***Supporting Information for***

# A Luminescent and Biocompatible PhotoCORM

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**Contents**

**Syntheses:**

All manipulations were done under an argon atmosphere using standard Schlenk line and glovebox techniques. All solvents were freshly distilled, and all other reagents were used without further purification, unless otherwise noted.

***THP:*** The water-soluble phosphine tris(hydroxymethyl)phosphine (THP) was prepared from tetra(hydroxymethyl)phosphonium chloride (THPC) (80% in water) by a modification of a literature procedure[1]. An 80% solution of THPC (10 mL, 55 mmol) was dried under vacuum, then the solid THPC was recrystallized from isopropanol. To the solid THPC, dry triethylamine (100 mL) was added, then the mixture was heated to 60 °C, and allowed to stir for 4 h. The resultant solid was removed by filtering the solution over a medium porosity sintered glass frit and washed with excess triethylamine. The filtrate was collected, and the excess solvent was removed *in vacuo*, leaving a pale yellow oil, which was recrystallized from acetonitrile/diethyl ether at -20°C. Spectral data matched reported values.[2] The white crystalline material was used for all subsequent reactions.

***Bipyridinetricarbonyltris(hydroxymethyl)phosphine rhenium(I) trifluoromethanesulfonate*: *fac*-[Re(bpy)(CO)$_3$(thp)](CF$_3$SO$_3$) (1): 1** was prepared by a modified literature procedure.[3] In a typical preparation, rhenium dodecacarbonyl (0.625 g, 2.0 mmol) was refluxed with 2,2' bipyridine (0.328 g, 4.1 mmol) in 2-chloroethanol (100 mL) for 18 h. Upon cooling the reaction solution to -40°C, a yellow solid, Re(bpy)(CO)$_3$Cl, precipitated and was collected. To 50 mL dry THF, Re(bpy)(CO)$_3$Cl (0.231 g, 0.5 mmol) and Ag(CF$_3$SO$_3$) (0.136 g, 0.53 mmol) were added, and this mixture was allowed to reflux for 6 h to give a solution of Re(bpy)(CO)$_3$(THF)$^+$ and insoluble AgCl. The latter was removed by filtering the solution through a fine porosity sintered glass frit. To the resulting solution, THP (0.620 g, 5.0 mmol) was added and the mixture was refluxed for 18 h. The remaining THF was removed *in vacuo*, to give the yellow, air-stable *fac*-[Re(bpy)(CO)$_3$(thp)](CF$_3$SO$_3$) (**1**) in moderate yield (66%). This salt was then purified by flash chromatography over octadecyl-functionalized silica gel. X-ray suitable crystals were grown from acetonitrile/diethyl ether solution at -20°C (*vide infra*). The mass spectrum (ESI+) showed peaks at 551.04 m/z [M]$^+$, and 523.04 m/z [M-CO]$^+$. An acetonitrile solution of **1** shows three

distinct $\nu$(CO) bands at 2037, 1949 and 1923 $cm^{-1}$. In water, **1** gives a light yellow solution, with a MLCT band at 345 nm ($\varepsilon$ = 3500 $M^{-1}cm^{-1}$).

**Instrumentation:**

Optical absorption spectra were recorded using a Shimadzu dual beam UV-2401 PC spectrometer with 1.0 cm path length quartz cells. Infrared spectra of solutions were recorded using a Mattson Research Series FTIR spectrometer. CO concentration was measured by GC using an Agilent 6890 gas chromatograph with a thermal conductivity detector. Solution-state NMR spectra were gathered on Varian Unity Inova 400 MHz and 500 MHz spectrometers. Exact mass data were gathered using a Micromass Q-ToF2 quadrupole/time-of-flight tandem mass spectrometer with an electrospray ion source. The 96-well plates used for cell viability assays were read with a Tecan Infinite 200 PRO plate reader.

**Photochemical and photophysical procedures:**

Continuous photolysis was performed using a 200 W high-pressure mercury lamp mounted on an Oriel optical train. This light was passed through an infrared filter, then an interference filter isolating the mercury lines at 366, 405 or 436 nm. A system of lenses and a shutter was used to collimate the light and control the irradiation time. Photon flux was determined using chemical actinometry with iron ferrioxalate as the standard[4]. Photoreaction quantum yields were determined by plotting the incremental quantum yields for the growth of a new band at 405 nm versus the elapsed irradiation time over the initial 10% of the reaction. A linear fit of this data provided an equation with a y-intercept equal to the overall quantum yield, $\phi_{rxn}$. Phosphorescence spectra were gathered using a Photon Technology International fluorometer with a model 814 PMT detection system at 1 nm resolution. Phosphorescence quantum yields were calculated using standard methods[5] with rhodamine B in ethanol as the standard. Phosphorescence lifetimes were measured using a custom built fluorescence lifetime instrument consisting of a sample holder mounted in front of a SPEX monochromator and a 1P28 photomultiplier tube (PMT) detector, signals from which were collected on a Tektronix 500 MHz

digital oscilloscope. Excitation was from the third harmonic (355 nm) output of a Spectra-Physics Quanta Ray INDY Nd:YAG pulsed laser.

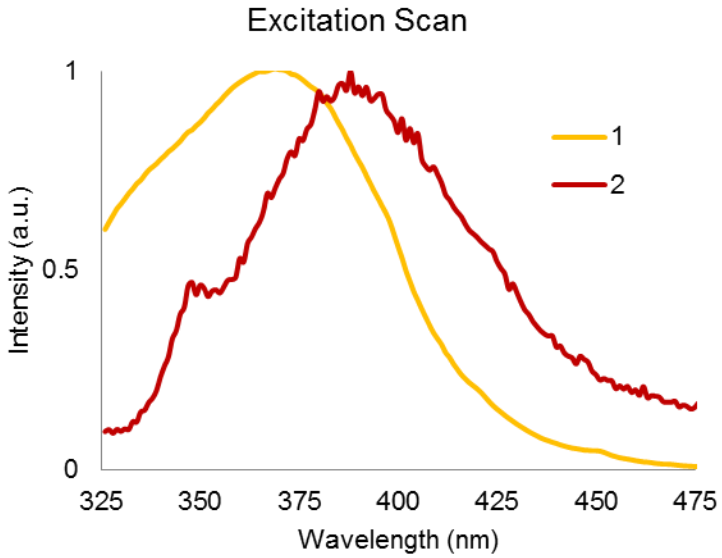


**Figure S-1:** Excitation scans for **1** and **2** in phosphate buffered saline (PBS, pH=7.2) at room temperature; $\lambda_{em}$ = 515 nm and 585 nm for **1** and **2**, respectively. Excitation spectra are normalized for clarity (nb: these spectra were not recorded for the same solutions).

**Further comments on the apparent non-toxicity of 1 and 2 in cell culture experiments.**

In addition to the PrestoBlue™ assay for cell viability during the course of an experiment, the long-term toxicity of **1** and **2** was assessed by qualitatively observing cell health using optical microscopy. To a well containing adhered cells maintained in DMEM supplemented with 10% FBS, an aliquot of a solution of **1** in PBS was added, giving a final concentration of 50 μM in the well. After three days incubation at 37°C (5% $CO_2$) in the dark, there were no obvious differences between the cells incubated with PBS only and those with 50 μM **1**. This suggests that 1 does not interfere with cellular replication, and thus is relatively benign to isolated cells. Furthermore, the results were similar when this experiment was carried out again with wells containing cells maintained in DMEM supplemented with 10% FBS and **1** (50 μM) but repeatedly exposed to ambient light, ostensibly producing **2** and CO. After three days incubation, there was no apparent toxicity when compared to a similarly exposed well with only PBS.

## X-ray Crystallography

The solid state crystal structure of **1** was determined by X-ray diffraction on a Kappa Apex II single-crystal diffractometer.

**Table S-1.** Crystal data and structure refinement for [Re(bpy)(CO)$_3$(thp)]OTf (**1**).

| | |
|---|---|
| Empirical formula | C17 H17 F3 N2 O9 P Re S |
| Formula weight | 699.56 |
| Temperature | 100(2) K |
| Wavelength | 0.71073 A |
| Crystal system, space group | Monoclinic, P2(1)/c |
| Unit cell dimensions | a = 12.7662(8) A alpha = 90 deg.<br>b = 12.1448(8) A beta = 94.3050(10) deg.<br>c = 14.8497(10) A gamma = 90 deg. |
| Volume | 2295.8(3) A^3 |
| Z, Calculated density | 4, 2.024 Mg/m^3 |
| Absorption coefficient | 5.529 mm^-1 |
| F(000) | 1352 |
| Crystal size | 0.25 x 0.15 x 0.10 mm |
| Theta range for data collection | 1.60 to 33.14 deg. |
| Limiting indices | -19<=h<=19, -18<=k<=18, -12<=l<=22 |
| Reflections collected / unique | 23091 / 8759 [R(int) = 0.0154] |
| Completeness to theta = 33.14 | 100.0 % |
| Absorption correction | Semi-empirical from equivalents |
| Max. and min. transmission | 0.7489 and 0.6257 |
| Refinement method | Full-matrix least-squares on F^2 |
| Data / restraints / parameters | 8759 / 0 / 310 |
| Goodness-of-fit on F^2 | 1.560 |
| Final R indices [I>2sigma(I)] | R1 = 0.0160, wR2 = 0.0290 |
| R indices (all data) | R1 = 0.0212, wR2 = 0.0296 |
| Largest diff. peak and hole | 0.698 and -0.647 e.A^-3 |

**Table S-2**. Atomic coordinates ( x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for **1**. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| C(1) | 843(1) | 982(1) | 8806(1) | 20(1) |
| C(2) | 829(1) | 3050(1) | 7968(1) | 17(1) |
| C(3) | 2563(1) | 1721(1) | 7997(1) | 19(1) |
| C(4) | 3438(1) | 4099(1) | 8875(1) | 17(1) |
| C(5) | 3964(1) | 5064(1) | 9124(1) | 21(1) |
| C(6) | 3604(1) | 5700(1) | 9806(1) | 24(1) |
| C(7) | 2724(1) | 5362(1) | 10225(1) | 21(1) |
| C(8) | 2221(1) | 4394(1) | 9944(1) | 15(1) |
| C(9) | 1259(1) | 3991(1) | 10332(1) | 14(1) |
| C(10) | 792(1) | 4515(1) | 11031(1) | 19(1) |
| C(11) | -129(1) | 4095(1) | 11327(1) | 20(1) |

| | | | | |
|---|---|---|---|---|
| C(12) | -579(1) | 3174(1) | 10909(1) | 21(1) |
| C(13) | -74(1) | 2679(1) | 10226(1) | 18(1) |
| C(14) | 2099(1) | 999(1) | 11086(1) | 24(1) |
| C(15) | 3350(1) | 30(1) | 9698(1) | 20(1) |
| C(16) | 3987(1) | 2044(1) | 10585(1) | 20(1) |
| C(17) | 6300(1) | 2635(1) | 7546(1) | 29(1) |
| F(1) | 5610(1) | 1824(1) | 7614(1) | 45(1) |
| F(2) | 5941(1) | 3520(1) | 7953(1) | 48(1) |
| F(3) | 6337(1) | 2865(1) | 6672(1) | 48(1) |
| N(1) | 2576(1) | 3769(1) | 9269(1) | 14(1) |
| N(2) | 840(1) | 3059(1) | 9945(1) | 14(1) |
| O(1) | 315(1) | 210(1) | 8798(1) | 34(1) |
| O(2) | 338(1) | 3493(1) | 7409(1) | 25(1) |
| O(3) | 3061(1) | 1407(1) | 7435(1) | 28(1) |
| O(4) | 2767(1) | 719(1) | 11854(1) | 25(1) |
| O(5) | 3998(1) | -538(1) | 10368(1) | 26(1) |
| O(6) | 4674(1) | 2266(1) | 9900(1) | 22(1) |
| O(7) | 7397(1) | 1963(1) | 8965(1) | 28(1) |
| O(8) | 7867(1) | 1302(1) | 7508(1) | 24(1) |
| O(9) | 8217(1) | 3208(1) | 7953(1) | 33(1) |
| P | 2810(1) | 1330(1) | 10092(1) | 13(1) |
| Re(1) | 1707(1) | 2273(1) | 8912(1) | 12(1) |
| S | 7594(1) | 2238(1) | 8046(1) | 18(1) |

**Table S-3** Bond lengths [A] and angles [deg] for **1**.

| | |
|---|---|
| C(1)-O(1) | 1.1537(18) |
| C(1)-Re(1) | 1.9164(15) |
| C(2)-O(2) | 1.1383(17) |
| C(2)-Re(1) | 1.9685(14) |
| C(3)-O(3) | 1.1516(17) |
| C(3)-Re(1) | 1.9270(15) |
| C(4)-N(1) | 1.3453(18) |
| C(4)-C(5) | 1.386(2) |
| C(4)-H(4) | 0.9500 |
| C(5)-C(6) | 1.379(2) |
| C(5)-H(5) | 0.9500 |
| C(6)-C(7) | 1.387(2) |
| C(6)-H(6) | 0.9500 |
| C(7)-C(8) | 1.389(2) |
| C(7)-H(7) | 0.9500 |
| C(8)-N(1) | 1.3616(17) |
| C(8)-C(9) | 1.478(2) |
| C(9)-N(2) | 1.3598(17) |
| C(9)-C(10) | 1.390(2) |
| C(10)-C(11) | 1.383(2) |
| C(10)-H(10) | 0.9500 |
| C(11)-C(12) | 1.383(2) |
| C(11)-H(11) | 0.9500 |
| C(12)-C(13) | 1.380(2) |

| | |
|---|---|
| C(12)-H(12) | 0.9500 |
| C(13)-N(2) | 1.3493(18) |
| C(13)-H(13) | 0.9500 |
| C(14)-O(4) | 1.4129(17) |
| C(14)-P | 1.8341(15) |
| C(14)-H(14A) | 0.9900 |
| C(14)-H(14B) | 0.9900 |
| C(15)-O(5) | 1.4232(17) |
| C(15)-P | 1.8358(15) |
| C(15)-H(15A) | 0.9900 |
| C(15)-H(15B) | 0.9900 |
| C(16)-O(6) | 1.4181(18) |
| C(16)-P | 1.8393(14) |
| C(16)-H(16A) | 0.9900 |
| C(16)-H(16B) | 0.9900 |
| C(17)-F(3) | 1.331(2) |
| C(17)-F(1) | 1.330(2) |
| C(17)-F(2) | 1.3325(19) |
| C(17)-S | 1.8248(18) |
| N(1)-Re(1) | 2.1746(11) |
| N(2)-Re(1) | 2.1782(12) |
| O(4)-H(4O) | 0.8400 |
| O(5)-H(5O) | 0.8400 |
| O(6)-H(6O) | 0.8400 |
| O(7)-S | 1.4452(11) |
| O(8)-S | 1.4475(11) |
| O(9)-S | 1.4340(12) |
| P-Re(1) | 2.4478(4) |

| | |
|---|---|
| O(1)-C(1)-Re(1) | 175.82(13) |
| O(2)-C(2)-Re(1) | 178.47(13) |
| O(3)-C(3)-Re(1) | 178.33(13) |
| N(1)-C(4)-C(5) | 122.06(14) |
| N(1)-C(4)-H(4) | 119.0 |
| C(5)-C(4)-H(4) | 119.0 |
| C(6)-C(5)-C(4) | 119.20(15) |
| C(6)-C(5)-H(5) | 120.4 |
| C(4)-C(5)-H(5) | 120.4 |
| C(5)-C(6)-C(7) | 119.32(14) |
| C(5)-C(6)-H(6) | 120.3 |
| C(7)-C(6)-H(6) | 120.3 |
| C(8)-C(7)-C(6) | 119.15(15) |
| C(8)-C(7)-H(7) | 120.4 |
| C(6)-C(7)-H(7) | 120.4 |
| N(1)-C(8)-C(7) | 121.40(14) |
| N(1)-C(8)-C(9) | 115.76(12) |
| C(7)-C(8)-C(9) | 122.83(13) |
| N(2)-C(9)-C(10) | 121.30(13) |
| N(2)-C(9)-C(8) | 115.02(12) |
| C(10)-C(9)-C(8) | 123.67(13) |
| C(11)-C(10)-C(9) | 119.38(13) |
| C(11)-C(10)-H(10) | 120.3 |
| C(9)-C(10)-H(10) | 120.3 |

| | |
|---|---|
| C(12)-C(11)-C(10) | 119.38(14) |
| C(12)-C(11)-H(11) | 120.3 |
| C(10)-C(11)-H(11) | 120.3 |
| C(13)-C(12)-C(11) | 118.68(15) |
| C(13)-C(12)-H(12) | 120.7 |
| C(11)-C(12)-H(12) | 120.7 |
| N(2)-C(13)-C(12) | 122.75(14) |
| N(2)-C(13)-H(13) | 118.6 |
| C(12)-C(13)-H(13) | 118.6 |
| O(4)-C(14)-P | 113.42(11) |
| O(4)-C(14)-H(14A) | 108.9 |
| P-C(14)-H(14A) | 108.9 |
| O(4)-C(14)-H(14B) | 108.9 |
| P-C(14)-H(14B) | 108.9 |
| H(14A)-C(14)-H(14B) | 107.7 |
| O(5)-C(15)-P | 114.03(10) |
| O(5)-C(15)-H(15A) | 108.7 |
| P-C(15)-H(15A) | 108.7 |
| O(5)-C(15)-H(15B) | 108.7 |
| P-C(15)-H(15B) | 108.7 |
| H(15A)-C(15)-H(15B) | 107.6 |
| O(6)-C(16)-P | 109.59(9) |
| O(6)-C(16)-H(16A) | 109.8 |
| P-C(16)-H(16A) | 109.8 |
| O(6)-C(16)-H(16B) | 109.8 |
| P-C(16)-H(16B) | 109.8 |
| H(16A)-C(16)-H(16B) | 108.2 |
| F(3)-C(17)-F(1) | 107.58(14) |
| F(3)-C(17)-F(2) | 108.14(14) |
| F(1)-C(17)-F(2) | 108.03(15) |
| F(3)-C(17)-S | 110.83(13) |
| F(1)-C(17)-S | 111.00(12) |
| F(2)-C(17)-S | 111.13(11) |
| C(4)-N(1)-C(8) | 118.86(12) |
| C(4)-N(1)-Re(1) | 124.20(9) |
| C(8)-N(1)-Re(1) | 116.93(9) |
| C(13)-N(2)-C(9) | 118.42(12) |
| C(13)-N(2)-Re(1) | 124.33(9) |
| C(9)-N(2)-Re(1) | 117.25(10) |
| C(14)-O(4)-H(4O) | 109.5 |
| C(15)-O(5)-H(5O) | 109.5 |
| C(16)-O(6)-H(6O) | 109.5 |
| C(14)-P-C(15) | 106.75(7) |
| C(14)-P-C(16) | 102.82(7) |
| C(15)-P-C(16) | 102.52(7) |
| C(14)-P-Re(1) | 112.70(5) |
| C(15)-P-Re(1) | 112.60(5) |
| C(16)-P-Re(1) | 118.23(5) |
| C(1)-Re(1)-C(3) | 90.62(6) |
| C(1)-Re(1)-C(2) | 92.29(6) |
| C(3)-Re(1)-C(2) | 89.26(6) |
| C(1)-Re(1)-N(1) | 169.70(5) |
| C(3)-Re(1)-N(1) | 99.01(5) |

| | |
|---|---|
| C(2)-Re(1)-N(1) | 91.49(5) |
| C(1)-Re(1)-N(2) | 95.57(5) |
| C(3)-Re(1)-N(2) | 173.79(5) |
| C(2)-Re(1)-N(2) | 89.97(5) |
| N(1)-Re(1)-N(2) | 74.85(4) |
| C(1)-Re(1)-P | 88.44(5) |
| C(3)-Re(1)-P | 90.90(4) |
| C(2)-Re(1)-P | 179.25(4) |
| N(1)-Re(1)-P | 87.76(3) |
| N(2)-Re(1)-P | 89.79(3) |
| O(9)-S-O(7) | 114.74(7) |
| O(9)-S-O(8) | 115.69(7) |
| O(7)-S-O(8) | 113.97(7) |
| O(9)-S-C(17) | 103.58(8) |
| O(7)-S-C(17) | 103.27(8) |
| O(8)-S-C(17) | 103.33(7) |

**Table S-4** Anisotropic displacement parameters (A^2 x 10^3) for **1**.
The anisotropic displacement factor exponent takes the form:
-2 pi^2 [ h^2 a*^2 U11 + ... + 2 h k a* b* U12 ]

| | U11 | U22 | U33 | U23 | U13 | U12 |
|---|---|---|---|---|---|---|
| C(1) | 18(1) | 23(1) | 18(1) | -6(1) | 4(1) | -3(1) |
| C(2) | 15(1) | 20(1) | 17(1) | -2(1) | 4(1) | -3(1) |
| C(3) | 18(1) | 21(1) | 17(1) | 1(1) | 1(1) | -3(1) |
| C(4) | 14(1) | 20(1) | 17(1) | 5(1) | 1(1) | -1(1) |
| C(5) | 17(1) | 22(1) | 26(1) | 9(1) | -1(1) | -6(1) |
| C(6) | 23(1) | 17(1) | 32(1) | 2(1) | -5(1) | -7(1) |
| C(7) | 21(1) | 16(1) | 27(1) | -2(1) | -2(1) | -3(1) |
| C(8) | 14(1) | 13(1) | 17(1) | 2(1) | -2(1) | 0(1) |
| C(9) | 14(1) | 14(1) | 15(1) | 1(1) | -1(1) | 0(1) |
| C(10) | 21(1) | 16(1) | 19(1) | -2(1) | 0(1) | 3(1) |
| C(11) | 22(1) | 21(1) | 18(1) | -1(1) | 5(1) | 5(1) |
| C(12) | 17(1) | 23(1) | 22(1) | 2(1) | 5(1) | 2(1) |
| C(13) | 15(1) | 18(1) | 20(1) | 0(1) | 3(1) | -2(1) |
| C(14) | 20(1) | 34(1) | 19(1) | 8(1) | 7(1) | 6(1) |
| C(15) | 21(1) | 15(1) | 24(1) | 2(1) | 6(1) | 2(1) |
| C(16) | 15(1) | 21(1) | 22(1) | -1(1) | -2(1) | -1(1) |
| C(17) | 27(1) | 30(1) | 29(1) | -6(1) | -1(1) | 4(1) |
| F(1) | 22(1) | 55(1) | 58(1) | -13(1) | -1(1) | -10(1) |
| F(2) | 43(1) | 42(1) | 58(1) | -18(1) | -5(1) | 21(1) |
| F(3) | 61(1) | 50(1) | 29(1) | 6(1) | -11(1) | 12(1) |
| N(1) | 13(1) | 14(1) | 14(1) | 2(1) | -1(1) | -1(1) |
| N(2) | 12(1) | 14(1) | 14(1) | 0(1) | 1(1) | 0(1) |
| O(1) | 32(1) | 31(1) | 41(1) | -12(1) | 8(1) | -16(1) |
| O(2) | 20(1) | 36(1) | 20(1) | 6(1) | 1(1) | 3(1) |

| | | | | | | |
|---|---|---|---|---|---|---|
| O(3) | 29(1) | 36(1) | 22(1) | -3(1) | 11(1) | 2(1) |
| O(4) | 33(1) | 25(1) | 16(1) | 6(1) | 0(1) | -3(1) |
| O(5) | 26(1) | 24(1) | 30(1) | 12(1) | 12(1) | 10(1) |
| O(6) | 15(1) | 17(1) | 36(1) | 4(1) | 5(1) | 0(1) |
| O(7) | 33(1) | 32(1) | 19(1) | 1(1) | 5(1) | -5(1) |
| O(8) | 28(1) | 20(1) | 26(1) | -1(1) | 9(1) | 2(1) |
| O(9) | 32(1) | 27(1) | 42(1) | -6(1) | 15(1) | -15(1) |
| P | 12(1) | 14(1) | 14(1) | 0(1) | 3(1) | -1(1) |
| Re(1) | 11(1) | 14(1) | 11(1) | -1(1) | 1(1) | -2(1) |
| S | 17(1) | 18(1) | 19(1) | -1(1) | 5(1) | -4(1) |

**Table S-5.** Hydrogen coordinates ( x 10^4) and isotropic displacement parameters (A^2 x 10^3) for **1**.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| H(4) | 3695 | 3657 | 8412 | 21 |
| H(5) | 4564 | 5285 | 8829 | 26 |
| H(6) | 3956 | 6362 | 9987 | 29 |
| H(7) | 2468 | 5788 | 10699 | 26 |
| H(10) | 1103 | 5156 | 11303 | 22 |
| H(11) | -449 | 4437 | 11812 | 24 |
| H(12) | -1223 | 2886 | 11090 | 25 |
| H(13) | -382 | 2045 | 9941 | 21 |
| H(14A) | 1666 | 1641 | 11233 | 29 |
| H(14B) | 1618 | 375 | 10936 | 29 |
| H(15A) | 2761 | -457 | 9484 | 24 |
| H(15B) | 3767 | 186 | 9177 | 24 |
| H(16A) | 3781 | 2743 | 10866 | 23 |
| H(16B) | 4349 | 1579 | 11059 | 23 |
| H(4O) | 2594 | 100 | 12047 | 38 |
| H(5O) | 3620 | -923 | 10684 | 39 |
| H(6O) | 5053 | 1712 | 9827 | 33 |

**Table S-6**. Torsion angles [deg] for **a02222012_0m**.

| | |
|---|---|
| N(1)-C(4)-C(5)-C(6) | 1.0(2) |
| C(4)-C(5)-C(6)-C(7) | -0.2(2) |
| C(5)-C(6)-C(7)-C(8) | -0.4(2) |
| C(6)-C(7)-C(8)-N(1) | 0.3(2) |
| C(6)-C(7)-C(8)-C(9) | -178.00(13) |
| N(1)-C(8)-C(9)-N(2) | -1.59(17) |
| C(7)-C(8)-C(9)-N(2) | 176.78(13) |
| N(1)-C(8)-C(9)-C(10) | 179.19(12) |
| C(7)-C(8)-C(9)-C(10) | -2.4(2) |

| | |
|---|---|
| N(2)-C(9)-C(10)-C(11) | -1.5(2) |
| C(8)-C(9)-C(10)-C(11) | 177.72(13) |
| C(9)-C(10)-C(11)-C(12) | -1.2(2) |
| C(10)-C(11)-C(12)-C(13) | 2.0(2) |
| C(11)-C(12)-C(13)-N(2) | -0.2(2) |
| C(5)-C(4)-N(1)-C(8) | -1.1(2) |
| C(5)-C(4)-N(1)-Re(1) | 179.71(10) |
| C(7)-C(8)-N(1)-C(4) | 0.47(19) |
| C(9)-C(8)-N(1)-C(4) | 178.86(11) |
| C(7)-C(8)-N(1)-Re(1) | 179.69(10) |
| C(9)-C(8)-N(1)-Re(1) | -1.92(14) |
| C(12)-C(13)-N(2)-C(9) | -2.4(2) |
| C(12)-C(13)-N(2)-Re(1) | 177.20(10) |
| C(10)-C(9)-N(2)-C(13) | 3.24(19) |
| C(8)-C(9)-N(2)-C(13) | -176.00(12) |
| C(10)-C(9)-N(2)-Re(1) | -176.41(10) |
| C(8)-C(9)-N(2)-Re(1) | 4.35(15) |
| O(4)-C(14)-P-C(15) | 71.23(13) |
| O(4)-C(14)-P-C(16) | -36.26(13) |
| O(4)-C(14)-P-Re(1) | -164.64(9) |
| O(5)-C(15)-P-C(14) | -55.94(13) |
| O(5)-C(15)-P-C(16) | 51.76(12) |
| O(5)-C(15)-P-Re(1) | 179.88(9) |
| O(6)-C(16)-P-C(14) | 174.52(10) |
| O(6)-C(16)-P-C(15) | 63.84(11) |
| O(6)-C(16)-P-Re(1) | -60.64(11) |
| O(1)-C(1)-Re(1)-C(3) | 146(2) |
| O(1)-C(1)-Re(1)-C(2) | -124(2) |
| O(1)-C(1)-Re(1)-N(1) | -13(2) |
| O(1)-C(1)-Re(1)-N(2) | -34(2) |
| O(1)-C(1)-Re(1)-P | 55(2) |
| O(3)-C(3)-Re(1)-C(1) | 98(5) |
| O(3)-C(3)-Re(1)-C(2) | 5(5) |
| O(3)-C(3)-Re(1)-N(1) | -86(5) |
| O(3)-C(3)-Re(1)-N(2) | -78(5) |
| O(3)-C(3)-Re(1)-P | -174(100) |
| O(2)-C(2)-Re(1)-C(1) | -101(5) |
| O(2)-C(2)-Re(1)-C(3) | -10(5) |
| O(2)-C(2)-Re(1)-N(1) | 89(5) |
| O(2)-C(2)-Re(1)-N(2) | 164(5) |
| O(2)-C(2)-Re(1)-P | 93(6) |
| C(4)-N(1)-Re(1)-C(1) | 160.3(3) |
| C(8)-N(1)-Re(1)-C(1) | -18.9(3) |
| C(4)-N(1)-Re(1)-C(3) | 1.33(11) |
| C(8)-N(1)-Re(1)-C(3) | -177.84(9) |
| C(4)-N(1)-Re(1)-C(2) | -88.16(11) |
| C(8)-N(1)-Re(1)-C(2) | 92.67(10) |
| C(4)-N(1)-Re(1)-N(2) | -177.72(11) |
| C(8)-N(1)-Re(1)-N(2) | 3.11(9) |
| C(4)-N(1)-Re(1)-P | 91.89(10) |
| C(8)-N(1)-Re(1)-P | -87.29(9) |
| C(13)-N(2)-Re(1)-C(1) | -7.52(12) |
| C(9)-N(2)-Re(1)-C(1) | 172.12(10) |

| | |
|---|---|
| C(13)-N(2)-Re(1)-C(3) | 167.7(4) |
| C(9)-N(2)-Re(1)-C(3) | -12.7(5) |
| C(13)-N(2)-Re(1)-C(2) | 84.78(11) |
| C(9)-N(2)-Re(1)-C(2) | -95.59(10) |
| C(13)-N(2)-Re(1)-N(1) | 176.34(12) |
| C(9)-N(2)-Re(1)-N(1) | -4.03(9) |
| C(13)-N(2)-Re(1)-P | -95.93(11) |
| C(9)-N(2)-Re(1)-P | 83.71(9) |
| C(14)-P-Re(1)-C(1) | -62.99(8) |
| C(15)-P-Re(1)-C(1) | 57.84(7) |
| C(16)-P-Re(1)-C(1) | 177.18(7) |
| C(14)-P-Re(1)-C(3) | -153.58(8) |
| C(15)-P-Re(1)-C(3) | -32.75(7) |
| C(16)-P-Re(1)-C(3) | 86.59(7) |
| C(14)-P-Re(1)-C(2) | 104(3) |
| C(15)-P-Re(1)-C(2) | -136(3) |
| C(16)-P-Re(1)-C(2) | -16(3) |
| C(14)-P-Re(1)-N(1) | 107.44(7) |
| C(15)-P-Re(1)-N(1) | -131.73(6) |
| C(16)-P-Re(1)-N(1) | -12.39(6) |
| C(14)-P-Re(1)-N(2) | 32.59(7) |
| C(15)-P-Re(1)-N(2) | 153.42(6) |
| C(16)-P-Re(1)-N(2) | -87.24(6) |
| F(3)-C(17)-S-O(9) | 63.11(13) |
| F(1)-C(17)-S-O(9) | -177.41(11) |
| F(2)-C(17)-S-O(9) | -57.16(14) |
| F(3)-C(17)-S-O(7) | -176.94(12) |
| F(1)-C(17)-S-O(7) | -57.46(13) |
| F(2)-C(17)-S-O(7) | 62.79(14) |
| F(3)-C(17)-S-O(8) | -57.93(13) |
| F(1)-C(17)-S-O(8) | 61.55(13) |
| F(2)-C(17)-S-O(8) | -178.20(12) |

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