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Redetermination of tetraphenylarsonium *trans*-aquatetrachlorooxorhenate(V) at 100 K

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Key indicators

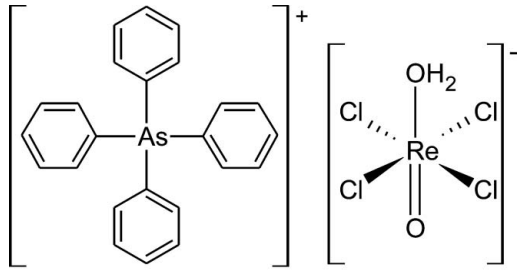
Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.002 Å
Disorder in main residue
R factor = 0.018
wR factor = 0.039
Data-to-parameter ratio = 35.1

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The crystal structure of the title compound, $(C_{24}H_{20}As)[ReCl_4O(H_2O)]$, has been re-examined at 100 K. The complex contains distorted octahedral $[ReOCl_4(H_2O)]^-$ anions, with the Re atom and disordered aqua ligand located on a fourfold axis, and $[Ph_4As]^+$ cations with the As atom located on a fourfold inversion axis. In the anion, the terminal Re=O bond, 1.662 (2) Å, is *trans* to the aqua ligand and the Re—(OH_2) and Re—Cl distances are 2.325 (3) and 2.3479 (5) Å, respectively.

Comment

Initial studies of the structure of $[Ph_4As][ReOCl_4(H_2O)]$, (I), were made by Lis & Jeżowska-Trzebiatowska (1977) and reinvestigated by Müller (1984). Müller proved, using the original data of Lis & Jeżowska-Trzebiatowska (1977), that this complex contains a water molecule. We now report an independent re-investigation of the title complex, (I), using data measured at 100 K.



(I)

The title complex (Fig. 1 and Table 1) comprises an $[ReOCl_4(OH_2)]^-$ anion and a $[Ph_4As]^+$ cation. In the anion, the Re, oxo O and water O atoms lie on a common fourfold rotation axis of symmetry, while in the cation the As atom is located on a fourfold inversion centre. In this model, the water H atoms take two positions, indicating disorder. The water molecule is not involved in any hydrogen bonds, an observation confirmed by its IR spectrum which shows a single sharp band at 933 cm^{-1}.

The Re atom has a distorted octahedral geometry with four Cl atoms in equatorial positions and with the water molecule and the oxo O atom occupying the axial positions. The Re atom deviates from the equatorial plane towards the oxo O atom by 0.415 (1) Å. The distortion from a regular octahedral geometry can also be noted in the value of the Cl—Re—O1 angle, *viz.* 100.18 (1)°. The Re—O1 bond distance is comparable with values observed in other oxorhenium complexes (Lis, 1979; Sergienko *et al.*, 1991).

The geometry of the $[Ph_4As]^+$ cation shows no significant difference compared to other compounds containing this species (Linden & James, 2002; Dean *et al.*, 2003).

Experimental

To obtain the title complex, the original procedure described by Lis & Jeżowska-Trzebiatowska (1977) was followed.

Crystal data

$(C_{24}H_{20}As)[ReCl_4O(H_2O)]$	Mo $K\alpha$ radiation
M_r = 745.34	Cell parameters from 20865 reflections
Tetragonal, $P4/n$	θ = 3.2–37.3°
a = 13.021 (2) Å	μ = 6.75 mm^{-1}
c = 7.236 (2) Å	T = 100 (2) K
V = 1226.8 (4) Å^3	Block, yellow
Z = 2	0.15 × 0.15 × 0.15 mm
D_x = 2.018 Mg m^{-3}	

Data collection

Kuma KM-4-CCD κ-geometry diffractometer	2671 independent reflections
ω scans	2535 reflections with $I > 2\sigma(I)$
Absorption correction: numerical (*CrysAlis RED*; Oxford Diffraction, 2003)	R_{int} = 0.043
T_{min} = 0.562, T_{max} = 0.686	θ_{max} = 35.0°
19131 measured reflections	$h = -20 \rightarrow 21$
	$k = -21 \rightarrow 21$
	$l = -9 \rightarrow 11$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0156P)^2 + 0.8991P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.018	$(\Delta/\sigma)_{max}$ = 0.002
$wR(F^2)$ = 0.039	$\Delta\rho_{max}$ = 1.52 e Å^{-3}
S = 1.06	$\Delta\rho_{min}$ = −0.98 e Å^{-3}
2671 reflections	
76 parameters	
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

Re—O1	1.662 (2)	Re—Cl	2.3479 (5)
Re—O2	2.325 (3)	As—C1	1.907 (2)
O1—Re—Cl	100.18 (1)	C1^{iv}—As—C1	106.31 (9)
Cl—Re—Cl^{i}	88.21 (1)	C1^{v}—As—C1	111.08 (5)

Symmetry codes: (i) $y, \frac{1}{2}-x, z$; (iv) $\frac{1}{2}-x, \frac{3}{2}-y, z$; (v) $-\frac{1}{2}+y, 1-x, -z$.

All H atoms were found in a difference map, but during the final refinement they were treated in a riding-model approximation, with C—H = 0.95 Å and O—H = 0.84 Å, and U_{iso}(H) = 1.2U_{eq}(C) and 1.5U_{eq}(O). The occupation factor for the water H atom was fixed at 0.5 as it is disordered over four symmetry-equivalent positions. The highest peak in the final difference map was 2.03 Å from atom C2.

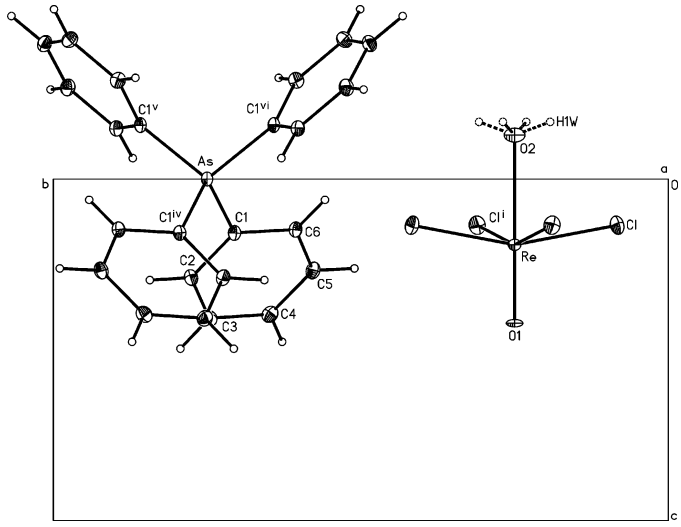


Figure 1
A view of the title complex, showing the atom-numbering scheme and disorder of the aqua ligand. Displacement ellipsoids are drawn at the 30% probability level. [Symmetry codes: (i) $y, \frac{1}{2}-x, z$; (iv) $\frac{1}{2}-x, \frac{3}{2}-y, z$; (v) $-\frac{1}{2}+y, 1-x, -z$; (vi) $1-y, \frac{1}{2}+x, -z$.]

Data collection: *CrysAlis CCD* (Oxford Diffraction 2003); cell refinement: *CrysAlis RED* (Oxford Diffraction 2003); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXL97*.

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