Supporting Information
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Synthesis and Reactivity of Metal Complexes with Acyclic (Amino)-(Ylide)Carbene Ligands**
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**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 ($\delta$) 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 $2d$, $2f$, $2e$, $7$ and $9$ 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. Phenylisocyanide was prepared by the method of Weber et al. from aniline.

**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.

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**Compound 4a:** Following the general procedure described above, a mixture of phenylisocyanide gold (l) 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.

$^1\text{H-NMR}$ (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. $^{13}\text{C-NMR}$ (101 MHz, CD$_2$Cl$_2$) $\delta = 200.9$ (d, $J_{CP} = 36.0$ Hz), 195.3 (d, $J_{CP} = 24.0$ Hz), 144.1, 134.5 (d, $J_{CP} = 8.6$ Hz), 133.5 (d, $J_{CP} = 3.0$ Hz), 129.8 (d, $J_{CP} = 12.3$ Hz), 129.1, 126.6, 125.8 (d, $J_{CP} = 91.6$ Hz), 123.7, 93.0 (d, $J_{CP} = 124.9$ Hz), 31.7 (d, $J_{CP} = 2.3$ Hz) ppm. $^{31}\text{P-NMR}$ (162 MHz, CD$_2$Cl$_2$) $\delta = 19.6$ ppm. HRMS calc’d. for C$_{28}$H$_{30}$NOAuClPNa: 676.084174; found 676.084426. 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 (l) 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.

$^1\text{H-NMR}$ (400 MHz, CD$_2$Cl$_2$) $\delta = 12.67$ (s, 1 H), 7.89-7.84 (m, 6 H), 7.67-7.63 (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. $^{13}\text{C-NMR}$ (101 MHz, CD$_2$Cl$_2$) $\delta = 200.6$ (d, $J_{CP} = 35.9$ Hz), 168.8 (d, $J_{CP} = 17.2$ Hz), 144.4, 134.1 (d, $J_{CP} = 9.0$ Hz), 133.0 (d, $J_{CP} = 2.6$ Hz), 129.4 (d, $J_{CP} = 12.7$ Hz), 129.1, 126.3, 126.2(d, $J_{CP} = 94.0$ Hz), 123.4, 79.4 (d, $J_{CP} = 134.4$ Hz), 60.0, 13.6 ppm. $^{31}\text{P-NMR}$ (162 MHz, CD$_2$Cl$_2$) $\delta = 22.0$ ppm. HRMS calc’d. for C$_{29}$H$_{30}$NO$_2$AuClPNa: 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 (l) chloride 1a (23 mg, 0.07 mmol) and the phosphorus ylide 2c (21 mg, 0.07 mmol) afforded pure 4c (38 mg, 88%) after a reaction time of 3 d at 35 °C.

$^1\text{H-NMR}$ (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. $^{13}\text{C-NMR}$ (101 MHz, CD$_2$Cl$_2$) $\delta = 201.9$ (d, $J_{CP} = 35.9$ Hz), 143.0, 134.8 (d, $J_{CP} = 9.8$ Hz), 134.4 (d, $J_{CP} = 2.9$ Hz), 129.8 (d, $J_{CP} = 13.0$ Hz), 129.3, 126.8, 123.1, 122.9 (d, $J_{CP} = 94.1$ Hz), 117.8 (d, $J_{CP} = 22.1$ Hz), 60.1 (d, $J_{CP} = 154.6$ Hz) ppm. $^{31}\text{P-NMR}$ (162 MHz, CD$_2$Cl$_2$) $\delta = 21.8$ ppm. HRMS calc’d. for C$_{27}$H$_{21}$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 (l) 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.

$^1\text{H-NMR}$ (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. $^{13}\text{C-NMR}$ (101 MHz,
CD$_2$Cl$_2$ $\delta$ = 188.9 (d, $J_{CP}$ = 32.0 Hz), 167.8 (d, $J_{CP}$ = 19.1 Hz), 144.4, 134.6 (d, $J_{CP}$ = 9.3 Hz), 133.4 (d, $J_{CP}$ = 2.9 Hz), 129.3 (d, $J_{CP}$ = 12.4 Hz), 129.0, 125.1 (d, $J_{CP}$ = 92.0 Hz), 125.0, 122.4, 86.1 (d, $J_{CP}$ = 132.3 Hz), 37.0 ppm.

$^{31}$P-NMR (162 MHz, CD$_2$Cl$_2$) $\delta$ = 18.1 ppm. HRMS calcld. for C$_{29}$H$_{27}$N$_2$O$_3$AuClPNa: 705.110727; found 705.110773.

IR (neat) $\tilde{\nu}$ = 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.

![4e](image)

$^1$H-NMR (400 MHz, CD$_2$Cl$_2$) $\delta$ = 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. $^{13}$C-NMR (101 MHz, CD$_2$Cl$_2$) $\delta$ = 189.6 (d, $J_{CP}$ = 35.7 Hz), 156.4 (d, $J_{CP}$ = 20.1 Hz), 149.6, 144.5, 136.3, 134.7 (d, $J_{CP}$ = 9.1 Hz), 133.0 (d, $J_{CP}$ = 2.6 Hz), 129.2 (d, $J_{CP}$ = 12.1 Hz), 128.8, 126.8 (d, $J_{CP}$ = 3.0 Hz), 126.2 (d, $J_{CP}$ = 91.8 Hz), 124.4, 121.7, 120.8, 87.9 (d, $J_{CP}$ = 134.4 Hz) ppm. $^{31}$P-NMR (162 MHz, CD$_2$Cl$_2$) $\delta$ = 20.0 ppm. HRMS calcld. for C$_{32}$H$_{25}$N$_2$O$_3$AuClPNa: 711.100164; found 711.100454.

IR (neat) $\tilde{\nu}$ = 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. $^1$H-NMR (400 MHz, CD$_2$Cl$_2$) $\delta$ = 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. $^{13}$C-NMR (101 MHz, CD$_2$Cl$_2$) $\delta$ = 187.1 (d, $J_{CP}$ = 37.7 Hz), 144.5, 134.8 (d, $J_{CP}$ = 9.0 Hz), 134.0 (d, $J_{CP}$ = 3.8 Hz), 133.1 (d, $J_{CP}$ = 2.8 Hz), 133.1 (d, $J_{CP}$ = 2.8 Hz), 132.3 (d, $J_{CP}$ = 9.9 Hz), 129.6 (d, $J_{CP}$ = 1.7 Hz), 129.1, 129.1 (d, $J_{CP}$ = 12.3 Hz), 129.0 (d, $J_{CP}$ = 12.1 Hz), 128.9, 127.9 (d, $J_{CP}$ = 2.3 Hz), 125.8 (d, $J_{CP}$ = 91.1 Hz), 124.0, 121.2, 88.9 (d, $J_{CP}$ = 132.1 Hz) ppm. $^{31}$P-NMR (162 MHz, CD$_2$Cl$_2$) $\delta$ = 21.0 ppm. HRMS calcld. for C$_{31}$H$_{31}$N$_2$O$_3$AuClPNa: 710.104912; found 710.105881. IR (neat) $\tilde{\nu}$ = 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.

$^1$H-NMR (400 MHz, CD$_2$Cl$_2$) $\delta$ = 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. $^{13}$C-NMR (101 MHz, CD$_2$Cl$_2$) $\delta$ = 206.6 (d, $J_{CP}$ = 34.8 Hz), 195.0 (d, $J_{CP}$ = 28.0 Hz), 142.6, 135.0, 134.6 (d, $J_{CP}$ = 8.6 Hz), 133.6 (d, $J_{CP}$ = 2.9 Hz), 129.7 (d, $J_{CP}$ = 12.4 Hz), 128.4, 127.6, 125.9 (d, $J_{CP}$ = 92.0 Hz), 91.1
(d, \( J_{C,P} = 124.3 \) Hz), 31.6 (d, \( J_{C,P} = 2.0 \) Hz), 19.1 ppm. \(^{31}\)P-NMR (162 MHz, CD\(_2\)Cl\(_2\)) \( \delta = 20.5 \) ppm. HRMS calcd. for C\(_{30}\)H\(_{30}\)NOAuClPNa: 704.115849; found 704.115049. IR (neat) \( \tilde{\nu} = 682, 693, 709, 720, 734, 755, 768, 781, 842, 873, 920, 982, 998, 1018, 1046, 1098, 1142, 1213, 1250, 1268, 1342, 1360, 1418, 1435, 1500, 1572, 2975 cm\(^{-1}\).

**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 calcd. for C\(_{31}\)H\(_{29}\)NO\(_2\)AuClPNa: 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 calcd. for C\(_{28}\)H\(_{27}\)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 calcd. for C\(_{31}\)H\(_{31}\)N\(_2\)O2AuClPNa:
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.

\[
\begin{align*}
\text{Ph}_3\text{P} & \quad \text{Au} \\
\text{N} & \quad \text{Cl}
\end{align*}
\]

\text{Compound 3e}

$^1$H-NMR (400 MHz, CD$_2$Cl$_2$) δ = 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, $J_{HP} = 7.9$ Hz, 1 H) ppm. $^{13}$C-NMR (101 MHz, CD$_2$Cl$_2$) δ = 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$_2$) δ = 28.3 ppm. HRMS calc'd. for C$_{35}$H$_{36}$N$_2$OAuClPNa: 733.142026; found 733.142583. IR (neat) ν = 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$_2$) δ = 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$_2$) δ = 198.0, 144.1, 133.3, 133.1, 130.5, 129.1, 129.0, 126.4, 123.4, 31.3 ppm HRMS calc'd. for C$_{28}$H$_{32}$AsAuClNO Na: 720.032365; found 720.033071. IR (neat) ν = 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$_2$) δ = 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$_2$) δ = 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 calc'd. for C$_{29}$H$_{36}$NOAuClPNa: 572.077435; found 572.078099. IR (neat) ν = 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).
\[ \begin{align*}
\text{1H-NMR (400 MHz, CD}_{2}\text{Cl}_2) \delta &= 12.09 (s, 1 H), 8.32 (d, J = 6.2 Hz, 1H), 8.22 (t, J = 7.9 Hz, 1H), 8.16 (d, J = 7.9 Hz, 1H), 7.78-7.76 (m, 2H), 7.66-7.63 (m, 1H), 7.35-7.32 (m, 2H), 7.18-7.14 (m, 1H), 4.17 (s, 3H), 4.13-4.04 (m, 2H), 1.15 (t, J = 7.0 Hz, 3H) ppm. 13C-NMR (101 MHz, CD}_{2}\text{Cl}_2) \delta = 189.6, 165.9, 162.2, 144.2, 143.1, 142.8, 135.3, 129.2, 125.0, 124.4, 122.0, 98.4, 59.5, 46.2, 14.8 ppm. HRMS calcld. for C_{17}H_{18}N_{2}O_{2}AuClNa: 537.061450; found 537.061877. IR (neat) \tilde{\nu} = 680, 694, 749, 759, 788, 893, 931, 954, 1028, 1074, 1091, 1175, 1218, 1256, 1299, 1336, 1373, 1447, 1493, 1527, 1588, 1622, 1640, 2975, 3055 cm\textsuperscript{-1}. 
\end{align*} \]

\[ \mathbf{10} \]

**Compound 11**: KOMe (5.5 mg, 0.078 mmol) and [Rh(COD)Cl]_2 (19.3 mg, 0.039 mmol) 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 \textit{in vacuo}. The yellow solid thus obtained was washed with small portions of DCM to afford 11 (54 mg, 72 %). \[ \text{1H-NMR (400 MHz, CDCl}_3) \delta = 8.06-8.01 (m, 6H), 7.67-7.57 (m, 9H), 7.13-7.09 (m, 2H), 6.97 (t, J = 7.4 Hz, 1H), 6.82 (d, J = 7.2 Hz, 2H), 4.18 (br, 2H), 3.33 (br, 2H), 2.44-2.35 (m, 4H), 1.83-1.72 (m, 4H), 1.49 (s, 3H) ppm. \]

\[ \text{13C-NMR (126 MHz, CDCl}_3) \delta = 197.6 (d, J= 35.0 Hz), 180.7 (d, J= 24.1 Hz), 155.5, 134.1 (d, J= 8.7 Hz), 133.1 (d, J= 2.8 Hz), 129.4 (d, J= 12.4 Hz), 128.2, 125.5 (d, J= 91.6 Hz), 124.8, 124.6, 95.3 (d, J= 126.6 Hz), 82.7 (d, J= 11.8 Hz), 75.1 (d, J= 11.8 Hz), 53.6, 31.6, 29.6, 28.7 ppm. 31P-NMR (162 MHz, CDCl}_3) \delta = 19.9 ppm. IR (neat) \tilde{\nu} = 687, 708, 725, 741, 759, 776, 800, 869, 911, 964, 998, 1023, 1070, 1096, 1142, 1187, 1218, 1262, 1350, 1362, 1404, 1440, 1483, 1494, 1591, 2228, 2838, 2858, 2942, 2994, 3052 cm\textsuperscript{-1}. HRMS calcld. for C_{38}H_{35}AuClNOPRh\textsuperscript{+}: 886.075756; found 886.076414. \]

\[ \mathbf{11} \]

**Compound 12**: [RhCp\textsuperscript{+}Cl\textsubscript{3}]_2 (5.0 mg, 0.009 mmol) and 4a (10.2 mg, 0.016 mmol) were dissolved in DCE (0.2 ml) and NEt\textsubscript{3} (0.04 ml, 0.272 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 \textit{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 crystallisations (2 times from DCM : pentane). Thus 11 was obtained as an orange solid (9.0 mg, 83 %). \[ \text{1H-NMR (400 MHz, CDCl}_3) \delta = 7.75-7.66 (m, 6H), 7.53-7.50 (m, 3H), 7.47-7.43 (m, 6H), 6.96-6.92 (m, 2H), 6.69 (t, J = 7.2 Hz, 1H), 6.54 (br, 2H), 1.36 (s, 3H), 1.34 (s, 15H) ppm. \]

\[ \text{13C-NMR (101 MHz, CDCl}_3) \delta = 200.5 (dd, J= 3.3 Hz, J= 35.0 Hz), 192.8 (d, J= 27.7 Hz), 149.2, 132.6 (d, J= 9.8 Hz), 130.1 (d, J= 3.3 Hz), 127.6 (d, J= 12.4 Hz), 126.6, 126.2 (d, J= 93.5 Hz), 121.1, 119.6, 93.6 (d, J= 6.7 Hz), 92.8 (d, J= 95.8 Hz), 22.1, 8.4 ppm. 31P-NMR (162 MHz, CDCl}_3) \delta = 11.4 (d, J= 2.9 Hz) ppm. IR (neat) \tilde{\nu} = 695, 708, 722, 742, 756, 768, 843, 905, 984, 994, 1021, 1062, 1105, 1119, 1154, 1187, 1225, 1278, 1309, 1353, 1392, 1435, 1473, 1554, 1588, 2911, 3044 cm\textsuperscript{-1}. HRMS calcld. for C_{38}H_{35}ClNOPRh: 694.150034; found 694.150324. \]

\[ \mathbf{12} \]
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) \(\delta = 7.72 \ (dd, \ J = 7.9, 12.3 \ Hz, \ 6 \ H), 7.56-7.55 \ (m, \ 3 \ H), 7.49-7.47 \ (m, \ 6 \ H), 7.00 \ (t, \ J = 7.4 \ Hz, \ 2 \ H), 6.78 \ (t, \ J = 7.4 \ Hz, \ 1 \ H), 6.40 \ (br, \ 2 \ H), 5.42 \ (d, \ J = 5.9 \ Hz, \ 1 \ H), 4.96 \ (d, \ J = 5.9 \ Hz, \ 1 \ H), 4.70 \ (d, \ J = 5.4 \ Hz, \ 1 \ H), 3.77 \ (d, \ J = 5.4 \ Hz, \ 1 \ H), 2.42 \ (quint. \ J = 6.8 \ Hz, \ 1 \ H), 1.94 \ (s, \ 3 \ H), 1.28 \ (s, \ 3 \ H), 1.08 \ (d, \ J = 6.8 \ Hz, \ 3 \ H), 1.03 \ (d, \ J = 6.8 \ Hz, \ 3 \ H) \ ppm. \ ^{13}\)C-NMR (101 MHz, CD_2Cl_2) \(\delta = 208.8 \ (d, \ J_{C\cdot P} = 2.8 \ Hz), 194.9 \ (d, \ J_{C\cdot P} = 28.1 \ Hz), 155.5, 134.2 \ (d, \ J_{C\cdot P} = 9.9 \ Hz), 132.1 \ (d, \ J_{C\cdot P} = 2.9 \ Hz), 128.9 \ (d, \ J_{C\cdot P} = 12.4 \ Hz), 128.1, 127.3 \ (d, \ J_{C\cdot P} = 93.0 \ Hz), 122.0, 120.8, 102.5, 98.0, 93.2 \ (d, \ J_{C\cdot P} = 97.3 \ Hz), 91.0, 84.2, 82.1, 76.6, 31.4, 23.2, 22.5, 22.2, 19.0 \ ppm. \ ^{31}\)P-NMR (162 MHz, CD_2Cl_2) \(\delta = 11.6 \ ppm. \) IR (neat) \(\tilde{\nu} = 692, 710, 726, 743, 801, 844, 864, 983, 1025, 1103, 1160, 1191, 1260, 1384, 1436, 1472, 1556, 1589, 1738, 2849, 1917, 2958, 3057 \ cm^{-1}. \) HRMS calcd. for C_{39}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
$^{31}\text{P-NMR (162 MHz, CD}_2\text{Cl}_2\text{)} 3e$
$^1$H-NMR (400 MHz, CD$_2$Cl$_2$) 4a

![NMR spectrum](image1)

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

![NMR spectrum](image2)
$^{31}$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
$^{31}\text{P-NMR (162 MHz, CD}_2\text{Cl}_2\text{) 4c}$
$^1$H-NMR (400 MHz, CD$_2$Cl$_2$) 4d

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

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

\[ \text{Structure Image} \]

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

\[ \text{NMR Spectrum Image} \]
$^{31}$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
$^{31}$P-NMR (162 MHz, CD$_2$Cl$_2$) 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
$^1$H-NMR (400 MHz, CD$_2$Cl$_2$) 4j

$^{13}$C-NMR (101 MHz, CD$_2$Cl$_2$) 4j
$^{31}$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$_2$) 11

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

$^{31}$P-NMR (162 MHz, CD$_2$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
X-Ray Analyses

**Compound 3e**

![Chemical Structure](image)

- **Empirical formula**: C_{25}H_{22}AuCl_{3}N_{3}P
- **Color**: colourless
- **Formula weight**: 670.72 g · mol^{-1}
- **Temperature**: 150 K
- **Wavelength**: 0.71073 Å
- **Crystal system**: MONOCLINIC
- **Space group**: C_{2}c, (no. 15)
- **Unit cell dimensions**:
  - \(a = 24.4111(6) \ \text{Å}\)
  - \(b = 8.577(2) \ \text{Å}\)
  - \(c = 23.0774(16) \ \text{Å}\)
- **Volume**: 4824.8(12) \ \text{Å}^3
- **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 mm^3
- **\(\theta\) range for data collection**: 3.05 to 33.10°
- **Index ranges**: -37 \leq h \leq 37, -13 \leq k \leq 13, -35 \leq l \leq 35
- **Reflections collected**: 67463
- **Independent reflections**: 9159 [\(R_{int} = 0.0532\)]
- **Reflections with I>2\(\sigma(I)\)**: 7901
- **Completeness to \(\theta = 27.50^\circ\)**: 99.9 %
- **Absorption correction**: Gaussian
- **Max. and min. transmission**: 0.81 and 0.39
- **Refinement method**: Full-matrix least-squares on \(F^2\)
- **Data / restraints / parameters**: 9159 / 0 / 289
- **Goodness-of-fit on \(F^2\)**: 1.086
- **Final R indices \([I>2\(\sigma(I)\)]\)**: \(R_1 = 0.0288\) \(wR^2 = 0.0665\)
- **R indices (all data)**: \(R_1 = 0.0378\) \(wR^2 = 0.0704\)
- **Largest diff. peak and hole**: 1.327 and -2.047 e · Å^{-3}
Empirical formula: $C_{31}H_{31}AuClN_{2}OP$

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, $\alpha = 90^\circ$.
- $b = 10.9240(5)$ Å, $\beta = 111.3890(10)^\circ$.
- $c = 16.4234(7)$ Å, $\gamma = 90^\circ$.

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$^\circ$.

Index ranges:
- $h$: $-18 \leq h \leq 20$.
- $k$: $-12 \leq k \leq 13$.
- $l$: $-19 \leq l \leq 19$.

Reflections collected: 67770

Independent reflections: 4988 ($R_{int} = 0.0481$)

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

Completeness to $\theta = 67.16^\circ$: 99.2 %

Absorption correction: Gaussian

Max. and min. transmission:
- Max. transmission: 0.54295.
- Min. transmission: 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$^{-3}$
Empirical formula: C_{28}H_{24}AuClNOP

Color: colourless

Formula weight: 653.87 g · mol^{-1}

Temperature: 100 K

Wavelength: 0.71073 Å

Crystal system: MONOCLINIC

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

Unit cell dimensions:
- a = 14.7905(9) Å, \( \alpha = 90^\circ \)
- b = 11.9586(10) Å, \( \beta = 115.796(4)^\circ \)
- c = 15.2551(7) Å, \( \gamma = 90^\circ \)

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

\( \theta \) 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 \( I > 2 \sigma (I) \): 9429

Completeness to \( \theta = 37.00^\circ \): 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 [\( I > 2 \sigma (I) \)]:
- \( R_I = 0.0329 \)
- \( wR^2 = 0.0548 \)

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}
**Empirical formula**

C_{31}H_{25}AuCl_4N_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**
P2₁/c (no. 14)

**Unit cell dimensions**
\[a = 8.7003(3) \text{ Å} \]
\[b = 19.0210(12) \text{ Å} \]
\[c = 17.8177(14) \text{ Å} \]
\[\alpha = 90^\circ \]
\[\beta = 90.348(4)^\circ \]
\[\gamma = 90^\circ \]

**Volume**
2948.6(3) Å³

**Z**
4

**Density (calculated)**
1.648 Mg · m⁻³

**Absorption coefficient**
5.248 mm⁻¹

**F(000)**
1428 e

**Crystal size**
0.17 × 0.10 × 0.09 mm³

**θ range for data collection**
3.17 to 34.94°

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

**Reflections collected**
95323

**Independent reflections**
12890 [R(int) = 0.0388]

**Reflections with I>2σ(I)**
11231

**Completeness to θ = 27.50°**
99.8 %

**Absorption correction**
Gaussian

**Max. and min. transmission**
0.66 and 0.49

**Refinement method**
Full-matrix least-squares on F²

**Data / restraints / parameters**
12890 / 0 / 345

**Goodness-of-fit on F²**
1.091

**Final R indices [I>2σ(I)]**
R₁ = 0.0244 \quad wR² = 0.0570

**R indices (all data)**
R₁ = 0.0326 \quad wR² = 0.0605

**Largest diff. peak and hole**
2.411 and -1.792 e · Å⁻³
Compound 6:

**Empirical formula**: C_{28}H_{24}AsAuClNO

**Color**: colourless

**Formula weight**: 697.82 g · mol⁻¹

**Temperature**: 100 K

**Wavelength**: 0.71073 Å

**Crystal system**: MONOCLINIC

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

**Unit cell dimensions**:
- $a = 14.9063(15)$ Å, $\alpha = 90^\circ$.
- $b = 12.0478(12)$ Å, $\beta = 115.743(3)^\circ$.
- $c = 15.3302(3)$ Å, $\gamma = 90^\circ$.

**Volume**: 2479.9(4) Å³

**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 mm³

**θ range for data collection**: 2.67 to 36.00°.

**Index ranges**: $-24 \leq h \leq 24$, $-19 \leq k \leq 19$, $-25 \leq l \leq 25$

**Reflections collected**: 62465

**Independent reflections**: 11702 [$R_{int} = 0.0491$]

**Reflections with I>2σ(I)**: 10436

**Completeness to θ = 27.50°**: 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>2σ(I)]**: $R_1 = 0.0294$, $wR_2 = 0.0693$

**R indices (all data)**: $R_1 = 0.0357$, $wR_2 = 0.0723$

**Largest diff. peak and hole**: 1.897 and −4.624 e · Å⁻³
**Compound 9**

Empirical formula: $\text{C}_{20}\text{H}_{19}\text{Au}\text{Cl}_{3}\text{N}_3\text{O}_3$

Color: yellow

Formula weight: 549.80 g · mol$^{-1}$

Temperature: 100 K

Wavelength: 0.71073 Å

Crystal system: MONOCLINIC

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

Unit cell dimensions:
- $a = 12.3447(8)$ Å
- $b = 11.9732(8)$ Å
- $c = 13.8197(9)$ Å
- $\alpha = 90^\circ$
- $\beta = 110.926(5)^\circ$
- $\gamma = 90^\circ$

Volume: $1907.9(2)$ Å$^3$

$Z$: 4

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 [$R_{int} = 0.0311$]

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

Completeness to $\theta = 34.99^\circ$: 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 [$I>2\sigma(I)$]: $R_F = 0.0157$, $wR^2 = 0.0432$

R indices (all data): $R_F = 0.0192$, $wR^2 = 0.0450$

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

Empirical formula: $\text{C}_{37}\text{H}_{37}\text{AuCl}_{3}\text{NOpRh}$

Color: orange

Formula weight: $948.87 \text{ g} \cdot \text{mol}^{-1}$

Temperature: 100 K

Wavelength: 0.71073 Å

Crystal system: MONOCLINIC

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

Unit cell dimensions:
- $a = 15.2528(12) \text{ Å}$
- $b = 12.7151(19) \text{ Å}$
- $c = 17.885(4) \text{ Å}$

Volume: $3394.7(10) \text{ Å}^3$

Density (calculated): 1.857 $\text{ Mg} \cdot \text{m}^{-3}$

Absorption coefficient: 5.118 mm$^{-1}$

$F(000)$: 1856 e

Crystal size: $0.14 \times 0.12 \times 0.06 \text{ mm}^3$

$\theta$ range for data collection: 2.729 to 33.218°

Index ranges:
- $-23 \leq h \leq 23$
- $-19 \leq k \leq 19$
- $-25 \leq l \leq 27$

Reflections collected: 58509

Independent reflections: 12950 [R$\text{int} = 0.0360$]

Reflections with $I > 2\sigma(I)$: 11849

Completeness to $\theta = 25.242°$: 99.8 %

Absorption correction: Gaussian

Max. and min. transmission: 0.75 and 0.51

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

Data / restraints / parameters: $12950 / 0 / 407$

Goodness-of-fit on $F^2$: 1.096

Final R indices [$I > 2\sigma(I)$]: $R_1 = 0.0294$, $wR^2 = 0.0676$

R indices (all data): $R_1 = 0.0342$, $wR^2 = 0.0698$

Extinction coefficient: n/a

Largest diff. peak and hole: 1.7 and $-3.4 \text{ e} \cdot \text{Å}^{-3}$
Empirical formula: C_{38}H_{38}Cl_{1}N_{1}O_{1}P_{1}Rh_{1}

Color: orange

Formula weight: 694.02 g · mol⁻¹

Temperature: 100 K

Wavelength: 0.71073 Å

Crystal system: MONOCLINIC

Space group: P2₁/c, (no. 14)

Unit cell dimensions:
- a = 10.7983(10) Å, α = 90°
- b = 13.1084(12) Å, β = 100.263(2)°
- c = 23.391(2) Å, γ = 90°

Volume: 3258.0(5) Å³

Z: 4

Density (calculated): 1.415 Mg · m⁻³

Absorption coefficient: 0.686 mm⁻¹

F(000): 1432 e

Crystal size: 0.15 x 0.12 x 0.08 mm³

θ range for data collection: 1.77 to 33.31°

Index ranges:
- h ≤ 16
- -20 ≤ k ≤ 20
- -35 ≤ l ≤ 36

Reflections collected: 107131

Independent reflections: 12543 [Rint = 0.0424]

Reflections with I > 2σ(I): 10641

Completeness to θ = 27.50°: 100.0 %

Absorption correction: Gaussian

Max. and min. transmission: 0.77 and 0.54

Refinement method: Full-matrix least-squares on F²

Data / restraints / parameters: 12543 / 0 / 394

Goodness-of-fit on F²: 1.114

Final R indices [I>2σ(I)]:
- R₁ = 0.0233
- wR² = 0.0596

R indices (all data):
- R₁ = 0.0333
- wR² = 0.0684

Largest diff. peak and hole: 0.535 and -0.498 e · Å⁻³