

Bis(triphenylphosphine)gold(I) Perrhenate

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Dedicated to Professor Bernd Krebs on the occasion of his 75th birthday

Bis(triphenylphosphine)gold(I) perrhenate $[\text{Ph}_3\text{PAuPPh}_3]^+\text{ReO}_4^-$ has been prepared in high yield from Ph_3PAuCl , Ph_3P and AgReO_4 in a mixed solvent. The compound is stable in air and decomposes at 235 °C. In the crystal structure, the two independent perrhenate anions are not approaching the gold centers of the two independent cations, but weak interionic interactions are entertained *via* π - π stacking of phenyl groups and C–H···O contacts. As three-blade chiral rotors, the Ph_3P ligands of the cations are in a staggered conformation at the gold atoms with only slightly bent P–Au–P axes. IR and NMR data show no anomalies and are close to those of alkali or onium perrhenates.

Key words: Gold(I), Rhenate(VII), Crystal Structure, IR Spectra, Phosphine

Introduction

The non-coordinated tetrahedral uninegative perrhenate anion ReO_4^- [1–4] is a component of many stable *ionic solids* where it appears surrounded by a large variety of metal or onium cations [5–18]. In *solutions* of these salts in protic or other polar solvents the perrhenate anion is solvated by establishing hydrogen bonds or ion-dipole interactions, respectively. Owing to similar structural and charge characteristics, there are many analogies not only with the corresponding permanganate MnO_4^- and pertechnetate TcO_4^- , but also with the perchlorate ClO_4^- or perbromate BrO_4^- anion. The similarities are so pronounced that *e. g.* the structures of CsReO_4 and CsBrO_4 are isotypical [19]. An increase of the coordination number of the rhenium(VII) center beyond 4 is observed only with strong nucleophiles, and also in the condensation of perrhenic acid HReO_4 and its hydrates $\text{Re}_2\text{O}_7(\text{H}_2\text{O})$ and $\text{Re}_2\text{O}_7(\text{H}_2\text{O})_4$ [20] to give the anhydride Re_2O_7 with alternating tetrahedral and octahedral coordination of the Re(VII) atoms [1–4]. Both perchloric or permanganic and pertechnetetic acid give dinuclear anhydrides with retention of the coordination number 4 of the Cl(VII), Mn(VII) and Tc(VII) atoms, respectively.

Covalently bonded perrhenates are rare and in most cases of limited stability [1–4]. This is particularly true for simple perrhenic acid *esters* R–O–ReO_3 , in analogy to the (explosive) esters of perchloric and perbromic acid on the one hand, and of permanganic and pertechnetetic acid on the other. An exceptional stability has been found, however, for silyl esters $\text{R}_3\text{SiOREO}_3$ which can be prepared by simple metathesis of AgReO_4 with chlorosilanes R_3SiCl or from siloxanes $\text{R}_3\text{SiOSiR}_3$ and Re_2O_7 [21]. The crystal structure of the trimethylsilyl compound ($\text{R} = \text{Me}$) has been determined [22] and shown to have a conventional ester structure with the Si atom attached specifically to one of the oxygen atoms in an angular array. This covalent bonding has also been confirmed by $^{185,187}\text{Re}$ NQR spectroscopy studies which showed a high asymmetry parameter η for the Re atom, while for alkali perrhenates with largely undistorted ReO_4^- tetrahedra the η value deviates only slightly from zero [23]. Similar characteristics have later been found for the corresponding germanium [24] and tin compounds [25, 26], the latter being aggregated through an increase in the coordination number of the tin atoms and with the ReO_4 units functioning as bridging units employing two of their four oxygen atoms. This work was complemented by diorganoindium and

other organometallic perrhenates, but structural details have not become available for most cases [27, 28].

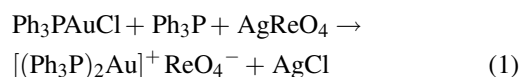
These compounds are examples for the *coordination chemistry of the perrhenate anion* (with ReO_4^- as a *ligand*), which is generally not very well developed [3]. A survey of the various types of coordination compounds with perrhenate as a ligand shows that there is a paucity of examples in particular for coinage metal complexes in general, and for gold complexes in particular. This situation called for some exploratory experiments, because the low coordination number of gold(I) may offer a largely unique type of bonding (L)Au–O– ReO_3 reminiscent of the bonding in esters of perrhenic acid R–O– ReO_3 (R = alkyl, aryl, silyl *etc.*). This type of bonding may also activate the perrhenate anion for stoichiometric or catalytic reactions [29–31].

Surprisingly, a literature search for gold perrhenates has afforded information only for compounds of gold(III) [32, 33], but no example for gold(I). The two examples with gold(III) have the metal cations chelated by two ethylenediamine (en) ligands in $[\text{Au}(\text{en})_2]^{3+}$, or by one diethylenetriamine (dien) in $[\text{Au}(\text{dien})\text{Cl}]^{2+}$, and the perrhenate anions have no coordinative contacts with the metal ion.

In the present report we describe some attempts to prepare gold(I) perrhenate complexes with tertiary phosphines as auxiliary ligands: $\text{R}_3\text{PAu–O–ReO}_3$. Strictly speaking, the experiments were not successful, but nevertheless provided us with a gold(I) perrhenate of the type $[\text{Au}(\text{PR}_3)_2]^+ \text{ReO}_4^-$ with R = Ph.

Results and Discussion

Attempts to prepare $\text{Ph}_3\text{PAuOREO}_3$ by metathesis of Ph_3PAuCl and AgReO_4 or NH_4ReO_4 in a 1 : 1 molar ratio in various solvents and at temperatures between 20 and 60 °C were not successful. There was either no reaction, or slow reaction led to partial decomposition and formation of the impure 2 : 1 complex. Therefore, the stoichiometry was changed by adding an extra equivalent of Ph_3P :



The mixing of a solution of Ph_3PAuCl and Ph_3P in dichloromethane with a solution of AgReO_4 in acetonitrile gave a colorless precipitate of AgCl which

was separated by filtration. Addition of diethyl ether to the concentrated filtrate gave a colorless precipitate consisting of the desired product in almost quantitative yield. Careful layering with diethyl ether allowed the growth of single crystals. The air-stable compound melts with decomposition at 235.4 °C (DSC). Elemental analysis has confirmed the proposed composition.

Solutions in CDCl_3 show a singlet resonance at $\delta = 44.8$ ppm in the $\{^1\text{H}\}^{31}\text{P}$ NMR spectrum at 25 °C, and the usual complex sets of ^1H and ^{13}C resonances of the phenyl groups are observed in the ^1H and $\{^1\text{H}\}^{13}\text{C}$ NMR spectra. The complexity of the latter is due to $A_n\text{XX}'A'_n$ and BXX' spin systems, respectively (A = H, B = C, X = P), already observed and assigned also in several previous studies and not duplicated here [34]. The chemical shifts and coupling constants indicate that there is no interference of the anions which would distort the cations and affect these parameters.

The IR spectrum of a powdered sample exhibits the pattern of bands characteristic of Ph_3P groups attached to gold(I) atoms. The prominent band of the anion (ν_3 , F_2 , for T_d symmetry) [14] appears at 900.5 cm^{-1} , which is close to reference data for anionic ReO_4^- in the literature [35, 36]. This band is known to be rather insensitive to the size and charge of innocent counterions like the alkali or alkaline earth cations ($\text{Li}^+ - \text{Cs}^+$, Ca^{2+}) for which its wavenumber falls in the narrow range of $895 - 920\text{ cm}^{-1}$. The second IR-active band of the ReO_4^- anion (ν_4 , F_2) is known to be located near 330 cm^{-1} . This region of the IR spectrum of the title compound is populated by other bands making assignments ambiguous [14].

A comparison of the solid state IR data of the title compound with those of known metal perrhenates M^+ReO_4^- shows that the compound has structure and bonding characteristics in between the extremes of $\text{Cs}^+\text{ReO}_4^-$ with large and thus innocent Cs^+ cations on the one hand and perrhenates with small and more strongly polarizing cations (Li^+ , Ag^+), or with cations with hydrogen bonding capabilities (NH_4^+). The IR spectra of the two modifications of the former (α -, β - CsReO_4) show the ν_3 band at 911 and 913 cm^{-1} , almost independent of temperature in the range from 300–470 K [10]. As an example for the latter, AgReO_4 shows its intense ν_3 band at 859 cm^{-1} indicating strong cation-anion interactions. A similar value may be anticipated for ligand-free Au^+ReO_4 , but this compound has not yet been prepared. By con-

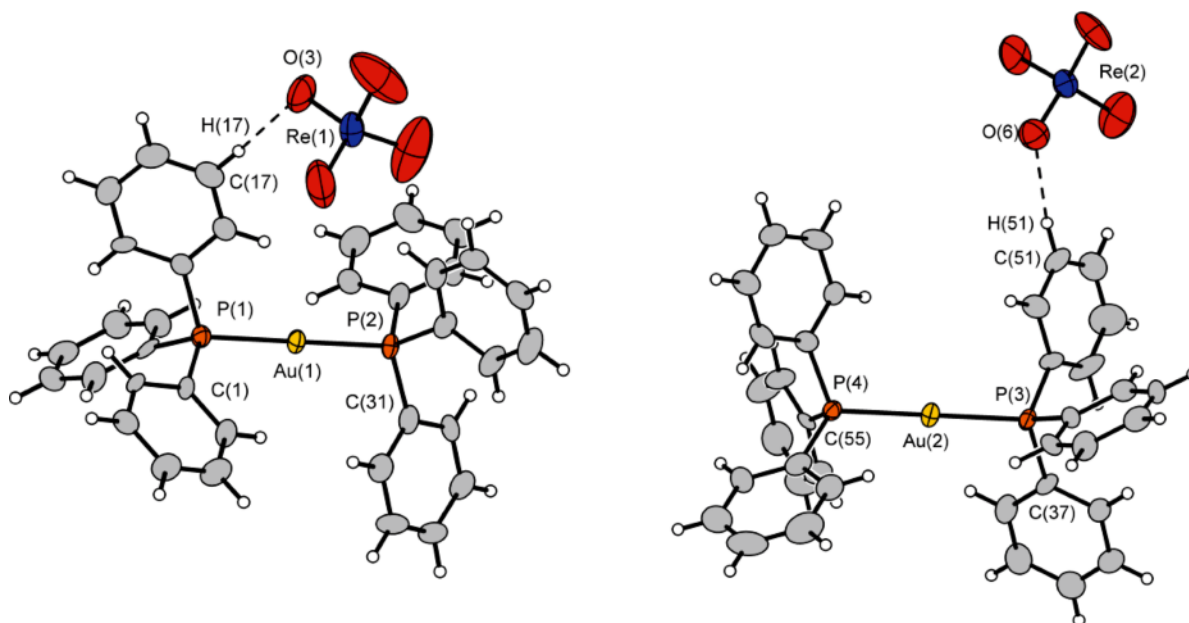


Fig. 1 (color online). The two independent $[\text{Ph}_3\text{PAuPPh}_3]^+$ and ReO_4^- units of the title compound. Displacement ellipsoids at the 70% probability level (at 123 K), H atoms isotropic. Two selected C-H \cdots O contacts are shown with dashed lines.

trast, $[\text{Au}(\text{en})_2](\text{ReO}_4)_2\text{Cl}$ with its ligand-covered trication [32, 33] has this band again at 903 cm^{-1} , the small shift relative to CsReO_4 (910 cm^{-1}) being probably due to hydrogen bonding $\text{O}\cdots\text{H}-\text{N}$ to the NH_2 groups of the en ligand of the cation. In ionic liquids with various imidazolium cations having only C-H \cdots O hydrogen bonding capabilities, the ν_3 band of the perrhenate anion is also found in the range $902-913\text{ cm}^{-1}$ [27, 28].

Crystals of the title compound are triclinic, space group $P\bar{1}$, with two independent cations and anions in the asymmetric unit (Fig. 1). In the unit cell shown in Fig. 2, equivalent units are related by centers of inversion. One of the two ReO_4^- anions has an only slightly distorted tetrahedral structure with Re(2)–O distances and O–Re(2)–O angles in the narrow ranges of $1.710(7)-1.716(7)\text{ \AA}$ and $108.7(4)-111.0(4)^\circ$, respectively. The broader range of the Re(1)–O distances and O–Re(1)–O angles for the second anion of $1.687(9)-1.737(9)\text{ \AA}$ and $104.4(5)-115.1(5)^\circ$, respectively, suggest a more significant distortion, but may also indicate a less satisfactory refinement of the atom positions. In the discussion of the ReO_4^- geometry, therefore, only the data of the Re(2)-centered tetrahedron are considered (Table 1).

Regarding the dimensions of the perrhenate anions given in Table 1, a comparison with published data for simple reference compounds gives the following details:

For LiReO_4 , the Re–O distances are in the range $1.69-1.78\text{ \AA}$ (average 1.716 \AA for three different anions) [7]. This value increases to $1.728(2)$ for NaReO_4 [8], $1.719(5) / 1.723(4) // 1.737(2)\text{ \AA}$ for KReO_4 (two by X-ray, and one by neutron diffraction) [9], and $1.722(6)\text{ \AA}$ for RbReO_4 [12, 13]. For the low-temperature phase $\beta\text{-CsReO}_4$ the average distance is $1.714(6)\text{ \AA}$, but for the high-temperature phase $\alpha\text{-ReO}_4$ (at 468 K) by far the lowest value has been detected: $1.683(7)\text{ \AA}$ [14]. The Re–O distance in $\text{NH}_4^+\text{ReO}_4^-$ is also temperature-dependent with values varying roughly between 1.72 and 1.74 \AA [17], probably owing to the flexibility of the extensive hydrogen bonding $\text{O}\cdots\text{H}-\text{N}$ in these crystals. As expected, the Re–O distances are longest in AgReO_4 , for which a value of $1.732(2)\text{ \AA}$ has been determined [18]. This distance indicated a strong polarizing influence of the silver cations which was already obvious from the IR characteristics.

In summary, this comparison suggests that the structures of the anions in the title compound are affected

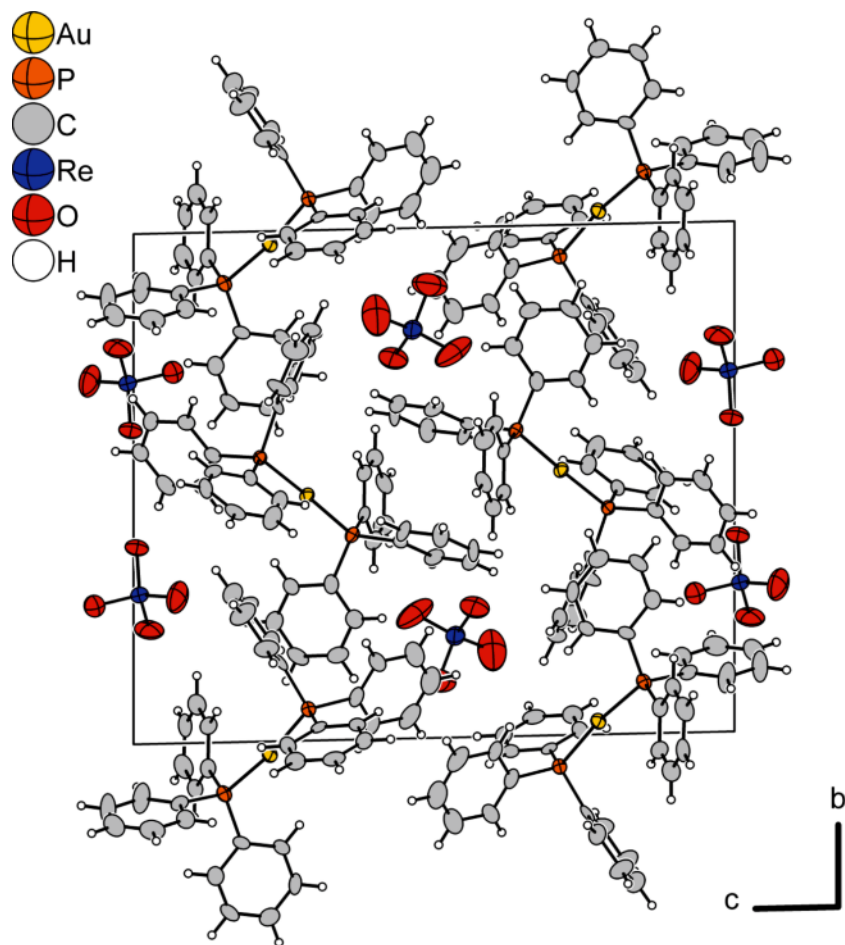


Fig. 2 (color online). Unit cell of the structure of the title compound viewed along the crystallographic *a* axis. Displacement ellipsoids are shown at the 70% probability level (at 123 K), H atoms as spheres with arbitrary radius.

very little by the $[\text{Au}(\text{PPh}_3)_2]^+$ cations. This result has been supported by an investigation of the structural details of these cations:

The two cations have almost linear axes P–Au–P with angles of $178.1(1)$ [Au(1)] and $172.58(7)^\circ$ [Au(2)]. The two Ph_3P ligands at each gold atoms are in a staggered conformation with dihedral angles close to 60° : $\text{C}(1)\text{--P}(1)\text{--P}(2)\text{--C}(31)$ 56.0° , $\text{C}(37)\text{--P}(3)\text{--P}(4)\text{--C}(61)$ 65.9° . Each Ph_3P ligand has its three phenyl groups in a three-bladed chiral propeller or rotor conformation [37]. These propellers appear in the crystal as both enantiomers (left- and right-handed), related by centers of inversion between pairs of equivalent cations. These centers of inversion are located between phenyl rings of neighboring cations. The distance between the planes of the phenyl rings is 3.62 \AA and therefore indicative of $\pi\text{--}\pi$ stacking effects

(center-to-center distance 4.29 \AA , center-to-corner distance 3.90 \AA ; Fig. 3). The general structural pattern of the cations in the title compound are very similar to those of the analogous nitrate and hexafluorophosphate showing that the anion has no marked influence on the geometry of the cations [38]. The result shows that the ReO_4^- anion also is a poor donor for gold(I). By contrast, with the halide anions Cl^- , Br^- and I^- compounds with tricoordinate gold atoms are obtained with strongly bent P–Au–P axes [39].

As listed in Table 2, there are $\text{O}\cdots\text{H}\cdots\text{C}$ contacts between anions and cations in which almost all oxygen atoms are involved [except O(7)]. The range of distances $\text{O}\cdots\text{H}$ from $2.40\text{--}2.57 \text{ \AA}$ and of angles $\text{O}\cdots\text{H}\cdots\text{C}$ from $123\text{--}167^\circ$ suggests only weak individual interactions, but in the sense of a “Gulliver effect” these contacts may in concert contribute significantly to the

Table 1. Selected interatomic distances (Å) and bond angles (deg) in the crystal structure of the title compound (with standard deviations in parentheses).

Atoms	Length (Å), Angle (deg)	Atoms	Length (Å), Angle (deg)
Au(1)–P(1)	2.311(2)	Au(1)–P(2)	2.311(2)
Au(2)–P(3)	2.312(2)	Au(2)–P(4)	2.308(2)
Re(1)–O(1)	1.687(9)	Re(1)–O(2)	1.737(9)
Re(1)–O(3)	1.712(7)	Re(1)–O(4)	1.693(7)
Re(2)–O(5)	1.714(6)	Re(2)–O(6)	1.713(6)
Re(2)–O(7)	1.710(7)	Re(2)–O(8)	1.716(7)
P(1)–C(1)	1.810(8)	P(1)–C(7)	1.813(9)
P(1)–C(13)	1.812(9)	P(2)–C(19)	1.826(8)
P(2)–C(25)	1.796(8)	P(2)–C(31)	1.810(9)
P(3)–C(37)	1.799(8)	P(3)–C(43)	1.802(8)
P(3)–C(49)	1.807(8)	P(4)–C(55)	1.814(8)
P(4)–C(61)	1.816(9)	P(4)–C(67)	1.810(8)
P(1)–Au(1)–P(2)	178.1(1)	P(3)–Au(2)–P(4)	172.6(1)
Au(1)–P(1)–C(1)	113.6(3)	Au(1)–P(1)–C(7)	111.4(3)
Au(1)–P(1)–C(13)	113.2(3)	Au(1)–P(2)–C(19)	111.0(3)
Au(1)–P(2)–C(25)	111.5(3)	Au(1)–P(2)–C(31)	108.1(4)
Au(2)–P(3)–C(37)	110.6(3)	Au(2)–P(3)–C(43)	116.8(3)
Au(2)–P(3)–C(49)	110.3(3)	Au(2)–P(4)–C(55)	108.8(3)
Au(2)–P(4)–C(61)	114.0(3)	Au(2)–P(4)–C(67)	111.8(3)

stability of the arrangement of cations and anions in the crystal.

Summary

The first gold(I) perrhenate has been prepared. The synthesis was successful only with two equivalents of a tertiary phosphine as auxiliary ligands. In the crystals of $[\text{Au}(\text{PPh}_3)_2]^+ \text{ReO}_4^-$ the perrhenate anions have no significant contact with the gold atoms of the cation. The array of the components is only supported by intercationic π - π stacking of phenyl groups and by weak C–H \cdots O contacts of phenyl hydrogen atoms with perrhenate oxygen atoms. The cations and anions have the structure and conformation known from salts with other anions and cations, respectively, as also demonstrated by IR and NMR investigations.

Experimental Part

Reagents: PPh_3 , $(\text{Ph}_3\text{P})\text{AuCl}$ and AgReO_4 are commercially available.

Instruments: NMR: Bruker AMX 400 spectrometer. 85% aqueous H_3PO_4 as reference for ^{31}P . IR: Varian 670 FT-IR spectrometer with PIKE Gladi ATR cell. DSC: Q2000 V24.4, TA Instruments. Crystal structure determination: Bruker Kappa APEX II CCD system, graphite monochromator, $\lambda = 0.71073 \text{ \AA}$.

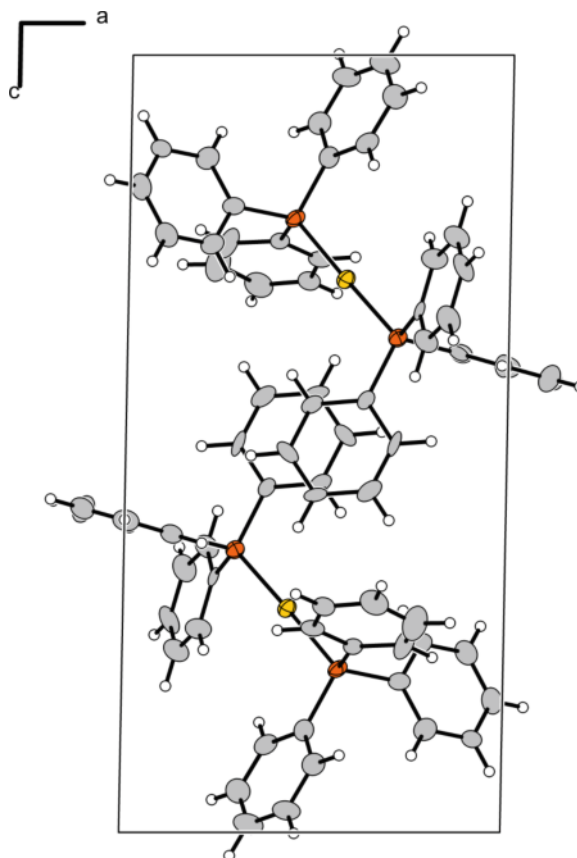


Fig. 3 (color online). A view along the crystallographic b axis of the structure of the title compound showing the π - π stacking. Displacement ellipsoids are shown at the 70% probability level (at 123 K), H atoms as spheres with arbitrary radius.

Preparation of $[\text{Au}(\text{PPh}_3)_2]\text{ReO}_4$

Ph_3PAuCl (52.2 mg, 0.105 mmol) and Ph_3P (27.9 mg, 0.106 mmol) are dissolved in dichloromethane (2.5 mL) at room temperature under an atmosphere of dry nitrogen. AgReO_4 (37.9 mg, 0.106 mmol) is dissolved in dry acetonitrile (3.5 mL), and the solution is added dropwise with stirring under nitrogen to the first solution. A colorless suspension is formed which is further stirred overnight and filtered. The filtrate is concentrated in a vacuum until a colorless precipitate occurs, which is separated by filtration and dried in a vacuum; yield 95 mg (92%); m. p. 235.4 °C (DSC). Single crystals can be grown by layering the native solution (5 mL of CH_2Cl_2 , CH_3CN) with diethyl ether (2 mL) and cooling in the refrigerator to 5 °C. The compound is soluble in dichloromethane, chloroform and acetonitrile. Elemental analysis, calculated / found (%): C 44.49 / 46.07, H 3.11 /

Table 2. Interionic C–H...O contacts in the crystal structure of the title compound. A complete atomic numbering scheme has been deposited (CCDC 952298). The C–H distance has been set to 0.95 Å (standard deviations in parentheses).

	H...O (Å)	O...Re (Å)	O–H...Re (deg)
C(2)–H(2)...O(3)	2.56	3.302(11)	135
C(9)–H(9)...O(8)	2.47	3.306(10)	147
C(15)–H(15)...O(4)	2.49	3.124(12)	124
C(17)–H(17)...O(3)	2.46	3.394(11)	167
C(33)–H(33)...O(5)	2.43	3.184(10)	137
C(48)–H(48)...O(1)	2.57	3.247(11)	128
C(51)–H(51)...O(6)	2.40	3.334(12)	166
C(69)–H(69)...O(2)	2.54	3.161(12)	123

3.44, Au 20.27 / 20.01, Re 19.16 / 18.34. – $\{^1\text{H}\}^{31}\text{P}$ NMR (CDCl₃, 25 °C): δ 44.8 ppm. – Selected IR bands (solid): 1479.1 (w), 1434 (m), 1100 (m), 900.5 (vs), 747.8 (m), 713 (s), 521 (s), 504 (vs), 543 (m) cm⁻¹.

Crystal structure determination

Data were collected on a single crystal X-ray diffractometer equipped with a CCD detector (BrukerAPEX II, κ -CCD), a fine-focus sealed tube with MoK α radiation ($\lambda = 0.71073$ Å), and a graphite monochromator by using the APEX 2 software package [40]. The measurements were performed on a single crystal coated with perfluorinated ether (Fomblin 1800). The crystal was fixed on the top of a glass capillary and transferred to the diffractometer. The crystal was frozen under a stream of cold nitrogen to 123 K. A matrix scan was used to determine the initial lattice parameters. As all crystals appeared to be twinned, the two components were identified using CELL_NOW [41] (180° rotated, twin law: $-1\ 0\ 0, 0\ -1\ 0, 0\ 0\ 1$) and integrated and treated together. Reflections were merged and corrected for Lorentz and polarization effects, scan speed, and background using SAINT [42]. Absorption corrections, including odd and even ordered spherical harmonics were performed using TWINABS [42]. The space group assignment was based upon systematic absences, E statistics, and successful refinement of the structures. The structure was solved by Direct Methods with the aid of successive difference Fourier maps [40], and were refined against all data using SHELXL-97 [43] in conjunction with SHELXLE [44]. Non-hydrogen atoms were refined with anisotropic displacement parameters. Hydrogen

Table 3. Crystal and structure refinement data for [Au(PPh₃)₂]⁺ReO₄⁻.

	Title compound
Formula	C ₇₂ H ₆₀ Au ₂ O ₈ P ₄ Re ₂
Color	colorless
Habitus	fragment
Size/mm ³	0.07 · 0.12 · 0.17
M_r	1943.41
Crystal system	triclinic
Space group	$P\bar{1}$
a , Å	9.7629(3)
b , Å	17.0026(5)
c , Å	19.8745(5)
α , deg	91.117(1)
β , deg	90.957(1)
γ , deg	96.255(2)
V , Å ³	3278.2(2)
Z	2
$\rho_{\text{calcd.}}$, g·cm ⁻³	1.97
λ , Å	0.71073
T , /K	123
μ (MoK α), mm ⁻¹	8.3
R_{int}	0.072
$R(F)$ [$I > 2\ \sigma(I)$, all data]	0.035, 0.064
$wR(F^2)$ [$I > 2\ \sigma(I)$, all data]	0.055, 0.070
S (all data)	1.04
Data / parameters	11 817 / 794
$\Delta\rho_{\text{max.}}$, $\Delta\rho_{\text{min.}}$, e·Å ⁻³	1.35, -1.11

atoms were placed in ideal positions using the SHELXL riding model. Full-matrix least-squares refinements were carried out by minimizing $\Sigma w(F_o^2 - F_c^2)^2$ with SHELXL-97 [43] weighting scheme. Neutral atom scattering factors for all atoms and anomalous dispersion corrections for the non-hydrogen atoms were taken from *International Tables for Crystallography* [45]. Details are presented in Table 3.

CCDC 952298 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

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