Supporting information

for

A N-Heterocyclic Carbene Gold Hydroxide Complex: A Golden Synthon

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Part 1: Preparation of complexes **1-9** and **10a-10b**.

General Considerations. All reactions were carried out in air unless otherwise stated. In such exceptions, manipulations were performed using standard Schlenk techniques using inert atmosphere of dry Argon or in a MBraun glovebox containing dry Argon and less than 1ppm oxygen. Anhydrous solvents were either distilled from appropriate drying agents or purchased from Aldrich and degassed prior to use by purging with dry Argon and kept over molecular sieves. Solvents for NMR spectroscopy were degassed with Argon and dried over molecular sieves. NMR spectra were recorded on a 400 MHz Varian Gemini spectrometer. Elemental analyses were performed by St Andrews analytical services.



[Au(Cl)(IPr)] (1): [Au(OH)(IPr)] 2 (50 mg, 0.083 mmol) and chlorodimethylphenylsilane (28.2 mg, 0.166 mmol) were charged in a vial containing toluene (1.0 mL). The reaction was stirred at room temperature for 6h. Solvent was reduced under vacuum and pentane (3 mL) was added. The resulting precipitate was filtered using a collection frit, the solid was washed with pentane (3 x 3 mL) and dried under vacuum to afford 1 as a white microcrystalline solid (51.5 mg, quantitative).¹



General procedure for the synthesis of [Au(OH)(IPr)] (2): A scintillation vial was charged with [AuCl(IPr)] (100 mg, 0.161 mmol) and the alkali metal salt (0.322 mmol). To the vial was then added a mixture of toluene and THF 1:1 (3.2 mL). The reaction mixture was stirred at 60°C for 24h. The resulting mixture was filtered through a pad of Celite[®], the cake washed with THF (2 x 2 mL) and the filtrate reduced in volume under vacuum with the resulting precipitate isolated by decantation. This solid was washed with toluene (2 x 2 mL) and pentane (3 x 2 mL) and dried under vacuum to yield **2** as a microcrystalline white solid.²

¹H NMR (400 MHz, CDCl₃): δ 7.49 (t, J = 7.8 Hz, 2H, CH aromatic), 7.35 (d, J = 7.8 Hz, 4H, CH aromatic), 7.12 (s, 2H, CH imidazole), 2.57 (sept, J = 6.9 Hz, 4H, CH(CH₃)₂), 1.35 (d, J = 6.9 Hz, 12H, CH(CH₃)₂), 1.21 (d, J = 6.9 Hz, 12H, CH(CH₃)₂) ppm. ¹³C NMR (125 MHz, CDCl₃): δ 171.9 (s, C carbene), 146.2 (s, C aromatic), 134.8 (s, C aromatic), 130.7 (s, CH imidazole), 124.5 (s, CH aromatic), 29.1 (s, CH(CH₃)₂), 24.4 (s, CH(CH₃)₂), 24.1 (s, CH(CH₃)₂) ppm. IR (KBr) 3678, 3627, 3392 (br), 3158, 3133, 3075, 2961, 2928, 2868, 1469, 1417, 1362, 1330, 806,

¹ M. R. Fructos, T. R. Belderrain, P. de Frémont, N. M. Scott, S. P. Nolan, M. M. Díaz-Requejo and P. J. Pérez, *Angew. Chem.*, *Int. Ed.* 2005, **44**, 5284-5288.

 $^{^{2}}$ The deuterated solvents used in the preparation of NMR samples were passed through basic alumina to remove any traces of acid that could partially reconvert 2 into 1.

763 cm⁻¹; Anal. Calcd for C₂₇H₃₈AuN₂O: C, 53.82; H, 6.19; N, 4.65. Found: C, 53.78; H, 6.13; N, 4.60.



[Au(CN)(IPr)] (3): [Au(OH)(IPr)] 2 (50 mg, 0.083 mmol) and trimethysilyl cyanide³ (22 μ L, 0.166 mmol) were charged in a vial containing toluene (0.8 mL). The reaction was stirred at room temperature for 7h. Solvent was reduced under vacuum and the residue triturated with pentane (3 mL). The resulting suspension was filtered using a collection frit, the solid was washed with pentane (3 x 3 mL) and dried under vacuum to afford **3** as a white microcrystalline solid (47.7 mg, 94 %).

¹H NMR (400 MHz, CDCl₃): δ 7.52 (t, J = 7.8 Hz, 2H, CH aromatic), 7.29 (d, J = 7.8 Hz, 4H, CH aromatic), 7.19 (s, 2H, CH imidazole), 2.48 (sept, J = 6.9 Hz, 4H, CH(CH₃)₂), 1.30 (d, J = 6.9 Hz, 12H, CH(CH₃)₂), 1.21 (d, J = 6.9 Hz, 12H, CH(CH₃)₂) ppm. ¹³C NMR (100 MHz, CDCl₃): δ 186.2 (s, C carbene), 152.3 (s, CN), 145.5 (s, C aromatic), 133.5 (s, C aromatic), 130.8 (s, CH imidazole), 124.2 (s, CH aromatic), 123.5 (s, CH aromatic), 28.8 (s, CH(CH₃)₂), 24.5 (s, CH(CH₃)₂), 24.0 (s, CH(CH₃)₂) ppm. Anal. Calcd for C₂₈H₃₆AuN₃: C, 54.99; H, 5.93; N, 6.87. Found: C, 54.61; H, 5.98; N, 6.57.



[Au(OAc)(IPr)] (4): [Au(OH)(IPr)] 2 (30 mg, 0.050 mmol) and acetic acid (6.0 mg, 0.10 mmol) were charged into a vial containing toluene (0.5 mL). The reaction was stirred at room temperature for 6h. Pentane (3 mL) was added and the resulting precipitate was collected on a frit. The solid was washed with pentane (3 x 2 mL) and dried under vacuum to afford 4 as a white microcrystalline solid (30.1 mg, 94%).

¹H NMR (400 MHz, CD₂Cl₂): δ 7.56 (t, J = 7.8 Hz, 2H, CH aromatic), 7.35 (d, J = 7.8 Hz, 4H, CH aromatic), 7.24 (s, 2H, CH imidazole), 2.56 (sept, J = 6.9 Hz, 4H, CH(CH₃)₂), 1.65 (s, 3H, CH₃C(O)O), 1.35 (d, J = 6.9 Hz, 12H, CH(CH₃)₂), 1.23 (d, J = 6.9 Hz, 12H, CH(CH₃)₂) ppm. ¹³C NMR (100 MHz, CD₂Cl₂): δ 175.9 (s, C carbene), 168.6 (s, C=O acetate), 146.2 (s, C aromatic), 134.5 (s, C aromatic), 131.0 (s, CH imidazole), 124.6 (s, CH aromatic), 123.8 (s, CH aromatic), 29.2 (s, CH(CH₃)₂), 24.4 (s, CH(CH₃)₂), 24.1 (s, CH(CH₃)₂), 23.9 (s, CH₃ acetate) ppm. Anal. Calcd for C₂₉H₃₉AuN₂O₂: C, 54.04; H, 6.10; N, 4.35. Found: C, 54.07; H, 5.97; N, 4.01.



³ CAUTION: The use of trimethylsilyl cyanide should be conducted in a well-ventilated hood.

[Au(CH(CO₂Me)₂)(IPr)] (**5**): [Au(OH)(IPr)] **2** (50 mg, 0.083 mmol) and dimethyl malonate (11.5 mg, 0.083 mmol) were charged into a vial containing benzene (0.5 mL). The reaction was stirred at room temperature for 6h. Pentane (3 mL) was added and the resulting precipitate was collected on a frit. The solid was washed with pentane (3 x 3 mL) and dried under vacuum to afford **5** as a white microcrystalline solid (54.2 mg, 91%).

¹H NMR (400 MHz, CDCl₃): δ 7.50 (t, J = 7.8 Hz, 2H, CH aromatic), 7.28 (d, J = 7.8 Hz, 4H, CH aromatic), 7.15 (s, 2H, CH imidazole), 3.29 (s, 6H, OCH₃ malonate), 3.18 (s, 1H, CH malonate), 2.50 (sept, J = 6.9 Hz, 4H, CH(CH₃)₂), 1.30 (d, J = 6.9 Hz, 12H, CH(CH₃)₂), 1.21 (d, J = 6.9 Hz, 12H, CH(CH₃)₂) ppm. ¹³C NMR (100 MHz, CDCl₃): δ 186.8 (s, C carbene), 172.6 (s, C=O malonate), 145.7 (s, C aromatic), 133.9 (s, C aromatic), 130.4 (s, CH imidazole), 124.0 (s, CH aromatic), 122.9 (s, CH aromatic), 50.0 (s, OCH₃ malonate), 43.0 (s, CH malonate), 28.7 (s, CH(CH₃)₂), 24.3 (s, CH(CH₃)₂), 23.9 (s, CH(CH₃)₂) ppm. Anal. Calcd for C₃₂H₄₃AuN₂O₄: C, 53.63; H, 6.05; N, 3.91. Found: C, 53.80; H, 5.78; N, 4.14.



Synthesis from boronic acid: [Au(Ph)(IPr)] (6): [Au(OH)(IPr)] 2 (30 mg, 0.050 mmol) and phenyl boronic acid (6.7 mg, 0.055 mmol) were charged in a vial containing benzene (0.5 mL). The reaction was stirred at room temperature for 7h. The solution was filtered through Celite[®], the solvent was removed from the filtrate under vacuum and pentane (6 mL) was added. The resulting precipitate was collected on a frit, washed with pentane (3 x 3 mL) and dried under vacuum to afford 6 as a microcrystalline white solid (32.9 mg, 100%).

Synthesis from trifluoroborate salt: [Au(Ph)(IPr)] (6): [Au(OH)(IPr)] 2 (50 mg, 0.083 mmol) and potassium phenyltrifluoroborate (16.0 mg, 0.087 mmol) were charged in a vial containing benzene (0.8 mL). The reaction was stirred at room temperature for 7h. After this time, the solution was filtered through Celite[®], the solvent was removed from the filtrate under vacuum and pentane (6 mL) was added. The resulting precipitate was collected on a frit, washed with pentane (3 x 3 mL) and dried under vacuum to afford **6** as a white microcrystalline solid (50.0 mg, 91%).

¹H NMR (400 MHz, C₆D₆): δ 7.52 (dd, *J* = 7.8, 1.4 Hz, 2H, C*H* aromatic phenyl), 7.21 (m, 4H, 2 C*H* aromatic phenyl and 2 C*H* aromatic NHC), 7.07 (d, *J* = 7.8 Hz, 4H, C*H* aromatic NHC), 6.99 (tt, *J* = 7.5, 1.5 Hz, 1H, C*H* aromatic phenyl), 6.30 (s, 2H, C*H* imidazole), 2.66 (sept, *J* = 6.9 Hz, 4H, C*H*(CH₃)₂), 1.48 (d, *J* = 6.9 Hz, 12H, CH(CH₃)₂), 1.09 (d, *J* = 6.9 Hz, 12H, CH(CH₃)₂) ppm. ¹³C NMR (100 MHz, CDCl₃): δ 196.9 (s, *C* carbene), 169.7 (s, *C* phenyl), 145.7 (s, *C* aromatic), 140.5 (s, *C*H phenyl), 134.6 (s, *C* aromatic), 130.1 (s, *C*H imidazole), 126.6 (s, *C*H phenyl), 124.2 (s, *C*H phenyl), 123.9 (s, *C*H aromatic), 122.7 (s, *C*H aromatic), 28.8 (s, *C*H(CH₃)₂), 24.5 (s, CH(CH₃)₂), 23.9 (s, CH(CH₃)₂) ppm. Anal. Calcd for C₃₃H₄₁AuN₂: C, 59.81; H, 6.24; N, 4.23. Found: C, 59.92; H, 6.59; N, 4.52.



[Au(CCPh)(IPr)] (7): [Au(OH)(IPr)] **2** (50 mg, 0.083 mmol) and phenylacetylene (17 mg, 0.166 mmol) were charged in a vial containing toluene (0.8 mL). The reaction was stirred at room temperature for 6h. Solvent was removed under vacuum and pentane (6 mL) was added. The resulting precipitate was collected on a frit, washed with pentane (3 x 3 mL) and dried under vacuum to afford **7** as a white microcrystalline solid (51.5 mg, 90%).

¹H NMR (400 MHz, C₆D₆): δ 7.38 (t, J = 7.8 Hz, 2H, CH aromatic), 7.21-7.17 (m, 2H, CH phenylacetylide), 7.18 (d, J = 7.8 Hz, 4H, CH aromatic), 7.01 (s, 2H, CH imidazole), 7.01-6.91 (m, 3H CH phenylacetylide), 2.50 (sept, J = 6.9 Hz, 4H, CH(CH₃)₂), 1.27 (d, J = 6.9 Hz, 12H, CH(CH₃)₂), 1.10 (d, J = 6.9 Hz, 12H, CH(CH₃)₂) ppm. ¹³C NMR (100 MHz, CDCl₃): δ 190.9 (s, C carbene), 145.6 (s, C aromatic NHC), 134.3 (s, C aromatic acetylide), 132.2 (s, CH aromatic), 130.4 (s, CH imidazole), 129.2 (s, C acetylide), 127.4 (s, CH aromatic acetylide), 125.9 (s, C acetylide), 125.7 (s, CH acetylide), 124.1 (s, CH aromatic), 123.2 (s, CH aromatic), 103.7 (s, C acetylide), 28.8 (s, CH(CH₃)₂), 24.6 (s, CH(CH₃)₂), 24.0 (s, CH(CH₃)₂) ppm. Anal. Calcd for C₃₅H₄₁AuN₂: C, 61.22; H, 6.02; N, 4.08. Found: C, 61.50; H, 6.10; N, 4.28.



[Au(Ph)(IPr)] (8): In a glovebox, under an inert atmosphere of Argon, [Au(OH)(IPr)] 2 (30 mg, 0.050 mmol) and triethoxysilane (58 μ L, 0.050 mmol) were charged in a Schlenk flask containing benzene (0.5 mL). The reaction was stirred at room temperature for 14h. The solvent was then removed under vacuum to afford 8 as a white microcrystalline solid (28.3 mg, 97%).⁴

¹H NMR (400 MHz, CD₂Cl₂): δ 7.53 (t, *J* = 7.8 Hz, 2H, C*H* aromatic), 7.33 (d, *J* = 7.8 Hz, 4H, C*H* aromatic), 7.16 (s, 2H, C*H* imidazole), 3.39 (s, 1H, hydride), 2.61 (sept, *J* = 6.9 Hz, 4H, C*H*(CH₃)₂), 1.34 (d, *J* = 6.9 Hz, 12H, CH(CH₃)₂), 1.22 (d, *J* = 6.9 Hz, 12H, CH(CH₃)₂) ppm.



[Au(Ph)(IPr)] (9): [Au(OH)(IPr)] 2 (20 mg, 0.033 mmol) and HNTf₂ (10.3 mg, 0.365 mmol) were charged into a vial containing benzene (0.5 mL). The reaction was stirred at room temperature for 6h. Solvent was removed under vacuum and pentane (3 mL) was added. The resulting precipitate was

⁴ E. Y. Tsui, P. Müller, J. P. Sadighi, Angew. Chem., Int. Ed. 2008, 47, 8937-8940. Angew. Chem. 2008, 120, 9069-9072.

collected on a frit and washed with pentane (3 x 3 mL). The solid was dried under vacuum to afford **9** as a microcrystalline white solid (26 mg, 91%).⁵

¹H NMR (400 MHz, CDCl₃): δ 7.53 (t, J = 7.8 Hz, 2H, CH aromatic), 7.31 (d, J = 7.8 Hz, 4H, CH aromatic), 7.28 (s, 2H, CH imidazole), 2.48 (sept, J = 6.9 Hz, 4H, CH(CH₃)₂), 1.31 (d, J = 6.9 Hz, 12H, CH(CH₃)₂), 1.23 (d, J = 6.9 Hz, 12H, CH(CH₃)₂) ppm.



[Au(C₆F₅)(IPr)] (**11a**): [Au(OH)(IPr)] **2** (50 mg, 0.083 mmol) and pentafluorobenzene **10a** (27.9 mg, 0.166 mmol) were charged into a vial containing toluene (0.8 mL). The reaction was stirred at 60°C for 14h. The reaction mixture was allowed to cool to RT and solvent volume was reduced by half, under vacuum. Pentane (6 mL) was added and the resulting precipitate was collected on a frit and washed with pentane (3 x 3 mL). The solid was dried under vacuum to afford **11b** as a white microcrystalline solid (58.2 mg, 93%).

¹H NMR (400 MHz, C₆D₆): δ 7.18 (t, J = 7.8 Hz, 2H, CH aromatic), 7.05 (d, J = 7.8 Hz, 4H, CH aromatic), 6.29 (s, 2H, CH imidazole), 2.59 (sept, J = 6.9 Hz, 4H, CH(CH₃)₂), 1.49 (d, J = 6.9 Hz, 12H, CH(CH₃)₂), 1.08 (d, J = 6.9 Hz, 12H, CH(CH₃)₂) ppm. ¹⁹F (376.5 MHz, C₆D₆): δ -116.5 - -116.7 (m, 2F), -161.4 - -161.6 (m, 1F), -164.0 - -164.2 (m, 2F) ppm. ¹³C NMR (100 MHz, C₆D₆): δ (C pentafluorobenzyl not observed), 192.5 (s, C carbene), 151.0-150.6 (dm, ¹J_{C-F} = 226 Hz, C-F pentafluorobenzyl), 145.9 (s, C aromatic NHC), 140.1-137.2 (dm, ¹J_{C-F} = 243 Hz, C-F pentafluorobenzyl), 138.8-135.8 (dm, ¹J_{C-F} = 250 Hz, C-F pentafluorobenzyl), 134.5 (s, CH aromatic), 120.9 (s, CH imidazole), 124.2 (s, CH aromatic), 122.8 (s, CH aromatic), 29.1 (s, CH(CH₃)₂), 24.7 (s, CH(CH₃)₂), 23.9 (s, CH(CH₃)₂) ppm. Anal. Calcd for C₃₃H₃₆AuF₅N₂: C, 52.66; H, 4.82; N, 3.72. Found: C, 53.02; H, 4.55; N, 4.02.



[Au(C₆HF₄)(IPr)] (**11b**): [Au(OH)(IPr)] **2** (50 mg, 0.083 mmol) and tetrafluorobenzene **10b** (27.9 mg, 0.166 mmol) were charged into a vial containing toluene (0.8 mL). The reaction was stirred at 80°C for 24h. The reaction mixture was allowed to cool to room temperature and solvent volume was reduced by half, under vacuum. Pentane (6 mL) was added and the resulting precipitate was collected on a frit and washed with pentane (3 x 3 mL). The solid was dried under vacuum to afford **12b** as a white microcrystalline solid (52.4 mg, 86%).

⁵ a) N. Mezailles, L. Ricard, F. Gagosz, *Org. Lett.* **2005**, *7*, 4133-4136. b) L. Ricard, F. Gagosz, *Organometallics* **2007**, *26*, 4704-4707.

¹H NMR (400 MHz, C₆D₆): δ 7.17 (t, J = 7.8 Hz, 2H, *CH* aromatic), 7.04 (d, J = 7.8 Hz, 4H, *CH* aromatic), 6.29 (s, 2H, *CH* imidazole), 6.15 (tt, $J_{\text{H-F}} = 6.9$, 9.5 Hz, 1H, *CH* aromatic tetrafluoroaryl), 2.60 (sept, J = 6.9 Hz, 4H, *CH*(CH₃)₂), 1.49 (d, J = 6.9 Hz, 12H, CH(*CH*₃)₂), 1.08 (d, J = 6.9 Hz, 12H, CH(*CH*₃)₂) ppm. ¹⁹F (376.5 MHz, C₆D₆): δ -118.2 - -118.4 (m, 2F), -141.7 - -141.9 (m, 2F) ppm. ¹³C NMR (100 MHz, C₆D₆): δ (*C* tetrafluoroaryl not observed), 193.1 (s, *C* carbene), 151.4-148.8 (dm, ¹ $J_{\text{C-F}} = 226$ Hz, *C*-F tetrafluoroaryl), 147.4-144.6 (dm, ¹ $J_{\text{C-F}} = 248$ Hz, *C*-F tetrafluoroaryl), 145.8 (s, *C* aromatic NHC), 134.5 (s, *C*H aromatic), 130.8 (s, *C*H imidazole), 124.2 (s, *C*H aromatic), 122.8 (s, *C*H aromatic), 102.4 (t, ² $J_{\text{C-F}} = 23.5$ Hz, *C*H tetrafluoroaryl), 29.1 (s, *C*H(CH₃)₂), 24.7 (s, CH(CH₃)₂), 23.9 (s, CH(*C*H₃)₂) ppm. Anal. Calcd for C₃₃H₃₇AuF₄N₂: C, 53.95; H, 5.08; N, 3.81. Found: C, 54.25; H, 4.74; N, 4.09.

| | Au(OH)(IPr) 2 | Au(CN)(IPr) 3 | Au(OAc)(IPr) 4 | $Au(CH(CO_2Me)_2)(IPr)$ 5 | Au(Ph)(IPr) 6 | Au(CCPh)(IPr) 7 |
|--|----------------------|--|---|---------------------------|---|---|
| Formula | $C_{27}H_{37}AuN_2O$ | C ₂₈ H ₃₆ AuN ₃ .2CHCl ₃ | C ₂₉ H ₃₉ AuN ₂ O ₂ .0.5CHCl ₃ | $C_{32}H_{43}AuN_2O_4$ | C ₃₃ H ₄₁ AuN ₂ ,CHCl ₃ | C ₃₅ H ₄₁ AuN ₂ ,CHCl ₃ |
| M/g.mol ⁻¹ | 602.55 | 850.30 | 704.27 | 716.65 | 782.01 | 806.03 |
| Crystal system | Monoclinic | Triclinic | Monoclinic | Triclinic | Monoclinic | Monoclinic |
| Space group | $P2_{1}/c$ | <i>P</i> -1 | $P2_{1}/c$ | <i>P</i> -1 | $P2_1/n$ | $P2_1/n$ |
| a/ Å | 12.239(4) | 10.415(8) | 17.530(5) | 9.218(3) | 10.598(3) | 10.8026(15) |
| b/ Å | 9.380(2) | 12.852(5) | 23.690(6) | 10.0459(15) | 15.354(4) | 14.985(2) |
| c/ Å | 23.006(7) | 15.096(8) | 16.642(5) | 18.472(6) | 21.195(5) | 22.788(3) |
| α / ° | 90 | 68.80(5) | 90 | 79.63(3) | 90 | 90 |
| eta/ ° | 95.899(7) | 88.93(7) | 117.753(7) | 89.03(3) | 98.952(7) | 100.880(6) |
| $\gamma/$ ° | 90 | 77.34(6) | 90 | 66.719(16) | 90 | 90 |
| $V/ Å^3$ | 2627.3(12) | 1834.0(18) | 6116(3) | 1542.9(7) | 3406.9(15) | 3622.6(8) |
| Z | 4 | 2 | 8 | 2 | 4 | 4 |
| $\rho calcd$ / g.cm ⁻³ | 1.523 | 1.540 | 1.530 | 1.543 | 1.525 | 1.478 |
| μ (Mo K _a)/ mm ⁻¹ | 5.619 | 4.471 | 4.968 | 4.804 | 4.578 | 4.308 |
| T/ K | 93(2) | 93(2) | 93(2) | 293(2) | 366(2) | 366(2) |
| No of reflections | 16266 | 11540 | 11018 | 9756 | 21408 | 18875 |
| No of unique refl | 3627 | 6431 | 11111 | 5448 | 6204 | 6450 |
| $R_{\rm int}$ | 0.0822 | 0.1630 | 0.1439 | 0.0773 | 0.0906 | 0.0928 |
| $R1, wR_2 (I > 2\sigma(I))$ | 0.0546, 0.1347 | 0.1346, 0.2898 | 0.0825, 0.1571 | 0.0640, 0.1561 | 0.0653, 0.1342 | 0.0500, 0.1211 |
| $R1$, w R_2 (all data) | 0.0756, 0.1547 | 0.2083, 0.3484 | 0.1880, 0.1962 | 0.0763, 0.1832 | 0.1073, 0.1544 | 0.0605, 0.1295 |
| GOF | 1.087 | 1.000 | 0.976 | 1.075 | 1.082 | 1.085 |

2. Crystallographic data for compounds 2-7

















¹H NMR (400 MHz, $CDCl_3$)







¹H NMR (400 MHz, CD_2Cl_2)









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5

¹H NMR (400 MHz, CDCl₃)



S15













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¹H NMR (400 MHz, CDCl₃)

10a ¹⁹F NMR (376.5 MHz, C₆D₆)

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10b

 19 F NMR (376.5 MHz, C₆D₆)

