**Insights in the Mechanism for Gold Catalysis: Behaviour of Gold(I) Amide Complexes in Solution**

Mariusz Bobin, Iain J. Day, Mark Roe, Eddy M. E. Viseux*

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1. X-Ray crystallographic studies

![Crystal structures of 7 and 6b](image)

**Fig. 1** Crystal structures of 7 and 6b with thermal ellipsoids at the 50% probability level. Hydrogen atoms are omitted for clarity.
Diffraction data were collected at 173(2) K on an Enraf-Nonius CAD4 diffractometer using monochromated Mo-Kα radiation (λ = 0.71073 Å). Single crystals were coated in oil and then directly mounted on the diffractometer under a stream of cold nitrogen gas at -100°C. All structures were solved with SHELXS1 and were refined on \( F^2 \) with H atoms in riding mode, using SHELXL97. All solvent molecules were able to be refined anisotropically. Molecular drawings were made with ORTEP-3v2. Crystal data and refinement parameters are shown in Table Y. Crystallographic data for the structures reported in this paper have been deposited with the Crystallographic Data Centre.

### Table 1: Crystal and structure refinement data for 7 and 6b.

<table>
<thead>
<tr>
<th>Compound</th>
<th>7</th>
<th>6b</th>
</tr>
</thead>
<tbody>
<tr>
<td>CCDC</td>
<td>915634</td>
<td>916508</td>
</tr>
<tr>
<td>Formula</td>
<td>( C_{72}H_{72}Au_2F_6N_4O_6P_2S_2 )</td>
<td>( C_{46}H_{54}Au_2F_6N_4O_6P_2S_2 )</td>
</tr>
<tr>
<td>( M )</td>
<td>1723.33</td>
<td>696.46</td>
</tr>
<tr>
<td>Crystal system</td>
<td>Triclinic</td>
<td>Monoclinic</td>
</tr>
<tr>
<td>Space group</td>
<td>( P1 )</td>
<td>( P2_1 )</td>
</tr>
<tr>
<td>( a ) (Å)</td>
<td>10.5355(3)</td>
<td>10.4641(2)</td>
</tr>
<tr>
<td>( b ) (Å)</td>
<td>11.8539(4)</td>
<td>17.3566(5)</td>
</tr>
<tr>
<td>( c ) (Å)</td>
<td>15.7927(5)</td>
<td>14.2244(4)</td>
</tr>
<tr>
<td>( α ) (°)</td>
<td>94.235(2)</td>
<td>90</td>
</tr>
<tr>
<td>( β ) (°)</td>
<td>100.106(1)</td>
<td>90.861(1)</td>
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<tr>
<td>( γ ) (°)</td>
<td>112.490(2)</td>
<td>90</td>
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<tr>
<td>( V ) (Å³)</td>
<td>1772.4(1)</td>
<td>2583.2(1)</td>
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<tr>
<td>( Z )</td>
<td>1</td>
<td>4</td>
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<tr>
<td>Absorption coefficient (mm⁻¹)</td>
<td>4.31</td>
<td>5.89</td>
</tr>
<tr>
<td>Unique reflections, ( R_{int} )</td>
<td>14989, 0.040</td>
<td>11184, 0.073</td>
</tr>
<tr>
<td>Final ( R ) indices</td>
<td>0.031, 0.068</td>
<td>0.051, 0.105</td>
</tr>
<tr>
<td>( I &gt; 2\sigma(I) ) ( R_{int}, wR_2 )</td>
<td>0.031, 0.068</td>
<td>0.051, 0.105</td>
</tr>
<tr>
<td>( R ) indices (all data) ( R_{int}, wR_2 )</td>
<td>0.037, 0.071</td>
<td>0.067, 0.110</td>
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</tbody>
</table>

1 G. M. Sheldrick, *SHELXL-97, Program for refinement of crystal structures*, University of Göttingen, Germany, 1997
**Discussion**

<table>
<thead>
<tr>
<th></th>
<th>7</th>
<th>6b</th>
</tr>
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<tbody>
<tr>
<td><strong>Au-N (Å)</strong></td>
<td>2.091(7)</td>
<td>2.093(9)</td>
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<tr>
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<td>2.094(8)</td>
<td>2.076(9)</td>
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<tr>
<td><strong>Au-P (Å)</strong></td>
<td>2.236(3)</td>
<td>2.238(3)</td>
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<td>2.242(2)</td>
<td>2.232(2)</td>
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<td><strong>P-Au-N (°)</strong></td>
<td>173.9(2)</td>
<td>174.1(4)</td>
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<td>177.4(2)</td>
<td>176.1(4)</td>
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<tr>
<td><strong>Au-Au (Å)</strong></td>
<td>6.613</td>
<td>9.077</td>
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<tr>
<td><strong>Longest dimension (Å)</strong></td>
<td>18.803</td>
<td>20.255</td>
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2. NMR spectra

**Methyl 3-\{(2S)-2-[(diphenylphosphino)methyl]pyrrolidin-1-yl\} propanoate**

![NMR spectra image]
3-[(25)-2-[(Diphenylphosphino)methyl]pyrrolidin-1-yl]propanoic acid
(3-((2S)-2-((Diphenylphosphino)methyl)pyrrolidin-1-ium-1-yl)propanoyl)((trifluoromethyl)sulfonyl)amide (15)
(3-((2S)-2-(((Diphenylphosphino)methyl)pyrrolidin-1-ium-1-yl)propanoyl)((trifluoromethyl)sulfonyl)amide gold chloride (1)
(3-{(2S)-2-((Diphenylphosphino)methyl)pyrrolidin-1-ium-1-yl)propanoyl}((trifluoromethyl)sulfonyl)amide gold bistriflic amide (2)
Bidentate gold(I) complex (7)
(S)-Methyl 5-((diphenylphosphino)methyl)pyrrolidin-1-yl)pentanoate
(S)-5-(2-((Diphenylphosphino)methyl)pyrrolidin-1-yl)pentanoic acid
(5-((2S)-2-((Diphenylphosphino)methyl)pyrroolidin-1-ium-1-yl)pentanoyl)((trifluoromethyl)sulfonyl)amide (16)
Bidentate gold(I) complex (6a/6b)
(S)-Methyl 4-(2-((diphenylphosphino)methyl)pyrrolidin-1-yl)butanoate
(S)-4-(2-((Diphenylphosphino)methyl)pyrrolidin-1-yl)butanoic acid
(4-((2S)-2-((Diphenylphosphino)methyl)pyrrolidin-1-ium-1-yl)butanoyl)((trifluoromethyl)sulfonyl)amide (22)
Bidentate Gold(II) complex (5a/5b)
*N,N*-Dibenzyl-*N*[( trifluoromethyl)sulfonyl]-*D*-tryptophanamide

Triphenylphosphine gold *N,N*-dibenzyl-*N*[( trifluoromethyl)sulfonyl]-*D*-tryptophanamide (4)
\[\text{N}^2-\text{[(Benzyloxy)carbonyl]-N}^1-\text{[(trifluoromethyl)sulfonyl]-(L)-leucinamide}\]
Triphenylphosphine gold N²-[(benzyloxy)carbonyl]-N¹-[(trifluoromethyl)sulfonyl]-
(L)-leucinamide (3)
(S)-2-((Diphenylphosphino)methyl)pyrrolidine-1-sulfonic acid