Supporting Information

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Synthesis and Reactivity of Metal Complexes with Acyclic (Amino)-(Ylide)Carbene Ligands**

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<table>
<thead>
<tr>
<th>Table of Contents</th>
</tr>
</thead>
<tbody>
<tr>
<td>Experimental Procedures</td>
</tr>
<tr>
<td>Characterization of new compounds</td>
</tr>
<tr>
<td>NMR spectra</td>
</tr>
<tr>
<td>X-ray structure analyses</td>
</tr>
</tbody>
</table>
Experimental procedures:

General: All reactions were carried out in flame-dried glassware under Ar. All solvents were purified by distillation over the appropriate drying agents and were transferred under Ar. IR: Nicolet FT-7199 spectrometer, wavenumbers in cm$^{-1}$. MS (EI): Finnigan MAT 8200 (70 eV), ESIMS: Finnigan MAT 95, accurate mass determinations: Bruker APEX III FT-MS (7 T magnet). NMR Spectra were recorded on a Bruker AV 500, AV 400 or DPX 300; $^1$H and $^{13}$C chemical shifts (δ) are given in ppm relative to TMS, coupling constants (J) in Hz. The solvent signals were used as references and the chemical shifts converted to the TMS scale.

All commercially available compounds (Acros, Aldrich) were used as received. The ylides 2d$^5$, 2f$^5$, 2e$^5$, 7 and 9$^5$ were prepared according to literature procedures. Gold (I) isonitriles 1a and 1b were prepared using the method described by Hashmi et al. in quantitative yields$^6$. Phenylisocyanide was prepared by the method of Weber et al. from aniline$^7$.

General procedure for AAYC-gold complexes bearing phosphorus ylides:

In a typical procedure, Gold (I) isonitrile 1 is suspended in toluene (0.024M) followed by addition of ylide 2 at the indicated temperature. After stirring the reaction for the referred time, the mixture was allowed to reach room temperature and the solvents filtered out. The remaining white solid thus obtained was then washed with small portions of pentane and dried under vacuum.

Compound 4a: Following the general procedure described above, a mixture of phenylisocyanide gold (I) chloride 1a (40 mg, 0.12 mmol) and 1-(Triphenylphosphoranylidene)-2-propanone 2a (38 mg, 0.12 mmol) afforded pure 4a (66 mg, 85%) after a reaction time of 3 d at room temperature.

$^{1}$H-NMR (400 MHz, CD$_2$Cl$_2$) $\delta$ = 14.54 (s, 1 H), 7.97-7.92 (m, 6 H), 7.72-7.69 (m, 3 H), 7.65-7.59 (m, 8 H), 7.34-7.31 (m, 2 H), 7.24-7.21 (m, 1 H), 1.47 (s, 3 H) ppm. $^{13}$C-NMR (101 MHz, CD$_2$Cl$_2$) $\delta = 200.9$ (d, $J_{C,P} = 36.0$ Hz), 195.3 (d, $J_{C,P} = 24.0$ Hz), 144.1, 134.5 (d, $J_{C,P} = 12.8$ Hz), 133.5 (d, $J_{C,P} = 8.6$ Hz), 129.8 (d, $J_{C,P} = 91.6$ Hz), 123.7, 93.0 (d, $J_{C,P} = 124.9$ Hz), 31.7 (d, $J_{C,P} = 2.3$ Hz) ppm. $^{31}$P-NMR (162 MHz, CD$_2$Cl$_2$) $\delta = 19.6$ ppm. HRMS calcld. for C$_{28}$H$_{34}$NOAuPNa: 767.084174; found 767.084426. IR (neat) $\tilde{\nu} = 680, 690, 706, 721, 736, 749, 756, 875, 901, 983, 998, 1024, 1052, 1095, 1133, 1182, 1227, 1253, 1365, 1415, 1438, 1482, 1506, 1506, 1567, 1587, 3052 cm$^{-1}$.

Compound 4b: Following the general procedure described above, a mixture of phenylisocyanide gold (I) chloride 1a (41 mg, 0.12 mmol) and the phosphorus ylide 2b (45 mg, 0.12 mmol) afforded pure 4b (62 mg, 74%) after a reaction time of 1 d at 35 °C.

$^{1}$H-NMR (400 MHz, CD$_2$Cl$_2$) $\delta = 12.67$ (s, 1 H), 7.89-7.84 (m, 6 H), 7.63-7.67 (m, 5 H), 7.58-7.53 (m, 6 H), 7.34-7.30 (m, 2 H), 7.23-7.19 (m, 1 H), 3.73 (q, $J = 7.2$ Hz, 2 H), 0.59 (t, $J = 7.2$ Hz, 3 H) ppm. $^{13}$C-NMR (101 MHz, CD$_2$Cl$_2$) $\delta = 200.6$ (d, $J_{C,P} = 35.9$ Hz), 168.8 (d, $J_{C,P} = 17.2$ Hz), 144.4, 134.1 (d, $J_{C,P} = 9.0$ Hz), 133.0 (d, $J_{C,P} = 2.6$ Hz), 129.4 (d, $J_{C,P} = 12.7$ Hz), 129.1, 126.3, 126.2 (d, $J_{C,P} = 94.0$ Hz), 123.4, 79.4 (d, $J_{C,P} = 134.4$ Hz), 60.0, 13.6 ppm. $^{31}$P-NMR (162 MHz, CD$_2$Cl$_2$) $\delta = 22.0$ ppm. HRMS calcld. for C$_{29}$H$_{36}$NOAuClPNa: 706.094742; found 706.095558. IR (neat) $\tilde{\nu} = 688, 681, 698, 710, 748, 760, 799, 819, 849, 905, 937, 997, 1024, 1071, 1081, 1103, 1156, 1164, 1185, 1197, 1233, 1292, 1336, 1368, 1392, 1436, 1479, 1517, 1588, 1629, 2907, 2976, 3054 cm$^{-1}$.

Compound 4c: Following the general procedure described above, a mixture of phenylisocyanide gold (I) chloride 1a (23 mg, 0.07 mmol) and the phosphorous ylide 2c (21 mg, 0.07 mmol) afforded pure 4c (38 mg, 88%) after a reaction time of 3 d at 35 °C.

$^{1}$H-NMR (400 MHz, CD$_2$Cl$_2$) $\delta = 8.89$ (s, 1 H), 7.83-7.75 (m, 9 H), 7.71-7.69 (m, 2 H), 7.69-7.61 (m, 6 H), 7.38-7.34 (m, 2 H), 7.28-7.24 (m, 1 H) ppm. $^{13}$C-NMR (101 MHz, CD$_2$Cl$_2$) $\delta = 201.9$ (d, $J_{C,P} = 35.9$ Hz), 143.0, 134.8 (d, $J_{C,P} = 9.8$ Hz), 134.4 (d, $J_{C,P} = 2.9$ Hz), 129.8 (d, $J_{C,P} = 13.0$ Hz), 129.3, 126.8, 123.1, 122.9 (d, $J_{C,P} = 94.1$ Hz), 117.8 (d, $J_{C,P} = 22.1$ Hz), 60.1 (d, $J_{C,P} = 154.6$ Hz) ppm. $^{31}$P-NMR (162 MHz, CD$_2$Cl$_2$) $\delta = 21.8$ ppm. HRMS calcld. for C$_{28}$H$_{37}$N$_2$AuClPNa: 659.068858; found 659.069049. IR (neat) $\tilde{\nu} = 687, 715, 727, 748, 758, 788, 850, 900, 923, 996, 1026, 1073, 1102, 1120, 1190, 1225, 1284, 1300, 1319, 1343, 1436, 1491, 1529, 1594, 2175, 3242 cm$^{-1}$.

Compound 4d: Following the general procedure described above, a mixture of phenylisocyanide gold (I) chloride 1a (23 mg, 0.07 mmol) and phosphorus ylide 2d (24 mg, 0.07 mmol) afforded pure 4d (38 mg, 81%) after a reaction time of 6 h at 35 °C.

$^{1}$H-NMR (400 MHz, CD$_2$Cl$_2$) $\delta = 7.98$ (s, 1 H), 7.85-7.80 (m, 6 H), 7.69-7.65 (m, 3 H), 7.61-7.53 (m, 8 H), 7.29-7.25 (m, 2 H), 7.14-7.06 (m, 1 H), 2.88 (s, 6 H) ppm. $^{13}$C-NMR (101 MHz,
CD₂Cl₂ δ = 188.9 (d, J_C,P = 32.0 Hz), 167.8 (d, J_C,P = 19.1 Hz), 144.4, 134.6 (d, J_C,P = 9.3 Hz), 133.4 (d, J_C,P = 2.9 Hz), 129.3 (d, J_C,P = 12.4 Hz), 129.0, 125.1 (d, J_C,P = 92.0 Hz), 125.0, 122.4, 86.1 (d, J_C,P = 132.3 Hz), 37.0 ppm.

³¹P-NMR (162 MHz, CD₂Cl₂) δ = 18.1 ppm. HRMS calcld. for C₂₉H₂₅N₅O₂AuClPNa: 705.110727; found 705.110773.

IR (neat) ν = 691, 729, 144, 756, 841, 900, 937, 998, 1027, 1048, 1070, 1098, 1158, 1188, 1215, 1271, 1304, 1384, 1435, 1446, 1481, 1496, 1542, 1597, 3042, 3275 cm⁻¹.

**Compound 4e:** Following the general procedure described above, a mixture of phenylisocyanide gold (I) chloride 1a (89 mg, 0.26 mmol) and phosphorus ylide 2e (94 mg, 0.26 mmol) afforded pure 4e (180 mg, 98%) after a reaction time of 6 h at 35 °C.

1H-NMR (400 MHz, CD₂Cl₂) δ = 9.77 (s, 1 H), 8.45 (d, J = 4.6 Hz, 1H), 7.82 (dd, J = 12.1, 8.1 Hz, 6 H), 7.62-7.58 (m, 5 H), 7.52-7.47 (m, 6 H), 7.30-7.22 (m, 3 H), 7.06 (t, J = 7.1 Hz, 1H), 6.93-6.90 (m, 1 H), 6.81 (d, J = 7.9 Hz, 1 H) ppm. ¹³C-NMR (101 MHz, CD₂Cl₂) δ = 189.6 (d, J_C,P = 35.7 Hz), 156.4 (d, J_C,P = 20.1 Hz), 149.6, 144.5, 136.3, 134.7 (d, J_C,P = 9.1 Hz), 130.0 (d, J_C,P = 2.6 Hz), 129.2 (d, J_C,P = 12.1 Hz), 128.8, 126.8 (d, J_C,P = 3.0 Hz), 126.2 (d, J_C,P = 91.8 Hz), 124.4, 121.7, 120.8, 87.9 (d, J_C,P = 134.4 Hz) ppm. ³¹P-NMR (162 MHz, CD₂Cl₂) δ = 20.0 ppm. HRMS calcld. for C₃₀H₂₆N₅O₂AuClPNa: 711.100164; found 711.100454.

IR (neat) ν = 688, 711, 742, 793, 862, 900, 997, 1017, 1051, 1098, 1154, 1183, 1263, 1312, 1379, 1425, 1435, 1460, 1494, 1514, 1557, 1582, 3056 cm⁻¹.

**Compound 4f:** Phenylisocyanide gold (I) chloride 1a (44 mg, 0.12 mmol) is added to a cooled solution of the phosphorus ylide 2f (43 mg, 0.12 mmol) at -78 °C. After 2 h, it was allowed to warm up to room temperature overnight. Filtration of the obtained suspension afforded a white solid, which contains both 4f and the side product 3f. Consecutive crystallizations (3 times) in DCM/pentane allowed the isolation of pure 4f (4 mg) in 5 % yield. ¹H-NMR (400 MHz, CD₂Cl₂) δ = 7.71-7.66 (m, 5 H), 7.64-7.59 (m, 3 H), 7.52-7.45 (m, 9 H), 7.30 (s, 1 H), 7.22-7.13 (m,5 H), 7.06-6.70 (m, 3 H) ppm. ¹³C-NMR (101 MHz, CD₂Cl₂) δ = 187.1 (d, J_C,P = 37.7 Hz), 144.5, 134.8 (d, J_C,P = 9.0 Hz), 134.0 (d, J_C,P = 3.8 Hz), 133.1 (d, J_C,P = 2.8 Hz), 133.1 (d, J_C,P = 2.8 Hz), 132.3 (d, J_C,P = 9.9 Hz), 129.6 (d, J_C,P = 1.7 Hz), 129.1 (d, J_C,P = 12.3 Hz), 129.0 (d, J_C,P = 12.1 Hz), 128.9, 127.9 (d, J_C,P = 2.3 Hz), 125.8 (d, J_C,P = 91.1 Hz), 124.0, 121.2, 88.9 (d, J_C,P = 132.1 Hz) ppm. ³¹P-NMR (162 MHz, CD₂Cl₂) δ = 21.0 ppm. HRMS calcld. for C₃₁H₃₁N₅O₂AuClPNa: 710.104912; found 710.105881. IR (neat) ν = 689, 704, 716, 744, 784, 800, 853, 887, 913, 996, 1009, 1027, 1071, 1098, 1159, 1220, 1261, 1305, 1326, 1372, 1433, 1444, 1480, 1493, 1508, 1590, 2851, 2922, 2961, 3051, 3331, 3494, 3551 cm⁻¹.

**Compound 4g:** Following the general procedure described above, a mixture of 2,6-dimethyl-phenylisocyanide gold (I) chloride 1b (100 mg, 0.28 mmol) and phosphorus ylide 2a (88 mg, 0.28 mmol) afforded pure 4g (54 mg, 30%) after a reaction time of 3 d at 50 °C.

1H-NMR (400 MHz, CD₂Cl₂) δ = 13.70 (s, 1 H), 7.97-7.92 (m, 6 H), 7.73-7.68 (m, 3 H), 7.64-7.58 (m, 6 H), 7.13-7.06 (m, 3 H), 2.26 (s, 6 H), 1.48 (s, 3 H) ppm. ¹³C-NMR (101 MHz, CD₂Cl₂) δ = 206.6 (d, J_C,P = 34.8 Hz), 195.0 (d, J_C,P = 28.0 Hz), 142.6, 135.0, 134.6 (d, J_C,P = 8.6 Hz), 133.6 (d, J_C,P = 2.9 Hz), 129.7 (d, J_C,P = 12.4 Hz), 128.4, 127.6, 125.9 (d, J_C,P = 92.0 Hz), 91.1
Compound 4h: Following the general procedure described above, a mixture of 2,6-
dimethyl-phenylisocyanide gold(I) chloride 1b (46 mg, 0.13 mmol) and phosphorus ylide
2c (47 mg, 0.13 mmol) afforded after 4 d at 50 °C a white solid that was further purified by
crystallization from DCM: pentane. Thus, 4h was obtained in 37% yield (33 mg).

$^1$H-NMR (400 MHz, CD$_2$Cl$_2$) $\delta$ = 11.89 (s, 1 H), 7.89-7.83 (m, 6 H), 7.67-7.63 (m, 3 H),
7.57-7.53 (m, 6 H), 7.14-7.07 (m, 3 H), 3.75 (q, $J$ = 7.1 Hz, 2 H), 2.20 (s, 6H), 0.59 (t, $J$
= 7.1 Hz, 3 H) ppm. $^{13}$C-NMR (101 MHz, CD$_2$Cl$_2$) $\delta$ = 206.1 (d, $J_{C,P}$ = 34.8 Hz), 168.8 (d, $J_{C,P}$ = 16.9 Hz), 142.9,
135.5, 134.2 (d, $J_{C,P}$ = 9.2 Hz), 132.7 (d, $J_{C,P}$ = 3.0 Hz), 129.3 (d, $J_{C,P}$ = 12.3 Hz), 128.4, 127.5, 126.2 (d, $J_{C,P}$ =
93.6 Hz), 76.9 (d, $J_{C,P}$ = 135.0 Hz), 59.6, 19.0, 13.6 ppm. $^{31}$P-NMR (162 MHz, CD$_2$Cl$_2$) $\delta$ = 22.4 ppm. HRMS calcld.
for C$_9$H$_9$NOAuClPNa: 734.126042; found 734.125744. IR (neat) $\tilde{\nu}$ = 683, 698, 709, 722, 748, 776, 802, 938, 997,
1025, 1078, 1103, 1162, 1182, 1216, 1258, 1300, 1339, 1368, 1390, 1436, 1480, 1521, 1635, 2982, 3063 cm$^{-1}$.

Compound 4i: Following the general procedure described above, a mixture of 2,6-
dimethyl-phenylisocyanide gold(I) chloride 1b (86 mg, 0.24 mmol) and phosphorus ylide
2c (71 mg, 0.24 mmol) afforded after 4 d at 50 °C a white solid that was further purified by
3 consecutive crystallizations from DCM: pentane. Thus, 4i was obtained in 25% yield (40 mg).

$^1$H-NMR (400 MHz, CD$_2$Cl$_2$) $\delta$ = 8.29 (s, 1 H), 7.82-7.75 (m, 9 H), 7.65-7.61 (m, 6 H), 7.20-
7.17 (m, 1 H), 7.12-7.11 (m, 2 H), 2.36 (s, 6 H) ppm. $^{13}$C-NMR (101 MHz, CD$_2$Cl$_2$) $\delta$ = 207.0 (d, $J_{C,P}$ = 29.5 Hz),
141.2, 136.1, 134.7 (d, $J_{C,P}$ = 9.9 Hz), 134.3 (d, $J_{C,P}$ = 2.7 Hz), 129.6 (d, $J_{C,P}$ = 13.1 Hz), 128.7, 128.4, 123.1 (d, 
$J_{C,P}$ = 95.0 Hz), 117.8 (d, $J_{C,P}$ = 22.2 Hz), 57.3 (d, $J_{C,P}$ = 154.6 Hz), 19.0 ppm. $^{31}$P-NMR (162 MHz, CD$_2$Cl$_2$) $\delta$
= 22.1 ppm. HRMS calcld. for C$_{29}$H$_{28}$N$_2$AuClPNa: 687.100159; found 687.100556. IR (neat) $\tilde{\nu}$ = 687, 697, 716, 749,
780, 804, 907, 927, 952, 997, 1025, 1105, 1123, 1165, 1186, 1217, 1260, 1312, 1328, 1375, 1436, 1482, 1505,
2180, 2962, 3282 cm$^{-1}$.

Compound 4j: 2,6-dimethyl-phenylisocyanide gold(I) chloride 1b (94 mg, 0.26 mmol) was
added to a cooled solution of the phosphorus ylide 2d (90 mg, 0.26 mmol) in toluene (11
ml) at -78 °C. After 2 h at this temperature, the reaction mixtures was allowed to reach room temperature overnight. Filtration of the obtained suspension afforded a white solid which was purified by recrystallization (3 times from DCM: pentane). Thus, 4j was
obtained as colourless crystals (21 mg, 12%). $^1$H-NMR (400 MHz, CD$_2$Cl$_2$) $\delta$ = 7.86-7.81
(m, 6 H), 7.70-7.65 (m, 3 H), 7.58-7.54 (m, 6 H), 7.24 (s, 1 H), 7.14-7.07 (m, 3 H), 2.95 (s, 6 H), 2.33 (s, 6 H) ppm.

$^{13}$C-NMR (101 MHz, CD$_2$Cl$_2$) $\delta$ = 194.3 (d, $J_{C,P}$ = 31.0 Hz), 168.0 (d, $J_{C,P}$ = 19.2 Hz), 142.4, 136.6, 134.5 (d, $J_{C,P}$ =
9.1 Hz), 133.3 (d, $J_{C,P}$ = 2.6 Hz), 129.2 (d, $J_{C,P}$ = 12.3 Hz), 128.5, 127.6, 125.5 (d, $J_{C,P}$ = 92.7 Hz), 82.1 (d, $J_{C,P}$ =
134.1 Hz), 37.0, 19.3 ppm. $^{31}$P-NMR (162 MHz, CD$_2$Cl$_2$) $\delta$ = 18.8 ppm. HRMS calcld. for C$_{31}$H$_{31}$N$_2$OAuClPNa:
Compound 3e: Phenylisocyanide gold (I) chloride 1a (230 mg, 0.63 mmol) was added to a cooled solution of phosphorus ylide 2e (224 mg, 0.63 mmol) in toluene (26 ml) at -78 °C. After 2 h at this temperature, the reaction mixture was allowed to reach room temperature overnight. Filtration of the obtained suspension afforded a white solid (306 mg, 83%) which corresponds to 3e.

$^1$H-NMR (400 MHz, CD$_2$Cl$_2$) $\delta$ = 7.64 (ddd, $J$ = 0.9, 1.8, 5.0 Hz, 1 H), 7.90-7.85 (m, 6 H), 7.66-7.61 (m, 3 H), 7.52-7.45 (m, 7 H), 7.23-7.21 (m, 1 H), 6.82-6.79 (m, 1 H), 4.5 (d, $J_{NH} = 7.9$ Hz, 1 H) ppm. $^{13}$C-NMR (101 MHz, CD$_2$Cl$_2$) $\delta$ = 161.0 (d, $J_{CP} = 5.5$ Hz), 147.9, 136.5, 134.5 (d, $J_{CP} = 9.2$ Hz), 133.3 (d, $J_{CP} = 12.1$), 125.9 (d, $J_{CP} = 87.9$), 122.7 (d, $J_{CP} = 13.1$ Hz), 119.3, 29.6 (d, $J_{CP} = 49.1$ Hz) ppm. $^{31}$P-NMR (162 MHz, CD$_2$Cl$_2$) $\delta$ = 28.3 ppm. HRMS calcld. for C$_{31}$H$_{30}$N$_2$O$_2$AgClPNa: 733.142026; found 733.142583. IR (neat) $\tilde{\nu}$ = 694, 712, 743, 755, 767, 854, 918, 946, 997, 1028, 1051, 1102, 1160, 1192, 1212, 1263, 1293, 1357, 1384, 1436, 1488, 1586, 1606, 2848, 2915, 2951, 3007, 3059, 3269 cm$^{-1}$.

Compound 6: A suspension of Phenylisocyanide gold (I) chloride 1a (38 mg, 0.11 mmol) in toluene (4.7 ml) was cooled at -10 °C and then the arsenic ylide 5 (41 mg, 0.11 mmol) was added. After stirring the obtained suspension for 1 d, the reaction mixture was allowed to reach room temperature. The solvents were then filtered out and the remaining a white solid washed with small portions of toluene and dried under vacuum. Thus, 6 was obtained as a white solid (32 mg, 40%). $^1$H-NMR (400 MHz, CD$_2$Cl$_2$) $\delta$ = 14.45 (s, 1 H), 7.85 (d, $J = 7.2$ Hz, 6 H), 7.70-7.60 (m, 11 H), 7.31 (t, $J = 7.7$ Hz, 2H), 7.21 (t, $J = 7.3$ Hz, 1 H), 1.61 (br, 3H) ppm. $^{13}$C-NMR (75 MHz, CD$_2$Cl$_2$) $\delta$ = 198.0, 144.1, 133.3, 133.1, 130.5, 129.1, 129.0, 126.4, 123.4, 31.3 ppm HRMS calcld. for C$_{28}$H$_{36}$AsAuClINaO: 720.032365; found 720.033071. IR (neat) $\tilde{\nu}$ = 691, 741, 756, 793, 865, 1014, 1078, 1259, 1371, 1441, 1459, 1509, 1565, 1589, 2853, 2922, 2955 cm$^{-1}$.

Compound 9: A mixture of phenylisocyanide gold (I) chloride 1a (25 mg, 0.08 mmol) and diaminoalkene 7 (16 mg, 0.08 mmol) was suspended in toluene (3 ml) and warmed to 35 °C. After 8 h the mixture was allowed to reach room temperature. Elimination of the solvents by filtration afforded pure 9 as a yellow solid (40 mg, 95% yield). $^1$H-NMR (400 MHz, CD$_2$Cl$_2$) $\delta$ = 14.73 (s, 1 H), 7.86 (d, $J = 7.8$ Hz, 2 H), 7.43-7.40 (m, 2 H), 7.35-7.32 (m, 1 H), 7.29-7.25 (m, 3 H), 7.06 (s, 2 H), 7.05-7.03 (m, 2 H) 3.63 (s, 6 H) ppm. $^{13}$C-NMR (101 MHz, CD$_2$Cl$_2$) $\delta$ = 194.6, 187.4, 151.5, 143.7, 142.3, 130.2, 129.3, 128.9, 126.2, 125.9, 122.7, 121.3, 98.4, 36.0 ppm. HRMS calcld. for C$_{30}$H$_{32}$N$_2$O$_2$AuClPNa: 572.077435; found 572.078099. IR (neat) $\tilde{\nu}$ = 691, 704, 728, 760, 792, 906, 1025, 1071, 1155, 1173, 1235, 1278, 1384, 1446, 1488, 1519, 1595, 3125 cm$^{-1}$.

Compound 10: A mixture of phenylisocyanide gold (I) chloride 1a (26 mg, 0.08 mmol) and diaminoalkene 9 (14 mg, 0.08 mmol) was suspended in toluene (3.3 ml) and warmed to 35 °C. After 18 h the mixture was allowed to reach room temperature. Elimination of the solvents by filtration afforded pure 10 as a yellow solid (35 mg, 87% yield).
\[ ^1H-NMR \ (400 \text{ MHz, } CD_2Cl_2) \delta = 12.09 \ (s, 1 \text{ H}), 8.32 \ (d, J = 6.2 \text{ Hz, } 1 \text{ H}), 8.22 \ (t, J = 7.9 \text{ Hz, } 1 \text{ H}), 8.16 \ (d, J = 7.9 \text{ Hz, } 1 \text{ H}), 7.78-7.76 \ (m, 2\text{ H}), 7.66-7.63 \ (m, 1\text{ H}), 7.35-7.32 \ (m, 2\text{ H}), 7.18-7.14 \ (m, 1 \text{ H}), 4.17 \ (s, 3 \text{ H}), 4.13-4.04 \ (m, 2 \text{ H}), 1.15 \ (t, J = 7.0 \text{ Hz, } 3 \text{ H}) \text{ ppm.} \]

\[ ^{13}C-NMR \ (101 \text{ MHz, } CD_2Cl_2) \delta = 189.6, 165.9, 162.2, 144.2, 143.1, 142.8, 135.3, 129.2, 125.0, 124.4, 122.0, 98.4, 59.5, 46.2, 14.8 \text{ ppm.} \]

HRMS calcd. for C_{17}H_{18}N_{2}O_{2}AuClNa: 537.061450; found 537.061877.

\[ \text{IR (neat) } \tilde{\nu} = 680, 694, 749, 759, 788, 893, 931, 954, 1028, 1074, 1091, 1175, 1218, 1256, 1299, 1336, 1373, 1447, 1493, 1527, 1588, 1622, 1640, 2975, 3055 \text{ cm}^{-1}. \]

\[ \text{Compound 11 : } \text{KOMe} \ (5.5 \text{ mg, } 0.078 \text{ mmol}) \text{ and } [\text{Rh(COD)}\text{Cl}]_2 \ (19.3 \text{ mg, } 0.039 \text{ mmol)} \text{ were suspended in THF (2 ml) and stirred for 10 min, at 5 °C. Then, 4a was added and the mixture stirred for 36 h. Along this time a light yellow precipitate was slowly formed. The reaction was then allowed to reach room temperature and the solvent evaporated in vacuo. The yellow solid thus obtained was washed with small portions of DCM to afford 11 \ (54 \text{ mg, } 72 \text{ %).} \]

\[ ^{1}H-NMR \ (400 \text{ MHz}, \text{CDCl}_3) \delta = 8.06-8.01 \ (m, 6 \text{ H}), 7.67-7.57 \ (m, 9 \text{ H}), 7.13-7.09 \ (m, 2 \text{ H}), 6.97 \ (t, J = 7.4 \text{ Hz, } 1 \text{ H}), 6.82 \ (d, J = 7.2 \text{ Hz}, 2 \text{ H}), 4.18 \ (br, 2 \text{ H}), 3.33 \ (br, 2 \text{ H}), 2.44-2.35 \ (m, 4 \text{ H}), 1.83-1.72 \ (m, 4 \text{ H}), 1.49 \ (s, 3 \text{ H}) \text{ ppm.} \]

\[ ^{13}C-NMR \ (126 \text{ MHz, } \text{CDCl}_3) \delta = 197.6 \ (d, J_{C-P} = 35.0 \text{ Hz}), 180.7 \ (d, J_{C-P} = 24.1 \text{ Hz}), 155.5, 134.1 \ (d, J_{C-P} = 8.7 \text{ Hz}), 133.1 \ (d, J_{C-P} = 2.8 \text{ Hz}), 129.4 \ (d, J_{C-P} = 12.4 \text{ Hz}), 128.2, 125.5 \ (d, J_{C-P} = 91.6 \text{ Hz}), 124.8, 124.6, 95.3 \ (d, J_{C-P} = 126.6 \text{ Hz}), 82.7 \ (d, J_{C-Rh} = 11.8 \text{ Hz}), 75.1 \ (d, J_{C-Rh} = 11.8 \text{ Hz}), 53.6, 31.6, 29.6, 28.7 \text{ ppm.} \]

\[ ^3P-NMR \ (162 \text{ MHz, } \text{CDCl}_3) \delta = 19.9 \text{ ppm.} \]

IR (neat) \[ \tilde{\nu} = 687, 708, 725, 741, 759, 776, 800, 869, 911, 964, 998, 1023, 1070, 1096, 1142, 1187, 1218, 1262, 1350, 1362, 1404, 1440, 1483, 1494, 1591, 2228, 2838, 2858, 2942, 2994, 3052 \text{ cm}^{-1}. \]

HRMS calcd. for C_{38}H_{35}AuCINO\text{PRhNa}^+: 886.075756; found 886.076414.

\[ \text{Compound 12: } [\text{RhCp}^*\text{Cl}]_2 \ (5.0 \text{ mg, } 0.009 \text{ mmol}) \text{ and 4a (10.2 mg, 0.016 mmol) were dissolved in DCE (0.2 ml) and NEt}_3 \ (0.04 \text{ ml, } 0.272 \text{ mmol) was dropwise added. This solution was heated at 50 °C and stirred at this temperature for 4 d. Then, the reaction mixture was allowed to cool down to room temperature and filtered in order to remove the formed precipitate. The bright red solution thus obtained was evaporated in vacuo affording a red solid that was redissolved in a small amount of toluene and filtrated again. Evaporation of the toluene produced an orange solid that could be further purified by consecutive crystallizations (2 times from DCM : pentane). Thus 11 was obtained as an orange solid (9.0 mg, 83 %).} \]

\[ ^1H-NMR \ (400 \text{ MHz, } \text{CDCl}_3) \delta = 7.75-7.66 \ (m, 6 \text{ H}), 7.53-7.50 \ (m, 3 \text{ H}), 7.47-7.43 \ (m, 6 \text{ H}), 6.96-6.92 \ (m, 2 \text{ H}), 6.69 \ (t, J = 7.2 \text{ Hz, } 1 \text{ H}), 6.54 \ (br, 2 \text{ H}), 1.36 \ (s, 3 \text{ H}), 1.34 \ (s, 15 \text{ H}) \text{ ppm.} \]

\[ ^{13}C-NMR \ (101 \text{ MHz, } \text{CDCl}_3) \delta = 200.5 \ (dd, J_{C-Rh} = 3.3 \text{ Hz}, J_{C-P} = 35.0 \text{ Hz}), 192.8 \ (d, J_{C-P} = 27.7 \text{ Hz}), 149.2, 132.6 \ (d, J_{C-P} = 9.8 \text{ Hz}), 130.1 \ (d, J_{C-P} = 3.3 \text{ Hz}), 127.6 \ (d, J_{C-P} = 12.4 \text{ Hz}), 126.6, 126.2 \ (d, J_{C-P} = 93.5 \text{ Hz}), 121.1, 119.6, 93.6 \ (d, J_{C-Rh} = 6.7 \text{ Hz}), 92.8 \ (d, J_{C-P} = 95.8 \text{ Hz}), 22.1, 8.4 \text{ ppm.} \]

\[ ^3P-NMR \ (162 \text{ MHz, } \text{CDCl}_3) \delta = 11.4 \ (d, J_{P-Rh} = 2.9 \text{ Hz}) \text{ ppm.} \]

IR (neat) \[ \tilde{\nu} = 695, 708, 722, 742, 756, 768, 843, 905, 984, 994, 1021, 1062, 1105, 1119, 1154, 1187, 1225, 1278, 1309, 1353, 1392, 1435, 1473, 1554, 1588, 2911, 3044 \text{ cm}^{-1}. \]

HRMS calcd. for C_{38}H_{35}CINO\text{PRh}: 694.150034; found 694.150324.
Compound 13: [Ru(cym)Cl]_2 (10.6 mg, 0.017 mmol) and 4a (22.6 mg, 0.035 mmol) were dissolved in DCE (0.7 ml) and then NEt_3 (0.08 ml, 0.595 mmol) was added drop wise. The reaction mixture was heated at 50 °C and stirred for 1 d. Then, the mixture was allowed to cool down to room temperature and filtered. The bright red solution was evaporated in vacuo and the remaining orange solids purified by consecutive crystallizations (two times from DCM : pentane) to afford 13 (10.0 mg, 41 %) as an orange solid. ^1^H-NMR (400 MHz, CD_2Cl_2) δ = 7.72 (dd, J = 7.9, 12.3 Hz, 6 H), 7.56-7.55 (m, 3 H), 7.49-7.47 (m, 6 H), 7.00 (t, J = 7.4 Hz, 2 H), 6.78 (t, J = 7.4 Hz, 1 H), 6.40 (br, 2 H), 5.42 (d, J = 5.9 Hz, 1 H), 4.96 (d, J = 5.9 Hz, 1 H), 4.70 (d, J = 5.4 Hz, 1 H), 3.77 (d, J = 5.4 Hz, 1 H), 2.42 (quint. J = 6.8 Hz, 1 H), 1.94 (s, 3 H), 1.28 (s, 3 H), 1.08 (d, J = 6.8 Hz, 3 H), 1.03 (d, J = 6.8 Hz, 3 H) ppm. ^1^3^C-NMR (101 MHz, CD_2Cl_2) δ = 208.8 (d, J_C-P = 2.8 Hz), 194.9 (d, J_C-P = 28.1 Hz), 155.5, 134.2 (d, J_C-P = 9.9 Hz), 132.1 (d, J_C-P = 2.9 Hz), 128.9 (d, J_C-P = 12.4 Hz), 128.1, 127.3 (d, J_C-P = 93.0 Hz), 122.0, 120.8, 102.5, 98.0, 93.2 (d, J_C-P = 97.3 Hz), 91.0, 84.2, 82.1, 76.6, 31.4, 23.2, 22.5, 22.2, 19.0 ppm. ^3^1^P-NMR (162 MHz, CD_2Cl_2) δ = 11.6 ppm. IR (neat) ν ~ = 692, 710, 726, 743, 801, 844, 864, 983, 1025, 1103, 1160, 1191, 1260, 1384, 1436, 1472, 1556, 1589, 1738, 2849, 1917, 2958, 3057 cm^{-1}. HRMS calcd. for C_{38}H_{38}ClNOPRu: 692.141123; found 692.141970.
Selected NMR spectra:

$^1$H-NMR (400 MHz, CD$_2$Cl$_2$) 3e

$^{13}$C-NMR (101 MHz, CD$_2$Cl$_2$) 3e
$^{31}\text{P-NMR (162 MHz, CD}_2\text{Cl}_2)$ 3e
$^1$H-NMR (400 MHz, CD$_2$Cl$_2$) 4a

$^{13}$C-NMR (101 MHz, CD$_2$Cl$_2$) 4a
$^{31}$P-NMR (162 MHz, CD$_2$Cl$_2$) 4a
$^1$H-NMR (400 MHz, CD$_2$Cl$_2$) 4b

$^{13}$C-NMR (101 MHz, CD$_2$Cl$_2$) 4b
$^{31}\text{P-NMR (162 MHz, CD}_2\text{Cl}_2)$ 4b
$^1$H-NMR (400 MHz, CD$_2$Cl$_2$) 4c

$^{13}$C-NMR (101 MHz, CD$_2$Cl$_2$) 4c
$^{31}$P-NMR (162 MHz, CD$_2$Cl$_2$) 4c
$^1$H-NMR (400 MHz, CD$_2$Cl$_2$) 4d

$^{13}$C-NMR (101 MHz, CD$_2$Cl$_2$) 4d
$^{31}\text{P-NMR (162 MHz, CD}_{2}\text{Cl}_{2})\ 4d$
$^1$H-NMR (400 MHz, CD$_2$Cl$_2$) 4e

$^{13}$C-NMR (101 MHz, CD$_2$Cl$_2$) 4e
$^{31}\text{P-NMR (162 MHz, CD}_2\text{Cl}_2\text{)}$ 4e
$^1$H-NMR (400 MHz, CD$_2$Cl$_2$) 4f

$^{13}$C-NMR (101 MHz, CD$_2$Cl$_2$) 4f
$^{31}$P-NMR (162 MHz, CD$_2$Cl$_2$) 4f
$^1$H-NMR (400 MHz, CD$_2$Cl$_2$) 4g

$^{13}$C-NMR (101 MHz, CD$_2$Cl$_2$) 4g
$^{31}P$-NMR (162 MHz, CD$_2$Cl$_2$) 4g
$^1$H-NMR (400 MHz, CD$_2$Cl$_2$) 4h

$^{13}$C-NMR (101 MHz, CD$_2$Cl$_2$) 4h
$^{31}\text{P-NMR (162 MHz, CD}_2\text{Cl}_2\text{)} ~ 4h$
$^1$H-NMR (400 MHz, CD$_2$Cl$_2$) 4i

$^{13}$C-NMR (101 MHz, CD$_2$Cl$_2$) 4i
$^{31}$P-NMR (162 MHz, CD$_2$Cl$_2$) 4i
$\text{^1H-NMR (400 MHz, CD}_2\text{Cl}_2\text{) 4j}$

![1H-NMR spectrum]

$\text{Me}_2\text{N CO}$

$\text{Ph}_3\text{P Au}$

$\text{Cl}$

$\text{4j}$

$\text{^13C-NMR (101 MHz, CD}_2\text{Cl}_2\text{) 4j}$

![13C-NMR spectrum]
$^{31}$P-NMR (162 MHz, CD$_2$Cl$_2$) 4j
$^1$H-NMR (400 MHz, CD$_2$Cl$_2$) 6

$^{13}$C-NMR (75 MHz, CD$_2$Cl$_2$) 6
$^1$H-NMR (400 MHz, CD$_2$Cl$_2$) 8

$^{13}$C-NMR (101 MHz, CD$_2$Cl$_2$) 8
$^1$H-NMR (400 MHz, CD$_2$Cl$_2$) **10**

$^{13}$C-NMR (101 MHz, CD$_2$Cl$_2$) **10**
$^1$H-NMR (400 MHz, CD$_2$Cl$_2$) 11

$^{13}$C-NMR (101 MHz, CD$_2$Cl$_2$) 11
$^{31}$P-NMR (162 MHz, CD$_2$Cl$_2$) 11
^1\text{H-}NMR\ (400\ \text{MHz, CD}_2\text{Cl}_2)\ 12

$\text{Ph}_3\text{P}^\text{-Rh}^\text{N-Ph}$

31\text{P-}NMR\ (162\ \text{MHz, CD}_2\text{Cl}_2)\ 12
$^1$H-NMR (400 MHz, CD$_2$Cl$_2$) 13

$^{13}$C-NMR (101 MHz, CD$_2$Cl$_2$) 13
$^{31}$P-NMR (162 MHz, CD$_2$Cl$_2$) 13
Empirical formula
Cₐ₀H₂₂AuCl₃NP

Color
colourless

Formula weight
670.72 g · mol⁻¹

Temperature
150 K

Wavelength
0.71073 Å

Crystal system
MONOCLINIC

Space group
C2/c, (no. 15)

Unit cell dimensions
a = 24.4111(6) Å  \( \alpha = 90^\circ \),
b = 8.577(2) Å  \( \beta = 93.067(5)^\circ \),
c = 23.0774(16) Å  \( \gamma = 90^\circ \).

Volume
4824.8(12) Å³

Z
8

Density (calculated)
1.847 Mg · m⁻³

Absorption coefficient
6.511 mm⁻¹

F(000)
2592 e

Crystal size
0.30 x 0.12 x 0.07 mm³

θ range for data collection
3.05 to 33.10°.

Index ranges
-37 ≤ h ≤ 37, -13 ≤ k ≤ 13, -35 ≤ l ≤ 35

Reflections collected
67463

Independent reflections
9159 \([R_{int} = 0.0532]\)

Reflections with I>2σ(I)
7901

Completeness to θ = 27.50°
99.9 %

Absorption correction
Gaussian

Max. and min. transmission
0.81 and 0.39

Refinement method
Full-matrix least-squares on F²

Data / restraints / parameters
9159 / 0 / 289

Goodness-of-fit on F²
1.086

Final R indices [I>2σ(I)]
R₁ = 0.0288
wR² = 0.0665

R indices (all data)
R₁ = 0.0378
wR² = 0.0704

Largest diff. peak and hole
1.327 and -2.047 e · Å⁻³
**Empirical formula**  
C$_{31}$H$_{31}$AuClN$_2$O$_2$P

**Color**  
colourless

**Formula weight**  
710.96 g·mol$^{-1}$

**Temperature**  
100 K

**Wavelength**  
1.54178 Å

**Crystal system**  
MONOCLINIC

**Space group**  
p 21/c, (no. 14)

**Unit cell dimensions**  
a = 16.8854(7) Å  
b = 10.9240(5) Å  
c = 16.4234(7) Å  
$\alpha$ = 90°,  
$\beta$ = 111.3890(10)°,  
$\gamma$ = 90°.

**Volume**  
2820.7(2) Å$^3$

**Z**  
4

**Density (calculated)**  
1.674 Mg·m$^{-3}$

**Absorption coefficient**  
11.412 mm$^{-1}$

**F(000)**  
1400 e

**Crystal size**  
0.14 x 0.13 x 0.10 mm$^3$

**$\theta$ range for data collection**  
2.81 to 67.16°.

**Index ranges**  
-18 ≤ h ≤ 20, -12 ≤ k ≤ 13, -19 ≤ l ≤ 19

**Reflections collected**  
67770

**Independent reflections**  
4988 [R$_{int}$ = 0.0481]

**Reflections with I>2$\sigma$(I)**  
4859

**Completeness to $\theta$ = 67.16°**  
99.2 %

**Absorption correction**  
Gaussian

**Max. and min. transmission**  
0.54295 and 0.22715

**Refinement method**  
Full-matrix least-squares on F$^2$

**Data / restraints / parameters**  
4988 / 0 / 338

**Goodness-of-fit on F$^2$**  
1.123

**Final R indices [I>2$\sigma$(I)]**  
R$_1$ = 0.0186  
wR$^2$ = 0.0442

**R indices (all data)**  
R$_1$ = 0.0193  
wR$^2$ = 0.0445

**Largest diff. peak and hole**  
0.496 and -0.717 e·Å$^{-3}$
**Compound 4a**

- **Empirical formula**: $\text{C}_{28}\text{H}_{24}\text{AuClNO}_2\text{P}$
- **Color**: colourless
- **Formula weight**: 653.87 g·mol$^{-1}$
- **Temperature**: 100 K
- **Wavelength**: 0.71073 Å
- **Crystal system**: MONOCLINIC
- **Space group**: $\text{P2}_1/\text{c}$, (no. 14)
- **Unit cell dimensions**:
  - $a = 14.7905(9)$ Å
  - $b = 11.9586(10)$ Å
  - $c = 15.2551(7)$ Å
  - $\alpha = 90^\circ$
  - $\beta = 115.796(4)^\circ$
  - $\gamma = 90^\circ$
- **Volume**: 2429.3(3) Å$^3$
- **Z**: 4
- **Density (calculated)**: 1.788 Mg·m$^{-3}$
- **Absorption coefficient**: 6.253 mm$^{-1}$
- **F(000)**: 1272 e
- **Crystal size**: 0.27 x 0.21 x 0.10 mm$^3$
- **θ range for data collection**: 2.68 to 37.00°
- **Index ranges**: $-24 \leq h \leq 25$, $-20 \leq k \leq 20$, $-25 \leq l \leq 25$
- **Reflections collected**: 60767
- **Independent reflections**: 12321  [R$_{int}$ = 0.0518]
- **Reflections with I>2σ(I)**: 9429
- **Completeness to θ = 37.00°**: 99.8 %
- **Absorption correction**: Gaussian
- **Max. and min. transmission**: 0.55184 and 0.21375
- **Refinement method**: Full-matrix least-squares on F$^2$
- **Data / restraints / parameters**: 12321 / 0 / 299
- **Goodness-of-fit on F$^2$**: 1.037
- **Final R indices [I>2σ(I)]**: $R_I = 0.0329$, $wR^2 = 0.0548$
- **R indices (all data)**: $R_I = 0.0580$, $wR^2 = 0.0605$
- **Largest diff. peak and hole**: 1.425 and -2.498 e·Å$^{-3}$
Compound 4e:

Empirical formula: $C_{31}H_{25}AuCl_2N_2P·0.5CH_2Cl_2$

Color: yellow

Formula weight: 731.38 g · mol$^{-1}$

Temperature: 100 K

Wavelength: 0.71073 Å

Crystal system: MONOCLINIC

Space group: $P2_1/c$, (no. 14)

Unit cell dimensions:

- a = 8.7003(3) Å
- b = 19.0210(12) Å
- c = 17.8177(14) Å
- $\alpha = 90^\circ$
- $\beta = 90.348(4)^\circ$
- $\gamma = 90^\circ$

Volume: 2948.6(3) Å$^3$

Z: 4

Density (calculated): 1.648 Mg · m$^{-3}$

Absorption coefficient: 5.248 mm$^{-1}$

$F(000)$: 1428 e

Crystal size: 0.17 x 0.10 x 0.09 mm$^3$

$\theta$ range for data collection: 3.17 to 34.94$^\circ$

Index ranges: -14 ≤ h ≤ 13, -28 ≤ k ≤ 30, -28 ≤ l ≤ 28

Reflections collected: 95323

Independent reflections: 12890 [$R_{int} = 0.0388$]

Reflections with $I>2\sigma(I)$: 11231

Completeness to $\theta = 27.50^\circ$: 99.8 %

Absorption correction: Gaussian

Max. and min. transmission: 0.66 and 0.49

Refinement method: Full-matrix least-squares on $F^2$

Data / restraints / parameters: 12890 / 0 / 345

Goodness-of-fit on $F^2$: 1.091

Final R indices [$I>2\sigma(I)$]: $R_1 = 0.0244$  $wR^2 = 0.0570$

R indices (all data): $R_1 = 0.0326$  $wR^2 = 0.0605$

Largest diff. peak and hole: 2.411 and -1.792 e · Å$^{-3}$
**Compound 6:**

- **Empirical formula:** C\(_{28}\)H\(_{24}\)AsAuClN\(_2\)
- **Color:** colourless
- **Formula weight:** 697.82 g · mol\(^{-1}\)
- **Temperature:** 100 K
- **Wavelength:** 0.71073 Å
- **Space group:** \(\text{P2}_1/c\), (no. 14)
- **Unit cell dimensions:**
  - \(a = 14.9063(15)\) Å, \(\alpha = 90^\circ\)
  - \(b = 12.0478(12)\) Å, \(\beta = 115.743(3)\)°
  - \(c = 15.3302(3)\) Å, \(\gamma = 90^\circ\)
- **Volume:** 2479.9(4) Å\(^3\)
- **Z:** 4
- **Density (calculated):** 1.869 Mg · m\(^{-3}\)
- **Absorption coefficient:** 7.385 mm\(^{-1}\)
- **F(000):** 1344 e
- **Crystal size:** 0.20 x 0.17 x 0.07 mm\(^3\)
- **\(\theta\) range for data collection:** 2.67 to 36.00°.
- **Index ranges:** -24 ≤ h ≤ 24, -19 ≤ k ≤ 19, -25 ≤ l ≤ 25
- **Reflections collected:** 62465
- **Independent reflections:** 11702 \([R_{\text{int}} = 0.0491]\)
- **Reflections with I>2\(\sigma(I)\):** 10436
- **Completeness to \(\theta = 27.50^\circ\):** 99.8 %
- **Absorption correction:** Gaussian
- **Max. and min. transmission:** 0.62 and 0.27
- **Refinement method:** Full-matrix least-squares on \(F^2\)
- **Data / restraints / parameters:** 11702 / 0 / 299
- **Goodness-of-fit on \(F^2\):** 1.094
- **Final R indices \([I>2\sigma(I)]\):** \(R_1 = 0.0294\) \(\quad wR^2 = 0.0693\)
- **R indices (all data):** \(R_1 = 0.0357\) \(\quad wR^2 = 0.0723\)
- **Largest diff. peak and hole:** 1.897 and -4.624 e · Å\(^{-3}\)
Compound 9

Empirical formula: \( \text{C}_{20}\text{H}_{19}\text{AuCl}_3\text{N}_3\text{O} \)

Color: yellow

Formula weight: 549.80 g · mol\(^{-1}\)

Temperature: 100 K

Wavelength: 0.71073 Å

Crystal system: MONOCLINIC

Space group: \( \text{P2}_1/\text{n}, \text{ (no. 14)} \)

Unit cell dimensions:
- \( a = 12.3447(8) \text{ Å} \)
- \( b = 11.9732(8) \text{ Å} \)
- \( c = 13.8197(9) \text{ Å} \)

Volume: 1907.9(2) Å\(^3\)

Density (calculated): 1.914 Mg · m\(^{-3}\)

Absorption coefficient: 7.864 mm\(^{-1}\)

\( F(000) \): 1056 e

Crystal size: 0.27 x 0.26 x 0.05 mm\(^3\)

\( \theta \) range for data collection: 2.76 to 34.99°

Index ranges:
- \(-19 \leq h \leq 19\)
- \(-19 \leq k \leq 19\)
- \(-22 \leq l \leq 22\)

Reflections collected: 55520

Independent reflections: 8346 [\( R_{\text{int}} = 0.0311 \)]

Reflections with \( I > 2 \sigma(I) \): 7679

Completeness to \( \theta = 34.99° \): 99.4 %

Absorption correction: Gaussian

Max. and min. transmission: 0.69 and 0.14

Refinement method: Full-matrix least-squares on \( F^2 \)

Data / restraints / parameters: 8346 / 0 / 237

Goodness-of-fit on \( F^2 \): 1.172

Final \( R \) indices \([I > 2 \sigma(I)]\):
- \( R_1 = 0.0157 \)
- \( wR^2 = 0.0432 \)

\( R \) indices (all data):
- \( R_1 = 0.0192 \)
- \( wR^2 = 0.0450 \)

Largest diff. peak and hole: 0.668 and -1.638 e · Å\(^{-3}\)
Compound 11:

Empirical formula: C_{37}H_{37}AuCl_3NOPRh

Color: orange

Formula weight: 948.87 g · mol⁻¹

Temperature: 100 K

Wavelength: 0.71073 Å

Crystal system: MONOCLINIC

Space group: P2_1/c (no. 14)

Unit cell dimensions:
- a = 15.2528(12) Å, α = 90°.
- b = 12.7151(19) Å, β = 101.855(11)°.
- c = 17.885(4) Å, γ = 90°.

Volume: 3394.7(10) Å³

Z: 4

Density (calculated): 1.857 Mg · m⁻³

Absorption coefficient: 5.118 mm⁻¹

F(000): 1856 e

Crystal size: 0.14 x 0.12 x 0.06 mm³

θ range for data collection: 2.729 to 33.218°.

Index ranges:
- -23 ≤ h ≤ 23
- -19 ≤ k ≤ 19
- -25 ≤ l ≤ 27

Reflections collected: 58509

Independent reflections: 12950 [R(int) = 0.0360]

Reflections with I>2σ(I): 11849

Completeness to θ = 25.242°: 99.8 %

Absorption correction: Gaussian

Max. and min. transmission: 0.75 and 0.51

Refinement method: Full-matrix least-squares on F²

Data / restraints / parameters: 12950 / 0 / 407

Goodness-of-fit on F²: 1.096

Final R indices [I>2σ(I)]: R₁ = 0.0294, wR² = 0.0676

R indices (all data): R₁ = 0.0342, wR² = 0.0698

Extinction coefficient: n/a

Largest diff. peak and hole: 1.7 and -3.4 e · Å⁻³
Compound 13:

Empirical formula: $C_{38}H_{38}Cl_1N_1O_1P_1Rh_1$

Color: orange

Formula weight: 694.02 g · mol$^{-1}$

Temperature: 100 K

Wavelength: 0.71073 Å

Crystal system: MONOCLINIC

Space group: $P2_1/c$, (no. 14)

Unit cell dimensions:
- $a = 10.7983(10)$ Å, $\alpha = 90^\circ$
- $b = 13.1084(12)$ Å, $\beta = 100.263(2)^\circ$
- $c = 23.391(2)$ Å, $\gamma = 90^\circ$

Volume: 3258.0(5) Å$^3$

$Z$: 4

Density (calculated): 1.415 Mg · m$^{-3}$

Absorption coefficient: 0.686 mm$^{-1}$

$F(000)$: 1432 e

Crystal size: $0.15 \times 0.12 \times 0.08$ mm$^3$

$\theta$ range for data collection: 1.77 to 33.31$^\circ$

Index ranges:
- $-16 \leq h \leq 16$, $-20 \leq k \leq 20$, $-35 \leq l \leq 36$

Reflections collected: 107131

Independent reflections: 12543 [$R_{int} = 0.0424$]

Reflections with $I > 2\sigma(I)$: 10641

Completeness to $\theta = 27.50^\circ$: 100.0 %

Absorption correction: Gaussian

Max. and min. transmission: 0.77 and 0.54

Refinement method: Full-matrix least-squares on $F^2$

Data / restraints / parameters: 12543 / 0 / 394

Goodness-of-fit on $F^2$: 1.114

Final R indices [$I > 2\sigma(I)$]: $R_1 = 0.0233$, $wR^2 = 0.0596$

R indices (all data): $R_1 = 0.0333$, $wR^2 = 0.0684$

Largest diff. peak and hole: 0.535 and -0.498 e · Å$^{-3}$