ligand *o*-[(diphenylphosphino)benzylidene]aniline (PNN) in methanol using a method analogous to that reported previously [32]. Complexes $[\text{ReX}(\text{CO})_3(\text{PNO})]$ (**1**, X=Cl; **2**, X=Br) were isolated as air-stable red–brown needles while complexes $[\text{ReX}(\text{CO})_3(\text{PNN})]$ (**3**, X=Cl; **4**, X=Br) were separated as yellowish blocks. In the rhenium(I) carbonyl stretching region of the infrared spectra, the complexes show three strong peaks consistent with a facial arrangement of the three carbonyl groups [33,34], which are assigned as the A'(1), A'' and A'(2) vibrations, respectively [35]. The IR spectra of the rhenium(I) aldehyde phosphine complexes **1** and **2** show a single strong band at ca. 1645 cm^{-1} attributable to the ligand $\nu(\text{C}=\text{O})$ vibrations. This stretching band is shifted to lower frequencies than in the free ligand PNO. The presence of the iminophosphine ligand coordinated to Re(I) in complexes **3** and **4** is detected by a strong band at ca. 1590 cm^{-1} attributed to the $\text{C}=\text{N}$ stretching vibration, which is also red-shifted with comparison to the free Schiff base ligand PNN [36]. The typical *fac*-tricarbonyl unit in complexes **3** and **4** is evidenced by the CO stretching frequencies as detected in the IR spectra and is confirmed by the X-ray crystal structures. In the case of iminophosphine complexes, the CO stretching bands were observed at ca. 2020, 1930 and 1925 cm^{-1} , which are significantly lower than in the corresponding phosphine aldehyde complexes (ca. 2035, 1940 and 1935 cm^{-1}). This can undoubtedly be explained by the stronger metal-to-carbonyl back-bonding in the imine phosphine complexes [37]. The appreciable coupling to the phosphorus atom [38] exhibited by the aldehyde proton (complexes **1** and **2**) and imine proton (complexes **3** and **4**) expected as doublets) is the most remarkable aspect of the ^1H NMR spectra.

Selected bond distances and angles for complexes **1** and **2** are given in Table 2. A perspective view of complex **1** with the atom numbering is given in Fig. 1. The coordination geometry at the Re atom is distorted octahedral with three carbonyl ligands arranged in the facial fashion. The *trans* angles at the Re(I) site are in the range of $171.1(2)–178.1(2)^\circ$, showing a slight deviation from an ideal octahedral arrangement. In complexes **1** and **2**, the PNO ligand forms a six-membered chelate ring with the Re(I) center. The O(1)–Re(1)–P(1) bite angle of $85.7(1)^\circ$ for complex **1** and $82.3(1)^\circ$ for complex **2** only slightly deviate from the expected ideal 90° for an octahedron. The Re–C linkage *trans* to the Re–P linkage is significantly longer than that *trans* to the Re–Cl and Re–O linkages; 1.966(4) versus 1.909(4) and 1.904(4) Å for complex **1**, and 1.945(5) versus 1.887(5) and 1.910(5) Å in complex **2**, respectively. This indicates stronger *trans* influence of the P donor atom toward the carbonyl C atom than halide and oxygen donors. The torsion angles of 4.2° for P(1)–C(1)–C(6)–C(7) and -20.8° for Re(1)–P(1)–C(1)–C(6) units point to a perturbed envelope conformation of the six membered ring chelate.

Selected bond distances and angles for complexes **3** and **4** are given in Table 3. An ORTEP view of the complex **3** with the atom numbering is given in Fig. 2. The metal center has an almost ideal octahedral coordination sphere with a facial arrangement of three carbonyl groups. The atoms P(1), N(1), C(26), C(28) and Re(1) lie on the same plane within experimental error. The N(1)–Re(1)–P(1) angle of 83.3(2)° is significantly smaller than 90°, as a result of the small bite angle between imine group and phosphine group. Due to the stronger π -backbonding induced by the coordinated phosphine P atom in the *trans* position, the Re(1)–C(26) bond length (1.948(10) Å) is longer than the Re–C bonds *trans* to halide and imine nitrogen atoms (1.889(11) and 1.926(10) Å). The C(7)–N(1) distance is 1.288(11) Å for complex **3** and 1.304(11) Å for complex **4**, this value is slightly longer than that observed for the C=N bond in normal Schiff base ligands.

4. Conclusions

The compound [Re(CO)₅X] provides a useful starting material for the development of the chemistry of {Re(CO)₃}⁺ core compounds. Bidentate ligands react readily with the precursor to give complexes of the type [Re(CO)₃(LL)X] (where LL = bidentate ligand), which may be further elaborated for potential radiopharmaceutical applications.

5. Supplementary material

All atomic and thermal parameters and all interatomic angles are available from the author upon request. Crystallographic data (excluding structure factors) for the structure reported in this paper have been deposited with the Cambridge Crystallographic Data Center as publication no. CCDC 152 368 to CCDC 152 371. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB21EQ, UK. Fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk or www.ccdc.cam.ac.uk

Supplementary Material

Refer to Web version on PubMed Central for supplementary material.

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Fig. 1. Perspective view of the complex $[\text{ReCl}(\text{CO})_3(\text{P}(\text{O})\text{Ph}_2)]$ (**1**): ORTEP drawing with 50% probability ellipsoids.

Fig. 2. Perspective view of the complex $[\text{ReCl}(\text{CO})_3(\text{P N})]$ (3): ORTEP drawing with 30% probability ellipsoids.

Table 1

Summary of crystal data, intensity, collection, and refinement data^a for complexes [ReCl(CO)₃(P ∩ O)] · 1/2C₆H₁₄ (**1**), [ReBr(CO)₃(P ∩ O)] (**2**), [ReCl(CO)₃(P ∩ N)] (**3**), and [ReBr(CO)₃(P ∩ N)] (**4**)

	1	2	3	4
Empirical formula	C ₂₂ H ₁₅ ClO ₄ Pre·1/2C ₆ H ₄	C ₂₂ H ₁₅ BrO ₄ Pre	C ₂₈ H ₂₀ ClNO ₃ Pre	C ₂₈ H ₂₀ BrNO ₃ Pre
Formula weight	639.05	640.42	671.07	715.53
Crystal size (mm)	0.2×0.2×0.2	0.2×0.1×0.1	0.2×0.2×0.15	0.15×0.15×0.2
Crystal system	Triclinic	Monoclinic	Monoclinic	Monoclinic
Space group	<i>P</i> 1	<i>C</i> 2/ <i>c</i>	<i>C</i> 2/ <i>c</i>	<i>C</i> 2/ <i>c</i>
<i>a</i> (Å)	8.7430(4)	31.6800(16)	22.675(4)	23.0349(12)
<i>b</i> (Å)	9.5767(4)	8.8880(4)	8.803(2)	8.7561(4)
<i>c</i> (Å)	13.9449(6)	18.4517(9)	28.218(5)	28.2685(14)
<i>α</i> (°)	93.651(1)			
<i>β</i> (°)	101.265(1)	124.990(1)	100.192(3)	100.811(1)
<i>γ</i> (°)	93.048(1)			
<i>V</i> (Å ³)	1140.20(9)	4256.4(4)	5543(2)	5600.4(5)
<i>Z</i>	2	8	8	8
<i>D</i> _{calc} (g cm ⁻³)	1.861	1.999	1.608	1.697
<i>F</i> (000)	622	2432	2608	2752
<i>μ</i> (mm ⁻¹)	5.546	7.688	4.566	5.851
Final indices (2σ data), <i>R</i> ₁ (<i>wR</i> ₂)	0.0282 (0.0772)	0.0325 (0.0673)	0.0505 (0.1790)	0.0514 (0.1707)
All data, <i>R</i> ₁ (<i>wR</i> ₂)	0.0298 (0.0781)	0.0508 (0.0727)	0.0779 (0.1950)	0.0693 (0.1793)

^aWhere $R_1 = \Sigma(|F_o| - |F_c|) / \Sigma F_o$, $wR_2 = [\sum [w(F_o^2 - F_c^2)^2] / \sum [w(F_o^2)^2]]^{1/2}$

Table 2Selected bond distances (Å) and angles (°) for complexes **1** and **2**

	1	2
Re(1)–C(20)	1.909(4)	1.887(5)
Re(1)–C(21)	1.904(4)	1.910(5)
Re(1)–C(22)	1.966(4)	1.945(5)
Re(1)–O(1)	2.171(3)	2.184(4)
Re(1)–P(1)	2.439(1)	2.449(1)
Re(1)–X	2.472(1)	2.620(1)
O(1)–C(7)	1.230(5)	1.239(5)
C(21)–Re(1)–C(20)	90.2(2)	90.9(2)
C(21)–Re(1)–C(22)	91.7(2)	90.8(2)
C(20)–Re(1)–C(22)	89.2(2)	88.5(2)
C(20)–Re(1)–O(1)	91.0(2)	92.1(2)
C(21)–Re(1)–O(1)	175.8(1)	176.9(2)
C(22)–Re(1)–O(1)	92.4(1)	89.0(2)
C(20)–Re(1)–P(1)	91.4(1)	93.5(2)
C(21)–Re(1)–P(1)	90.2(1)	97.8(2)
C(22)–Re(1)–P(1)	178.1(1)	171.1(2)
O(1)–Re(1)–P(1)	85.7(1)	82.3(1)
C(20)–Re(1)–X	172.3(1)	176.2(2)
C(21)–Re(1)–X	97.4(1)	92.1(2)
C(22)–Re(1)–X	89.4(1)	89.1(1)
O(1)–Re(1)–X	81.6(1)	82.3(1)
P(1)–Re(1)–X	89.8(1)	88.4(1)

Table 3Selected bond distances (Å) and angles (°) for complexes **3** and **4**

	3	4
Re(1)–C(26)	1.948(10)	1.949(10)
Re(1)–C(27)	1.889(11)	1.894(10)
Re(1)–C(28)	1.926(10)	1.885(8)
Re(1)–N(1)	2.238(7)	2.193(7)
Re(1)–P(1)	2.446(2)	2.441(2)
Re(1)–X	2.489(2)	2.628(1)
N(1)–C(7)	1.288(11)	1.304(11)
C(27)–Re(1)–C(26)	90.5(4)	89.9(4)
C(27)–Re(1)–C(28)	86.4(5)	89.2(4)
C(26)–Re(1)–C(28)	89.8(5)	88.9(4)
C(26)–Re(1)–N(1)	93.5(4)	94.3(4)
C(27)–Re(1)–N(1)	95.1(4)	94.7(4)
C(28)–Re(1)–N(1)	176.4(4)	176.3(3)
C(26)–Re(1)–P(1)	175.6(3)	175.0(3)
C(27)–Re(1)–P(1)	92.8(3)	93.7(3)
C(28)–Re(1)–P(1)	93.3(3)	94.8(3)
C(26)–Re(1)–X	89.4(3)	87.9(3)
C(27)–Re(1)–X	179.8(4)	177.8(3)
C(28)–Re(1)–X	93.8(3)	92.8(3)
N(1)–Re(1)–X	84.7(2)	85.4(2)
P(1)–Re(1)–X	87.3(1)	88.5(1)