Triethylammonium bis(oxalato)-oxido(triphenylphosphane)rhenate(V)

Philipp Grimminger and Peter Klüfers*

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The anionic part of the title compound, (C_{15}H_{16}N)\[Re(C_2O_4)2O(C_{18}H_{15}P)], is a substituted derivative of trans-trichloridoxoixidobis(triphenylphosphane)rhenium(V) with oxalate. In the structure of the anion, an oxidorhenium(V) unit defines a molecular axis in a distorted octahedral coordination about the central atom. The second axial position is occupied by an O atom of one of the chelating oxalate ligands. The distances of the oxalate O atoms to the central Re atom vary from 2.003 (3) to 2.092 (3) Å, the longest bond being the one trans to the oxide ligand. The anions and cations are connected by a bifurcated hydrogen bond from a triethylammonium NH donor to two oxalate O-atom acceptors.

Related literature

The title compound was synthesized by analogy with a published procedure (Kettler et al., 1994). For the crystal structure of a related compound with oxalate as the ligand, see: Chiozzone et al. (2001).

Experimental

Crystal data

(C_{15}H_{16}N)[Re(C_2O_4)2O(C_{18}H_{15}P)]

Orthorhombic, Pbc a

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Refinement

R[F^2 > 2σ(F^2)] = 0.033
wR(F^2) = 0.079
S = 1.02
6646 reflections

Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan
(SADABS; Sheldrick, 2001)

Refinement

R[F^2 > 2σ(F^2)] = 0.033
wR(F^2) = 0.079
S = 1.02
6646 reflections

Table 1

Hydrogen-bond geometry (Å, °).

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Data collection: COLLECT (Nonius, 2004); cell refinement: SCALEPACK (Otwinowski & Minor 1997); data reduction: SCALEPACK (Otwinowski & Minor 1997) and SCALEPACK; program(s) used to solve structure: SIR97 (Altomare et al., 1999); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEPIII (Burnett & Johnson, 1996) and SCHAKAL99 (Keller, 1999); software used to prepare material for publication: SHELXL97.

The authors thank Dr Peter Mayer for professional support and Richard Betz for helpful discussions.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: EZ2113).

References

supplementary materials
Triethylammonium bis(oxalato)oxido(triphenylphosphane)rhenate(V)

P. Grimminger and P. Klüfers

Comment

The title compound (I) was prepared as a precursor in a study on hydrolytically stable rhenium(V) compounds.

Fig. 1 shows the anion with its two chelating oxalato ligands. The anion's charge is counterbalanced by a triethylammonium cation. In the crystal structure, hydrogen bonds are formed between non-Re-bonded O32 and O42 atoms of one of the oxalato ligands and the protonated triethylamine (Fig. 2).

Experimental

The title compound was prepared in analogy to a published procedure (Kettler et al., 1994). 0.833 g (1.0 mmol) of the rhenium(V)-oxo-complex trans-ReOCl3(PPh3)2 was stirred with 0.360 g (4.0 mmol) oxalic acid and 0.405 g (4.0 mmol) triethylamine in 100 ml methanol for 3 h at 60 °C. Then the volume was reduced in vacuo to 20 ml. Violet crystals suitable for X-ray analysis were obtained by slow evaporation of a solution of the compound in methanol at room temperature.

Refinement

All H atoms were located in a difference map and refined as riding on their parent atoms. One common isotropic displacement parameter for all H atoms was refined.

Figures

Fig. 1. The structure of ion pairs in (I), with atom labels and anisotropic displacement ellipsoids (drawn at 50% probability level) for non-H atoms. Hydrogen bonds are indicated by dotted lines.

Fig. 2. The packing of (I), viewed along [1 0 0].
Triethylammonium bis(oxalato)oxido(triphenylphosphane)rhenate(V)

Crystal data

\( (\text{C}_6\text{H}_{16}\text{N})[\text{Re(C}_2\text{O}_4)_2\text{O}(\text{C}_{18}\text{H}_{15}\text{P})]\)  
\( M_r = 742.71 \)

Orthorhombic, \( Pbc\alpha \)

Hall symbol: -P 2ac 2ab

\( a = 12.4146 (2) \text{ Å} \)
\( b = 15.3531 (2) \text{ Å} \)
\( c = 30.3448 (5) \text{ Å} \)

\( V = 5783.80 (15) \text{ Å}^3 \)
\( Z = 8 \)

Data collection

Nonius KappaCCD diffractometer

Radiation source: rotating anode

Monochromator: MONTEL, graded multilayered X-ray optics

\( T = 200(2) \text{ K} \)

CCD; rotation images; thick slices scans

Absorption correction: multi-scan

\( T_{\text{min}} = 0.631, T_{\text{max}} = 0.772 \)

60925 measured reflections

6646 independent reflections

Refinement

Refinement on \( F^2 \)

Least-squares matrix: full

\( R(F^2 > 2\sigma(F^2)) = 0.033 \)

\( wR(F^2) = 0.079 \)

\( S = 1.02 \)

6646 reflections

365 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

\( H\text{-atom parameters constrained} \)

\( w = 1/[\sigma^2(F_o^2) + (0.0284P)^2 + 5.1452P] \)

where \( P = (F_o^2 + 2F_c^2)/3 \)

\( (\Delta\sigma)_{\text{max}} = 0.001 \)

\( \Delta\rho_{\text{max}} = 1.08 \text{ e Å}^{-3} \)

\( \Delta\rho_{\text{min}} = -0.57 \text{ e Å}^{-3} \)

Extinction correction: none

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

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supplementary materials

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C15  0.067 (4)  0.050 (3)  0.047 (3)  0.021 (3)  0.010 (3)  −0.008 (3)
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C17  0.032 (3)  0.028 (2)  0.025 (3)  −0.006 (2)  −0.002 (2)  −0.005 (2)
C18  0.035 (3)  0.035 (2)  0.032 (3)  −0.007 (2)  −0.003 (2)  0.001 (2)
C19  0.031 (3)  0.055 (3)  0.037 (3)  −0.008 (2)  0.003 (3)  −0.011 (3)
C20  0.048 (4)  0.052 (3)  0.037 (3)  −0.025 (3)  0.011 (3)  −0.011 (3)
C21  0.058 (4)  0.029 (3)  0.042 (3)  −0.014 (3)  0.009 (3)  −0.005 (2)
C22  0.040 (3)  0.030 (2)  0.031 (3)  −0.006 (2)  0.009 (2)  −0.008 (2)
N    0.030 (2)  0.047 (2)  0.030 (2)  −0.0107 (19)  0.0010 (18)  0.0067 (19)
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C26  0.063 (4)  0.062 (4)  0.046 (3)  −0.010 (3)  0.010 (3)  −0.014 (3)
C27  0.040 (3)  0.071 (4)  0.041 (3)  −0.008 (3)  −0.001 (3)  0.019 (3)
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Geometric parameters (Å, °)

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Re—O11 2.003 (3)  C15—C16  1.391 (6)
Re—O21 2.030 (3)  C15—H15  0.9500
Re—O31 2.046 (3)  C16—H16  0.9500
Re—O41 2.092 (3)  C17—C18  1.390 (6)
Re—P  2.4626 (11) C17—C22  1.402 (6)
C1—O12  1.187 (5)  C18—C19  1.387 (6)
C1—O11  1.337 (5)  C18—H18  0.9500
C1—C2  1.554 (6)  C19—C20  1.390 (7)
C2—O22  1.191 (5)  C19—H19  0.9500
C2—O21  1.312 (5)  C20—C21  1.360 (7)
C3—O32  1.212 (5)  C20—H20  0.9500
C3—O31  1.310 (5)  C21—C22  1.386 (6)
C3—C4  1.536 (6)  C21—H21  0.9500
C4—O42  1.214 (5)  C22—H22  0.9500
C4—O41  1.312 (5)  N—C27  1.498 (6)
P—C5  1.818 (4)  N—C23  1.511 (5)
P—C17  1.819 (4)  N—C25  1.514 (6)
P—C11  1.824 (4)  N—H71  0.9300
C5—C10  1.385 (6)  C23—C24  1.506 (7)
C5—C6  1.397 (6)  C23—H231  0.9900
C6—C7  1.370 (6)  C23—H232  0.9900
C6—H6  0.9500  C24—H241  0.9800
C7—C8  1.374 (8)  C24—H242  0.9800
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C8—C9  1.378 (7)  C25—C26  1.505 (6)
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supplementary materials

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supplementary materials

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Hydrogen-bond geometry (Å, °)

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Fig. 1
supplementary materials

Fig. 2