

Co(2)—Ru(1)—Co(3)	54.4 (1)	Ru(1)—Co(3)—C(13)	85.8 (3)
Co(3)—Ru(1)—Co(1)	54.5 (1)	Co(1)—Co(3)—C(13)	49.6 (3)
Co(3)—Ru(1)—P(1)	117.8 (1)	C(9)—Co(3)—C(13)	94.5 (5)
Co(2)—Ru(1)—C(3)	99.7 (4)	Au—Co(1)—Ru(1)	115.0 (1)
Co(1)—Ru(1)—C(3)	94.8 (4)	Ru(1)—Co(1)—Co(2)	61.7 (1)
Co(2)—Ru(1)—C(1)	145.5 (4)	Ru(1)—Co(1)—Co(3)	61.6 (1)
Co(1)—Ru(1)—C(1)	92.9 (4)	Au—Co(1)—C(5)	72.3 (4)
C(3)—Ru(1)—C(1)	94.0 (5)	Co(2)—Co(1)—C(5)	126.3 (4)
Co(3)—Ru(1)—C(6)	87.9 (3)	Au—Co(1)—C(4)	165.3 (4)
P(1)—Ru(1)—C(6)	83.2 (3)	Co(2)—Co(1)—C(4)	128.7 (4)
C(1)—Ru(1)—C(6)	172.9 (4)	C(5)—Co(1)—C(4)	93.3 (5)
Co(3)—Ru(1)—C(8)	40.5 (3)	Ru(1)—Co(1)—C(12)	83.1 (3)
P(1)—Ru(1)—C(8)	83.2 (3)	Co(3)—Co(1)—C(12)	108.0 (3)
C(1)—Ru(1)—C(8)	81.4 (4)	C(4)—Co(1)—C(12)	97.2 (5)
Au—Co(2)—Ru(1)	117.3 (1)	Ru(1)—Co(1)—C(13)	84.4 (3)
Ru(1)—Co(2)—Co(3)	62.7 (1)	Co(3)—Co(1)—C(13)	49.8 (3)
Ru(1)—Co(2)—Co(1)	63.6 (1)	C(4)—Co(1)—C(13)	98.5 (5)
Au—Co(2)—C(6)	176.9 (4)	Ru(1)—C(6)—Co(2)	77.8 (5)
Co(3)—Co(2)—C(6)	113.8 (4)	Co(2)—C(23)—Co(3)	79.1 (5)
Au—Co(2)—C(7)	82.8 (4)	Co(3)—C(13)—Co(1)	80.7 (4)
Co(2)—Au—Co(1)	55.4 (1)	Co(1)—Co(2)—C(7)	132.6 (4)
Co(2)—Au—P(4)	149.0 (1)	Au—Co(2)—C(23)	79.5 (4)
Co(1)—Au—P(4)	151.7 (1)	Co(3)—Co(2)—C(23)	50.0 (4)
Co(2)—Ru(1)—Co(1)	54.7 (1)	C(6)—Co(2)—C(23)	97.4 (5)
Co(2)—Ru(1)—P(1)	116.9 (1)	Au—Co(2)—C(12)	85.2 (3)
Co(1)—Ru(1)—P(1)	170.5 (1)	Co(3)—Co(2)—C(12)	111.7 (3)
Co(3)—Ru(1)—C(3)	146.9 (4)	C(6)—Co(2)—C(12)	97.8 (5)
P(1)—Ru(1)—C(3)	91.1 (4)	C(23)—Co(2)—C(12)	160.2 (5)
Co(3)—Ru(1)—C(1)	99.1 (3)	Au—Co(3)—Co(2)	61.8 (1)
P(1)—Ru(1)—C(1)	94.1 (4)	Au—Co(3)—Co(1)	62.1 (1)
Co(2)—Ru(1)—C(6)	40.0 (3)	Co(2)—Co(3)—Co(1)	60.6 (1)
Co(1)—Ru(1)—C(6)	90.6 (3)	Ru(1)—Co(3)—C(8)	58.7 (3)
C(3)—Ru(1)—C(6)	79.6 (5)	Co(1)—Co(3)—C(8)	116.7 (3)
Co(2)—Ru(1)—C(8)	87.5 (3)	Ru(1)—Co(3)—C(9)	154.8 (4)
Co(1)—Ru(1)—C(8)	91.6 (3)	Co(1)—Co(3)—C(9)	133.1 (4)
C(3)—Ru(1)—C(8)	172.3 (4)	Au—Co(3)—C(23)	78.4 (3)
C(6)—Ru(1)—C(8)	104.7 (4)	Co(2)—Co(3)—C(23)	50.9 (3)
Au—Co(2)—Co(3)	64.1 (1)	C(8)—Co(3)—C(23)	94.7 (5)
Au—Co(2)—Co(1)	62.7 (1)	Au—Co(3)—C(13)	84.5 (3)
Co(3)—Co(2)—Co(1)	60.2 (1)	Co(2)—Co(3)—C(13)	110.2 (3)
Ru(1)—Co(2)—C(6)	62.2 (4)	C(8)—Co(3)—C(13)	101.1 (5)
Co(1)—Co(2)—C(6)	118.5 (4)	C(23)—Co(3)—C(13)	159.1 (5)
Ru(1)—Co(2)—C(7)	159.9 (4)	Au—Co(1)—Co(2)	61.9 (1)
Co(3)—Co(2)—C(7)	132.8 (4)	Au—Co(1)—Co(3)	63.4 (1)
C(6)—Co(2)—C(7)	97.7 (6)	Co(2)—Co(1)—Co(3)	59.1 (1)
Ru(1)—Co(2)—C(23)	90.2 (3)	Ru(1)—Co(1)—C(5)	171.9 (4)
Co(1)—Co(2)—C(23)	109.7 (4)	Co(3)—Co(1)—C(5)	122.1 (4)
C(7)—Co(2)—C(23)	93.4 (5)	Ru(1)—Co(1)—C(4)	79.5 (4)
Ru(1)—Co(2)—C(12)	85.8 (3)	Co(3)—Co(1)—C(4)	129.5 (4)
Co(1)—Co(2)—C(12)	51.5 (3)	Au—Co(1)—C(12)	83.2 (3)
C(7)—Co(2)—C(12)	97.0 (5)	Co(2)—Co(1)—C(12)	48.8 (3)
Au—Co(3)—Ru(1)	115.7 (1)	C(5)—Co(1)—C(12)	101.6 (5)
Ru(1)—Co(3)—Co(2)	63.0 (1)	Au—Co(1)—C(13)	85.5 (3)
Ru(1)—Co(3)—Co(1)	63.8 (1)	Co(2)—Co(1)—C(13)	108.9 (4)
Au—Co(3)—C(8)	171.4 (4)	C(5)—Co(1)—C(13)	93.2 (5)
Co(2)—Co(3)—C(8)	109.8 (4)	C(12)—Co(1)—C(13)	157.7 (5)
Au—Co(3)—C(9)	89.3 (4)	Ru(1)—C(8)—Co(3)	80.8 (4)
Co(2)—Co(3)—C(9)	138.6 (4)	Co(2)—C(12)—Co(1)	79.7 (4)
C(8)—Co(3)—C(9)	96.8 (5)		

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A New Phase of Bis[2-(Diphenylphosphino- κP)phenylazanido- κN](ethanolato)(oxo)-rhenium(V)

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Abstract

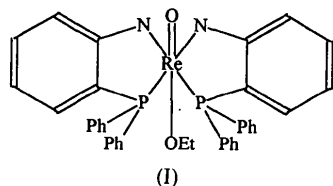
The title compound, [Re(C₁₈H₁₅NP)₂(C₂H₅O)O], was synthesized by the oxidation–substitution reaction of [Re^{III}(PNH)₂(PNH₂)]Cl, where PNH₂ is 2-(diphenylphosphino)phenylamine, with NEt₃ in EtOH. The X-ray diffraction study of this new triclinic phase, designated β , confirms the molecular structural details for the monoclinic α isomer, which crystallized in space group $P2_1/c$ with $a = 12.056$ (3), $b = 26.303$ (6), $c = 11.005$ (3) Å, $\beta = 102.32$ (2)° [Refosco, Tisato, Bandoli, Bolzati, Dolmella, Moresco & Nicolini (1993). *J. Chem. Soc. Dalton Trans.* pp. 605–618].

Comment

The structure contains discrete monomeric neutral units (I), with the coordination geometry around the Re atom being highly distorted octahedral. The fitted

All crystallographic calculations were performed using the *SHELXTL-Plus* program package (Sheldrick, 1987). The structure was determined by direct methods and subsequent ΔS calculations. Metal, P and O atoms were refined anisotropically as were C atoms in the methyl groups, C(111) and C(112), and in the carbonyl groups. Phenyl rings were refined as rigid groups with anisotropic displacement parameters. H atoms were placed in calculated positions (C—H = 0.96 Å, $U_{iso} = 0.09$ Å²).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71630 (43 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AB1078]



octahedron atoms of the α and β phases, overlapped using the *BMFIT* program (Nyburg, 1974), show a weighted root-mean-square deviation of only 0.05 Å. In fact, in the $P_2N_2O_2$ coordination polyhedron of the β phase the Re atom is 1.13 (1) Å from the plane formed by the atoms N(1), N(2) and O(2), and -1.10 (1) Å from the plane formed by atoms P(1), P(2) and O(1), the angle between the two triangles being 13.8 (1)°; the corresponding values in the α phase are 1.17 (1), -1.09 (1) Å and 14.6 (1)°. The two PCCN coordinating rings are bent away from the oxo group and make dihedral angles of 12.5 (3) and 2.9 (3)° with the P_2N_2 mean equatorial plane; the corresponding values are 23.6 (4) and 9.8 (4)° in the α isomer. However, the two isomers differ significantly in the periphery, in particular in the orientation of the phenyl rings, as evidenced by the pertinent torsion angles, *i.e.* Re—P(1)—C(13)—C(14), Re—P(1)—C(19)—C(20), Re—P(2)—C(25)—C(26) and Re—P(2)—C(31)—C(32) which have values of -61.6 (2), -18.6 (2), 50.0 (2) and -107.5 (2)°, respectively, in the α isomer, and -56.6 (2), -55.6 (2), -60.9 (2) and 146.5 (2)°, respectively, in the β isomer. It should be noted also that the closely related chlorooxobis[*o*-amido-phenyl]diphenylphosphine]rhenium(V) complex presents two different phases in the solid state (Refosco *et al.*, 1993).

IR spectra of the title compound and its α phase in KBr pellets show two major differences; in the β phase the band at 1098 cm^{-1} becomes very strong and a new absorption appears at 625 cm^{-1} . On the other hand, the stretching vibration associated with the N—H group (3300 cm^{-1}) remains unchanged and only a minor shift (from 887 to 883 cm^{-1}) is observed for $\nu_{\text{Re}=\text{O}}$. A detailed analysis of the structural data reveals that the α and β phases show slight but significant differences for the rhenium-donor distances; these differences could arise from the different packing of the two phases. In fact, in the β isomer, the intermolecular contact (*via* hydrogen bonding) between the O(1) and HN(1a) atoms (2.41 Å) weakens both the Re=O(1) [from 1.692 (7) to 1.717 (8) Å] and Re—N(1) bonds [from 1.990 (8) to 2.033 (7) Å]; such a lengthening is accompanied, within the same coordinated ligand, by a shortening of the Re—P(1) bond [from 2.495 (2) to 2.467 (2) Å]. In contrast, the other PNH⁻ chelate is coordinated to the metal by a weakened Re—P(2) bond [from 2.493 (3) to 2.535 (3) Å] and a reinforced Re—N(2)

bond [from 2.003 (7) to 1.959 (7) Å]. A similar interaction between the oxo O atom and the H atom bonded to an amino N atom of the ligand skeleton of a neighbouring unit has been described for a neutral rhenium diaminoxodithiolato complex (Jackson, Kojima & Lambrecht, 1992). In spite of the different nature of the N_2S_2 and *bis*-NP backbones, as well as the coordinating geometry of the resulting complexes (square pyramidal *versus* distorted octahedral), the relevant spectroscopic data for [ReO(te)], where te is 3,10-diethyl-5,8-diaza-3,10-dodecanedithiolate, follow the trend of those of the title compound. In particular, the Re=O bond length is 1.709 Å and $\nu_{\text{Re}=\text{O}}$ is 920 cm^{-1} , showing a slight bathochromic shift with respect to the analogous complex *syn*-[ReO(nmtm)], where nmtm is 2,4,9-trimethyl-4,7-diaza-2,9-decanedithiolate (Mahmood, Baidoo & Lever, 1990).

The β -[ReO(OEt)(PNH)₂] complex can also be described as a *syn* species, the O(1)⋯(H1a)—N(1a) and O(1a)⋯H(1)—N(1) angles being approximately 160°. When dissolved in CHCl_3 solution, the ethoxo group in β -[ReO(OEt)(PNH)₂] is easily exchanged with water, the complex rearranging to the hydroxo derivative [ReO(OH)(PNH)₂]; both α - and β -isomers of [ReO(OEt)(PNH)₂] are still present in solution as traces.

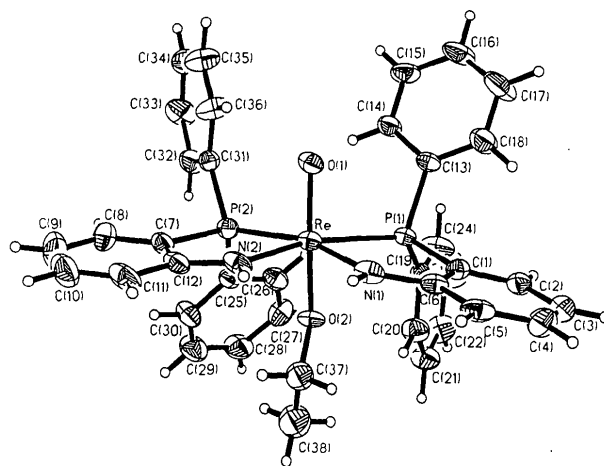


Fig. 1. ORTEP (Johnson, 1965) view of the title complex showing the atom-labelling scheme with atom contours at the 50% probability level.

Experimental

[Re^{III}(PNH)₂(PNH₂)]Cl (Refosco *et al.*, 1993) was dissolved in CH_2Cl_2 . The brown solution turned red-brown upon addition of NEt_3 . Brown prisms of [ReO(OEt)(PNH)₂] formed on slow diffusion of EtOH into the CH_2Cl_2 solution.

Crystal data

[Re(C₁₈H₁₅NP)₂(C₂H₅O)O] $D_x = 1.56 \text{ Mg m}^{-3}$
 $M_r = 799.8$ $D_m = 1.55 \text{ Mg m}^{-3}$

Triclinic	Mo K α radiation
$P\bar{1}$	$\lambda = 0.71073 \text{ \AA}$
$a = 11.335 (3) \text{ \AA}$	Cell parameters from 50 reflections
$b = 13.208 (3) \text{ \AA}$	$\theta = 9-13^\circ$
$c = 13.288 (4) \text{ \AA}$	$\mu = 3.7 \text{ mm}^{-1}$
$\alpha = 61.06 (2)^\circ$	$T = 296 \text{ K}$
$\beta = 79.11 (2)^\circ$	Prismatic
$\gamma = 80.71 (2)^\circ$	$0.20 \times 0.15 \times 0.10 \text{ mm}$
$V = 1703.9 (9) \text{ \AA}^3$	Brown
$Z = 2$	

Data collection

Nicolet Siemens R3m/V diffractometer	$R_{\text{int}} = 0.022$
$\omega-2\theta$ scans (2.5–14.5° min ⁻¹ in ω , range 1.35°)	$\theta_{\text{max}} = 22.5^\circ$
Absorption correction: empirical	$h = 0 \rightarrow 12$
$T_{\text{min}} = 0.73$, $T_{\text{max}} = 0.98$	$k = -14 \rightarrow 14$
4714 measured reflections	$l = -14 \rightarrow 14$
4447 independent reflections	2 standard reflections monitored every 100 reflections
3393 observed reflections	intensity variation: $\pm 2.1\%$
$[F_o > 3\sigma(F_o)]$	

Refinement

Refinement on F	$\Delta\rho_{\text{max}} = 1.1$
$R = 0.037$	(0.95 $\text{\AA}$ from Re)
$wR = 0.044$	$\Delta\rho_{\text{min}} = -1.3 \text{ e \AA}^{-3}$
$S = 0.76$	Atomic scattering factors from <i>International Tables for X-ray Crystallography</i> (1974, Vol. IV), inlaid in <i>SHELXTL-Plus</i> (Sheldrick, 1987)
3393 reflections	
397 parameters	
Only H-atom U 's refined	
$w = [\sigma^2(F) + 0.02F^2]^{-1}$	
$(\Delta/\sigma)_{\text{max}} = 0.16$	

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters ($\text{\AA}^2$)

	x	y	z	U_{eq}
Re	0.3573 (1)	0.0750 (1)	0.1188 (1)	0.034 (1)
P(1)	0.3203 (2)	-0.0790 (2)	0.3209 (2)	0.031 (1)
P(2)	0.1882 (2)	0.2357 (2)	0.0735 (2)	0.038 (1)
O(1)	0.2800 (6)	0.0058 (5)	0.0737 (5)	0.042 (3)
O(2)	0.3946 (5)	0.1529 (5)	0.2046 (5)	0.038 (3)
N(1)	0.5192 (7)	-0.0198 (7)	0.1434 (7)	0.042 (4)
N(2)	0.4265 (7)	0.1948 (7)	-0.0306 (7)	0.046 (4)
C(1)	0.4703 (8)	-0.1549 (8)	0.3434 (8)	0.036 (5)
C(2)	0.5082 (9)	-0.2467 (8)	0.4489 (9)	0.047 (5)
C(3)	0.6242 (10)	-0.3039 (9)	0.4576 (10)	0.057 (6)
C(4)	0.7043 (9)	-0.2673 (10)	0.3612 (11)	0.061 (7)
C(5)	0.6730 (9)	-0.1742 (10)	0.2562 (11)	0.053 (6)
C(6)	0.5555 (8)	-0.1152 (8)	0.2466 (8)	0.038 (5)
C(7)	0.2490 (9)	0.3407 (8)	-0.0678 (8)	0.045 (5)
C(8)	0.1881 (11)	0.4440 (10)	-0.1431 (8)	0.062 (6)
C(9)	0.2422 (15)	0.5188 (12)	-0.2474 (11)	0.089 (8)
C(10)	0.3598 (15)	0.4837 (12)	-0.2761 (11)	0.086 (8)
C(11)	0.4250 (12)	0.3801 (10)	-0.2068 (9)	0.068 (6)
C(12)	0.3692 (10)	0.3039 (9)	-0.0989 (8)	0.047 (5)
C(13)	0.2224 (8)	-0.1809 (8)	0.3354 (8)	0.037 (5)
C(14)	0.1081 (8)	-0.1370 (8)	0.2976 (9)	0.049 (5)
C(15)	0.0288 (10)	-0.2105 (10)	0.3112 (10)	0.059 (6)
C(16)	0.0628 (10)	-0.3262 (10)	0.3600 (11)	0.070 (7)
C(17)	0.1757 (11)	-0.3736 (10)	0.3989 (13)	0.086 (9)
C(18)	0.2552 (10)	-0.2991 (9)	0.3840 (11)	0.066 (6)

C(19)	0.2743 (7)	-0.0576 (7)	0.4485 (7)	0.033 (4)
C(20)	0.3398 (10)	0.0034 (9)	0.4737 (8)	0.058 (6)
C(21)	0.3104 (10)	0.0240 (9)	0.5684 (9)	0.058 (6)
C(22)	0.2133 (11)	-0.0192 (11)	0.6395 (9)	0.067 (6)
C(23)	0.1463 (11)	-0.0806 (12)	0.6181 (10)	0.085 (8)
C(24)	0.1756 (10)	-0.1001 (10)	0.5249 (9)	0.066 (7)
C(25)	0.1610 (8)	0.3031 (8)	0.1678 (8)	0.039 (5)
C(26)	0.1283 (9)	0.2335 (9)	0.2842 (9)	0.052 (5)
C(27)	0.1059 (10)	0.2746 (10)	0.3639 (9)	0.060 (6)
C(28)	0.1112 (11)	0.3927 (10)	0.3254 (11)	0.071 (7)
C(29)	0.1426 (11)	0.4625 (10)	0.2102 (11)	0.073 (7)
C(30)	0.1686 (10)	0.4166 (8)	0.1320 (9)	0.058 (6)
C(31)	0.0365 (8)	0.2251 (8)	0.0501 (7)	0.039 (4)
C(32)	-0.0659 (9)	0.2817 (9)	0.0834 (10)	0.057 (6)
C(33)	-0.1781 (9)	0.2677 (10)	0.0659 (11)	0.069 (7)
C(34)	-0.1907 (11)	0.2013 (10)	0.0149 (10)	0.065 (6)
C(35)	-0.0900 (11)	0.1477 (12)	-0.0144 (11)	0.076 (8)
C(36)	0.0237 (10)	0.1594 (11)	-0.0012 (10)	0.067 (7)
C(37)	0.4976 (10)	0.2090 (9)	0.1806 (9)	0.057 (3)
C(38)	0.4874 (12)	0.2740 (11)	0.2467 (11)	0.082 (4)

Table 2. Selected geometric parameters ($\text{\AA}$, $^\circ$)

Re—P(1)	2.467 (2)	Re—P(2)	2.535 (3)
Re—O(1)	1.717 (8)	Re—O(2)	1.997 (8)
Re—N(1)	2.033 (7)	Re—N(2)	1.959 (7)
P(1)—C(1)	1.827 (9)	P(2)—C(7)	1.795 (8)
N(1)—C(6)	1.42 (1)	N(2)—C(12)	1.41 (1)
O(1)—Re—N(1)	103.2 (4)	O(1)—Re—N(2)	100.7 (4)
P(1)—Re—N(1)	77.4 (2)	P(2)—Re—N(2)	78.9 (2)
P(1)—Re—O(1)	90.4 (2)	P(2)—Re—O(1)	89.3 (2)
P(1)—Re—O(2)	79.2 (2)	P(2)—Re—O(2)	79.5 (2)
P(1)—Re—P(2)	110.8 (1)	N(1)—Re—N(2)	90.9 (3)
P(1)—Re—N(2)	165.6 (3)	P(2)—Re—N(1)	165.2 (3)
O(1)—Re—O(2)	160.8 (3)	Re—O(2)—C(37)	125.6 (7)
Re—P(1)—C(1)	101.2 (3)	Re—P(2)—C(7)	100.5 (3)
Re—N(1)—C(6)	128.5 (6)	Re—N(2)—C(12)	126.0 (6)

Integrated intensities were corrected for Lorentz and polarization effects. The structure was solved by the heavy-atom method and the H atoms were included in calculated positions with a common variable isotropic displacement parameter. Refinement was by full-matrix least squares with anisotropic displacement parameters for non-H atoms. Structure determination and refinement were performed with the *SHELXTL-Plus* program system (Sheldrick, 1987).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71651 (20 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: NA1047]

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