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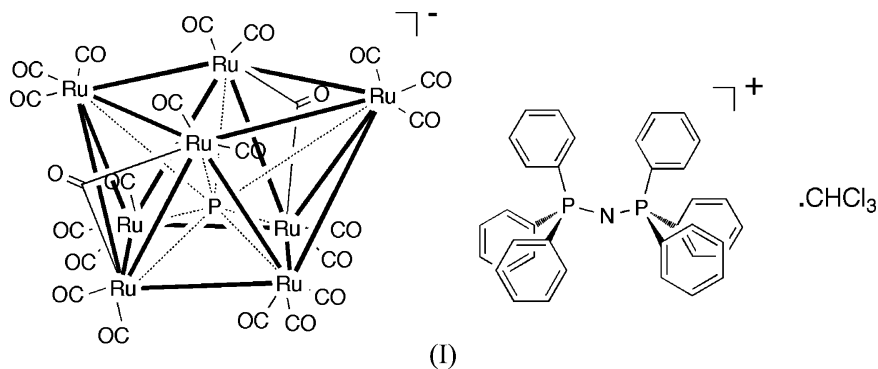
Key indicators

Single-crystal X-ray study
 $T = 200$ K
Mean $\sigma(\text{C}-\text{C}) = 0.010$ Å
Disorder in main residue
 R factor = 0.024
 wR factor = 0.023
Data-to-parameter ratio = 12.0For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.Bis(triphenylphosphoranylidene)ammonium docosacarbonyl(μ_8 -phosphido)octaruthenate chloroform solvateThe title compound, $(\text{C}_{36}\text{H}_{30}\text{NP}_2)[\text{Ru}_8\text{P}(\text{CO})_{22}]\cdot\text{CHCl}_3$ or $[\text{N}(\text{PPh}_3)_2][\text{Ru}_8(\mu_8\text{-P})(\text{CO})_{22}]\cdot\text{CHCl}_3$, comprises a square antiprismatic octaruthenium cluster anion with an interstitial phosphido ligand, two bridging carbonyl ligands, and 20 terminal carbonyl ligands, together with a disordered PPN cation and a disordered chloroform solvent molecule.

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Comment

The title salt, (I), was obtained from thermolysis of $\text{Ru}_5(\mu\text{-H})(\mu\text{-NC}_5\text{H}_4)(\text{CO})_{10}$ and PClPh_2 in chlorobenzene (Cifuentes *et al.*, 1998), and was only the second octaruthenium cluster carbonyl complex. The structural study of the non-solvated title compound has been reported (Cifuentes *et al.*, 1998). The majority of the Ru–Ru bond lengths and Ru–Ru–Ru angles in the solvated cluster anion from the present study differ by more than 3 s.u.'s from the analogous distances and angles in the non-solvated anion. The largest respective deviations occur for Ru1–Ru4 [2.9375 (6) versus 2.8984 (8) Å], Ru3–Ru6 [2.9041 (6) versus 2.938 (1) Å] and Ru7–Ru8 [2.9168 (6) versus 2.9486 (7) Å].

Experimental

The compound was prepared by the literature procedure (Cifuentes *et al.*, 1998). Purple crystals suitable for the structural study were obtained by liquid diffusion of heptane into a chloroform solution.

Crystal data

 $(\text{C}_{36}\text{H}_{30}\text{NP}_2)[\text{Ru}_8\text{P}(\text{CO})_{22}]\cdot\text{CHCl}_3$ $M_r = 2113.73$ Orthorhombic, $Pna2_1$ $a = 19.1786$ (2) Å $b = 10.5448$ (1) Å $c = 32.9569$ (4) Å $V = 6665.02$ (12) Å³ $Z = 4$ $D_x = 2.106$ Mg m⁻³Mo $K\alpha$ radiation $\mu = 2.03$ mm⁻¹ $T = 200$ K

Plate, purple

0.27 × 0.16 × 0.02 mm

Data collection

Nonius KappaCCD diffractometer
 φ and ω scans
 Absorption correction: integration
 Gaussian integration (Coppens,
 1970)
 $T_{\min} = 0.644$, $T_{\max} = 0.959$

78313 measured reflections
 15035 independent reflections
 10343 reflections with $I > 3\sigma(I)$
 $R_{\text{int}} = 0.038$
 $\theta_{\text{max}} = 27.5^\circ$

Refinement

Refinement on F
 $R[F^2 > 3\sigma(F^2)] = 0.024$
 $wR[F^2 > 3\sigma(F^2)] = 0.023$
 $S = 1.11$
 10343 reflections
 863 parameters
 H-atom parameters constrained
 $w = [1 - (F_o - F_c)^2 / 36\sigma^2(F)]^2 /$
 $[0.282T_0(x) - 0.0449T_1(x)$
 $+ 0.108T_2(x)]$
 where T_i are the Chebyshev

polynomials and $x = F_c/F_{\text{max}}$
 (Prince, 1982; Carruthers &
 Watkin, 1979)
 $(\Delta/\sigma)_{\text{max}} = 0.04$
 $\Delta\rho_{\text{max}} = 2.01 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -1.26 \text{ e } \text{\AA}^{-3}$
 Absolute structure: Flack (1983);
 7252 Friedel pairs, partially
 inversion twinned
 Flack parameter: 0.43 (2)

Table 1

Selected bond lengths (\AA).

Ru1—Ru2	2.9643 (6)	Ru3—C33	2.078 (6)
Ru1—Ru4	2.9375 (6)	Ru4—Ru7	2.9107 (6)
Ru1—Ru5	2.8215 (6)	Ru4—Ru8	2.9005 (6)
Ru1—Ru8	2.9389 (6)	Ru4—P1	2.3986 (13)
Ru1—P1	2.4346 (12)	Ru5—Ru6	2.9367 (6)
Ru1—C13	2.059 (6)	Ru5—Ru8	2.9547 (6)
Ru2—Ru3	2.9294 (6)	Ru5—P1	2.4160 (14)
Ru2—Ru5	2.9281 (6)	Ru5—C13	2.050 (6)
Ru2—Ru6	2.9086 (5)	Ru6—Ru7	2.9759 (6)
Ru2—P1	2.4013 (12)	Ru6—P1	2.3960 (12)
Ru3—Ru4	2.9695 (6)	Ru7—Ru8	2.9168 (6)
Ru3—Ru6	2.9041 (6)	Ru7—P1	2.4098 (12)
Ru3—Ru7	2.8064 (6)	Ru7—C33	2.053 (6)
Ru3—P1	2.4031 (14)	Ru8—P1	2.3970 (13)

The crystallographic asymmetric unit consists of one $[\text{N}(\text{PPh}_3)_2]^+$ cation, one $[\text{Ru}_8(\mu_8\text{-P})(\text{CO})_{22}]^-$ anion and a chloroform solvent molecule. Within the cation, one phenyl group was found to be disordered over two locations. Each atom of this ring was split over two sites and refined with isotropic displacement parameters which were constrained to be equal for each pair; the relative populations were refined to 0.545 (9):0.455 (9). The anion was well behaved. Within the chloroform solvate, one chlorine and the carbon atom were found to be disordered over two sites, the relative populations of which were refined to 0.654 (7):0.346 (7). The Cl atoms were refined anisotropically while the C atoms were treated isotropically. H atoms were included at idealized positions and allowed to ride on the atoms to which they are bonded, with $\text{C—H} = 1.00 \text{ \AA}$ and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. The extrema in the final difference electron density map (-1.26 and $2.01 \text{ e } \text{\AA}^{-3}$) were located close to the Cl atoms.

Data collection: *COLLECT* (Nonius, 1997); cell refinement: *DENZO/SCALEPACK*; data reduction: *DENZO/SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure:

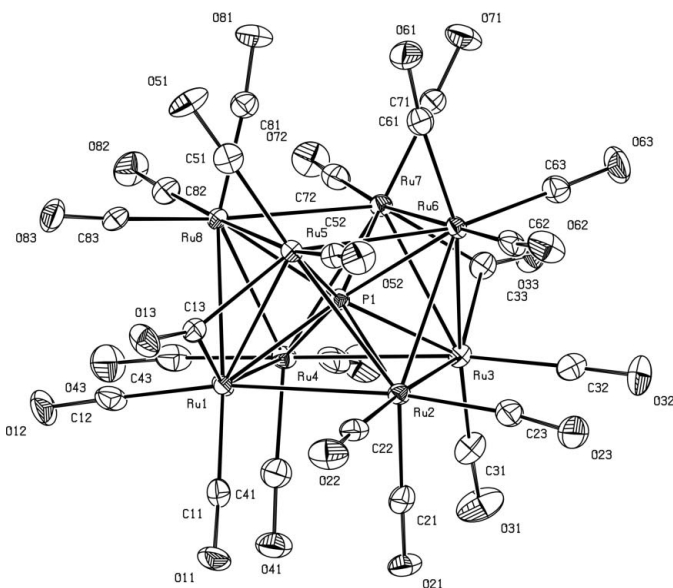


Figure 1

A view of the anion of the title compound, showing the atom-numbering scheme. The counter-cation and the chloroform solvent molecule are not shown. Displacement ellipsoids are drawn at the 30% probability level.

SIR92 (Altomare *et al.*, 1994); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *CRYSTALS*.

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