S(3)	−0.0476 (2)	0.3270 (2)	0.3083 (2)	0.0491 (8)
S(4)	−0.0258 (2)	0.0701 (2)	0.3123 (2)	0.0616 (11)
C(1)	−0.0944 (7)	0.1824 (7)	0.3644 (7)	0.0551 (32)
N(1)	−0.1816 (8)	0.1670 (7)	0.4457 (7)	0.0809 (41)
C(2)	−0.2503 (12)	0.2580 (10)	0.4789 (11)	0.0931 (64)
C(3)	−0.3917 (12)	0.1890 (13)	0.3697 (15)	0.1121 (80)
C(6)	0.2286 (6)	0.4415 (7)	0.0047 (5)	0.0417 (26)
N(2)	0.2805 (5)	0.5316 (6)	−0.0582 (5)	0.0458 (24)
C(7)	0.2857 (7)	0.6784 (8)	−0.0152 (8)	0.0566 (36)
C(8)	0.4311 (9)	0.8042 (10)	0.0795 (14)	0.1040 (59)
C(9)	0.3465 (7)	0.4999 (9)	−0.1692 (7)	0.0595 (39)
C(10)	0.2183 (12)	0.3951 (13)	−0.2994 (9)	0.1063 (64)
C(4)	−0.2792 (26)	0.0201 (26)	0.4578 (24)	0.0341 (46)
C(5)	−0.2116 (30)	0.0810 (32)	0.6071 (31)	0.0749 (67)
C(42)	−0.1957 (15)	0.0560 (16)	0.5208 (15)	0.1007 (37)
C(52)	−0.3258 (16)	−0.0983 (18)	0.4319 (17)	0.1015 (43)

Table 2. *Geometric parameters* (Å, °)

Te—I(1)	2.835 (2)	C(2)—N(1)	1.480 (17)
Te—I(2)	3.048 (2)	C(4)—N(1)	1.500 (26)
Te—S(1)	2.726 (2)	C(42)—N(1)	1.623 (22)
Te—S(2)	2.544 (3)	C(2)—C(3)	1.564 (17)
Te—S(3)	2.556 (2)	C(6)—N(2)	1.306 (9)
Te—S(4)	2.788 (3)	N(2)—C(7)	1.500 (11)
Te—I(2^{i})	3.380 (2)	N(2)—C(9)	1.481 (10)
S(1)—C(6)	1.717 (9)	C(7)—C(8)	1.513 (10)
S(2)—C(6)	1.736 (7)	C(9)—C(10)	1.579 (11)
S(3)—C(1)	1.737 (9)	C(4)—C(5)	1.537 (40)
S(4)—C(1)	1.696 (9)	C(42)—C(52)	1.524 (16)
C(1)—N(1)	1.326 (11)		
I(1)—Te—I(2)	173.8 (1)	Te—S(3)—C(1)	90.8 (3)
I(1)—Te—S(1)	90.5 (1)	Te—S(4)—C(1)	84.1 (3)
I(1)—Te—S(2)	90.1 (1)	S(1)—C(6)—S(2)	117.2 (4)
I(1)—Te—S(3)	94.0 (1)	S(1)—C(6)—N(2)	122.6 (5)
I(1)—Te—S(4)	88.1 (1)	S(2)—C(6)—N(2)	120.1 (5)
I(1)—Te—I(2^{i})	86.5 (1)	C(6)—N(2)—C(7)	121.4 (5)
I(2)—Te—S(1)	86.3 (1)	C(6)—N(2)—C(9)	122.0 (5)
I(2)—Te—S(2)	93.6 (1)	N(2)—C(7)—C(8)	110.2 (7)
I(2)—Te—S(3)	91.7 (1)	N(2)—C(9)—C(10)	106.8 (6)
I(2)—Te—S(4)	91.8 (1)	C(7)—N(2)—C(9)	116.4 (5)
I(2)—Te—I(2^{i})	87.5 (1)	N(1)—C(4)—C(5)	93.9 (18)
S(1)—Te—S(2)	67.9 (1)	C(2)—N(1)—C(4)	107.5 (11)
S(1)—Te—S(3)	145.0 (1)	C(2)—N(1)—C(42)	120.8 (8)
S(1)—Te—S(4)	148.2 (1)	C(4)—N(1)—C(42)	34.3 (11)
S(1)—Te—I(2^{i})	72.8 (1)	S(3)—C(1)—S(4)	118.1 (4)
S(2)—Te—S(3)	77.3 (1)	S(3)—C(1)—N(1)	117.8 (5)
S(2)—Te—S(4)	143.8 (1)	S(4)—C(1)—N(1)	124.1 (5)
S(2)—Te—I(2^{i})	140.6 (1)	C(1)—N(1)—C(2)	122.4 (7)
S(3)—Te—S(4)	66.7 (1)	C(1)—N(1)—C(4)	123.4 (10)
S(3)—Te—I(2^{i})	142.1 (1)	C(1)—N(1)—C(42)	116.5 (7)
S(4)—Te—I(2^{i})	75.4 (1)	N(1)—C(2)—C(3)	111.2 (9)
Te—S(1)—C(6)	84.7 (3)	N(1)—C(42)—C(52)	105.4 (11)
Te—S(2)—C(6)	90.1 (3)		

Symmetry code: (i) $-x, -y, -z$.

$Te^{IV}(dedtc)_2I_2$ was synthesized by controlled oxidative displacement of the dithiocarbamate:

$$TeL_4 + I_2 \rightarrow TeL_2I_2 + L{-}L$$

where *L-L* is tetraethylthiuram disulfide. 1 m*M* (0.254 g) of iodine, dissolved in dichloromethane, was added to 1 m*M* (0.677 g) of TeL_4, dissolved in 25 ml of dichloromethane, with constant stirring for a few minutes. The solution gave a gummy mass on slow evaporation. Washing with carbon tetrachloride removed the much more soluble disulfide. Recrystallization of TeL_2I_2 using a mixture of dichloromethane and ethyl acetate yielded tiny deep-red crystals. Elemental analysis: found C 18.10, H 2.90, N 4.04, S 18.64, Te 19.12 wt%; calculated [for $Te(C_{10}H_{20}N_2S_4)I_2$]: C 17.72, H 2.97, N 4.13, S 18.91, Te 18.82 wt%. The structure was solved using *SHELX*76 (Sheldrick, 1976). Two of the ethyl carbon atoms, namely C(4) and C(5), showed positional disorder. They were isotropically refined with two positions for each [C(4), C(42) and C(5), C(52)]. The refined site occupancy factors are C(4) 0.2, C(42) 0.8, C(5) 0.3 and C(52) 0.7. H atoms could not be located. Peaks with heights > 1 e Å^{-3} in $\Delta\rho$ maps turned out to be ripples near the heavy atoms.

Lists of structure factors and anisotropic thermal parameters, as well as a unit-cell packing diagram have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55902 (17 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AB1024]

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Bis(4-hydroxymethyl-1,5-dimethylimidazole-N^3)gold(I) Chloride

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Abstract

The complex, bis(1,5-dimethylimidazole-4-methanol-N^3)gold(I) chloride, is a salt in which the ligand molecules are bound through N(3) atoms of the imidazole rings to the Au atom, which has an approximate inversion centre. Bond lengths and angles are normal.

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Comment

Gold(I) is relatively soft and is stabilized by soft-ligand atoms such as S and P or light atoms involved in π bonding to other atoms, as in cyanide, isonitriles and carbenes. Ligands bound through O or N atoms are much less effective at stabilizing Au^I (Cotton & Wilkinson, 1988), unless the N atom is part of an aromatic ring system (Rosopulos, Nagel & Beck, 1985; Lock & Turner, 1987). We have investigated the reaction of aromatic amine-substituted phosphines with Au^I compounds and, generally, obtained complexes in which the Au atom is bound to the P atom (Howard-Lock, Lock, Penny & Turner, 1989). In one case, when tris(4,5-dimethylimidazol-2-yl)phosphine was reacted with tetrachloroauric acid, a bis(carbene)gold(I) salt was obtained, where the imidazole group had been cleaved from the phosphine and trapped as the carbene, rather than as the tautomeric imidazole (Britten, Lock & Wang, 1992). We were unable to isolate any product from the direct reaction of imidazole with Au^I compounds or from the reaction with tetrachloroauric acid in the presence of a reducing agent. We have, however, been successful in isolating a product from the reaction of 4-hydroxymethyl-1,5-dimethylimidazole with chloro(dimethyl sulfide-*S*)gold(I) in methanol. Slow evaporation of a 1:1 methanol–toluene solution gave crystals suitable for X-ray crystallography. The ligand is not a particularly good complexing agent since, in water, the complex very soon starts to deposit Au metal.

The complex cation is shown in Fig. 1. The imidazole rings are bound to the Au atom through the N(3) atoms, rather than through C(2) of the tautomeric carbene. The rings attached to the Au atom are arranged such that the cation has very approximate C_{2h} symmetry, although this is destroyed by the disorder of the O atoms attached to C(41′). As such, the cation is roughly planar; the imidazole rings are at 12.4° to each other, which is achieved by the rings twisting about the N(3)···N(3′) axis rather than the cation bending at the Au atom [N(3)—Au—N(3′) 178.8 (4)°]. Each imidazole ring is planar, and the exocyclic atoms bound to the rings are displaced only slightly from the planes (up to 0.1 Å), although the O atoms are displaced about 1 Å from the rings [O(4) 1.03 (1), O(41′) 0.86 (2), O(42′) −1.28 (2) Å].

The Au—N bond lengths [Au—N(3) 2.011 (5), Au—N(3′) 2.000 (5) Å] are shorter than the values quoted for similar Au^I compounds, although the large errors in most previous determinations mean there is no significant difference (Bovio, Bonati, Burini & Pietroni, 1984; Adams, Hiller & Strähle, 1982; Fehlhammer & Finck, 1991). The distances observed here are even shorter than the Au^{III}—N distances in the dimethyl(tri-2-pyridylmethane-*N*,*N*′)gold(III) cation (Canty, Minchin, Healy & White, 1982), although they are longer than the Au^{III}—N distances in trichloropyridinegold(III) and the dichlorodi(pyridine)gold(III) cation (Adams & Strähle, 1982). The bond lengths and angles within the rings are remarkably uniform; there is little indication of single bond–double bond localization. They agree well with values for various imidazoles summarized by Bovio *et al.* (1984).

The packing is shown in Fig. 2. The major axes of the cations are along [101] and the cations are arranged in a staggered array, like bricks in a wall, such that the ring at one end of a cation is interleaved between the rings at the opposite ends of two other cations, thus maximizing π–π interactions between the rings. Because of the disorder of the O(41′)/O(42′) O atom, four layers comprise the repeat, so that we would expect the *b* axis to be 4 × 3.4 Å, close to the observed 13.998 (1) Å. This arrangement means that the cations form layers in the (101) planes. The Cl ions lie halfway between these layers. They bind the cation layers together through ionic interactions and quite strong O(4)···Cl···O(41′)/O(42′) hydrogen bonds [Cl···O(4) 3.08 (1), Cl···O(41′) 3.04 (2) Cl···O(42′) 3.09 (2) Å].

As noted above, the cations have nearly C_{2h} symmetry, with the Au atom lying on a pseudo-inversion centre. It is the requirements of the hydrogen bonding, and the resultant O-atom disorder, which lowers

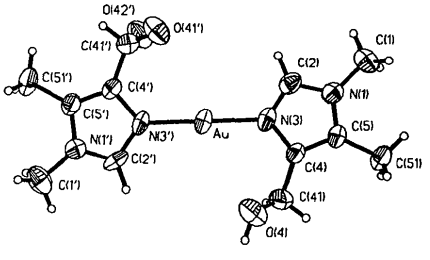

Fig. 1. The molecular complex, showing the atom numbering.

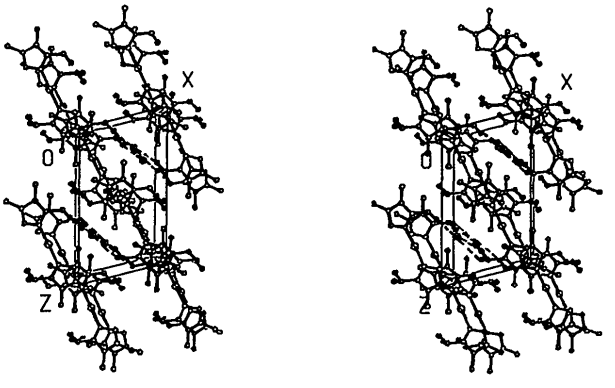

Fig. 2. The packing within the unit cell. **a*** and **c** are parallel to the bottom and sides of the page, respectively, and the view is down **b**. Hydrogen bonds are indicated by broken lines.

this symmetry. If one considers only the cations, and rejects the disorder, one can envisage a cell in which the origin inversion centre is moved to an Au atom and the b axis is halved. With minor reorientation of the cation the space group would be $C2/m$.

Experimental

Crystal data

$[Au(C_6H_{10}N_2O)_2]Cl$
M_r = 484.7
Monoclinic
$P2_1/n$
a = 8.115 (1) Å
b = 13.998 (1) Å
c = 13.919 (1) Å
β = 104.190 (7)°
V = 1532.8 (2) Å^3
Z = 4
D_x = 2.101 Mg m^{-3}
D_m = 2.12 (1) Mg m^{-3}
Density measured by suspension in $CCl_4/CHBr_3$
Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 39 reflections
θ = 4.85–12.45°
μ = 9.779 mm^{-1}
T = 294 (1) K
Needle
0.40 × 0.16 × 0.14 mm
Colourless

Data collection

Siemens $P4$ (rotating) diffractometer
θ-2θ scans
Absorption correction: empirical ψ scans
T_{min} = 0.0362, T_{max} = 0.0657
2790 measured reflections
2547 independent reflections
2547 observed reflections [$I \geq -3\sigma(I)$]
R_{int} = 0.0197
θ_{max} = 22.5°
h = 2 → 10
k = −1 → 18
l = −18 → 16
3 standard reflections monitored every 97 reflections
intensity variation: ±1.7%

Refinement

Refinement on F
Final R = 0.044
wR = 0.033
S = 1.81
190 parameters
H-atom parameters not refined
$w = 1/(\sigma_F)^2$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.65 e Å^{-3}
$\Delta\rho_{min}$ = −0.46 e Å^{-3}

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement coefficients* (Å^2)

Euivalent isotropic U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor. O(41′) and O(42′) represent a disordered O atom and were given occupancies of 0.5.

	x	y	z	U_{eq}
Au	0.1717 (1)	0.6160 (1)	0.2014 (1)	0.046 (1)
Cl	0.6638 (2)	0.5694 (2)	0.2014 (2)	0.062 (1)
N(1)	−0.1397 (6)	0.6284 (6)	−0.0815 (5)	0.044 (2)
C(2)	−0.1075 (8)	0.6178 (8)	0.0173 (5)	0.047 (3)
N(3)	0.0611 (7)	0.6171 (7)	0.0554 (4)	0.046 (2)
C(4)	0.1349 (7)	0.6261 (8)	−0.0226 (5)	0.046 (3)
C(5)	0.0096 (7)	0.6343 (6)	−0.1073 (5)	0.040 (2)
C(1)	−0.3113 (8)	0.6258 (8)	−0.1458 (6)	0.058 (3)
C(41)	0.3234 (8)	0.6226 (10)	−0.0078 (6)	0.064 (4)
C(51)	0.0163 (9)	0.6451 (8)	−0.2135 (5)	0.059 (3)
O(4)	0.4080 (6)	0.6966 (6)	0.0570 (5)	0.072 (3)
N(1′)	0.4787 (6)	0.6254 (6)	0.4840 (5)	0.045 (2)
C(2′)	0.4552 (9)	0.6179 (8)	0.3889 (6)	0.051 (3)
N(3′)	0.2816 (7)	0.6179 (7)	0.3466 (4)	0.045 (2)
C(4′)	0.2049 (7)	0.6289 (7)	0.4256 (5)	0.045 (3)
C(5′)	0.3272 (7)	0.6337 (7)	0.5109 (5)	0.044 (3)
C(1′)	0.6489 (8)	0.6237 (9)	0.5533 (7)	0.062 (3)
C(41′)	0.0148 (8)	0.6276 (10)	0.4070 (6)	0.063 (4)
O(41′)	−0.0704 (12)	0.6908 (12)	0.3408 (10)	0.069 (5)
O(42′)	−0.0491 (13)	0.5413 (12)	0.3916 (10)	0.073 (6)
C(51′)	0.3143 (10)	0.6471 (9)	0.6137 (6)	0.060 (3)

Table 2. *Bond lengths* (Å) *and angles* (°)

Au—N(3)	2.011 (5)	Au—N(3′)	2.000 (5)
N(1)—C(2)	1.34 (1)	N(1′)—C(2′)	1.30 (1)
C(2)—N(3)	1.340 (8)	C(2′)—N(3′)	1.387 (8)
N(3)—C(4)	1.37 (1)	N(3′)—C(4′)	1.40 (1)
C(4)—C(5)	1.360 (8)	C(4′)—C(5′)	1.349 (8)
C(5)—N(1)	1.347 (9)	C(5′)—N(1′)	1.376 (9)
N(1)—C(1)	1.460 (8)	N(1′)—C(1′)	1.478 (8)
C(4)—C(41)	1.493 (9)	C(4′)—C(41′)	1.501 (9)
C(41)—O(4)	1.44 (1)	C(41′)—O(41′)	1.34 (2)
		C(41′)—O(42′)	1.31 (2)
C(5)—C(51)	1.50 (1)	C(5′)—C(51′)	1.47 (1)
Au—N(3)—C(2)	124.0 (5)	Au—N(3′)—C(2′)	125.7 (5)
Au—N(3)—C(4)	129.0 (4)	Au—N(3′)—C(4′)	128.5 (4)
C(5)—N(1)—C(2)	108.5 (5)	C(5′)—N(1′)—C(2′)	111.5 (5)
N(1)—C(2)—N(3)	109.2 (6)	N(1′)—C(2′)—N(3′)	108.3 (7)
C(2)—N(3)—C(4)	106.8 (5)	C(2′)—N(3′)—C(4′)	105.6 (5)
N(3)—C(4)—C(5)	108.4 (5)	N(3′)—C(4′)—C(5′)	108.9 (6)
C(4)—C(5)—N(1)	107.1 (6)	C(4′)—C(5′)—N(1′)	105.7 (6)
C(1)—N(1)—C(2)	122.8 (6)	C(1′)—N(1′)—C(2′)	123.1 (6)
C(1)—N(1)—C(5)	128.6 (7)	C(1′)—N(1′)—C(5′)	125.4 (6)
N(3)—C(4)—C(41)	121.4 (6)	N(3′)—C(4′)—C(41′)	120.0 (6)
C(5)—C(4)—C(41)	130.2 (7)	C(5′)—C(4′)—C(41′)	131.0 (7)
C(4)—C(41)—O(4)	112.3 (8)	C(4′)—C(41′)—O(41′)	116 (1)
		C(4′)—C(41′)—O(42′)	112.3 (9)
C(4)—C(5)—C(51)	131.5 (6)	C(4′)—C(5′)—C(51′)	130.5 (6)
N(1)—C(5)—C(51)	121.4 (5)	N(1′)—C(5′)—C(51′)	123.8 (5)
O(41′)—C(41′)—O(42′)	112.3 (9)	N(3)—Au—N(3′)	178.8 (4)

Reflections with $-3\sigma_I \leq I \leq 3\sigma_I$ were treated by the method of French & Wilson (1978). O(41′) and O(42′), the disordered O atoms, were each refined with an occupancy of 0.5. H atoms were placed in calculated positions, with fixed temperature factors, and constrained to ride on the atoms to which they were attached. *International Tables for X-ray Crystallography* (1974, Vol. IV) provided the atomic scattering factors (Table 2.2B) as well as anomalous-dispersion corrections for N, O, Cl and Au atoms (Table 2.3.1). Calculations employed *SHELXTL PC* (Sheldrick, 1990) and Laser 386 or IBM 486 computers.

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Lists of structure factors, anisotropic displacement coefficients, H-atom coordinates and best planes and the dihedral angles between them, have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71035 (11 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: BR1023]

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(*tert*-Butyl isocyano-*C*)(carbonyl)(η^5-cyclopentadienyl)(triphenylphosphine-*P*)-ruthenium(II) Hexafluorophosphate

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Abstract

The crystal structure of the title compound consists of discrete $[Ru(CN^tBu)(CO)(PPh_3)(\eta^5\text{-}C_5H_5)]^+$ cations and $[PF_6]^-$ anions. The cation has pseudo-octahedral symmetry with the cyclopentadienyl group occupying three sites in a facial arrangement.

Comment

The chemistry of the $[Ru(CN^tBu)(PPh_3)(\eta^5\text{-}C_5H_5)]^+$ moiety has been extensively studied as it is able to ligate molecules such as dihydrogen, iodoalkanes and mercaptans (Conroy-Lewis, Redhouse & Simpson, 1989; Conroy-Lewis & Simpson, 1986, 1991); these molecules normally undergo oxidative addition at late-transition-metal centres. The variation in structural parameters of the triphenylphosphine and *tert*-butyl isocyanide ligands for a series of compounds containing this moiety might illuminate the unusual electronic character responsible for this reactivity. We previously reported the crystal structure of $[Ru(CN^tBu)(PPh_3)(NH_3)(\eta^5\text{-}C_5H_5)]PF_6$, where the additional ligand was a pure σ-donor (Conroy-Lewis & Simpson, 1990), and we present here the structure of $[Ru(CN^tBu)(CO)(PPh_3)(\eta^5\text{-}C_5H_5)]PF_6$, where the carbonyl ligand is an archetypal good π-acceptor and poor σ-donor. The compound has been reported previously (Conroy-Lewis, Redhouse & Simpson, 1989).

The cation has the expected pseudo-octahedral geometry with the cyclopentadienyl group occupying three *fac* sites. Both the Ru—P bond length, 2.328 (1) Å, and Ru—C(N) distance, 1.961 (4) Å, are longer than the 2.290 (1) and 1.934 (5) Å, respectively, found in $[Ru(CN^tBu)(PPh_3)(NH_3)(\eta^5\text{-}C_5H_5)]PF_6$ (Conroy-Lewis & Simpson, 1990). This reflects the increased competition for π-electron density at the metal as a result of replacing an ammine group with a carbonyl group. The Ru—C(O) bond length of 1.901 (6) Å in $[Ru(CN^tBu)(CO)(PPh_3)(\eta^5\text{-}C_5H_5)]PF_6$ is at the high end of the range reported for this class of molecule: 1.880 (6) Å for $[Ru(CO)\{P(OMe)_3\}_2(\eta^5\text{-}C_5H_5)]BF_4$ (Frank & Selegue, 1991), 1.869 (2) Å for $[Ru(CO)(PPh_3)_2(\eta^5\text{-}C_5H_5)]BPh_4$ (Wisner, Bartczak & Ibers, 1985) and 1.890 (2) Å for $[Ru(CO)(PPh_3)_2(\eta^5\text{-}C_5H_5)][Co(CO)_4]$ (Doyle & Van Engen, 1985). The carbonyl stretching frequencies in the infrared spectra of these cations, at 2014, 2022, 1987 and 1978 cm^{-1}, respectively, illustrate more clearly the electron density at the metal centre available for back donation. The values of $\nu(C{\equiv}N)$ for $[Ru(CN^tBu)(PPh_3)(CO)(\eta^5\text{-}C_5H_5)]PF_6$ and $[Ru(CN^tBu)(PPh_3)(NH_3)(\eta^5\text{-}C_5H_5)]PF_6$ are 2186 and 2126 cm^{-1}, respectively, confirming the effect of the carbonyl group.

It seems likely that the ability of the $[Ru(CN^tBu)(PPh_3)(\eta^5\text{-}C_5H_5)]^+$ moiety to stabilize unusual ligands results from the presence of both a good σ-donor (^{t}BuNC) and a good π-acceptor (PPh_3) in the coordination sphere.

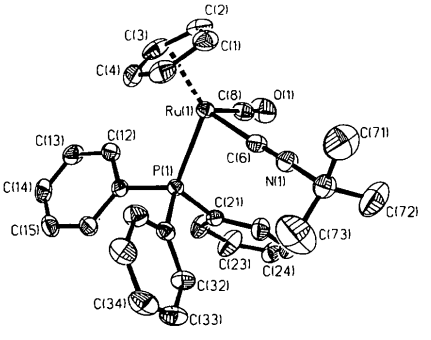

Fig. 1. Plot showing 30% thermal ellipsoids for the cation present in $[Ru(CN^tBu)(CO)(PPh_3)(\eta^5\text{-}C_5H_5)]PF_6$.

Experimental

Crystal data

$[Ru(C_{18}H_{15}P)(C_5H_9N)(C_5H_5)(CO)][PF_6]$	$Z = 2$
	$D_x = 1.505$ Mg m^{-3}

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