1,1,1,1-Tetrakis[triorganylphosphineaurio(I)]ethanium(+) Tetrafluoroborates – Hypercoordinated Species Containing [H₃C—C(AuL)₄]+ Cations

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Auriophilicity, Gold(I) Phosphine Complexes, Ethane-tetraaurio(I) Compounds, X-Ray

Hypercoordinated, tetraaurated carbon-complexes of the type [H₃C—C(AuPR₃)_4]+BF₄⁻, R = Ph (1), R = C₆H₅ (2), (PR₃) = 1,2-C₆H₅(CH₂(CH₂PPh₂)₃ (3) have been prepared by the reaction of the appropriate (phosphane)gold(I) chlorides with 1,1,1-tris(dimethylboryl)ethane H,C—C[B(OCH₃)₃]3 in the presence of CsF. The products have been characterized by standard analytical and spectroscopic methods, including single crystal X-ray analyses of 1 and 2. In each case the pentacoordinated carbon atoms have been located at the centre of a square pyramid built up by a methyl group at the apex and four gold atoms forming the base with short Au···Au distances of about 2.85 Å, which strongly contribute to the formation and stability of these species.

Introduction

The special position of gold in the Periodic Table is largely based on relativistic effects [1], which modify significantly the properties of the valence electrons of Post-Lanthanide elements. The relativistic contraction of the 5s orbitals of the heavy elements reaches a pronounced maximum for gold and leads to a break-up of the 5d orbitals of electrons in the Au(I) oxidation state. A wealth of evidence has been obtained from structural and spectroscopic studies of both mononuclear gold(I) compounds for the existence of the resulting metal-metal bonding between Au⁺ centers [2]. Intra- and intermolecular metal-metal interactions give rise not only to the aggregation of complex units L—Au—X of phosphane gold(I) compounds [3], but also to a clustering of gold(I) atoms at or around a main group element like carbon, nitrogen, phosphorus, arsenic, oxygen, sulfur, and selenium [4–10]. Apart from species with a more conventional stoichiometry [(AuL)E]+ (E = O [8], S [9a], Se [10a]), [(AuL)N]+ [5a–c, 7], [(AuL)₂NR]⁺ [5e–g], [(AuL)₂PR]⁺ [6], or C(AuL)₄ [6c], novel cations [(AuL)₂As]+ [7] with a nonclassical square-pyramidal structure and the unprecedented hypercoordinated cations [(AuL)₃Cl]⁺ [4d], [(AuL)₃Cl]⁺ [4c, e–h], [(AuL)₂N]⁺ [5d], and [(AuL)₃P]²⁺ [6a] have been discovered.

While the number of homoleptic species A–D with the interstitial atom in the center of trigonal bipyramids or octahedra of gold atoms has been steadily increasing, only two examples are known...
to date where the coordination sphere around the hypercoordinate central atom contains also a non-gold substituent (E, F): These two dications [RP(AuL)]$_2$$^+$ [6b] (R = o-tolyl) and (dimeric) [RC(AuL)]$_4$$^+$ [4b] (R = oxazolinyl) feature square pyramidal structures with, respectively, phosphorus or carbon at the center, the R substituent at the apex, and the four gold atoms at the base of the polyhedron, with short Au−Au contacts, indicative of the origin of this unusual clustering of gold in both cases.

We now report on simple compounds with five-coordinated carbon atoms in cationic CH$_3$−C[AuPR$_3$]$_4$ units.

**Results**

In preceding work tetrakis(dimethoxyboryl)methane C[B(OMe)$_2$]$_4$ and bis(dimethoxyboryl)methane H$_2$C[B(OMe)$_2$]$_2$ were found to be excellent starting materials for the synthesis of highly aurated homoletic carbon centered clusters [4c, d, e−h]. Thus, the reactions of C[B(OMe)$_2$]$_4$ with (triorganylphosphine)gold(I) chlorides LAuCl in the presence of CsF lead to the formation of hypercoordinated hexakis(phosphineaurio(I))-methanium (2+) dications of the type [(AuL)$_6$C]$^2+$. The degree of auration could be reduced from six to five and four by the use of gold chloride complexes with bulky phosphanes [6c].

When 1,1,1-tris(dimethoxyboryl)ethane was chosen as the precursor, which is readily obtained from the reaction of 1,1,1-trichloromethane and CIB(OMe)$_2$ with lithium powder in tetrahydrofuran, as described by Matteson et al. [11], the analogous reaction led to the title compounds. The gold(I) chloride complexes of PPh$_3$, P(C$_6$H$_5$)$_3$, and 1,2-C$_6$H$_4$(CH$_2$CH$_2$PPh$_2$)$_2$ required, were readily available via published methods [12, 13, 4e].

Treatment of the individual (phosphine)gold(I) chloride with 1,1,1-tris(dimethoxyboryl)ethane in the presence of cesium fluoride in hexamethylphosphoronic triamide (HMPT) produced yellow solutions. After filtration and precipitation with pentane/benzene crystalline complexes [H$_3$C−C(AuPPh$_3$)$_4$]$^+$BF$_4^-$ (1), [H$_3$C−C(AuP(C$_6$H$_5$)$_3$)$_4$]$^+$BF$_4^-$ (2) and [H$_3$C−C((AuPPh$_2$CH$_2$CH$_2$)$_2$C$_6$H$_4$)$_2$]$^+$BF$_4^-$ (3) were obtained.

The products 1, 2 (yellow) and 3 (colourless) are airstable, hygroscopic substances, insoluble in non-polar solvents like pentane, slightly soluble in benzene and readily soluble in dichloromethane or HMPT. The compounds decompose on melting. Characterization is achieved by elemental analysis, mass spectrometry and NMR spectroscopy. The data are summarized in the Experimental Section.

The existence of the hypercoordinated species [CH$_3$−C(AuPR$_3$)$_4$]$^+$ was first established by high-resolution field desorption (FD) mass spectrometry. In the mass spectral studies the parent peaks at $m/z = 1864.7$ (1), 1936.1 (2), and 1821.1 (3) can readily be assigned to the appropriate cations.
Table I. \( ^1{\text{H}} \)\( ^3{\text{P}} \) NMR chemical shifts \( \delta \) [ppm] for compounds \( \text{R}_3 \text{PAuCl} \) and \( \text{[CH}_3\text{C(AuPR}_3)_4]\text{BF}_4 \).

<table>
<thead>
<tr>
<th>PR(_3)</th>
<th>( \text{R}_3 \text{PAuCl} )</th>
<th>( \text{[CH}_3\text{C(AuPR}_3)_4]\text{BF}_4 )</th>
</tr>
</thead>
<tbody>
<tr>
<td>PPh(_3)</td>
<td>33.4</td>
<td>32.1</td>
</tr>
<tr>
<td>P(C(_6)H(_5)H(_3))(_3)</td>
<td>54.9</td>
<td>49.5</td>
</tr>
<tr>
<td>( %\text{(PPh,PCH}_3\text{CH}_3)\text{C}_6\text{H}_4)</td>
<td>29.2</td>
<td>27.5</td>
</tr>
</tbody>
</table>

The \( ^1{\text{H}} \)\( ^3{\text{P}} \)-NMR spectra at room temperature of compounds 1–3 in dichloromethane-\( d_2 \) show one sharp singlet resonance in each case, which is not only indicative of a virtual fourfold symmetry of the cations of 1 and 2, but also demonstrates the equivalence of the ligand halves in the chelated cation of 3. When compared to the data of the corresponding (phosphine)gold(I) chlorides, upfield shifts can be stated (Table I), as already reported for the conversion of (phosphine)gold(I) compounds to homoleptic hypercoordinated species \( \text{[(AuL)}_5\text{C}]^+ \) [4d], \( \text{[(AuL)}_6\text{C}^2 \text{]}^+ \) [4c, e–h]. The equivalence of the LAu units is further established by the number and multiplicity of the signals in the \( ^1{\text{H}} \) NMR spectra. Besides the resonances of the (phosphine) ligands, symmetrical 1:4:6:4:1 quintets could be observed for the methyl hydrogen atoms at \( \delta = 3.55 \) (1), 3.22 (2) and 3.65 ppm (3) due to a \( ^4J_{HP} \) coupling with the phosphorus atoms of the phosphines (Fig. 1). The signals appear in a region downfield from the resonances of \( \beta \)-hydrogen atoms in alkylgold(I) complexes like \( \text{(H}_3\text{C)}_3\text{CAuPPh}_3 \) (\( \delta = 1.97 \) ppm) [14] attesting a deshielding with an increasing degree of auration. The absolute values of the coupling constants \( ^4J_{HP} \) (between 4.3 and 4.7 Hz) are also in good agreement with that found for \( \text{(H}_3\text{C)}_2\text{C-AuPPh}_3 \) (\( ^4J_{HP} = 6.5 \text{ Hz} \)).

In the \( ^1{\text{H}} \)\( ^3{\text{C}} \) NMR spectrum of \( \text{[H}_3\text{C-C(AuPPh}_3)_4]\text{BF}_4 \) the resonances of the phenyl carbon atoms show a pattern also common for homoleptic carbon centered gold cluster cations. The doublet for the ipso C atom is shifted downfield as compared to the corresponding resonance in Ph\(_3\)PAuCl, whereas the other phenyl signals are largely unchanged. The o- and m-carbon atoms give rise to centered doublets caused by long range P–P' coupling. The resonance of the terminal H\(_3\)C group appears as a singlet at \( \delta = 28.2 \) ppm. The signal of the interstitial carbon could not be detected, and partial enrichment with \( ^3{\text{C}} \) will probably be necessary for a direct observation of the nucleus, a procedure already successfully applied for the \( \text{[C(AuL)}_6\text{]}^+ \) species [4f].

**Crystal Structure Determinations**

Because of strong crystal disorder in all samples of 1 so far investigated, only the C–C(Au-P)\(_4\) skeleton could be determined for the cation in \( \text{[H}_3\text{C-C(AuPPh}_3)_4]\text{BF}_4 \), which crystallizes in the triclinic space group \( \text{P}\overline{1} \) with two stoichiometric equivalents in the unit cell excluding any crystallographic symmetry for the individual cation. Nevertheless, by using a split model and refining Au–P moieties at two positions, the coordination geometry at the interstitial carbon atom could be established beyond any doubt. The penta-coordinated cluster C atom is capping a square of gold atoms and is associated with an apical methyl group (Fig. 2). The structure solution is a preliminary result.
The resulting tetragonal-pyramidal environment of the central carbon atom was fully confirmed, however, for the [H_2C - C - C(AuP(C_5H_11)Ph_3)]^{2+} cation in 2, which crystallizes in the triclinic space group PT with two formula units in the unit cell. The carbon-carbon bond distance center-apex corresponds to a standard C-C single bond. The average Au-C-Au angle of 83° and gold-carbon distances between 2.14 and 2.19 Å give rise to short intramolecular Au···Au contacts of 2.816 Å (average). The local geometrical situation at the interstitial cluster atom is thus comparable to that found for the central carbon atom in the [Au(oxazolinyl)(C(AuPPh_3))_3]^{2+} cation [4b]. The increasing sterical demand of the tricyclohexylphosphine ligands in 1 as compared to the PPh_3 substituents in the oxazolinyl complex is reflected in an elongation of the Au-P bonds (average 2.30 Å vs. 2.16 Å) and in larger deviations of the C-Au-P angles (average 169.9° vs. 175.0°) from linearity.

Fractional atomic coordinates and equivalent isotropic displacement parameters for 2 are given in Table II, selected structural parameters in the cation to Fig. 3 [15].

Table II. Fractional atomic coordinates and equivalent isotropic displacement parameters for 2, \( \langle U(\text{eq.}) \rangle = \langle U(1) \cdot U(2) \cdot U(3) \rangle^{1/2} \), where \( U(1) \), \( U(2) \), \( U(3) \) are the eigenvalues of the \( U(\text{ij}) \) matrix. E.s.d.'s in parentheses.

<table>
<thead>
<tr>
<th>Atom</th>
<th>( x/a )</th>
<th>( y/b )</th>
<th>( z/c )</th>
<th>( U(\text{eq.}) )</th>
</tr>
</thead>
<tbody>
<tr>
<td>Au1</td>
<td>0.0221(8)</td>
<td>0.7365(9)</td>
<td>0.2886(3)</td>
<td>0.043</td>
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<tr>
<td>Au2</td>
<td>-0.1628(8)</td>
<td>0.7513(9)</td>
<td>0.2767(5)</td>
<td>0.042</td>
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<tr>
<td>Au3</td>
<td>0.0008(6)</td>
<td>0.9228(9)</td>
<td>0.2832(8)</td>
<td>0.044</td>
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<tr>
<td>Au4</td>
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<td>0.9301(7)</td>
<td>0.2969(8)</td>
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<tr>
<td>P1</td>
<td>0.1352(5)</td>
<td>0.6352(6)</td>
<td>0.2753(5)</td>
<td>0.046</td>
</tr>
<tr>
<td>P2</td>
<td>-0.2551(6)</td>
<td>0.6595(6)</td>
<td>0.2423(6)</td>
<td>0.047</td>
</tr>
<tr>
<td>P3</td>
<td>0.1000(6)</td>
<td>1.0356(6)</td>
<td>0.2499(6)</td>
<td>0.049</td>
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<tr>
<td>P4</td>
<td>-0.2894(6)</td>
<td>1.0388(6)</td>
<td>0.2863(6)</td>
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<tr>
<td>C1</td>
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<td>0.821(2)</td>
<td>0.326(2)</td>
<td>0.035</td>
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<td>C2</td>
<td>-0.082(2)</td>
<td>0.793(2)</td>
<td>0.401(2)</td>
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<tr>
<td>C11</td>
<td>0.113(2)</td>
<td>0.526(2)</td>
<td>0.323(2)</td>
<td>0.048</td>
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<tr>
<td>C112</td>
<td>0.100(3)</td>
<td>0.526(3)</td>
<td>0.405(2)</td>
<td>0.072</td>
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<tr>
<td>C113</td>
<td>0.080(3)</td>
<td>0.427(3)</td>
<td>0.446(2)</td>
<td>0.072</td>
</tr>
<tr>
<td>C114</td>
<td>0.000(4)</td>
<td>0.394(4)</td>
<td>0.417(3)</td>
<td>0.107</td>
</tr>
<tr>
<td>C115</td>
<td>0.016(3)</td>
<td>0.393(3)</td>
<td>0.342(3)</td>
<td>0.097</td>
</tr>
<tr>
<td>C116</td>
<td>0.035(3)</td>
<td>0.491(3)</td>
<td>0.296(2)</td>
<td>0.078</td>
</tr>
<tr>
<td>C121</td>
<td>0.156(3)</td>
<td>0.625(3)</td>
<td>0.186(2)</td>
<td>0.068</td>
</tr>
<tr>
<td>C122</td>
<td>0.224(3)</td>
<td>0.540(3)</td>
<td>0.173(3)</td>
<td>0.100</td>
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<tr>
<td>C123</td>
<td>0.219(5)</td>
<td>0.541(5)</td>
<td>0.093(4)</td>
<td>0.146</td>
</tr>
<tr>
<td>C124</td>
<td>0.251(4)</td>
<td>0.626(4)</td>
<td>0.047(3)</td>
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<tr>
<td>C125</td>
<td>0.185(4)</td>
<td>0.700(4)</td>
<td>0.060(3)</td>
<td>0.123</td>
</tr>
<tr>
<td>C126</td>
<td>0.187(3)</td>
<td>0.711(4)</td>
<td>0.142(3)</td>
<td>0.103</td>
</tr>
</tbody>
</table>
Fig. 3. Molecular structure of the \( [\text{H}_3\text{C}-\text{C}(\text{AuP(C}_6\text{H}_{11})_3)]^+ \) cation of 2 (SCHAKAL, only the \( \alpha \)-carbon atoms of the cyclohexyl groups are shown).

Selected bond distances [Å] and angles [°]:

- \( \text{Au}_1 - \text{C}1 \): 2.17(3), \( \text{Au}_2 - \text{C}1 \): 2.14(3), \( \text{Au}_3 - \text{C}1 \): 2.14(3), \( \text{Au}_4 - \text{C}1 \): 2.19(3), \( \text{Au}_1 - \text{P}1 \): 2.30(1), \( \text{Au}_2 - \text{P}2 \): 2.31(1), \( \text{Au}_3 - \text{P}3 \): 2.28(1), \( \text{Au}_4 - \text{P}4 \): 2.30(1), \( \text{Au}_1 - \text{Au}_2 \): 2.849(2), \( \text{Au}_1 - \text{Au}_3 \): 2.865(2), \( \text{Au}_4 - \text{Au}_2 \): 2.883(2), \( \text{Au}_4 - \text{Au}_3 \): 2.846(2), \( \text{C}_1 - \text{C}2 \): 1.44(5), \( \text{Au}_1 - \text{Au}_2 \): 2.83(1), \( \text{Au}_1 - \text{C}1 \): 3.83(1), \( \text{Au}_4 - \text{C}1 \): 3.83(1), \( \text{Au}_2 - \text{P}2 \): 2.31(1), \( \text{Au}_3 - \text{P}3 \): 2.17(3), \( \text{Au}_2 - \text{C}1 \): 2.14(3), \( \text{Au}_3 - \text{C}1 \): 2.14(3).

Conclusions

The present study has shown that partially dimethoxyboryl substituted alkanes can be used as starting materials for the syntheses of polyaurio(I)alkanes in much the same way like tetraakis(dimethoxyboryl)methane for the synthesis of auration methanes [4c, e–h]. The reaction does not come to a halt at the triaurated \( [\text{H}_3\text{C}-\text{C}(\text{AuPR}_3)_3]^+ \) stage, but proceeds to novel penta-coordinated cationic species \( [\text{H}_3\text{C}-\text{C}(\text{AuPR}_3)_4]^+ \). This result again demonstrates the tendency of polyaurio-carbon aggregates to accept further \( \text{LAu}^+ \) units. In this context it is quite remarkable that for carbon-centered clusters precursors like \( \text{C}_2\text{AuL}_4 \) or \( \text{H}_3\text{C}-\text{C}(\text{AuL})_3 \) are transient species for smaller ligands L, whereas in the case of the ammonium and phosphonium analogues \( [\text{N}(\text{AuL})_3]^+ \) [5d] and \( [\text{RP}(\text{AuL})_3]^+ \) [6b] the auration can be carried out in well defined steps [16].

According to X-ray analyses the monocations \( [\text{H}_3\text{C}-\text{C}(\text{AuPR}_3)_4]^+ \) feature a square-pyramidal geometry with an interstitial carbon, four gold atoms at the base and the methyl group at the apex. The short \( \text{Au} \cdots \text{Au} \) contacts of ca. 2.8 Å along the edges of a distorted square reflect bonding interactions between neighbouring gold atoms, which are important for the stabilization of the system. Neglecting the \( \text{Au} \cdots \text{Au} \) bonding, the electron-deficient nature of the hypercoordinated pentagonal-pyramidal framework with eight valence electrons for five covalent bonds can be illustrated by a simplified molecular orbital diagram under idealized \( \text{C}_4 \) symmetry. Symmetry allowed linear combinations of the three donor electron pairs of the (formal) methylcarbide trianion \( \text{H}_3\text{C}-\text{C}^- \) and the \( \sigma \) acceptor orbitals (sp hybrids) of the four \( \text{LAu}^+ \) units lead to three bonding states, which are filled by the six electrons available to give a diamagnetic ground state. Therefore the bond order for each of the \( \text{Au} - \text{C} \) bonds is 3/4.

The synthesis of the novel compounds shows the way to the preparation of other polyaurio(I) alkane species starting from materials like \( \text{R}_3\text{C}[\text{B(OMe)}_2]_3 \). Preliminary results have indicated, however, that the auration sometimes is accompanied by dealkylation of the boryl ester, which parallels the dealkylation of alkylamines observed in analogous auration reactions. Further work will be necessary to mark scope and limitations of the hyperauration process.

Experimental Part

All experiments were carried out in an atmosphere of dry and purified nitrogen. Solvents and glassware were dried and saturated/filled with nitrogen. – NMR: [D]_4-dichloromethane as solvent, tetramethylsilane and phosphoric acid as reference compounds, measurement temperature 25 °C, Jeol JNM GX 270 and GX 400 spectrometers. – MS: Varian MAT 311 (FD). – 1,1,1-Tris(dimethoxyboryl)ethane was prepared as described [11]. \( \text{Ph}_3\text{PAuCl}, (\text{C}_6\text{H}_{11})_2\text{PAuCl} \) and \( \text{C}_6\text{H}_4(\text{CH}_2\text{CH}_2\text{PPH}_3\text{AuCl})_2 \) were synthesized following literature procedures [12, 13, 4e]. All other reagents were obtained commercially and used without further purification.

\[1,1,1,1\text{-Tetrakis(triphenylphosphineaurio(I))}-\text{ethanum}(+)\text{ tetrafluoroborate(I)}\]

(Triphenylphosphine)gold(I) chloride (1.00 g, 2.0 mmol) and CsF (3.42 g, 22.6 mmol) in HMPT (30 ml) were treated with a solution of \( \text{H}_3\text{C}-\text{C}^- \) \( \text{B}([\text{OMe})_2]_3 \) (0.17 g, 0.70 mmol) in the same solvent (5 ml) for 24 h at 25 °C under nitrogen.
The yellow solution was filtered and the product precipitated with pentane. Repeated extraction with CH₂Cl₂ and reprecipitation with pentane/benzene afforded yellow crystals of 1: yield 0.41 g (42%); m.p. 154 °C (decomp.). - ¹H NMR (CD₂Cl₂): δ = 3.55 (quin, J_HP = 4.9 Hz, 3H, H3-C), 7.12 (m, 24H, m-C₆H₅), 7.31–7.36 (m, 36H, o-/p-C₆H₅). - (¹H)¹³P NMR (CD₂Cl₂): δ = 32.1 (s). - (¹H)¹¹C NMR (CD₂Cl₂): δ = 28.2 (s, C(CH₃)), 131.5 (d, J_CP = 51.8 Hz, C-1), 134.4 (d, C(CH₃)), 16.669 (s), N = 13.0 Hz, C-2). - ¹H NMR (CD₂Cl₂): δ = 3.22 (quin, J_HP = 4.3 Hz, 3H, H3-C), 1.15–2.00 (m, 132H, C), 2.66–2.70 (m, 132H, C). - MS (FD, CH₂Cl₂): m/z (%) = 1936.1 (100), M⁺ (cation).

Analysis for C₇₄H₆₃Au₄BF₄P₄ (1906.88)
Calcd C 44.93 H 3.40%.
Found C 44.93 H 3.40%.

Crystal structure determination of compound 1, [H₂C–C(AuPPh₃)₄]/BF₄

Enraf-Nomius CAD4 diffractometer, Mo-Kα radiation, λ = 0.71069 Å, graphite monochromator, T = −27 °C.

Crystal data: C₇₄H₆₃Au₄BF₄P₄, M = 1950.89, triclinic, space group P₁, a = 15.167(2), b = 16.669(3), c = 19.119(3) Å, α = 112.62(2), β = 96.46(1), γ = 90.61(1), V = 4452.8 Å³, Z = 2, Dcalcd. = 1.464 g cm⁻³, μ(Mo–Kα) = 66.99 cm⁻¹, F(000) = 1848, 13800 reflections, 13800 unique, and 6234 “observed” with Fo ≥ 4σ(Fo), data corrected for Lorentz polarization and adsorption effects (empirically, T = 0.73–1.00). The structure was solved by direct methods (SHELXS 86 [16]), completed by difference Fourier syntheses, and refined by full-matrix, least-squares techniques (SHELX 76 [17]). Au, P were refined anisotropically, phenyl-C, B, F, H were neglected.

Analysis for C₇₄H₆₃Au₄BF₄P₄ (1906.88)
Calcd C 44.93 H 3.40%.
Found C 43.62 H 3.42%.
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[15] Supplementary material on the X-ray structure determination may be obtained from the Fachinformationszentrum Karlsruhe, Gesellschaft für wissenschaftlich-technische Information mbH, D-W-7514 Eggenstein-Leopoldshafen 2, on quoting the depository number CSD 56807, the names of the authors, and the journal citation.