Supporting Information

Cu-free 1,3-dipolar cycloaddition click reactions to form isoxazole linkers in chelating ligands for *fac*-$[M^{I}(CO)_3]^+$ centers (M = Re, ^{99m}Tc)

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Experimental

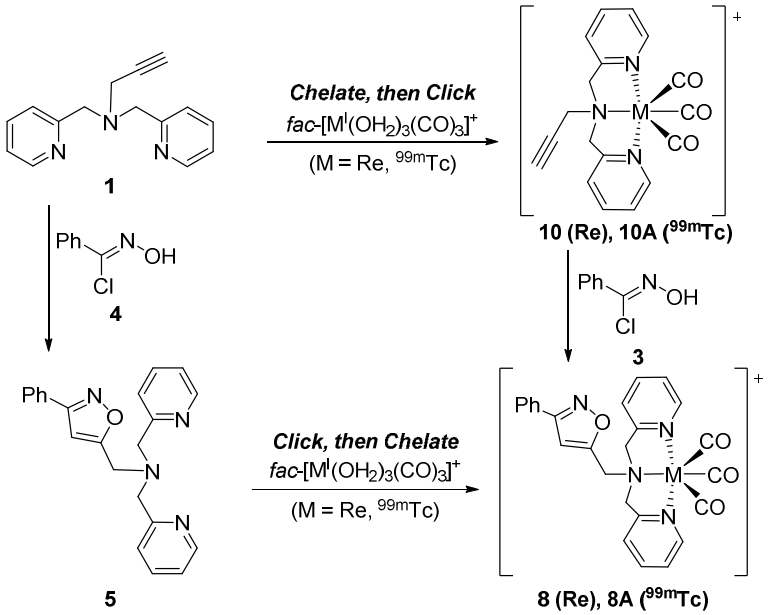
All reagents and organic solvents were of reagent grade or better and used as purchased from Aldrich, Acros, or Fluka without further purification. Rhenium starting material *fac*-$[Re^{I}(OH_2)_3(CO)_3](OSO_2CF_3)$, was prepared by literature methods from $Re_2(CO)_{10}$ purchased from Strem.[1] The preparations of *N,N*-bis(pyridin-2-ylmethyl)prop-2-yn-1-amine, (**1**), methyl 2-(prop-2-ynyl(pyridine-2-ylmethyl)amino)acetate (**2**), 2-(prop-2-yn-1-yl(pyridin-2-ylmethyl)amino)acetic acid (**3**), *fac*-$[Re^{I}(CO)_3(\mathbf{3})]$ (**11**),[2] *N*-hydroxybenzimidoyl chloride (**4**),[3] and [*fac*-$Re^{I}(CO)_3(\mathbf{1})](OSO_2CF_3)$ (**10**),[4] were performed as previously reported. UV-Vis spectra were obtained using a Varian Cary 50 spectrophotometer (1 cm path-length). ^{1}H and ^{13}C NMR spectra were recorded on a Varian 300 MHz instrument at 25 ºC. Elemental analyses were performed by Intertek Pharmaceutical Services, NJ or Atlantic Microlab, Inc. Norcross, GA. Analytical separation and identification of compounds were conducted on a Perkin Elmer Series 200 High Pressure Liquid Chromatograph (HPLC) equipped with a UV/VIS Series 200 detector and a Radiomatic 610TR detector. Utilizing a Varian Pursuit XRS 5μm particle and 250 x 4.6 mm C18 column, the compounds were separated with a reverse phase gradient system beginning with 0.1% trifluoroacetic acid (TFA) aqueous eluent gradually shifting to methanol according to the following method: 0-3.0 min (100% TFA), 3.0-9.0 min (75% TFA, 25% MeOH), 9.0-20.0 min (25% to 100% MeOH linear gradient), 20.0-30.0 min (100% MeOH) at a flow rate of 1.0 mL/min. Preparatory separations of compounds were conducted on a Hitachi D-7000 High Pressure Liquid Chromatograph (HPLC) equipped with a UV/VIS Series L-7400 detector and a L-7150 pump.

Utilizing a Phenomenex Gemini 5µm particle and 250 x 21.20 mm C18 column, the compounds were separated with a reverse phase gradient system beginning with 0.1% TFA aqueous eluent gradually shifting to methanol according to the following method: 0-5.0 min (75% TFA, 25% MeOH), 5.0-9.1 min (25% MeOH to 34% MeOH linear gradient), 9.1-20.0 min (34% to 100% MeOH linear gradient), 20.0-30.0 min (100% MeOH) at a flow rate of 10.0 mL/min. FT-IR spectra were obtained on a Thermo Nicolet 6700 FT-IR with an ATR cell and analyzed with OMNIC 7.1 software. Mass spectra were obtained on a Thermo-Finnigan LCQ Advantage ESI-MS.

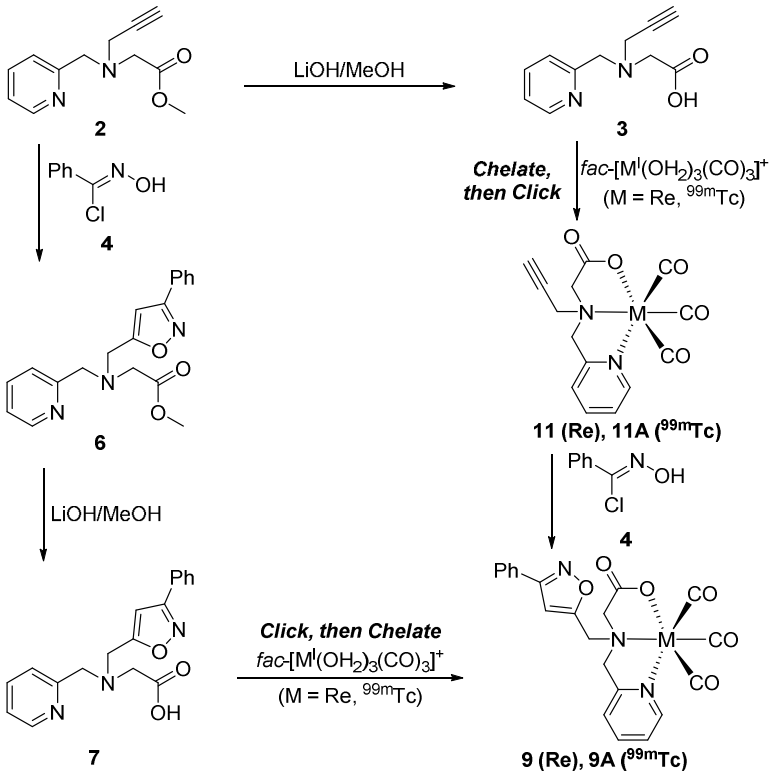
X-Ray Crystallography

Crystals of compound **9** were analyzed at 293(2) K on a Nonius KappaCCD diffractometer using MoK_α irradiation (λ = 0.71073 Å) in a series of phi and omega scans. Data collection, cell refinement and data reduction were performed using *Collect*[5] and *HKL Scalepack*/*Denzo*[6], respectively. The crystal structures were solved by direct methods and refined by least squares method on F^2 using SHELXS-97 and SHELXL-97, respectively. All non-hydrogen atoms were refined anisotropically. All hydrogen atoms bonded to carbon atoms were located in the Fourier-difference electron-density map, fixed in geometrically constrained riding positions and isotropically refined. Crystallographic data of the investigated compounds are available in the cif file. All bond distances (Å) and angles (°) are listed in Table S1.

Scheme S1. Synthesis of the symmetric complexes



Scheme S2. Synthesis of the asymmetric complexes



1-(3-phenylisoxazol-5-yl)-*N*,*N*-bis(pyridin-2-ylmethyl)methanamine, 5.

A 20 mL scintillation vial was charged with **1** (66.7 mg, 0.28 mmol), **4** (87.0 mg, 0.56 mmol) and methylene chloride (5 mL). The dropwise addition of $NaHCO_{3(aq)}$ (1.0 M, 0.56 mmol) produced a colorless heterogeneous solution. The solution was vigorously stirred at rt for 1 h and purified by prep RP-HPLC to yield **5** as a yellow oil (44.9 mg; 45%). ^{1}H NMR [δ (ppm), CD_3OD] 8.63 (d, 2H, J = 2.42), 8.22 (t, 2H, J = 7.81), 7.85 (d, 2H, J = 7.97), 7.66 (t, 2H, J = 6.67), 7.54 – 7.52 (m, 2H), 7.25 – 7.22 (m, 3H), 6.63 (s, 1H), 4.20 (s, 4H), 3.87 (s, 2H); ^{13}C NMR [δ (ppm), CD_3OD] 169.5, 163.9, 155.1, 146.5, 144.2, 131.4, 130.1, 129.7, 127.9, 127.8, 126.9, 104.4, 57.7, 49.9; $\lambda_{max}(CH_3OH)$/nm (ε/dm^3 mol^{-1} cm^{-1})**:** 231 **(**11,978); υ_{max}/cm^{-1}: 1620 (Isox Ring) 1179 (Isox C-H), 1128 (Isox Ring); m/z 379.3 ($[M+Na]^+$ 100 %) 357.3 ($[M+H]^+$ 30 %).

***fac*-$[Re^{I}(CO)_3(5)](OSO_2CF_3)$, 8.**

***Chelate, then Click*:** A 20 mL scintillation vial was charged with $[Re^{I}(CO)_3(\mathbf{1})]^+$, **10**, (10.29 mg, 0.015 mmol) and **4** (9.53 mg, 0.06 mmol) in methylene chloride (1 mL). The dropwise addition of $NaHCO_{3(aq)}$ (1.0 M, 31 μL, 0.03 mmol) produced a colorless heterogeneous solution. The solution was vigorously stirred at rt for 15 h. The reaction mixture was dissolved in 6 mL of 0.1% TFA/MeOH solution (25% MeOH) and purified by prep RP-HPLC to yield **8**•TFA as a yellow oil (6.16 mg; 53%). Analytical data (^{1}H, ^{13}C NMR, MS) were identical to the product formed in the following procedure.

***Click, then Chelate*:** To a methanolic solution of **5** (40.42 mg, 0.11 mmol) in a 20 mL scintillation vial, 0.1 M *fac*-$[Re^{I}(OH_2)_3(CO)_3](OSO_2CF_3)_{(aq)}$ (1.4 mL, 0.14 mmol) was added slowly while stirring at rt. The vial was sealed and stirred for 12 h producing an off-white precipitate that was collected via vacuum filtration. (65.4 mg; 75%). ^{1}H NMR [δ (ppm), CD_3OD] 8.84 (d, 2H, J = 5.6), 7.91 – 7.85 (m, 2H), 7.50 – 7.47 (m, 5H), 7.33 (t, 2H, J = 6.8), 7.28 (s, 1H), 5.14 (s, 2H), 4.91 (ABq, 4H, $\Delta\delta_{AB}$ = 0.16, J

= 16.5); ^{13}C NMR [δ (ppm), CD_3OD] 166.8, 164.4, 161.3, 153.1, 141.7, 131.6, 130.2, 129.7, 128.0, 127.0, 124.8, 107.6, 69.5, 63.5; $\lambda_{max}(CH_3OH)$/nm (ε/dm^3 mol^{-1} cm^{-1})**:** 230 **(**25,523), 295 (6,755); υ_{max}/cm^{-1}: 2029 and 1900 (CO), 1611 (Isox Ring), 1160 (C-H); m/z 627.2 ([M$^+$] 100 %); Calc for $C_{26}H_{20}F_3N_4O_7SRe{\cdot}CH_2Cl_2$: C, 37.67; H, 2.58; N, 6.51. Found: C, 37.30; H, 2.61; N, 6.53.

methyl 2-(((3-phenylisoxazol-5-yl)methyl)(pyridin-2-ylmethyl)amino)acetate, 6.

A 20 mL scintillation vial was charged with **2** (77.7 mg, 0.35 mmol), **4** (107.6 mg, 0.69 mmol) and methylene chloride (2 mL). The dropwise addition of $NaHCO_{3(aq)}$ (1.0 M, 0.69 mmol) produced a colorless heterogeneous solution. The solution was vigorously stirred at rt for 12 h and purified by prep RP-HPLC to yield **6** as a yellow oil (58.7 mg, 50%). ^{1}H NMR [δ (ppm), CD_3OD] 8.73 (d, 1H, J = 5.9), 8.45 (td, 1H, J = 1.6, 7.9), 8.00 (d, 1H, J = 8.1), 7.87 (t, 1H, J = 6.4), 7.76 – 7.73 (m, 2H), 7.48 – 7.45 (m, 3H), 6.79 (s, 1H), 4.43 (s, 2H), 4.20 (s, 2H), 3.81 (s, 2H), 3.73 (s, 3H); ^{13}C NMR [δ (ppm), CD_3OD] 172.9, 170.7, 163.8, 156.6, 146.8, 142.9, 131.3, 130.1, 129.8, 127.7, 127.2, 126.6, 103.5, 56.5, 56.1, 52.3, 50.4; $\lambda_{max}(CH_3OH)$/nm (ε/dm^3 mol^{-1} cm^{-1})**:** 242 (16,400); υ_{max}/cm^{-1}: 1609 (Isox Ring), 1171 (Isox C-H), 1126 (Isox Ring); m/z 338.2 ([M+H$^+$] 100%).

2-(((3-phenylisoxazol-5-yl)methyl)(pyridin-2-ylmethyl)amino)acetic acid, 7.

Compound **6** (58.7 mg, 0.17 mmol) was dissolved in methanol (1.5 mL). After dropwise addition of $LiOH_{(aq)}$ (0.5 mL, 1.0 M), the reaction was stirred 15 h at rt and purified by prep RP-HPLC to yield **7** as a light yellow oil (27.6 mg, 49 %). ^{1}H NMR [δ (ppm), CD_3OD] 8.73 (d, 1H, J = 5.7), 8.43 (td, 1H, J = 1.5, 7.9), 7.97 (d, 1H, J = 8.1), 7.85 (t, 1H, J = 6.8), 7.78 – 7.72 (m, 2H), 7.49 – 7.44 (m, 3H), 6.80 (s, 1H), 4.42 (s, 2H), 4.21 (s, 2H), 3.78 (s, 2H); ^{13}C NMR [δ (ppm), CD_3OD] 174.4, 170.7, 163.9, 156.6, 147.1, 142.7, 131.4, 130.1, 129.9, 127.8, 127.3, 126.7, 103.7, 56.7, 56.6, 50.5; $\lambda_{max}(CH_3OH)$/nm (ε/dm^3

mol^{-1} cm^{-1})**:** 245 (5,147); υ_{max}/cm^{-1}: 1620 (Isox Ring), 1180 (Isox C-H), 1127 (Isox Ring); m/z 324.2 ([M+H^+] 100%).

***fac*-[$Re^{I}(CO)_3$(7)], 9.**

***Chelate, then Click*:** A 20 mL scintillation vial was charged with [$Re^{I}(CO)_3$(**3**)], **11**, (20.4 mg, 0.04 mmol) and **4** (14.5 mg, 0.09 mmol) in methylene chloride (2 mL). The dropwise addition of $NaHCO_{3(aq)}$ (1.0 M, 0.09 mmol) produced a colorless heterogeneous solution. Methanol (1 mL) was added dropwise to facilitate solubility. The solution was vigorously stirred at rt for 12 h producing a white precipitate that was collected via vacuum filtration. The filtered material was re-dissolved in 3:1 DCM:MeOH and recrystallized via slow evaporation (18.7 mg; 73 %).

***Click, then Chelate*:** To a methanolic solution of **7** (44.8 mg, 0.13 mmol) in a 20 mL scintillation vial, 0.1 M *fac*-[$Re^{I}(OH_2)_3(CO)_3$]$(OSO_2CF_3)_{(aq)}$ (1.524 mL, 0.15 mmol) was added slowly while stirring. 1.0 M solution of $NaHCO_{3(aq)}$ was added dropwise to neutralize the solution to pH 6. The vial was sealed and stirred for 12 h at 40 °C producing a white precipitate that was collected via vacuum filtration. The filtered material was re-dissolved in 2:1 DCM:MeOH and X-ray quality crystals of **9** were grown by slow evaporation of DCM (45.1 mg; 59 %). ^{1}H NMR [δ (ppm), $(CD_3)_2SO$] 8.76 (d, 1H, J = 5.0), 8.12 (td, 1H, J = 1.3, 7.8), 7.90 – 8.87 (m, 2H), 7.72 (d, 1H, J = 7.7), 7.59 – 7.52 (m, 4H), 7.39 (s, 1H), 4.88 (ABq, 2H, $\Delta\delta_{AB}$ = 0.07, J = 14.5), 4.69 (ABq, 2H, $\Delta\delta_{AB}$ = 0.05, J = 15.5), 3.71 (ABq, 2H, $\Delta\delta_{AB}$ = 0.38, J = 16.7); ^{13}C NMR [δ (ppm), $(CD_3)_2SO$] 178.21, 166.34, 162.01, 158.81, 152.07, 140.59, 130.50, 129.28, 128.25, 126.67, 126.03, 124.23, 105.78, 99.56, 68.18, 61.18; $\lambda_{max}(CH_3OH)$/nm (ε/dm^3 mol^{-1} cm^{-1})**:** 243 (19,231), 305 (3,573); υ_{max}/cm^{-1}: 2013, 1903, and 1873 (CO), 1609 (Isox Ring), 1160 (Isox C-H); m/z 594.2 ([M+H^+] 100 %); Calc for $C_{21}H_{16}N_3O_6Re{\cdot}CH_3OH$: C, 42.30; H, 3.22; N, 6.27. Found: C, 42.07; H, 2.73; N, 6.59.

Cysteine and Histidine Challenge Studies

Complex stability studies were conducted on purified samples of **8** and **9**. The Re complexes (100 μL, 100 μM) were added to an equal volume of cysteine or histidine solution (100 μL, 2 mM) dissolved in a phosphate buffer (10 mM, pH 7.4) to give a final amino acid concentration of 1 mM. The solutions were incubated at 37 °C in a temperature controlled water bath that were sampled at 1, 4, and 24 h and analyzed by analytical HPLC. The relative ratio of the product peak was compared to the additional peaks observed in the chromatogram to determine the stability of the product under the respective amino acid challenge conditions.

General *fac*-[^{99m}Tc(OH$_2$)$_3$(CO)$_3$]$^+$ radiolabeling procedure, 8A, 9A, 10A, 11A

The ligand **1, 3, 5, 7** (100 μL, 10^{-3}, 10^{-4} or 10^{-5} M) and phosphate buffer (800 μL, 0.01 M, pH 7.4) was added to a sealable labeling vial (5.0 mL). The vial was sealed and degassed with nitrogen for ~10 min. The *fac*-[^{99m}Tc(OH$_2$)$_3$(CO)$_3$]$^+$ precursor solution (100 μL) was prepared in an Isolink® kit following Tyco specifications. The solution was added to a degassed vial and the vial heated for 30 min at 70° C. The reaction mixture was then carefully allowed to cool on an ice bath prior to injection and analysis by radio-HPLC. Purified **10A** and **11A** were blown down under nitrogen to yield a solid residue that was dissolved in dry DCM (500 μL) prior to subsequent reactions.

Chelate, then Click

The oxime **4** (200 μL, 10^{-3} M in DCM) was added to a sealable labeling vial (5.0 mL). The vial was fitted with a micro stir bar, sealed and degassed with nitrogen for ~10 min, 400 μL of dry DCM was added and the vial degassed for one additional minute. Purified **10A** or **11A** (100 μL) were added to the

sealed vial followed by $NaHCO_3$ (20 μL, 1.0 M). The reaction was stirred vigorously at rt for 2.5, 5, 30, and 60 min then blown down to dryness with nitrogen. The residue was dissolved in methanol (500 μL) prior to injection and analysis by radio-HPLC.

Cysteine and Histidine Challenge Studies

Complex stability studies were conducted on HPLC purified samples of **8A** and **9A**. The ^{99m}Tc samples (50 μL, ~10 μCi) were added to an equal volume of cysteine or histidine solution (50 μL, 2 mM) dissolved in a phosphate buffer (10 mM, pH 7.4) to give a final amino acid concentration of 1 mM. The solutions were incubated at 37 °C in a temperature controlled water bath that were sampled at 1, 4, and 24 h and analyzed by radio (γ)-HPLC. The relative ratio of the product peak was compared to the additional peaks observed in the chromatogram to determine the stability of the product under the respective amino acid challenge conditions.

Table S1. All bond distances (Å) and bond angles (°) for *fac*-[$Re^{I}(CO)_3$(**7**)], **9**

Re1	O4	2.125(4)	O7	C22	1.400(9)	N2	C6	H6A	109.4(4)
Re1	N1	2.172(4)	C22	H22A	0.96(1)	N2	C6	H6B	109.4(4)
Re1	N2	2.233(4)	C22	H22B	0.960(6)	C7	C6	H6A	109.3(4)
Re1	C2	1.909(5)	C22	H22C	0.961(8)	C7	C6	H6B	109.4(4)
Re1	C3	1.915(5)	O4	Re1	N1	78.4(1)	H6A	C6	H6B	108.0(5)
Re1	C1	1.914(6)	O4	Re1	N2	78.6(1)	O6	C13	C14	107.6(5)
O1	C1	1.148(7)	O4	Re1	C2	174.7(2)	O6	C13	C12	116.9(4)
O2	C2	1.157(7)	O4	Re1	C3	96.8(2)	C14	C13	C12	135.4(5)
O3	C3	1.154(6)	O4	Re1	C1	94.7(2)	C8	C9	H9	120.4(6)
O4	C4	1.302(7)	N1	Re1	N2	77.1(1)	C8	C9	C10	119.2(5)
O5	C4	1.222(7)	N1	Re1	C2	97.7(2)	H9	C9	C10	120.4(6)
O6	N3	1.412(6)	N1	Re1	C3	173.4(2)	N2	C12	C13	115.5(4)
O6	C13	1.354(6)	N1	Re1	C1	96.4(2)	N2	C12	H12A	108.4(4)
N1	C11	1.348(5)	N2	Re1	C2	97.1(2)	N2	C12	H12B	108.4(4)
N1	C7	1.357(6)	N2	Re1	C3	97.5(2)	C13	C12	H12A	108.4(4)
N2	C6	1.498(6)	N2	Re1	C1	171.4(2)	C13	C12	H12B	108.4(4)
N2	C12	1.499(5)	C2	Re1	C3	86.7(2)	H12A	C12	H12B	107.4(5)
N2	C5	1.498(8)	C2	Re1	C1	89.3(2)	C16	C21	H21	119.9(6)
N3	C15	1.312(7)	C3	Re1	C1	88.6(2)	C16	C21	C20	120.2(6)
C14	H14	0.930(6)	Re1	O4	C4	118.3(3)	H21	C21	C20	119.9(6)
C14	C15	1.372(7)	N3	O6	C13	108.1(4)	C16	C17	H17	120.2(6)
C14	C13	1.348(9)	Re1	N1	C11	124.4(3)	C16	C17	C18	119.7(5)
C16	C15	1.468(8)	Re1	N1	C7	116.1(3)	H17	C17	C18	120.1(6)
C16	C21	1.394(7)	C11	N1	C7	118.6(4)	Re1	C1	O1	178.1(5)
C16	C17	1.387(8)	Re1	N2	C6	106.9(3)	O4	C4	O5	124.3(5)
C11	H11	0.930(5)	Re1	N2	C12	112.8(3)	O4	C4	C5	115.7(4)
C11	C10	1.380(7)	Re1	N2	C5	107.8(3)	O5	C4	C5	119.8(5)
C7	C8	1.390(7)	C6	N2	C12	109.6(4)	N2	C5	C4	113.6(4)
C7	C6	1.496(6)	C6	N2	C5	110.1(4)	N2	C5	H5A	108.8(5)
C8	H8	0.931(5)	C12	N2	C5	109.5(4)	N2	C5	H5B	108.8(5)
C8	C9	1.376(7)	O6	N3	C15	106.1(4)	C4	C5	H5A	108.8(5)
C6	H6A	0.970(6)	H14	C14	C15	126.0(6)	C4	C5	H5B	108.9(5)
C6	H6B	0.970(5)	H14	C14	C13	126.1(6)	H5A	C5	H5B	107.7(5)
C13	C12	1.490(7)	C15	C14	C13	107.9(5)	C17	C18	H18	119.4(6)
C9	H9	0.931(6)	Re1	C2	O2	177.3(5)	C17	C18	C19	121.2(6)
C9	C10	1.384(8)	Re1	C3	O3	177.5(4)	H18	C18	C19	119.4(7)
C12	H12A	0.971(5)	C15	C16	C21	120.2(5)	C21	C20	H20	119.6(6)
C12	H12B	0.970(6)	C15	C16	C17	120.4(5)	C21	C20	C19	120.6(6)
C21	H21	0.930(7)	C21	C16	C17	119.5(5)	H20	C20	C19	119.7(6)
C21	C20	1.38(1)	N1	C11	H11	118.8(5)	C18	C19	C20	118.8(6)
C17	H17	0.930(5)	N1	C11	C10	122.3(5)	C18	C19	H19	120.6(7)
C17	C18	1.39(1)	H11	C11	C10	118.9(5)	C20	C19	H19	120.6(7)

C4	C5	1.520(5)	N1	C7	C8	121.4(4)	C11	C10	C9	119.1(5)
C5	H5A	0.969(6)	N1	C7	C6	115.3(4)	C11	C10	H10	120.4(5)
C5	H5B	0.970(6)	C8	C7	C6	123.3(4)	C9	C10	H10	120.5(5)
C18	H18	0.929(7)	C7	C8	H8	120.2(5)	H7	O7	C22	109.5(6)
C18	C19	1.387(9)	C7	C8	C9	119.4(5)	O7	C22	H22A	109.5(7)
C20	H20	0.930(6)	H8	C8	C9	120.4(5)	O7	C22	H22B	109.4(7)
C20	C19	1.38(1)	N3	C15	C14	110.2(5)	O7	C22	H22C	109.4(7)
C19	H19	0.930(7)	N3	C15	C16	121.3(5)	H22A	C22	H22B	109.5(8)
C10	H10	0.930(5)	C14	C15	C16	128.4(5)	H22A	C22	H22C	109.5(8)
O7	H7	0.820(4)	N2	C6	C7	111.4(4)	H22B	C22	H22C	109.5(8)

Table S2. Stability studies of **8**, **8A**, **9** and **9A** in competition with histidine or cysteine (1 mM) in phosphate buffer (10mM, pH 7.4) at 37 °C.

Complex	Amino Acid	1 h	4 h	24 h
8	Cys	>99%	>99%	>99%
	His	>99%	>99%	>99%
8A	Cys	>99%	>99%	>99%
	His	>99%	>99%	>99%
9	Cys	>99%	>99%	>99%
	His	>99%	>99%	>99%
9A	Cys	>99%	>99%	>99%
	His	>99%	>99%	>99%

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