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Note

# Technetium and rhenium heterocomplexes containing the diphenylphosphinoferrocenyl fragment

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**Abstract**

Reactions of dppf with labile technetium and rhenium precursors such as $[M(N)Cl_4]^-$ and $[M(NPh)Cl_3(PPh_3)_2]$ yield the monomeric mono-substituted $[M(N)Cl_2(dppf)]$ and $[M(NPh)Cl_3(dppf)]$ complexes, where the diphosphinoferrocenyl fragment coordinates on the equatorial plane of a distorted square pyramid or of a distorted octahedron, respectively. © 2001 Elsevier Science B.V. All rights reserved.

*Keywords:* Technetium; Rhenium; Nitrido complexes; Mixed ligand complexes; Diphenylphosphinoferrocene; Crystal structures

## 1. Introduction

Radiopharmaceuticals containing the γ-emitting isotope $^{99m}$Tc continue to occupy a prominent role in diagnostic nuclear medicine owing to the favorable nuclear properties ($E_\gamma = 142$ keV, $t_{1/2} = 6.02$ h) and the availability of this radionuclide. In the last two decades, dramatic advancements of the studies of the basic chemistry of technetium complexes have led to the introduction of the clinical application of useful tracers for heart, brain and kidneys [1–3]. More recently, the advent of the two β-emitters $^{186}$Re ($t_{1/2} = 3.8$ day, $E_{\beta max} = 1.07$ MeV) and $^{188}$Re ($t_{1/2} = 0.7$ day, $E_{\beta max} = 2.11$ MeV) in the therapeutic field [4,5] has made the third-row congener rhenium studies attractive. Synthesis at the macroscopic level (millimolar concentration or 'carrier added', CA), devoted at the elucidation of the molecular structure of the agents injected in vivo at microscopic level (nanomolar concentrations or 'non-carrier added' NCA), are performed by utilizing the $^{99g}$Tc isotope (a soft β-emitter with $t_{1/2} = 2.12 \times 10^5$ year, $E_\beta = 292$ keV) and the 'cold' natural isotopic mixture of $^{185}$Re and $^{187}$Re.

In the 1990s a new concept has emerged in the design of substitution-inert technetium and rhenium agents. Thus, an essential requirement is generating a robust 'metal-fragment', to which small bifunctional ligands bearing specific pharmacophores can be eventually coordinated. The metal-fragment contains the ion stabilized in an appropriate oxidation state by means of a suitable ligand framework, which only partially fills the coordination sphere. Examples of application of this strategy include the recently developed low-valent Tc-tricarbonyl $[Tc^I(CO)_3]^+$ [6] and the high-valent 'super-nitrido' $[Tc^V(N)(PNP)]^{2+}$ [7] (PNP = tridentate aminediphosphine ligand) fragments. On the opposite side of the molecule, these substitution-inert moieties accommodate labile ligands, i.e. water molecules and/or related hydroxyl functions or halide groups. These

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monodentate ligands can be readily replaced by the incoming small chelate ligands, either bidentate or tridentate to remain with the examples detailed above, which may balance the positive charge of the moiety and fill the resulting coordination sphere completely.

Looking for additional stable metal-fragments, we have been exploring new chelates capable of stabilizing the nitrido and the imido groups. Phosphorus containing ligands are among the best donors for technetium and rhenium species characterized by the presence of multiple metal–nitrogen bonds, as demonstrated earlier in the case of the above-mentioned 'super-nitrido' fragment, or in the so-called 'HYNIC' compounds (HYNIC means hydrazinonicotinamine) [8,9], containing the [Tc = NNR] diazenido core.

According to the sufficiently large bite generated by the potentially bidentate diphenylphosphine-ferrocene (dppf) ligand [10], we thought of testing the reactivity of dppf towards substitution-labile precursors bearing the $[M{\equiv}N]^{2+}$ or $[M{=}NR]^{3+}$ groups (M = Tc, Re). In this study we report the synthesis and characterization of four novel hetero bi-metallic complexes, including the X-ray molecular structure of the representative compound $[Re(N)Cl_2(dppf)]$, **3**, which combine distinctive technetium and rhenium cores with the ferrocenyl diphosphine fragment.

## 2. Experimental

### 2.1. General

All the chemicals were of reagent grade and used as such without prior purification. The synthesis of 1,1′-bis(diphenylphosphino)ferrocene (dppf) was carried out according to the protocols published in Ref. [10]. Rhenium was purchased from Aldrich as $KReO_4$ and converted first to the labile precursors $[n\text{-}Bu_4N][Re(N)Cl_4]$ [11] and $[Re(NPh)Cl_3(PPh_3)_2]$ [12] according to the protocols published. Technetium starting complexes $[Ph_4As][Tc(N)Cl_4]$ [13] and $[Tc(NPh)Cl_3(PPh_3)_2]$ [14] were prepared by following the methods outlined in the literature. Proton and $^{31}$P-NMR spectra were recorded on a Bruker AMX-300 instrument, using $SiMe_4$ as the internal reference (for $^1$H) and 85% aq. $H_3PO_4$ as the external reference (for $^{31}$P). Samples were dissolved in $CDCl_3$ at a concentration of ca. 1–2%. Elemental analyses for C, H, and N were carried out on a Fisons EA1108 elemental analyzer.

**Caution!** $^{99g}$Tc is a weak β-emitter ($E_\beta = 0.292$ MeV, $t_{1/2} = 2.12 \times 10^5$ year). All manipulations were carried out in laboratories approved for low-level radioactivity using monitored hoods and gloveboxes. When handled in milligram amounts, $^{99g}$Tc does not present a serious health hazard as common laboratory glassware provides adequate shielding. Bremsstrahlung is not a significant problem caused by the low-energy of the β-particles. However, normal radiation safety procedures must be used at all times, especially with solid samples, to prevent contamination and inhalation.

### 2.2. Synthesis of technetium complexes

#### 2.2.1. *[Tc(N)Cl$_2$(dppf)]* (**1**)

To a suspension containing $[Ph_4As][Tc(N)Cl_4]$ (40.0 mg, 0.063 mmol) in benzene (5 ml) solid dppf (35.0 mg, 0.063 mmol) was added with continuous stirring. The orange mixture was then refluxed under a dinitrogen atmosphere. After 2 h the color turned yellow–orange with an appearance of a solid. The mixture was filtered, and the pale-orange solid was washed with benzene (3 × 1 ml), diethylether (10 ml) and dried under a dinitrogen stream (Yield: 24 mg (65%)). The solid is soluble in MeCN, benzene and chlorinated solvents, insoluble in alcohols and $Et_2O$. Anal. Found: C, 55.45; H, 3.67; N, 2.01. Calc. for $C_{34}H_{28}NP_2Cl_2FeTc$: C, 55.31; H, 3.82; N, 1.90%. $^1$H-NMR (300 MHz, $CDCl_3$, $\delta$ ppm): 4.38, 4.56, 4.64, 4.85 (m, 2H each, Cp*H*), 7.15–7.97 (20H, P*Ph*). $^{31}$P-NMR (300 MHz, $CDCl_3$, $\delta$ ppm): 47.2 (bs, $\gamma_{1/2} = 340$ Hz).

#### 2.2.2. *[Tc(NPh)Cl$_3$(dppf)]* (**2**)

To a suspension of $[Tc(NPh)Cl_3(PPh_3)_2]$ (27.0 mg, 0.032 mmol) in benzene–$CH_2Cl_2$ (4:1, ml/ml) solid dppf (18.0 mg, 0.032 mmol) was added with continuous stirring. The light-green mixture was then refluxed for 2 h under a nitrogen atmosphere. The color turned green–brown and a solid appeared with time. The mixture was filtered, and the green solid was washed with MeOH (3 × 1 ml), $Et_2O$ (10 ml) and dried under a dinitrogen stream (Yield: 19 mg (85%)). The solid is soluble in chlorinated solvents, insoluble in MeCN, alcohols and $Et_2O$. Anal. Found: C, 56.72; H, 3.77; N, 1.75. Calc. for $C_{40}H_{33}NP_2Cl_3FeTc$: C, 56.46; H, 3.90; N, 1.64%. $^1$H-NMR (300 MHz, $CDCl_3$, $\delta$ ppm): 4.61 (m, 2H), 4.66 (m, 4H), 5.35 (m, 2H) [Cp*H*], 6.78 (t, 2H-β), 7.14 (dd, 2H-α), 7.42 (t, 1H-γ) [N*Ph*], 7.02 (m, 6H-α/γ), 7.83 (t, 4H-β) [P*Ph-anti*], 7.32 (m, 6H-α/γ), 8.03 (t, 4H-β) [P*Ph-syn*]. $^{31}$P-NMR (300 MHz, $CDCl_3$, $\delta$ ppm): 22.4 (bs, $\gamma_{1/2} = 1100$ Hz).

### 2.3. Synthesis of rhenium complexes

Rhenium compounds were prepared as detailed above for the technetium analogs starting from $[n\text{-}Bu_4N][Re(N)Cl_4]$ and $[Re(NPh)Cl_3(PPh_3)_2]$, respectively. Spectroscopic data are reported below.

#### 2.3.1. *[Re(N)Cl$_2$(dppf)]* (**3**)

Anal. Found: C, 49.05; H, 3.76; N, 1.81. Calc. for $C_{34}H_{28}NP_2Cl_2FeRe$: C, 49.47; H, 3.82; N, 1.70%. $^1$H-NMR (300 MHz, $CDCl_3$, $\delta$ ppm): 4.40 (m, 2H), 4.57

(m, 2H), 4.80 (m, 4H) [Cp*H*], 7.18–7.86 (20H, P*Ph*). $^{31}$P-NMR (300 MHz, $CDCl_3$, $\delta$ ppm): 27.7 (s).

### 2.3.2. [Re(NPh)Cl$_3$(dppf)] (4)

Anal. Found: C, 52.01; H, 3.45; N, 1.50. Calc. for $C_{28}H_{25}NP_2Cl_3FeRe$: C, 51.21; H, 3.54; N, 1.49%. $^1$H-NMR (300 MHz, $CDCl_3$, $\delta$ ppm): 4.59, 4.64, 4.68, 5.27 (m, 2H each; Cp*H*), 6.79 (t, 2H-β), 7.11 (dd, 2H-α), 7.40 (t, 1H-γ) [N*Ph*], 6.68 (d, 2H-α′), 7.02 (m, 4H-α/γ), 7.83 (t, 4H-β) [P*Ph-anti*], 7.33 (m, 6H-α/γ), 8.00 (t, 4H-β) [P*Ph-syn*]. $^{31}$P-NMR (300 MHz, $CDCl_3$, $\delta$ ppm): −17.1 (s).

Table 1
Crystal data for compound **3**

| | |
|---|---|
| Empirical formula | $C_{34}H_{28}NP_2Cl_2FeRe$ |
| Formula weight | 825.46 |
| Temperature (K) | 293(2) |
| Wavelength (Å) | 0.71073 |
| Crystal system | Monoclinic |
| Space group | $P2_1/c$ |
| Unit cell dimensions | |
| $a$ (Å) | 9.885(4) |
| $b$ (Å) | 18.347(7) |
| $c$ (Å) | 17.321(7) |
| $\beta$ (°) | 97.38 |
| $V$ (Å$^3$) | 3115(2) |
| $Z$ | 4 |
| $\rho_{calcd}$ (g cm$^{-3}$) | 1.760 |
| Absorption coefficient (mm$^{-1}$) | 4.65 |
| $F$(000) | 1616 |
| Max/min transmission | 0.714, 0.563 |
| Theta range for data collection, $\Theta$ (°) | 2.2–25.1 |
| Observed reflections [$I \geq 2\sigma(I)$] | 4142 |
| Independent reflections | 4538 |
| Final $R$ indices [$I \geq 2\sigma(I)$] | $R_1$[a] = 0.038 |
| | $wR_2$[b] = 0.094 |
| Goodness-of-fit on $F^2$ | 1.056 |

[a] $R_1 = \Sigma| |F_o| - |F_c| | / \Sigma |F_o|$.
[b] $wR_2 = \{\Sigma[w(F_o^2 - F_c^2)^2] / \Sigma[w(F_o^2)^2]\}^{1/2}$.

Table 2
Selected bond lengths (Å) and bond angles (°) for compound **3**

| | | | |
|---|---|---|---|
| *Bond lengths* | | | |
| Re–N | 1.603(6) | Re–Cl(1) | 2.417(2) |
| Re–P(1) | 2.433(2) | Re–Cl(2) | 2.409(2) |
| Re–P(2) | 2.422(2) | Fe–C$_{average}$ | 2.071(5) |
| P(1)–C(1) | 1.803(6) | P(2)–C(6) | 1.822(6) |
| P(1)–C(1A) | 1.835(8) | P(2)–C(1C) | 1.832(6) |
| P(1)–C(1B) | 1.842(6) | P(2)–C(1D) | 1.858(6) |
| *Bond angles* | | | |
| N–Re–Cl(1) | 110.6(2) | Cl(1)–Re–P(2) | 149.6(1) |
| N–Re–Cl(2) | 111.3(2) | Cl(2)–Re–P(1) | 151.6(1) |
| N–Re–P(1) | 96.7(2) | Cl(2)–Re–P(2) | 83.2(1) |
| N–Re–P(2) | 99.4(2) | P(1)–Re–P(2) | 97.1(1) |
| Cl(1)–Re–Cl(2) | 81.7(1) | Re–P(1)–C(1) | 106.3(2) |
| Cl(1)–Re–P(1) | 84.4(1) | Re–P(2)–C(6) | 107.1(2) |

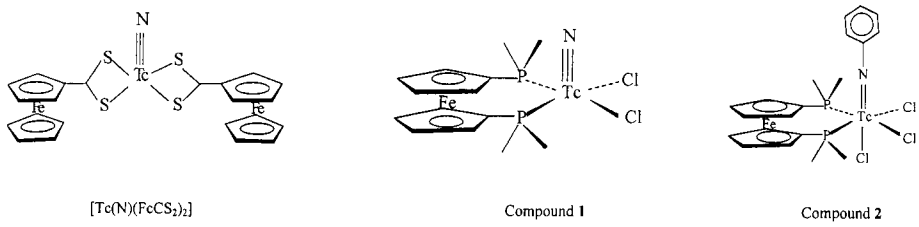
### 2.4. Crystallographic structure determination of [Re(N)Cl$_2$(dppf)] (3)

Bright orange crystals of **3** were grown from $CH_2Cl_2$–MeOH solutions. Data were collected by using a Bruker Siemens R3m/V diffractometer. Crystal data and refinement parameters are summarized in Table 1. Selected interatomic distances and angles are cumulated in Table 2. The structure was solved by heavy-atom methods, completed by subsequent Fourier syntheses, and refined on $F^2$ with full-matrix least-squares methods. All non-hydrogen atoms were refined with anisotropic displacement coefficients and all hydrogen atoms were treated as idealized contributions. Features of the final difference map showed peaks (up to 1.01 e Å$^{-3}$) in chemically unreasonable positions and were considered to be noise. All scattering factors and anomalous dispersion coefficients are contained in the SHELXTL-NT 5.10 program library [15].

## 3. Results and discussion

Despite the rich chemistry exhibited by most transition metals with ferrocenyl ligands incorporating a variety of donor atoms [16], mixed technetium–iron complexes have appeared only recently in the literature [17]. According to the well-established ferrocene/ferrocenium reversible one-electron couple, the incorporation of such moiety through appropriate coordinating group onto Tc and Re centers would produce species showing single or multiple electron exchange at suitable potentials. Redox active complexes of technetium have been demonstrated to show specific in vivo biodistribution [18] because of their participation in some biological processes involving the transfer of one or more electrons. Therefore, studies aiming at the synthesis of mixed technetium–iron or rhenium–iron compounds may be relevant to nuclear medicine, both for diagnostic (Tc) and therapeutic (Re) purposes. In this connection, we have recently prepared the di-substituted nitrido technetium complex [Tc(N)(FcCS$_2$)$_2$] [17] outlined in Scheme 1. Although this species exhibits an interesting redox behavior, no significant biological data were recovered when this agent, prepared at NCA level, was injected in vivo [19].

These results prompted us to introduce the ferrocene unit into technetium and rhenium complexes by using a different donor group like phosphine, as in dppf. Examples of low-valent mixed rhenium carbonyl–dppf complexes appeared in the literature mostly in the beginning of the 1990s [20–25]. In these compounds, the diphosphine ligand acts as mono-dentate, μ-bridging and/or bidentate ligand. The large bite generated in the latter coordination mode could promote the formation of novel metal-fragments of the type




Scheme 1.

$[M(N)(dppf)]^{2+}$ and $[M(NR)(dppf)]^{3+}$. In fact, reactions of labile precursors such as $[M(N)Cl_4]^-$ or $[M(NPh)Cl_3(PPh_3)_2]$ yield the monomeric mono-substituted complexes $[M(N)Cl_2(dppf)]$ (**1**, **3**) and $[M(NPh)Cl_3(dppf)]$ (**2**, **4**). Elemental analyses and spectroscopic data, as reported in Section 2, are in agreement with the proposed formulation, which is further supported by the X-ray structural determination of the representative complex **3**. The diffractometric analysis shows a five-coordinate structure (Fig. 1) realizing a square-pyramidal environment (trigonality index of 0.03 [26]), with the nitrido group at the apex and the remaining $P_2Cl_2$ donors at the base of the pyramid. The Re atom is displaced by 0.59 Å from the mean equatorial plane toward the nitrido nitrogen and the four basal donors are displaced by only $\pm 0.02$ Å. The dppf acts as $\eta^2$-diphoshine ligand that represents its preferred coordination mode. In fact, more than 70% of the mononuclear and polynuclear dppf containing complexes exhibit the $\eta^2$-coordination mode, as retrieved from a search through the CCDB [27]. The twist angle $\tau$, defined as the C(1)···$X_A$···$X_B$···C(6) torsion angle ($X_A$ is the Cp ring 'A' centroid and $X_B$ is the Cp ring 'B' centroid), is 3.2°. Thus, this structure shows dppf in the '*syn*-periplanar eclipsed' conformation with $\tau$ confined between 0 and 18°, while in the other structures the '*syn*-clinal staggered' arrangement ($\tau$ between 18 and 54°) is largely preferred [16]. The two Cp rings are virtually parallel (dihedral angle between the mean planes of 6.2°) and the $X_A$···Fe···$X_B$ angle is 174.7°. Bond lengths and bond angles (Table 2) do not show any unusual features [27]. In fact, comparison with the sole monomeric Re–dppf complex previously determined, i.e. fac-$[ReCl(CO)_3(dppf)]$ [21] shows the same P···P separation (3.64 Å), the same P···Fe···P angle (63.2°) and P–Re–P bite angle (97.1° in **3** and 96.9°), while, as expected, the Re–P and Re–Cl distances lengthen by ca. 0.07 Å on going from coordination number five (in **3**) to six.

NMR data confirm the structure of neutral nitrido complexes **1** and **3** in solution to be identical to that established in the solid state for the rhenium species. According to the equatorial *cis*-P coordination of dppf, the phenyl substituents at P become magnetically inequivalent because of the asymmetry introduced in the molecule by the nitrido group. Consequently, such phenyl proton signals are spread over a region (7.1–8.0 ppm) wider than that exhibited by uncoordinated dppf (7.25–7.35 ppm), and the Cp protons give rise to four distinct multiplets. A similar pattern is observed for phenylimido complexes **2** and **4**, indicating an equatorial dppf coordination as well. In the latter compounds neutrality is achieved by the coordination of a further halide group in the position *trans* with respect to the imido linkage (Scheme 1) giving rise to a distorted octahedral geometry. As already shown by similar phosphine containing nitrido compounds [7,28,29], the strong acid character of the $[M(N)]^{2+}$ groups induces a significant downfield shift of the $^{31}P$ signal of dppf on coordination, the pertinent values being $\delta = 47.2$, 27.7

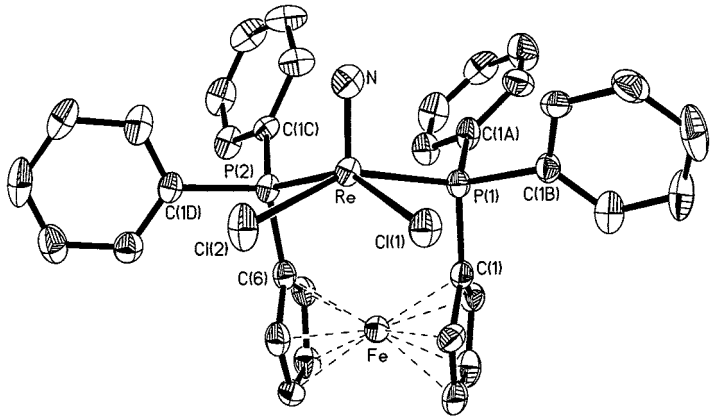



Fig. 1. ORTEP view of complex **3**. The thermal ellipsoids are drawn at a 40% probability. Hydrogen atoms have been omitted for clarity.

and −16.4 ppm for **1**, **3** and the uncoordinated dppf, respectively. Less pronounced deshielding is observed in complexes containing the $[M(NPh)]^{3+}$ groups ($\delta = 22.4$ and −17.1 ppm for **2** and **4**, respectively), according to the diminished acid character of the imido core. $^{31}$P-NMR signals of technetium complexes **1** and **2** show very broad profiles at ambient temperature, which become somewhat narrower on lowering the temperature. Such behavior was attributed earlier to the coupling of the $^{31}$P nuclei with the quadrupolar $^{99}$Tc center [29].

## 4. Supplementary material

Crystallographic data for structural analysis have been deposited with the Cambridge Crystallographic Data Centre, CCDC no. 155601 for compound **3**. Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (Fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

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