Data Article

Dataset of polyoxometalate-assisted N-heterocyclic carbene gold(I) complexes

Kenji Nomiya*, Yuichi Murata, Yuta Iwasaki, Hidekazu Arai, Takuya Yoshida, Noriko Chikaraishi Kasuga, Toshiaki Matsubara**

Department of Chemistry, Faculty of Science, Kanagawa University, Hiratsuka, Kanagawa 259-1293, Japan

ARTICLE INFO

Article history:
Received 28 February 2019
Received in revised form 9 May 2019
Accepted 9 May 2019
Available online 24 May 2019

Keywords:
Polyoxometalate
N-Heterocyclic carbene ligand
Gold(I) complex
Homogeneous catalysis
Hydration of diphenylacetylene

ABSTRACT

The present paper is the Supplemental materials for our original paper entitled “highly active, homogeneous catalysis by polyoxometalate-assisted N-heterocyclic carbene gold(I) complexes for hydration of diphenylacetylene. The present article refers to the preparations of several monomeric, N-heterocyclic (NHC) carbene/carboxylate (RS-pyrrld)/gold(I) complexes, [Au(RS-pyrrld)(NHC)] (NHC = IMes (6), BIPr (7), IF3 (8), fBu (9)), which were used for homogenous catalysis of the hydration reaction of diphenylacetylene to afford deoxybenzoin. The article also includes the preparations of the precursor complexes, [AuCl(NHC)] (NHC = IPr, IMes, BIPr, IF3, fBu), and novel X-ray crystallography of the separately prepared [Au(IPr)(H2O)]3[a-PW12O40]7Et2O (2), summary of crystal data of (2), and selected bond distances (Å) and angles (deg) of (2). Also presented are Cartesian coordinates of the optimized structures in the quantum-mechanical calculations.

© 2019 The Author(s). Published by Elsevier Inc. This is an open access article under the CC BY license (http://creativecommons.org/licenses/by/4.0/).

DOI of original article: https://doi.org/10.1016/j.mcat.2019.02.014.

* Corresponding author.
** Corresponding author.
E-mail addresses: nomiyk01@jindai.jp (K. Nomiya), matsubara@kanagawa-u.ac.jp (T. Matsubara).

https://doi.org/10.1016/j.dib.2019.104002
2352-3409/© 2019 The Author(s). Published by Elsevier Inc. This is an open access article under the CC BY license (http://creativecommons.org/licenses/by/4.0/).
Data presented in this article displays the preparations of several precursors used for homogenous catalysis of the hydration reaction of diphenylacetylene to afford deoxybenzoin; monomeric, N-heterocyclic carbene/carboxylato/gold(I) complexes, \([\text{Au}((R)-\text{pyriddy})(\text{NHC})]\) (NHC = IMes (6), BIPr (7), IF\(^3\) (8), ItBu (9)), as well as the precursor complexes, \([\text{AuCl}((\text{NHC})]\) (NHC = IPr, IMes, BIPr, IF\(^3\), ItBu) [1]. Also presented are summary of crystal data of the separately prepared \([\text{Au}(\text{IPr})(\text{H}_2\text{O})]\)(\(\text{a-PW}_{12}\text{O}_{40}\))\(\cdot\)7Et\(_2\text{O}\) (2) (Table 1), selected bond distances (Å) and angles (deg) of (2) (Table 2), and Cartesian coordinates of the optimized structures in the quantum-mechanical calculations (Table 3). The \(^1\text{H}\) NMR and IR spectra of \([\text{Au}((R)-\text{pyriddy})(\text{IPr})]\) (1) and \(^1\text{H}\) NMR spectrum of \([\text{Au}(\text{H}_2\text{O})(\text{IPr})]\)(\(\text{a-PW}_{12}\text{O}_{40}\))\(\cdot\)7Et\(_2\text{O}\) (2) are shown in Figs. 1–3, respectively.
Table 1
Summary of crystal data of \([\text{Au(H}_2\text{O})(\text{IPr})\text{]}_3[\text{PW}_{12}\text{O}_{40}]\cdot 7\text{Et}_2\text{O}\ (2)\).

| Property                        | Value                        |
|---------------------------------|------------------------------|
| Empirical formula               | \(\text{C}_{109}\text{H}_{178}\text{Au}_3\text{N}_6\text{O}_{50}\text{PW}_{12}\) |
| Formula weight                  | 1268.75                      |
| Crystal system                  | Orthorhombic                 |
| Space group                     | \(\text{P}_2_1\text{2}_1\text{2}_1\) (No.19) |
| \(a/\text{Å}\)                  | 21.4563 (2)                  |
| \(b/\text{Å}\)                  | 21.6021 (2)                  |
| \(c/\text{Å}\)                  | 31.3646 (3)                  |
| \(\alpha^\prime\)              | 90                           |
| \(\beta^\prime\)               | 90                           |
| \(\gamma^\prime\)              | 90                           |
| \(V/\text{Å}^3\)               | 14537.5 (2)                  |
| \(D_{\text{calc/d}}/\text{g-cm}^{-3}\) | 2.376                        |
| \(Z\)                           | 4                            |
| \(\mu/\text{mm}^{-1}\)         | 12.553                       |
| \(T/\text{K}\)                  | 100                          |
| No. of reflections              | Total 196846                  |
|                                | Unique 33392                  |
|                                | \((I > 2\sigma(I))\)         | 32699                        |
| \(R_{\text{int}}\)             | 0.0595                       |
| \(R_1 (I > 2\sigma(I))\)       | 0.0246                       |
| \(wR_2 (I > 2\sigma(I))\)      | 0.0626                       |
| \(\text{GOF}\)                 | 1.068                        |

\(R_1 = \Sigma[(|\text{Fo}| - |\text{Fc}|)]/\Sigma|\text{Fo}|, \ wR_2 = [\Sigma\omega(|\text{Fo}| - |\text{Fc}|)^2/\Sigma\omega|\text{Fo}|^2]^{1/2}, \ \text{GOF} = [\Sigma\omega(|\text{Fo}|-|\text{Fc}|)^2/(m-n)]^{1/2} m; \ \text{No. of reflections, n; No. of parameters.}

Table 2
Selected bond distances (Å) and angles (deg) of \([\text{Au(H}_2\text{O})(\text{IPr})\text{]}_3[\text{PW}_{12}\text{O}_{40}]\cdot 7\text{Et}_2\text{O}\ (2)\).

| Bond            | Distance (Å) |
|-----------------|--------------|
| Au (1)--O (41)  | 2.068 (5)    |
| Au (1)--C (1)   | 1.948 (7)    |
| Au (2)--O (42)  | 2.062 (5)    |
| Au (2)--C (28)  | 1.936 (5)    |
| Au (3)--O (43)  | 2.071 (5)    |
| Au (3)--C (55)  | 1.955 (6)    |
| C (1)--Au (1)--O (41) | 177.5 (3) |
| C (28)--Au (2)--O (42) | 179.2 (3) |
| C (55)--Au (3)--O (43) | 177.5 (3) |
| O (41) ... O (44) | 2.526 (14) |
| O (41) ... O (45) | 2.602 (11) |
| O (42) ... O (47) | 2.657 (10) |
| O (42) ... O (49) | 2.581 (10) |
| O (43) ... O (46) | 2.606 (10) |
| O (43) ... O (48) | 2.641 (11) |

Symmetry operations; \(i = -x, 0.5 + y, 0.5 - z\); \(ii = 1 - x, 0.5 + y, 0.5 - z\).

2. Experimental design, materials, and methods

CHN elemental analyses were carried out using a PerkinElmer 2400 CHNS Elemental Analyzer II (Kanagawa University). IR spectra were recorded on a Jasco 4100 FT-IR spectrometer in KBr disks at room temperature. TG/DTA was performed using a Rigaku Thermo Plus 2 series TG/DTA TG 8120 instrument.

The \(^1\text{H}\) NMR (400 MHz), \(^{31}\text{P}\{^1\text{H}\}\) NMR (161 MHz) and \(^{13}\text{C}\{^1\text{H}\}\) NMR (99 MHz) spectra of the samples were recorded in 5-mm-outer-diameter tubes on a JEOL NMR-ECA 400 FT-NMR or a JEOL JNM-ECS-400 FT-NMR spectrometer and a JEOL ECA-400 NMR or ECS-400 NMR data processing system, respectively. The \(^1\text{H}\) and \(^{13}\text{C}\{^1\text{H}\}\) NMR spectra were referenced to an internal TMS. The \(^{31}\text{P}\{^1\text{H}\}\) NMR spectra were
Table 3
Cartesian coordinates (in Å).

| Atom | x/C0 | y/C0 | z/C0 |
|------|------|------|------|
| Au   | 0.000097 | -0.000050 | -1.609283 |
| C    | 0.000038 | 0.000136 | 0.381573 |
| N    | -1.078422 | -0.026872 | 1.1811125 |
| C    | 0.107839 | 0.027269 | 1.181062 |
| C    | -0.679079 | -0.022403 | 2.504786 |
| N    | 0.679117 | 0.022963 | 2.504745 |
| C    | 1.398147 | -0.048544 | 3.309907 |
| C    | 1.398229 | 0.049211 | 3.309822 |
| C    | -2.441449 | -0.103760 | 0.709816 |
| C    | 2.441407 | 0.103860 | 0.709673 |
| C    | -3.187295 | 1.084250 | 0.650852 |
| C    | -4.489906 | 0.982350 | 0.159775 |
| C    | -5.008357 | -0.244514 | -0.248124 |
| C    | -4.238808 | -1.399557 | -0.167035 |
| C    | -2.928722 | -1.358232 | 0.319063 |
| C    | -2.595226 | 2.427872 | 1.047910 |
| H    | -5.109623 | 1.871079 | 0.095716 |
| C    | -6.024449 | -0.299816 | -0.626590 |
| C    | -4.663446 | -2.349385 | -0.480186 |
| C    | -2.108410 | -2.634645 | 0.416430 |
| C    | 3.186994 | 1.084311 | 0.650703 |
| C    | 4.489642 | -0.982690 | 0.159657 |
| C    | 5.008379 | 0.244064 | 0.248191 |
| C    | 4.239093 | 1.399284 | 0.167063 |
| C    | 2.928885 | 1.358240 | 0.318987 |
| C    | 2.594708 | 2.427814 | 1.047838 |
| C    | -1.791182 | 2.250272 | 1.772222 |
| C    | -1.976001 | 3.120627 | -0.175859 |
| C    | -1.537478 | 4.082515 | 0.111330 |
| C    | -1.186685 | 2.510648 | -0.623929 |
| C    | -2.741420 | 3.307319 | -0.937472 |
| C    | -3.108415 | 4.231141 | 2.125851 |
| C    | -4.367999 | 3.709199 | 1.011537 |
| C    | -4.150888 | 2.846603 | 2.543189 |
| C    | 3.615436 | 3.349967 | 1.722877 |
| C    | 1.790199 | 2.249997 | 1.771583 |
| C    | 1.976287 | -3.121024 | -0.176074 |
| C    | 3.107307 | -4.230677 | 2.126757 |
| C    | 4.367563 | 3.709074 | 1.013044 |
| C    | 4.133655 | -2.845934 | 2.544230 |
| C    | 1.537333 | -4.082785 | 0.111194 |
| C    | 1.187301 | -2.511189 | -0.633368 |
| C    | 2.742194 | -3.308043 | -0.937116 |
| C    | 1.118591 | 2.391084 | 0.821335 |
| C    | 2.763627 | 3.633200 | 1.380600 |
| C    | 1.899484 | 3.265971 | -0.965724 |
| C    | -1.118151 | -2.390691 | 0.821552 |
| C    | -2.762990 | -3.633155 | 1.380652 |
| C    | -1.898642 | -3.265736 | -0.965579 |
| H    | 2.138686 | 4.526018 | 1.482985 |
| H    | 2.903322 | 3.193781 | 2.373277 |
| H    | 3.744427 | 3.952358 | 1.013053 |
| H    | 1.414537 | 2.563718 | -1.65166 |
| H    | 1.268567 | 4.157118 | -0.887434 |
| H    | 2.852011 | 3.567478 | 1.414150 |
| H    | -2.137878 | -4.525848 | 1.483099 |
H \quad -2.902884\quad -3.193773\quad 2.373318
H \quad -3.743687\quad -3.952498\quad 1.012988
H \quad -1.413774\quad -2.563371\quad -1.655964
H \quad -1.267525\quad -4.156734\quad -0.887208
H \quad -2.851033\quad -3.567483\quad -1.414128

[(IPr)Au(C2Ph2)]^+

\begin{align*}
\text{Au} &\quad 0.000394\quad 0.588216\quad 0.000911 \\
\text{C} &\quad -0.000726\quad 1.418942\quad -0.001712 \\
\text{N} &\quad 1.034455\quad 2.231477\quad 0.286980 \\
\text{N} &\quad 1.032137\quad 2.231779\quad -0.292623 \\
\text{C} &\quad 0.657220\quad 3.557563\quad 0.173318 \\
\text{C} &\quad 0.653501\quad 3.557764\quad -0.182669 \\
\end{align*}

C 2.349801\quad -1.757922\quad -0.635384 \\
C 2.688036\quad -1.673634\quad 1.992411 \\
C 3.962319\quad -1.191428\quad 2.298847 \\
C 4.848842\quad -0.820320\quad 2.90516 \\
C 4.477396\quad -0.914431\quad -0.405710 \\
C 3.212031\quad -1.384198\quad -0.049990 \\
C 1.701504\quad -2.041640\quad 3.090288 \\
C 4.271128\quad -1.111605\quad 3.336473 \\
C 5.840081\quad -0.460941\quad 1.552873 \\
C 5.179062\quad -0.617055\quad -0.821001 \\
C 2.827579\quad -1.478134\quad -1.878091 \\
C 2.686669\quad -1.669945\quad -1.995862 \\
C 3.961396\quad -1.187659\quad -2.300387 \\
C 4.848086\quad -0.820898\quad -1.290614 \\
C 4.476415\quad -0.919735\quad 0.045214 \\
C 3.210428\quad -1.389175\quad 0.407599 \\
C 1.700071\quad -2.033438\quad -3.095185 \\
H 4.270377\quad -1.104336\quad -3.376990 \\
H 5.839679\quad -0.461433\quad -1.551504 \\
H 5.178184\quad -0.625608\quad 0.821642 \\
H 2.825197\quad -1.486450\quad 1.875242 \\
H 2.372820\quad -2.727964\quad 4.284487 \\
H 0.973862\quad -2.748899\quad 2.675233 \\
H -0.928628\quad -0.796964\quad 3.533105 \\
H -0.215474\quad -1.063299\quad 4.340994 \\
H -0.307256\quad -0.335500\quad 2.730188 \\
H -1.618798\quad -0.047133\quad 3.956924 \\
H -1.609031\quad -3.102590\quad 4.972429 \\
H -3.002257\quad -2.032807\quad 4.849641 \\
H -2.994122\quad -3.571726\quad 3.968872 \\
C 2.371089\quad -2.716929\quad -4.291190 \\
H 0.971462\quad -2.741085\quad -2.682490 \\
C 0.928843\quad -0.786489\quad -3.554629 \\
H 1.607162\quad -3.088296\quad -4.980746 \\
H 3.001835\quad -2.080284\quad -4.853718 \\
H 2.991031\quad -3.562550\quad -3.977889 \\
H 0.216129\quad -1.049529\quad -4.344012 \\
H 0.370174\quad -0.327096\quad -2.730749 \\
H 1.620124\quad -0.036068\quad -3.955457 \\
H 1.809242\quad -1.894751\quad 1.947010 \\
C 3.759436\quad -2.445475\quad 2.624706 \\
C 2.815110\quad -0.101394\quad 2.535027 \\
H -1.811850\quad -1.886718\quad -1.951298 \\
C -3.762629\quad -2.435282\quad -2.628974 \\
C -2.817142\quad -0.091800\quad -2.535081 \\
H 3.431120\quad -2.557944\quad 3.662873 \\
H 3.772055\quad -3.435770\quad 2.158090 \\
H 4.787119\quad -2.067271\quad 2.639675 \\
H 2.096488\quad 0.568785\quad 2.048531
H 2.542281−0.187313 3.592121
H 3.800789 0.374373 2.476586
H 3.800789 0.374373 2.476586
H 3.800789 0.374373 2.476586
H 3.800789 0.374373 2.476586
H 3.800789 0.374373 2.476586
C 0.608991 2.829050 0.107363
C 0.606506 2.829697 0.099756
C 1.998472 3.030917 0.441925
C 1.995643 3.031377 −0.434768
C 3.011243 2.393890 −0.239229
C 4.341359 2.565489 0.098132
C 4.665875 3.463803 1.114469
C 3.657522 4.140193 1.801368
C 2.324008 3.926531 1.468544
H 2.755132 1.627085 1.022333
H 5.125698 2.038342 0.435704
H 5.706194 3.638278 1.371715
H 3.909847 4.837412 2.594773
H 1.531113 4.460446 1.981090
H −0.000561 0.023782 1.485084
C −0.000071 −0.009431 −0.492262
N 1.075729 0.007267 −1.302871
N 1.074310 −0.045101 −1.303227
C −0.679504 −0.011174 −2.627682
C 0.677599 −0.055525 −2.627871
H −1.398818 0.005115 −3.432688
C 1.396256 −0.089576 −3.432883
C −2.438517 0.087026 −0.837800
C 2.437651 −0.111578 −0.837043
C −3.186131 −1.099821 −0.773965
C −4.491589 −0.996174 −0.290848
C −5.013293 0.233114 0.105781
C −4.243510 1.387592 0.020338
C −2.930702 1.342981 −0.458333
C −2.590681 −2.445271 −1.158986
H −5.111685 −1.884687 −0.225803
H −6.032130 0.290731 0.476745
H −4.670253 2.339987 0.323000
C −2.112040 2.619722 −0.564597
C 3.179033 1.079542 −0.786128
C 4.483845 0.988350 −0.298134
C 5.011103 −0.233197 0.114113
C 4.247570 −1.392969 0.039984
C 2.935774 −1.360394 −0.441972
C 2.580174 2.417327 −1.192281
H 5.098595 1.881173 −0.241653
H 6.029468 −0.281084 0.487770
H 4.679090 −2.339703 0.353657
C 2.122371 −2.641576 −0.533495
C −3.609325 −3.378644 −1.822091

\[
[(\text{IPr})\text{Au(H}_2\text{O)}]^{+}
\]
| X  | Y  | Z         |
|----|----|-----------|
| H  | -1.789205 | -2.270726 -1.887492 |
| C  | -1.964080 | -3.123329 0.068307 |
| H  | -1.518041 | -4.083983 -0.211984 |
| H  | -1.180925 | -2.501596 0.518750 |
| H  | -2.728923 | -3.311101 0.830700 |
| H  | -3.098925 | -4.262246 -2.216932 |
| H  | -4.359268 | -3.732122 -1.106926 |
| H  | -4.103708 | -2.820245 -2.708905 |
| C  | 1.191660  | 2.515385 0.502838 |
| H  | 1.768712  | 2.228705 1.905181 |
| C  | 1.971501  | 3.123499 0.028807 |
| H  | 3.078575  | 4.207955 2.298135 |
| H  | 4.350897  | 3.702270 1.190691 |
| H  | 4.103708  | 2.820245 2.708905 |
| C  | 3.592334  | 3.333029 1.888579 |
| H  | 1.116128  | 2.369827 -0.950920 |
| C  | 2.756044  | 3.599037 1.555118 |
| H  | 1.922073  | 3.275432 0.808940 |
| C  | 2.157266  | -4.533405 -1.995441 |
| H  | 2.909252  | -3.196846 -2.494028 |
| H  | 3.762386  | -3.949933 -1.383329 |
| C  | 1.432574  | 2.570983 1.537175 |
| H  | 1.297829  | -4.168659 0.773861 |
| C  | 2.879043  | -3.567727 1.294266 |
| H  | 2.133011  | 4.492628 -1.663541 |
| C  | 2.880003  | 3.142899 -2.542422 |
| H  | 3.743405  | 3.920286 1.206928 |
| H  | 1.446628  | 2.586865 1.518263 |
| H  | 1.290400  | 4.165400 0.721105 |
| H  | 2.881175  | 3.587537 1.236111 |
| O  | -0.012789 | 0.110453 3.685545 |
| H  | 0.825203  | -0.050442 4.147939 |
| H  | -0.703174 | -0.386546 4.152821 |

\[
\text{[(PPh}_3\text{)Au]}^+ \\
\text{Au} = -0.004449 -0.011261 -2.044328 \\
P = -0.00622 0.001979 0.238992 \\
C = 1.153850 -1.247090 0.876880 \\
C = 0.510092 1.629023 0.863995 \\
C = -1.660838 -0.366992 0.877151 \\
C = 2.064052 -0.850550 1.866761 \\
C = 2.956235 -1.769293 2.409294 \\
C = 2.938043 -3.088157 1.963519 \\
C = 2.026210 -3.482676 0.987347 \\
C = 1.118066 -2.583279 0.421732 \\
C = 2.075410 0.176241 2.220857 \\
C = 3.654953 -1.453933 3.177218 \\
C = 3.629454 -3.814465 2.379299 \\
C = 2.012069 -4.516810 0.653954 \\
C = 0.429798 -2.940035 -0.338310 \\
C = -0.281643 2.227023 1.854613 \\
C = 0.074406 3.461054 2.388439 \\
C = 1.226071 4.097679 1.933967 \\
C = 2.016226 3.497832 0.956137 \\
C = 1.684028 2.260537 0.398433 \\
H = -1.175452 1.726795 2.216543 \\
H = -0.542530 3.915536 3.156660 \\
H = 1.516350 5.060027 2.344120 \\
H = 2.918721 3.998195 0.615537 \\
H = 2.331696 1.836802 -0.363388 
\]
C −1.774720−1.345802 1.874267
C −3.017223−1.653034 2.418385
C −4.148603−0.978488 1.967354
C −4.032088 0.00317 0.983249
C −2.799039 0.329827 0.415426
H −0.891675−1.866506 2.233269
H −3.095918−2.410238 3.191516
H −5.124141−2.08043 2.384746
H −4.919707 0.527978 0.644887
H −2.761675 1.098146−0.351180

\[(\text{PPh}_3\text{Au(C}_2\text{Ph}_2))^+\]

Au 0.791400 0.070986 0.048181
P −1.479093−0.266220−0.014319
C −1.941770−1.946346 −0.560908
C −2.253202−0.018372 1.620302
C −2.282750 0.898845−1.170516
C −2.941275−2.627848 0.145041
C −3.344124−3.900650−0.249163
C −2.748590−4.495085 −1.357590
C −1.762083−3.813044−2.065958
C −1.336895−2.535701−1.690789
H −3.413815−2.162272 1.005235
H −4.121683−4.417320 0.304068
H −3.055482−5.485903−1.678020
H −1.310312−4.278675−2.937852
H −0.568578−2.044136−2.280237
C −3.368034 0.822552 1.719909
C −3.992527 1.027429 2.947559
C −3.503655 0.384467 4.080437
C −2.400701−0.460854 3.979151
C −1.751820−0.680416 2.761258
H −3.751713 1.317726 0.834575
H −4.850949 1.680297 3.011210
H −3.982398 0.532015 5.043604
H −2.032568−0.969140 4.866560
H −0.898081−1.350993 2.732069
C −3.192076 0.390602−2.107469
C −3.828518 1.237700−3.010048
C −3.561969 2.602877−2.972397
C −2.668482 3.110289−2.031826
C −2.009908 2.282765−1.118342
H −3.410533−0.673052−2.131292
H −4.531096 0.830821−3.730010
H −4.053490 3.276480−3.667739
H −2.475137 4.179376−1.999272
H −1.319247 2.733823−0.411992
C 2.936435 1.139206 0.090020
C 3.202043−0.060758 0.076396
C 2.737770 2.564921 0.068145
C 3.655100−1.427293 0.053851
C 2.416350 3.252057 1.249209
C 2.185130 4.622415 1.204766
C 2.266074 5.307927−0.088431
C 2.585221 4.624620−1.182541
C 2.817790 3.253757−1.150854
H 2.353523 2.708723 2.187605
H 1.943919 5.157352 2.117751
H 2.086041 6.378094−0.037501
H 2.655179 5.160200−2.123961
H 3.064301 2.711200−2.058392
C 3.903714−2.055471−1.175822
C 4.363508−3.367211−1.196449
C 4.570848−4.055433−0.000276
C 4.318544−3.432047 1.222443
referenced to an external standard, 25% H₃PO₄ in H₂O in a sealed capillary. The ³¹P{¹H} NMR data with the usual 85% H₃PO₄ reference were shifted +0.544 ppm from these data.

The high-performance liquid chromatography (HPLC) apparatus and conditions are as follows: Shimadzu LC-20AD with Shimadzu SPD-20A detector (wavelength 260 nm), column VP-ODS (150 mm × 4.6 mm), flow rate 0.7 mL per min, and solvent MeOH: water (30 : 17).

2.1. Preparation of [AuCl(NHC)] complexes (NHC = IPr, IMes, BIPr, IF₃, lBu)

The [AuCl(NHC)] complexes (NHC = IPr, IMes, BIPr, IF₃ and lBu) were prepared by reaction of H[AuCl₄]·4H₂O with the NHC ligands (HIPr⁺Cl⁻, HIMes⁺Cl⁻) and Na₂CO₃, or by reaction of [AuCl(THT)] (THT = tetrahydrothiophene) with the NHC precursors (HBIPr⁺Cl⁻, HIF₃⁺Cl⁻, HlBu⁺Cl⁻) and K₂CO₃, according to the cited references.
Fig. 1. $^1$H NMR spectrum of $[\text{Au(RS-pyrrld)}](\text{IPr})$ (1) in CDCl$_3$ at 22.6 °C.

Fig. 2. IR spectrum of $[\text{Au(RS-pyrrld)}](\text{IPr})$ (1).
2.1.1. \([\text{AuCl}(\text{IPr})]\) [3]

To a solution of \(\text{H}[\text{AuCl}_4]\cdot 4\text{H}_2\text{O}\) (1.00 g, 2.43 mmol) in 9 mL of 3-chloropyridine, HIPr\(^+\)/CO\([3]\) (1.03 g, 2.43 mmol) and then Na\(_2\)CO\(_3\) (1.03 g, 12.2 mmol) were sequentially added. The mixture was stirred for 24 h in an oil bath at ca 80 °C, then cooled to room temperature, and 18 mL of CH\(_2\)Cl\(_2\) was added. The resulting brown suspension was filtered through a folded filter paper (Whatman #5). Dichloromethane was removed from the filtrate with a folded filter paper (Whatman #5). A yellow-white suspension was obtained. Filtration on a membrane filter (JV 0.1 \(\mu\)m) gave a yellow-white powder, which was washed with MeOH (10 mL x 2) and hexane (30 mL x 2), dried thoroughly by suction, and dried in vacuo for 2 h. Yield 0.366 g (24.3%).

Anal. Calcd for C\(_{27}\)H\(_{36}\)N\(_2\)ClAu or \([\text{AuCl}(\text{IPr})]\): C, 52.22; H, 5.84; N, 4.51. Found: C, 52.28; H, 6.24; N, 4.43%. TG/DTA under atmospheric conditions: a weight loss of 79.46% due to decomposition at below 500.0 °C was observed with an endothermic peak at 353.5 °C and an exothermic peak at 432.9 °C. IR (KBr, cm\(^{-1}\)) \([\text{AuCl}(\text{IPr})]\): 1683 (w), 1581 (w), 1550 (w), 1470 (vs), 1456 (vs), 1415 (s), 1384 (m), 1364 (m), 1327 (m), 1254 (w), 1212 (w), 1177 (w), 1116 (w), 1058 (w), 976 (vw), 937 (w), 808 (s), 764 (s), 742 (s), 705 (w), 450 (vw). \(^1\)H NMR (22.0 °C, CDCl\(_3\)) \([\text{AuCl}(\text{IPr})]\): \(\delta_H\) 1.22 (12H, d, \(J\) 7.2 Hz, H\(_6\)), 1.34 (12H, d, \(J\) 7.2 Hz, H\(_6\)), 2.55 (4H, sept, \(J\) 6.8 Hz, H\(_5\)), 7.12 (2H, s, H\(_2\)), 7.28 (4H, d, \(J\) 7.6 Hz, H\(_6\)), 1.34 (12H, d, \(J\) 7.2 Hz, H\(_6\)), 2.55 (4H, sept, \(J\) 6.8 Hz, H\(_5\)), 7.12 (2H, s, H\(_2\)), 7.28 (4H, d, \(J\) 7.6 Hz, H\(_6\)), 1.34 (12H, d, \(J\) 7.2 Hz, H\(_6\)), 2.55 (4H, sept, \(J\) 6.8 Hz, H\(_5\)), 7.12 (2H, s, H\(_2\)), 7.28 (4H, d, \(J\) 7.6 Hz, H\(_6\)), 1.34 (12H, d, \(J\) 7.2 Hz, H\(_6\)).

2.1.2. \([\text{AuCl}(\text{IMes})]\) [4]

The complex \([\text{AuCl}(\text{IMes})]\) was prepared by reaction of a solution of \(\text{H}[\text{AuCl}_4]\cdot 4\text{H}_2\text{O}\) (1.00 g, 2.43 mmol) in 9 mL of 3-chloropyridine with HIMes\(^+\)/Cl\(^-\) [3,4] (0.83 g, 2.43 mmol) and Na\(_2\)CO\(_3\) (6.45 g, 60.9 mmol). Workup as described above for \([\text{AuCl}(\text{IPr})]\) [3] afforded a pale yellow powder. Yield 0.371 g (28.5%).

Anal. Calcd for C\(_{21}\)H\(_{24}\)N\(_2\)ClAu or \([\text{AuCl}(\text{IMes})]\): C, 46.98; H, 5.41; N, 5.22. Found: C, 46.69; H, 6.18; N, 5.23%. TG/DTA under atmospheric conditions: a weight loss of 68.10% due to decomposition at below 500.0 °C was observed with an endothermic peak at 319.9 °C and exothermic peaks at 365.7 and 438.3 °C. IR (KBr, cm\(^{-1}\)): 1745 (vw), 1705 (vw), 1609 (w), 1556 (w), 1488 (vs), 1444 (m), 1414 (m), 1378 (m), 1346 (w), 1293 (w), 1234 (s), 1167 (vw), 1122 (vw), 1091 (vw), 1079 (vw), 1036 (w), 1014 (vw), 979 (w).
(vw), 963 (vw), 931 (w), 865 (s), 749 (m), 731 (w), 705 (m), 646 (w), 596 (w), 575 (m), 430 (w). 1H NMR (21.3 °C, CDCl3): δH 2.10 (12H, s, H5), 2.34 (6H, s, H8), 6.99 (4H, s, H6), 7.09 (2H, s, H2). 13C{1H} NMR (22.3 °C, CDCl3): δC 17.76 (C5), 21.15 (C8), 122.19 (C2), 129.49 (C6), 134.64 (C4 or C7), 134.69 (C4 or C7), 139.77 (C3), 173.27 (C1).

2.1.3. [AuCl(BIPr)] [5]

The complex [AuCl(BIPr)] was prepared by reaction of HBIPr+Cl− [6] (0.291 g, 0.613 mmol) in 60 mL of acetone with [AuCl(THT)] [7,8] (0.271 g, 0.920 mmol) and K2CO3 (0.424 g, 3.07 mmol) in an oil bath at 20 °C for 2 h with stirring. The mixture was filtered through a membrane filter (JV 0.1 μm), and the filtrate was evaporated to dryness. The resulting pale purple solid was dissolved in 20 mL of CH2Cl2, and the solution was filtered through a folded filter paper (Whatman #5). The pale purple clear filtrate was added to 600 mL of hexane. Filtration on a membrane filter (JV 0.1 μm) gave a pale purple solid, which was washed with hexane (20 mL x 2), dried thoroughly by suction, and dried in vacuo for 2 h to afford a pale purple powder. Yield 0.1290 g (70.5%).

Anal. Calcld for C31H38Cl2Au: 55.48; H, 5.71; N, 4.17. Found: C, 55.59; H, 5.68; N, 4.06%. IR (KBr, cm−1): 1468 (s), 1455 (s), 1392 (vs), 1360 (vs), 1254 (w), 1225 (w), 1184 (w), 1160 (w), 1061 (w), 1009 (w), 938 (w), 903 (vw), 798 (s), 754 (vs), 638 (vw), 594 (w), 430 (w), 419 (w). 1H NMR (21.8 °C, CDCl3): δH 1.09 (d, J = 6.9 Hz, H10), 1.33 (d, J = 7.0 Hz, H10), 2.40 (sept, J = 6.9 Hz, H9), 7.09 (dd, J = 3.1, 6.1 Hz, H3 or H4), 7.37–7.42 (m, H3 or H4 and H7). 13C{1H} NMR (21.6 °C, CDCl3): δC 23.92 (s, C10), 24.66 (s, C10), 28.94 (s, C9), 112.02 (s, C3), 124.66 (s, C2), 125.41 (s, C8), 131.07 (s, C4 or C7), 131.21 (s, C4 or C7), 134.52 (s, C6), 146.45 (s, C5), 181.68 (s, C1).

2.1.4. [AuCl(II3)] [5]

The complex [AuCl(II3)] was prepared by reaction of a solution of HBIPr+Cl− [5,9] (0.10 g, 0.274 mmol) in 15 mL of acetone with [AuCl(THT)] [7,8] (0.132 g, 0.411 mmol) and K2CO3 (0.189 g, 1.37 mmol). Workup as described above for [AuCl(BIPr)] [5] afforded a white powder. Yield 0.053 g (34.7%).

Anal. Calcld for C15H38Cl2F6Au or [AuCl(II3)]: C, 32.14; H, 1.08; N, 5.00. Found: C, 31.28; H, 0.43; N, 4.62%. IR (KBr, cm−1): 1616 (vs), 1557 (w), 1523 (vs), 1458 (s), 1397 (w), 1364 (m), 1286 (w), 1251 (w), 1178 (m), 1129 (vs), 1104 (m), 1046 (vs), 999 (s), 978 (w), 847 (s), 768 (vw), 756 (w), 724 (vw), 695 (w), 657 (m), 621 (w), 509 (w), 446 (vw). 1H NMR (22.1 °C, CDCl3): δH 6.91–6.96 (m, H5), 7.29 (s, H2). 13C{1H} NMR (21.5 °C, CDCl3): δC 101.88 (ddd, J = 3.9, 25.0, 50.1 Hz, C6), 112.84 (ddd, J = 5.4, 15.8, 31.5 Hz, C3), 123.39 (s, C2), 156.91 (dd, J = 5.3, 30.5 Hz, C4), 159.47 (dd, J = 5.0, 30.5 Hz, C4), 162.02 (dd, J = 14.4, 28.5 Hz, C5), 164.57 (dd, J = 14.4, 28.8 Hz, C5), 178.61 (s, C1).

2.1.5. [AuCl(fBu)] [5,10]

The complex [AuCl(fBu)] was prepared by reaction of a solution of HF3+Cl− [10,11] (0.400 g, 1.845 mmol) in 60 mL of acetone with [AuCl(THT)] [7,8] (0.887 g, 2.767 mmol) and K2CO3 (1.275 g, 9.225 mmol). Workup as described above for [AuCl(BIPr)] [5] afforded a white powder. Yield 0.381 g (50.0%).

Anal. Calcld for C41H40N2Cl4Au or [AuCl(fBu)]: C, 32.01; H, 4.88; N, 6.79. Found: C, 32.32; H, 4.60; N, 6.64%. IR (KBr, cm−1): 1647 (w), 1559 (w), 1542 (m), 1518 (w), 1507 (w), 1473 (w), 1458 (w), 1438 (w), 1406 (m), 1378 (s), 1305 (w), 1236 (w), 1209 (vs), 1183 (s), 1156 (w), 1053 (vw), 1039 (vw), 1022 (vw), 981 (vw), 962 (vw), 931 (vw), 823 (vw), 720 (m), 693 (s), 626 (w), 418 (vw). 1H NMR (21.5 °C, CDCl3): δH 1.88 (18H, s, H4), 7.11 (2H, s, H2). 13C{1H} NMR (21.4 °C, CDCl3): δC 31.76 (s, C4), 58.97 (s, C3), 116.43 (s, C2), 168.03 (s, C1).

2.2. Preparation of [Au(RS-pyrrld)(NHC)] complexes (NHC = IMes (6), BIPr (7), IF3 (8), fBu (9))

The [Au(RS-pyrrld)(NHC)] complexes (NHC = IMes (6), BIPr (7), IF3 (8), fBu (9)) were prepared by reaction of [AuCl(NHC)] with Ag(RS-pyrrld)2 [12]. The 1H NMR and IR spectra of [Au(RS-pyrrld)(IPr)] (1) [1] are shown in Figs. 1 and 2.

2.2.1. [Au(RS-pyrrld)(IMes)] (6)

Compound (6) was prepared by reaction of [AuCl(IMes)] (0.403 g, 0.750 mmol) with Ag(RS-pyrrld)2 (0.533 g, 1.13 mmol). Workup as described above for [Au(RS-pyrrld)(IPr)] (1) afforded a pale yellow powder. Yield 0.119 g (50.4%).
2.2.2. [Au(RS-pyrrld)\{BPr\}] (7)

Compound (7) was prepared by reaction of [AuCl(BPr)] (0.227 g, 0.338 mmol) with \(\text{Ag}([\text{RS-pyrrld}]_{2}) (0.319 g, 0.676 mmol). Workup as described above for [Au(RS-pyrrld)\{IPr\}] (1) afforded a pale yellow powder. Yield 0.161 g (60.5%).

Anal. Calcd for C\(_{36}\)H\(_{44\,\text{b}}\)N\(_{3}\)O\(_{3}\)Au or [Au \((\text{RS-pyrrld})\{\text{Mes}\}\)]: C, 49.61; H, 4.80; N, 6.68. Found: C, 48.62; H, 6.31; N, 6.53%. TG/DTA under atmospheric conditions: a weight loss of 71.95% due to decomposition at below 500.0 °C was observed with exothermic peaks at 166.5 and 501.0 °C. IR (KBr, cm\(^{-1}\)): 1686 (vs), 1652 (s), 1488 (s), 1437 (w), 1415 (m), 1378 (m), 1274 (m), 1239 (m), 1035 (vw), 1014 (vw), 930 (vw), 749 (w), 704 (w), 577 (w), 422 (w). \(^{1}\)H NMR (20.9 °C, CDCl\(_{3}\)): \(\delta_{H}2.13\) (s, H5), 2.20–2.29 (m, CH\(_{2}\) pyrrld), 2.35 (s, H8), 3.97–4.01 (m, CH pyrrld), 5.79 (s, NH pyrrld), 7.02 (s, H6), 7.13 (s, H2). \(^{13}\)C\(^{1}\)H NMR (22.4 °C, CDCl\(_{3}\)): \(\delta_{C}17.82\) (s, C5), 21.19 (s, C8), 25.21 (s, CH\(_{2}\)CH pyrrld), 30.21 (s, CH\(_{2}\)CO pyrrld), 57.84 (s, CH pyrrld), 122.50 (s, C2), 129.54 (s, C6), 134.57 (s, C4 or C7), 134.70 (s, C4 or C7), 139.82 (s, C3), 165.38 (s, C1), 175.99 (s, COO pyrrld), 177.68 (s, CO).

2.2.3. [Au(RS-pyrrld)\{IPr\}\{\text{BFu}\}] (8)

Compound (8) was prepared by reaction of [AuCl(Ir)] (0.210 g, 0.374 mmol) with \(\text{Ag}([\text{RS-pyrrld}]_{2}) (0.299 g, 0.724 mmol). Workup as described above for [Au(RS-pyrrld)\{IPr\}] (1) afforded a white powder. Yield 0.117 g (46.2%).

Anal. Calcd for C\(_{29}\)H\(_{30}\)N\(_{3}\)O\(_{3}\)Au or [Au \((\text{RS-pyrrld})\{\text{BFu}\}\)]: C, 36.29; H, 1.83; N, 6.32. Found: C, 36.06; H, 1.53; N, 6.07%. TG/DTA under atmospheric conditions: a weight loss of 10.44% due to desorption of 0.1 CHCl\(_{3}\) at below 188.1 °C was observed; calcld 1.79% for 0.1 solvated CHCl\(_{3}\) molecules. Further, a weight loss of 66.79% due to decomposition was observed at below 500.0 °C with exothermic peaks at 201.4, 228.1, and 502.3 °C. IR (KBr, cm\(^{-1}\)): 1694 (vs), 1648 (vs), 1619 (vs), 1525 (vs), 1459 (s), 1394 (m), 1365 (s), 1261 (m), 1179 (m), 1130 (m), 1104 (m), 1049 (vs), 1000 (s), 845 (m), 740 (w), 697 (w), 668 (w), 657 (w), 621 (w), 510 (w), 448 (wv). \(^{1}\)H NMR (22.1 °C, CDCl\(_{3}\)): \(\delta_{H}2.39\) (s, CIO), 24.58 (s, C1O), 25.30 (s, CH\(_{2}\)CH pyrrld), 29.00 (s, C9), 30.29 (s, CH\(_{2}\)CO pyrrld), 57.78 (s, CH pyrrld), 112.02 (s, C3), 124.70 (s, C2), 125.47 (s, C8), 131.10 (s, C4 or C7), 131.18 (s, C4 or C7), 134.58 (s, C6), 146.48 (s, C5), 174.47 (s, C1), 175.53 (s, COO pyrrld), 177.54 (s, CO).

2.2.4. [Au(RS-pyrrld)\{\text{BFu}\}] (9)

Compound (9) was prepared by reaction of [AuCl(Ir)] (0.299 g, 0.724 mmol) with \(\text{Ag}([\text{RS-pyrrld}]_{2}) (0.683 g, 1.448 mmol). Workup as described above for [Au(RS-pyrrld)\{IPr\}] (1) afforded a white powder. Yield 0.262 g (71.5%).

Anal. Calcd for C\(_{56}\)H\(_{58}\)N\(_{3}\)O\(_{3}\)Au or [Au \((\text{RS-pyrrld})\{\text{BFu}\}\)]: C, 38.03; H, 5.19; N, 8.31. Found: C, 38.03; H, 5.10; N, 8.00%. TG/DTA under atmospheric conditions: a weight loss of 61.46% due to decomposition at below 500.0 °C was observed with an exothermic peak at 250.6 °C. IR (KBr, cm\(^{-1}\)): 1697 (vs), 1653 (s), 1604 (s), 1473 (m), 1457 (m), 1406 (s), 1378 (vs), 1305 (m), 1262 (m), 1234 (s), 1213 (vs), 1147 (w), 1038 (vw), 979 (vw), 930 (vw), 825 (vw), 731 (w), 697 (m), 631 (w), 568 (vw), 418 (vw). \(^{1}\)H NMR (21.8 °C, CDCl\(_{3}\)): \(\delta_{H}1.90\) (s, H4), 2.33–2.50 (m, CH\(_{2}\) pyrrld), 4.24–4.28 (m, CH pyrrld), 5.90 (s, NH pyrrld), 7.11 (s, H2). \(^{13}\)C\(^{1}\)H NMR (22.0 °C,
CDC(3)J: δ C 25.58 (s, CH2CH pyrrld), 30.29 (s, CH2CO pyrrld), 31.70 (s, C), 57.83 (s, CH pyrrld), 59.12 (s, C3), 116.67 (s, C2), 159.66 (s, C1), 176.68 (s, COO pyrrld), 177.77 (s, CO pyrrld).

2.3. X-ray crystallography of [Au(1Pr)(H2O)]2[a-PW12O40]·7Et2O (2)

Crystallization of (2), whose 1H NMR spectrum is shown in Fig. 3 [1], was carried out by liquid-liquid diffusion of an internal aqueous solution of the metal complex with an external solvent (ether) in a refrigerator. Single crystals of the metal complex were mounted on a loop and used for measurements of cell constants and for the collection of intensity data on a Rigaku VariMax with Saturn CCD diffractometer. The structure was solved by a direct method, followed by difference Fourier calculation; it was refined by a full-matrix least-squares method on F2 using the Yadokari program package [13]. All non-hydrogen atoms were refined anisotropically. The hydrogen atoms were placed geometrically or identified on a difference Fourier-map and were treated using a riding model. The crystal data of (2) are summarized in Table 1, and selected bond distances (Å) and angles (deg) are shown in Table 2. The details of the crystal data have been deposited with the Cambridge Crystallographic Data Centre as a supplementary publication (CCDC no. 1864226).

Acknowledgments

This work was supported by funds from the Strategic Research Base Development Program for Private Universities (S1201017) of the Ministry of Education, Culture, Sports, Science and Technology of Japan. Computations were performed in part at the Research Center for Computational Science, Okazaki, Japan.

Transparency document

Transparency document associated with this article can be found in the online version at https://doi.org/10.1016/j.dib.2019.104002.

References

[1] K. Nomiya, Y. Murata, Y. Iwasaki, H. Arai, T. Yoshida, N.C. Kasuga, T. Matsubara, Highly active, homogeneous catalysis by polyoxometalate-assisted N-heterocyclic carbene gold(I) complexes for hydration of diphenylacetylene, Mol. Catal. 469 (2019) 144–154.
[2] F. Sirindil, S.P. Nolan, S. Dagorne, P. Pale, A. Blanc, P. de Frémont, Synthesis, characterization and catalytic activity of NHC gold(I) polyoxometalate complexes, Chem. Eur J. 24 (2018) 12630–12637.
[3] J. Huang, S.P. Nolan, Efficient cross-coupling of aryl chlorides with aryl Grignard reagents (Kumada reaction) mediated by a palladium/imidazolium chloride system, J. Am. Chem. Soc. 21 (1999) 9889–9890.
[4] S. Zhu, R. Liang, H.A. Jiang, A direct and practical approach for the synthesis of N-heterocyclic carbene coinage metal complexes, Tetrahedron 68 (2012) 7949–7955.
[5] A. Collado, A. Gómez-Suárez, A.R. Martin, A.M.Z. Slawin, S.P. Nolan, Straightforward synthesis of [Au(NHC)X] (NHC = N-heterocyclic carbene, X = Cl, Br, I) complexes, Chem. Commun. 49 (2013) 5541–5543.
[6] G. Grieco, O. Blacque, H.A. Berke, A facile synthetic route to benzimidazolium salts bearing bulky aromatic N-substituents, Beilstein J. Org. Chem. 11 (2015) 1656–1666.
[7] G.A. Price, A.K. Bridson, K.R. Flower, R.G. Pritchard, P. Quayle, Solvent effect in gold-catalysed A1-coupling reactions, Tetrahedron Lett. 55 (2014) 151–154 (Supplementary data).
[8] N.A. Barnes, A.K. Bridson, F.R.W. Brown, W.I. Cross, I.R. Crossley, C. Fish, C.J. Herbert, R.G. Pritchard, J.E. Warren, “Synthesis of gold(I) fluoroalkyl and fluoroalkenyl-substituted phosphine complexes and factors affecting their crystal packing”, Dalton Trans. 40 (2011) 1743–1750.
[9] D.A.J. Harding, E.G. Hope, K. Singh, G.A. Solan, Bis-cyclometalation of fluorinated N-aryl NHCS, Organometallics 31 (2012) 1518–1523.
[10] C.J. Serpell, J. Cookson, A.L. Thompson, C.M. Brown, P.D. Beer, Haloururate and halopalladate imidazolium salts: structures, properties , and uses as precursors for catalytic metal nanoparticles, Dalton Trans. 42 (2013) 1385–1393.
[11] F. Medina, C. Michon, F. Agbossou-Niedercorn, Intermolecular mono- and dihydroamination of activated alkene using recoverable gold catalyst, Eur. J. Org. Chem. (2012) 6218–6227.
[12] K. Nomiya, S. Takahashi, R. Noguchi, S. Nemoto, T. Takayama, M. Oda, “Synthesis and characterization of water-soluble silver(I) complexes with L-histidine (H2his) and (S)-(-)-2-pyridolone-5-carboxylic acid (H2pyrrld) showing a wide spectrum of effective antibacterial and antifungal activities. Crystal structures of chiral helical polymers [Ag(Hhis)]n and ![Ag3(Hpyrrld)2]n in the solid-state”, Inorg. Chem. 39 (2000) 3301–3311.
[13] K. Wakita, Yadokari-XG, Software for crystal structure analyses (2001); Release of Software (Yadokari-XG 2009) for Crystal Structure Analyses, C. Kabuto, S. Akine, T. Nemoto, and E. Kwon, J. Crystallogr. Soc. Jpn. 51 (2009) 218–224.