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

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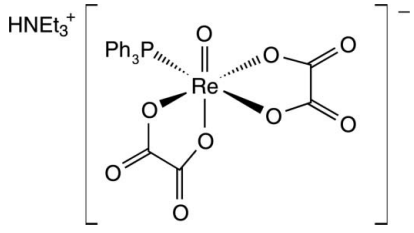
Key indicators: single-crystal X-ray study; T = 200 K; mean σ(C–C) = 0.007 Å; R factor = 0.033; wR factor = 0.079; data-to-parameter ratio = 18.2.

The anionic part of the title compound, $(C_6H_{16}N)[Re(C_2O_4)_2O(C_{18}H_{15}P)]$, is a substituted derivative of *trans*-trichloridooxidobis(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

$(C_6H_{16}N)[Re(C_2O_4)_2O(C_{18}H_{15}P)]$
M_r = 742.71
Orthorhombic, *Pbca*
a = 12.4146 (2) Å
b = 15.3531 (2) Å
c = 30.3448 (5) Å
V = 5783.80 (15) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 4.31 mm^{-1}
T = 200 (2) K
0.14 × 0.10 × 0.06 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)
T_{min} = 0.631, T_{max} = 0.772
60925 measured reflections
6646 independent reflections
3895 reflections with $I > 2\sigma(I)$
R_{int} = 0.086

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.079
S = 1.02
6646 reflections
365 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.08 e Å^{-3}
$\Delta\rho_{min}$ = −0.57 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N—H71···O42	0.93	2.25	3.022 (5)	140
N—H71···O32	0.93	2.29	3.029 (5)	136

Data collection: *COLLECT* (Nonius, 2004); cell refinement: *SCALEPACK* (Otwinowski & Minor 1997); data reduction: *DENZO* (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*.

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

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supplementary materials

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

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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 me thanol 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

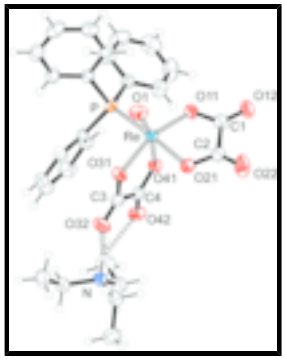

Fig. 1. The structure of ion pairs in (I), with atom labels and anisotropic displacement ellipsoids (drawn at 50% probability level) for non-H atoms. Hydrogen bonds are indicated by dotted lines.

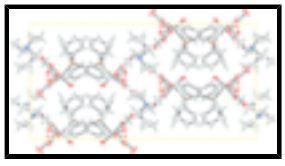

Fig. 2. The packing of (I), viewed along [1 0 0].

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

Crystal data

$(C_6H_{16}N)[Re(C_2O_4)_2O(C_{18}H_{15}P)]$	F_{000} = 2944
M_r = 742.71	D_x = 1.706 Mg m^{-3}
Orthorhombic, *Pbca*	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ac 2ab	Cell parameters from 7243 reflections
a = 12.4146 (2) Å	θ = 3.1–27.5º
b = 15.3531 (2) Å	μ = 4.31 mm^{-1}
c = 30.3448 (5) Å	*T* = 200 (2) K
V = 5783.80 (15) Å^3	Block, blue
Z = 8	0.14 × 0.10 × 0.06 mm

Data collection

Nonius KappaCCD diffractometer	6646 independent reflections
Radiation source: rotating anode	3895 reflections with $I > 2\sigma(I)$
Monochromator: MONTEL, graded multilayered X-ray optics	R_{int} = 0.086
T = 200(2) K	θ_{max} = 27.7º
CCD; rotation images; thick slices scans	θ_{min} = 3.2º
Absorption correction: multi-scan (SADABS; Sheldrick, 2001)	*h* = −16→16
T_{min} = 0.631, T_{max} = 0.772	*k* = −19→19
60925 measured reflections	*l* = −36→39

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.033	H-atom parameters constrained
$wR(F^2)$ = 0.079	$w = 1/[\sigma^2(F_o^2) + (0.0284P)^2 + 5.1452P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.02	$(\Delta/\sigma)_{max}$ = 0.001
6646 reflections	$\Delta\rho_{max}$ = 1.08 e Å^{-3}
365 parameters	$\Delta\rho_{min}$ = −0.57 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}

Re	0.498114 (15)	0.461665 (10)	0.363732 (5)	0.02656 (7)
O1	0.4955 (2)	0.53210 (17)	0.32118 (11)	0.0399 (8)
C1	0.6980 (4)	0.4910 (3)	0.40682 (16)	0.0349 (11)
C2	0.6141 (4)	0.5489 (3)	0.43108 (16)	0.0380 (12)
C3	0.2921 (4)	0.4200 (3)	0.40579 (15)	0.0334 (11)
C4	0.3717 (4)	0.3524 (3)	0.42411 (14)	0.0297 (11)
O11	0.6545 (2)	0.44017 (19)	0.37577 (10)	0.0357 (8)
O12	0.7913 (3)	0.4916 (2)	0.41544 (11)	0.0461 (9)
O21	0.5143 (2)	0.53762 (18)	0.41811 (10)	0.0352 (8)
O22	0.6417 (3)	0.5995 (3)	0.45864 (14)	0.0735 (13)
O31	0.3363 (2)	0.46982 (18)	0.37576 (10)	0.0312 (7)
O32	0.1993 (3)	0.4237 (2)	0.41803 (11)	0.0458 (9)
O41	0.4699 (2)	0.36089 (18)	0.40868 (9)	0.0284 (7)
O42	0.3414 (3)	0.29743 (19)	0.45003 (10)	0.0388 (8)
P	0.48011 (9)	0.33914 (7)	0.31188 (4)	0.0255 (3)
C5	0.4700 (3)	0.3804 (3)	0.25589 (14)	0.0270 (10)
C6	0.5531 (4)	0.4345 (3)	0.24132 (15)	0.0322 (11)
H6	0.6125	0.4461	0.2601	0.050 (3)*
C7	0.5497 (4)	0.4712 (3)	0.20018 (16)	0.0412 (13)
H7	0.6075	0.5071	0.1906	0.050 (3)*
C8	0.4639 (5)	0.4566 (3)	0.17257 (17)	0.0481 (15)
H8	0.4608	0.4841	0.1446	0.050 (3)*
C9	0.3824 (4)	0.4016 (3)	0.18586 (16)	0.0445 (13)
H9	0.3240	0.3897	0.1665	0.050 (3)*
C10	0.3850 (4)	0.3633 (3)	0.22762 (15)	0.0355 (12)
H10	0.3285	0.3256	0.2366	0.050 (3)*
C11	0.5896 (3)	0.2601 (3)	0.30916 (14)	0.0280 (10)
C12	0.6593 (4)	0.2481 (3)	0.34468 (17)	0.0414 (12)
H12	0.6523	0.2828	0.3704	0.050 (3)*
C13	0.7386 (5)	0.1852 (3)	0.34204 (19)	0.0552 (16)
H13	0.7860	0.1768	0.3662	0.050 (3)*
C14	0.7498 (5)	0.1348 (4)	0.3050 (2)	0.0595 (16)
H14	0.8054	0.0924	0.3036	0.050 (3)*
C15	0.6815 (5)	0.1454 (3)	0.27037 (18)	0.0546 (16)
H15	0.6888	0.1098	0.2449	0.050 (3)*
C16	0.6009 (4)	0.2083 (3)	0.27213 (15)	0.0417 (13)
H16	0.5535	0.2157	0.2478	0.050 (3)*
C17	0.3611 (4)	0.2749 (3)	0.32478 (14)	0.0284 (10)
C18	0.2593 (4)	0.3121 (3)	0.32160 (14)	0.0339 (11)
H18	0.2511	0.3684	0.3091	0.050 (3)*
C19	0.1697 (4)	0.2671 (3)	0.33670 (15)	0.0408 (13)
H19	0.0998	0.2913	0.3335	0.050 (3)*
C20	0.1833 (4)	0.1863 (3)	0.35658 (16)	0.0459 (14)
H20	0.1222	0.1561	0.3677	0.050 (3)*
C21	0.2827 (5)	0.1498 (3)	0.36032 (16)	0.0430 (13)
H21	0.2904	0.0945	0.3740	0.050 (3)*
C22	0.3729 (4)	0.1927 (3)	0.34439 (14)	0.0335 (11)
H22	0.4421	0.1667	0.3467	0.050 (3)*
N	0.1147 (3)	0.2970 (2)	0.48630 (12)	0.0359 (10)

H71	0.1640	0.3087	0.4640	0.050 (3)*
C23	0.0146 (4)	0.2623 (3)	0.46403 (16)	0.0394 (12)
H231	0.0337	0.2092	0.4473	0.050 (3)*
H232	−0.0383	0.2456	0.4869	0.050 (3)*
C24	−0.0374 (4)	0.3265 (4)	0.43311 (17)	0.0542 (15)
H241	0.0162	0.3475	0.4119	0.050 (3)*
H242	−0.0658	0.3758	0.4500	0.050 (3)*
H243	−0.0964	0.2979	0.4173	0.050 (3)*
C25	0.0978 (4)	0.3817 (3)	0.51086 (16)	0.0431 (13)
H251	0.1672	0.3996	0.5242	0.050 (3)*
H252	0.0769	0.4272	0.4894	0.050 (3)*
C26	0.0139 (4)	0.3782 (4)	0.54661 (18)	0.0572 (16)
H261	0.0372	0.3377	0.5697	0.050 (3)*
H262	−0.0546	0.3582	0.5341	0.050 (3)*
H263	0.0045	0.4364	0.5593	0.050 (3)*
C27	0.1657 (4)	0.2295 (4)	0.51527 (17)	0.0508 (15)
H271	0.1162	0.2167	0.5400	0.050 (3)*
H272	0.2326	0.2540	0.5279	0.050 (3)*
C28	0.1924 (5)	0.1457 (3)	0.49223 (18)	0.0585 (16)
H281	0.2344	0.1582	0.4656	0.050 (3)*
H282	0.1257	0.1157	0.4841	0.050 (3)*
H283	0.2347	0.1085	0.5120	0.050 (3)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Re	0.02523 (10)	0.02382 (9)	0.03063 (11)	−0.00210 (9)	0.00355 (10)	−0.00176 (7)
O1	0.049 (2)	0.0263 (15)	0.0449 (19)	−0.0042 (16)	0.0111 (18)	−0.0025 (14)
C1	0.035 (3)	0.030 (3)	0.039 (3)	0.002 (2)	0.000 (3)	−0.002 (2)
C2	0.035 (3)	0.039 (3)	0.040 (3)	−0.002 (2)	0.001 (2)	−0.009 (2)
C3	0.037 (3)	0.032 (3)	0.031 (3)	−0.002 (2)	−0.001 (2)	−0.005 (2)
C4	0.033 (3)	0.030 (2)	0.026 (3)	−0.003 (2)	−0.001 (2)	−0.003 (2)
O11	0.0246 (18)	0.0397 (19)	0.043 (2)	−0.0030 (14)	0.0013 (15)	−0.0151 (16)
O12	0.029 (2)	0.055 (2)	0.054 (2)	0.0027 (17)	−0.0083 (18)	−0.0160 (18)
O21	0.033 (2)	0.0357 (17)	0.0370 (18)	0.0009 (15)	0.0018 (15)	−0.0135 (14)
O22	0.047 (3)	0.082 (3)	0.092 (3)	0.010 (2)	−0.016 (2)	−0.063 (3)
O31	0.0251 (18)	0.0293 (17)	0.0393 (19)	0.0015 (14)	0.0011 (15)	0.0048 (14)
O32	0.028 (2)	0.057 (2)	0.052 (2)	0.0026 (17)	0.0079 (17)	0.0040 (18)
O41	0.0280 (19)	0.0294 (16)	0.0279 (16)	−0.0018 (12)	0.0003 (13)	0.0022 (13)
O42	0.047 (2)	0.0371 (19)	0.0319 (19)	−0.0014 (16)	0.0084 (16)	0.0059 (15)
P	0.0278 (7)	0.0235 (5)	0.0252 (6)	−0.0016 (5)	0.0003 (5)	0.0018 (5)
C5	0.030 (3)	0.026 (2)	0.025 (2)	0.0035 (18)	−0.0028 (19)	0.001 (2)
C6	0.034 (3)	0.034 (3)	0.028 (3)	0.000 (2)	0.001 (2)	0.003 (2)
C7	0.047 (3)	0.038 (3)	0.039 (3)	−0.001 (2)	0.007 (3)	0.009 (2)
C8	0.071 (4)	0.046 (3)	0.027 (3)	0.013 (3)	−0.003 (3)	0.010 (2)
C9	0.045 (3)	0.051 (3)	0.037 (3)	0.009 (3)	−0.012 (3)	0.001 (3)
C10	0.037 (3)	0.037 (3)	0.032 (3)	−0.003 (2)	−0.004 (2)	0.006 (2)
C11	0.029 (3)	0.027 (2)	0.028 (3)	−0.0005 (19)	0.000 (2)	0.005 (2)

C12	0.050 (3)	0.032 (3)	0.042 (3)	0.003 (3)	−0.007 (3)	0.005 (2)
C13	0.058 (4)	0.043 (3)	0.065 (4)	0.009 (3)	−0.028 (3)	0.007 (3)
C14	0.049 (4)	0.051 (3)	0.079 (4)	0.025 (3)	0.000 (3)	0.013 (3)
C15	0.067 (4)	0.050 (3)	0.047 (3)	0.021 (3)	0.010 (3)	−0.008 (3)
C16	0.050 (4)	0.046 (3)	0.030 (3)	0.009 (3)	−0.005 (2)	0.000 (2)
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 (2)	−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)
C23	0.035 (3)	0.049 (3)	0.034 (3)	−0.006 (2)	0.001 (2)	−0.001 (2)
C24	0.039 (3)	0.075 (4)	0.048 (3)	−0.007 (3)	−0.009 (3)	0.006 (3)
C25	0.041 (3)	0.051 (3)	0.038 (3)	−0.016 (2)	0.002 (2)	−0.004 (3)
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)
C28	0.055 (4)	0.053 (4)	0.067 (4)	−0.002 (3)	−0.005 (3)	0.019 (3)

Geometric parameters (Å, °)

Re—O1	1.685 (3)	C14—H14	0.9500
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
C7—H7	0.9500	C24—H243	0.9800
C8—C9	1.378 (7)	C25—C26	1.505 (6)
C8—H8	0.9500	C25—H251	0.9900
C9—C10	1.397 (6)	C25—H252	0.9900

C9—H9	0.9500	C26—H261	0.9800
C10—H10	0.9500	C26—H262	0.9800
C11—C16	1.384 (6)	C26—H263	0.9800
C11—C12	1.394 (6)	C27—C28	1.502 (7)
C12—C13	1.381 (7)	C27—H271	0.9900
C12—H12	0.9500	C27—H272	0.9900
C13—C14	1.371 (7)	C28—H281	0.9800
C13—H13	0.9500	C28—H282	0.9800
C14—C15	1.361 (7)	C28—H283	0.9800
O1—Re—O11	105.32 (14)	C14—C15—C16	120.1 (5)
O1—Re—O21	104.84 (14)	C14—C15—H15	120.0
O11—Re—O21	81.42 (12)	C16—C15—H15	120.0
O1—Re—O31	94.50 (13)	C11—C16—C15	120.2 (5)
O11—Re—O31	158.36 (13)	C11—C16—H16	119.9
O21—Re—O31	85.22 (12)	C15—C16—H16	119.9
O1—Re—O41	166.22 (13)	C18—C17—C22	119.6 (4)
O11—Re—O41	85.51 (12)	C18—C17—P	120.0 (3)
O21—Re—O41	84.92 (12)	C22—C17—P	119.6 (4)
O31—Re—O41	76.37 (11)	C19—C18—C17	120.1 (4)
O1—Re—P	89.93 (10)	C19—C18—H18	120.0
O11—Re—P	94.51 (9)	C17—C18—H18	120.0
O21—Re—P	165.22 (9)	C18—C19—C20	119.4 (5)
O31—Re—P	94.11 (9)	C18—C19—H19	120.3
O41—Re—P	80.59 (8)	C20—C19—H19	120.3
O12—C1—O11	123.6 (4)	C21—C20—C19	120.9 (5)
O12—C1—C2	123.0 (4)	C21—C20—H20	119.6
O11—C1—C2	113.4 (4)	C19—C20—H20	119.6
O22—C2—O21	124.6 (5)	C20—C21—C22	120.5 (5)
O22—C2—C1	120.9 (5)	C20—C21—H21	119.7
O21—C2—C1	114.5 (4)	C22—C21—H21	119.7
O32—C3—O31	125.7 (4)	C21—C22—C17	119.4 (5)
O32—C3—C4	122.1 (4)	C21—C22—H22	120.3
O31—C3—C4	112.1 (4)	C17—C22—H22	120.3
O42—C4—O41	126.0 (4)	C27—N—C23	111.5 (4)
O42—C4—C3	120.4 (4)	C27—N—C25	111.3 (4)
O41—C4—C3	113.7 (4)	C23—N—C25	114.1 (4)
C1—O11—Re	115.1 (3)	C27—N—H71	106.5
C2—O21—Re	114.4 (3)	C23—N—H71	106.5
C3—O31—Re	120.0 (3)	C25—N—H71	106.5
C4—O41—Re	117.5 (3)	C24—C23—N	113.6 (4)
C5—P—C17	109.5 (2)	C24—C23—H231	108.8
C5—P—C11	103.95 (19)	N—C23—H231	108.8
C17—P—C11	104.7 (2)	C24—C23—H232	108.8
C5—P—Re	109.73 (14)	N—C23—H232	108.8
C17—P—Re	110.51 (14)	H231—C23—H232	107.7
C11—P—Re	117.99 (15)	C23—C24—H241	109.5
C10—C5—C6	118.6 (4)	C23—C24—H242	109.5
C10—C5—P	124.5 (3)	H241—C24—H242	109.5
C6—C5—P	116.9 (3)	C23—C24—H243	109.5

C7—C6—C5	120.7 (5)	H241—C24—H243	109.5
C7—C6—H6	119.6	H242—C24—H243	109.5
C5—C6—H6	119.6	C26—C25—N	114.9 (4)
C6—C7—C8	120.8 (5)	C26—C25—H251	108.6
C6—C7—H7	119.6	N—C25—H251	108.6
C8—C7—H7	119.6	C26—C25—H252	108.6
C7—C8—C9	119.4 (5)	N—C25—H252	108.6
C7—C8—H8	120.3	H251—C25—H252	107.5
C9—C8—H8	120.3	C25—C26—H261	109.5
C8—C9—C10	120.4 (5)	C25—C26—H262	109.5
C8—C9—H9	119.8	H261—C26—H262	109.5
C10—C9—H9	119.8	C25—C26—H263	109.5
C5—C10—C9	120.0 (4)	H261—C26—H263	109.5
C5—C10—H10	120.0	H262—C26—H263	109.5
C9—C10—H10	120.0	N—C27—C28	114.3 (4)
C16—C11—C12	119.3 (4)	N—C27—H271	108.7
C16—C11—P	119.6 (3)	C28—C27—H271	108.7
C12—C11—P	121.0 (4)	N—C27—H272	108.7
C13—C12—C11	119.3 (5)	C28—C27—H272	108.7
C13—C12—H12	120.3	H271—C27—H272	107.6
C11—C12—H12	120.3	C27—C28—H281	109.5
C14—C13—C12	121.0 (5)	C27—C28—H282	109.5
C14—C13—H13	119.5	H281—C28—H282	109.5
C12—C13—H13	119.5	C27—C28—H283	109.5
C15—C14—C13	120.1 (5)	H281—C28—H283	109.5
C15—C14—H14	119.9	H282—C28—H283	109.5
C13—C14—H14	119.9		
O12—C1—C2—O22	2.9 (8)	O41—Re—P—C11	−72.59 (17)
O11—C1—C2—O22	−177.7 (5)	C17—P—C5—C10	−1.2 (4)
O12—C1—C2—O21	−178.0 (5)	C11—P—C5—C10	110.3 (4)
O11—C1—C2—O21	1.4 (6)	Re—P—C5—C10	−122.6 (4)
O32—C3—C4—O42	3.8 (7)	C17—P—C5—C6	177.2 (3)
O31—C3—C4—O42	−175.0 (4)	C11—P—C5—C6	−71.3 (4)
O32—C3—C4—O41	−176.8 (4)	Re—P—C5—C6	55.8 (4)
O31—C3—C4—O41	4.4 (5)	C10—C5—C6—C7	1.0 (7)
O12—C1—O11—Re	−173.7 (4)	P—C5—C6—C7	−177.5 (4)
C2—C1—O11—Re	6.9 (5)	C5—C6—C7—C8	1.0 (7)
O1—Re—O11—C1	94.0 (3)	C6—C7—C8—C9	−2.6 (8)
O21—Re—O11—C1	−9.1 (3)	C7—C8—C9—C10	2.1 (8)
O31—Re—O11—C1	−61.6 (5)	C6—C5—C10—C9	−1.5 (7)
O41—Re—O11—C1	−94.6 (3)	P—C5—C10—C9	176.9 (3)
P—Re—O11—C1	−174.8 (3)	C8—C9—C10—C5	−0.1 (7)
O22—C2—O21—Re	170.2 (5)	C5—P—C11—C16	−37.3 (4)
C1—C2—O21—Re	−8.9 (5)	C17—P—C11—C16	77.6 (4)
O1—Re—O21—C2	−93.8 (3)	Re—P—C11—C16	−159.1 (3)
O11—Re—O21—C2	9.9 (3)	C5—P—C11—C12	145.8 (4)
O31—Re—O21—C2	172.9 (3)	C17—P—C11—C12	−99.3 (4)
O41—Re—O21—C2	96.1 (3)	Re—P—C11—C12	24.1 (4)
P—Re—O21—C2	84.8 (5)	C16—C11—C12—C13	0.5 (7)

O32—C3—O31—Re	174.1 (4)	P—C11—C12—C13	177.4 (4)
C4—C3—O31—Re	−7.1 (5)	C11—C12—C13—C14	0.2 (8)
O1—Re—O31—C3	175.3 (3)	C12—C13—C14—C15	−0.9 (9)
O11—Re—O31—C3	−28.2 (5)	C13—C14—C15—C16	0.9 (9)
O21—Re—O31—C3	−80.2 (3)	C12—C11—C16—C15	−0.5 (7)
O41—Re—O31—C3	5.7 (3)	P—C11—C16—C15	−177.4 (4)
P—Re—O31—C3	85.0 (3)	C14—C15—C16—C11	−0.2 (8)
O42—C4—O41—Re	179.4 (3)	C5—P—C17—C18	−57.5 (4)
C3—C4—O41—Re	0.2 (5)	C11—P—C17—C18	−168.5 (3)
O1—Re—O41—C4	−52.3 (7)	Re—P—C17—C18	63.5 (4)
O11—Re—O41—C4	165.2 (3)	C5—P—C17—C22	132.4 (3)
O21—Re—O41—C4	83.5 (3)	C11—P—C17—C22	21.4 (4)
O31—Re—O41—C4	−2.8 (3)	Re—P—C17—C22	−106.6 (3)
P—Re—O41—C4	−99.4 (3)	C22—C17—C18—C19	−1.7 (6)
O1—Re—P—C5	−1.26 (18)	P—C17—C18—C19	−171.8 (3)
O11—Re—P—C5	−106.62 (18)	C17—C18—C19—C20	2.6 (7)
O21—Re—P—C5	−179.9 (4)	C18—C19—C20—C21	−1.8 (7)
O31—Re—P—C5	93.25 (17)	C19—C20—C21—C22	0.1 (7)
O41—Re—P—C5	168.69 (17)	C20—C21—C22—C17	0.8 (7)
O1—Re—P—C17	−122.10 (19)	C18—C17—C22—C21	0.0 (6)
O11—Re—P—C17	132.53 (18)	P—C17—C22—C21	170.2 (3)
O21—Re—P—C17	59.3 (4)	C27—N—C23—C24	−177.7 (4)
O31—Re—P—C17	−27.60 (18)	C25—N—C23—C24	55.1 (5)
O41—Re—P—C17	47.84 (17)	C27—N—C25—C26	−68.6 (5)
O1—Re—P—C11	117.46 (19)	C23—N—C25—C26	58.7 (6)
O11—Re—P—C11	12.09 (18)	C23—N—C27—C28	55.6 (5)
O21—Re—P—C11	−61.2 (4)	C25—N—C27—C28	−175.7 (4)
O31—Re—P—C11	−148.03 (18)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N—H71···O42	0.93	2.25	3.022 (5)	140
N—H71···O32	0.93	2.29	3.029 (5)	136

Fig. 1

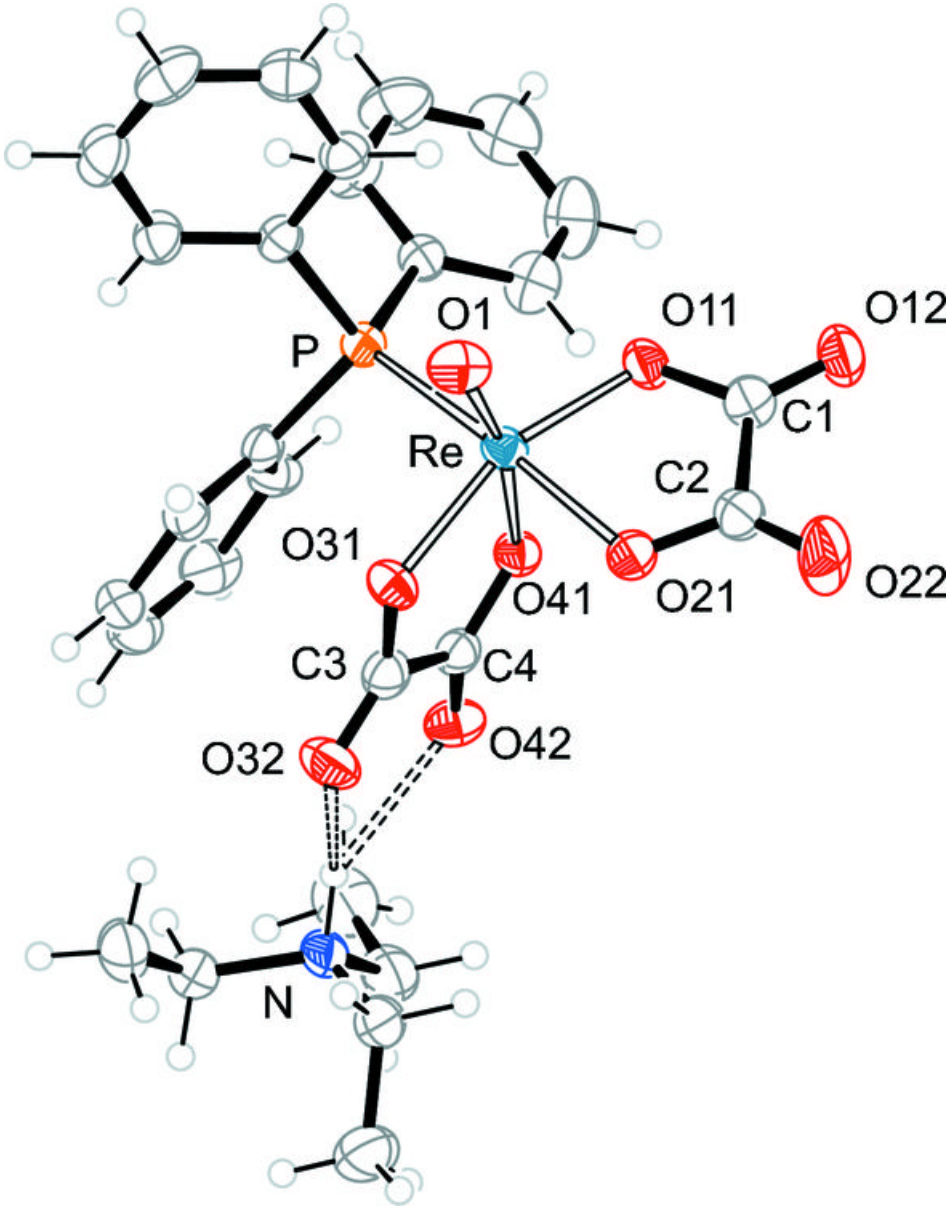

Fig. 2