

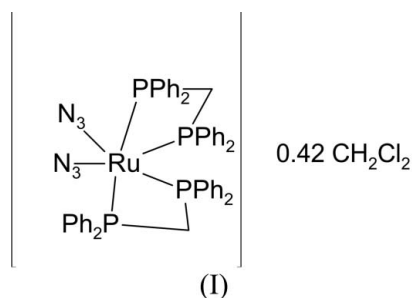
Luca Russo,^a Kalle Nättinen,^a
Viatcheslav Vertlib,^b João
Rodrigues^{b*} and Kari Rissanen^a^aNanoscience Center, Department of Chemistry,
University of Jyväskylä, PO Box 35, 40014
Jyväskylä, Finland, and ^bCentro de Química da
Madeira, LQCM, Departamento de Química
da Universidade da Madeira, Campus de
Penteada, 9000-390 Funchal, Portugal

Correspondence e-mail: krissane@cc.jyu.fi

Key indicators

Single-crystal X-ray study
T = 173 K
Mean $\sigma(\text{C}-\text{C}) = 0.009 \text{ \AA}$
Disorder in solvent or counterion
R factor = 0.062
wR factor = 0.162
Data-to-parameter ratio = 14.0For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.*cis*-Diazido[bis(diphenylphosphino)methane- κ^2P,P']-
ruthenium(II) dichloromethane 0.42-solvateThe title compound, $[\text{Ru}(\text{N}_3)_2(\text{C}_{25}\text{H}_{22}\text{P}_2)_2]$ or *cis*- $[\text{Ru}(\text{N}_3)_2(\text{dppm})_2]$ [dppm is bis(diphenylphosphino)methane], was obtained unexpectedly from our attempts to prepare new ruthenium molecular wires using organic bridging ligands. Both the azido ligands show a triple bond adjacent to the metal.Received 6 July 2006
Accepted 26 July 2006

Comment

Three structures of diazidoruthenium monomeric complexes are known from the literature and in all these the two azide ligands are in the *trans* configuration (Siebald *et al.*, 1996; Wehlan *et al.*, 2000; Buys *et al.*, 1995). In the title compound, (I), the Ru^{II} atom is in a distorted octahedral coordination environment. It is coordinated by two chelating bis(diphenylphosphino)methane ligands and two azido anions in a mutually *cis* geometry. Noteworthy features of the complex are the two N—N bonds adjacent to the metal, with bond lengths of 0.847 (7) and 0.823 (7) Å; this clearly defines them as triple bonds, thus locating a formal negative charge on the uncoordinated end of the azido group. 16 examples of metal-coordinated azido ligands, with N—N bonds adjacent to the metal shorter than 1 Å, can be found in the Cambridge Structural Database (CSD, Version 5.27; Allen, 2002), with refcodes AZNPNI, EBUXEE, EMOFOB, FASJAK, FIQLEX, HOJLIB, INOXAK, JAHSOA, LOZVOL, MENPUQ, NEHJEP, NEKLUK, REDKOA, WIVLOC, YETYUR and DAWTUR. There is no indication of disorder in the structure of the title complex of which the short N—N

bonds could be an artefact. Bond lengths and angles within the distorted octahedral coordination environment of the metal are reported in Table 1. Along with the neutral complex molecule, the asymmetric unit also contains dichloromethane, partially occupied and disordered about a twofold axis.

Experimental

The title complex was obtained from the reaction of a solution of *trans*- $[\text{RuCl}(\text{NO})(\text{dppm})_2](\text{BF}_4)_2$ (0.075 g, 0.12 mmol) and NaN_3

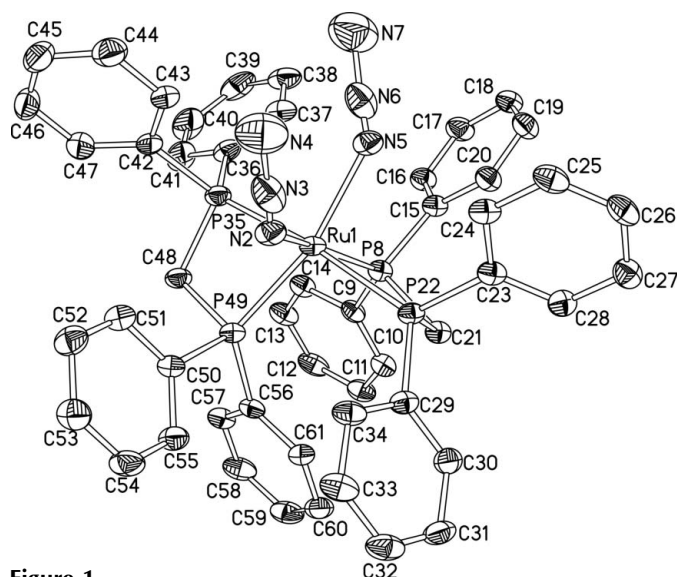


Figure 1

The molecular structure of (I) (H atoms omitted), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. The disordered solvent molecule has been omitted.

(0.008 g, 0.12 mmol) in 10 ml of acetone. The reaction mixture was refluxed for 2 h, then stirred for 20 h. A brown liquid fraction was removed by filtration and the recovered yellow residue dried under vacuum, washed with diethyl ether (2 × 5 ml) and dried again. Bright-yellow crystals suitable for X-ray diffraction were obtained from a dichloromethane/*n*-pentane (5:3) mixture.

Crystal data

[Ru(N₃)₂(C₂₅H₂₂P₂)₂].0.42CH₂Cl₂
M_r = 989.17
 Monoclinic, *C*2/*c*
a = 36.8818 (7) Å
b = 11.2016 (2) Å
c = 22.3275 (4) Å
 β = 97.235 (2)°
V = 9150.8 (3) Å³

Z = 8
D_x = 1.436 Mg m⁻³
 Mo *K*α radiation
 μ = 0.57 mm⁻¹
T = 173 (2) K
 Block, yellow
 0.45 × 0.40 × 0.40 mm

Data collection

Nonius KappaCCD diffractometer
 φ and ω scans
 Absorption correction: none
 42989 measured reflections

7920 independent reflections
 6115 reflections with *I* > 2σ(*I*)
*R*_{int} = 0.067
 θ_{\max} = 25.0°

Refinement

Refinement on *F*²
R [*F*² > 2σ(*F*²)] = 0.062
wR (*F*²) = 0.162
S = 1.06
 7920 reflections
 566 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0635P)^2 + 73.832P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 1.45 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.91 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (Å, °).

Ru1—N2	2.254 (5)	Ru1—P22	2.3455 (15)
Ru1—N5	2.220 (6)	Ru1—P35	2.3446 (16)
Ru1—P8	2.3050 (15)	Ru1—P49	2.3099 (15)
N2—Ru1—N5	84.41 (19)	N5—Ru1—P49	165.79 (15)
N2—Ru1—P8	161.90 (15)	P8—Ru1—P22	72.03 (5)
N2—Ru1—P22	90.18 (14)	P8—Ru1—P35	100.99 (6)
N2—Ru1—P35	97.07 (15)	P8—Ru1—P49	94.93 (5)
N2—Ru1—P49	91.76 (16)	P22—Ru1—P35	170.18 (5)
N5—Ru1—P8	92.83 (14)	P22—Ru1—P49	101.24 (5)
N5—Ru1—P22	92.48 (15)	P35—Ru1—P49	72.03 (5)
N5—Ru1—P35	94.81 (15)		

The occupancy of the dichloromethane molecule, disordered about a twofold axis, was refined freely to 0.417 (7). Geometrical restraints were applied on the dichloromethane molecule; on the solvent molecule, as well as on the two azido ligands, displacement parameter restraints were also applied. All H atoms were refined using a riding model, with C—H = 0.95 Å for aromatic atoms, C—H = 0.99 Å for CH₂ and *U*_{iso}(H) = 1.2*U*_{eq}(C). The maximum electron-density peak in the final difference map lies 0.46 Å from atom N2.

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 2001); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2001) and *CIFTAB* (Sheldrick, 1993).

We thank the Academy of Finland for a research grant (KR and LR). The Fundação para a Ciência e a Tecnologia (FCT, Portugal) is also gratefully acknowledged for partial support of this research through the project POCTI CTM/41495/2001.

References

- Allen, F. R. (2002). *Acta Cryst.* **B58**, 380–388.
 Buys, I. E., Field, L. D., George, A. V., Hambley, T. W. & Purches, G. R. (1995). *Aust. J. Chem.* **48**, 27–34.
 Hooft, R. W. (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.
 Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
 Sheldrick, G. M. (1993). *CIFTAB*. University of Göttingen, Germany. (Modified by W. Clegg, Newcastle University, England.)
 Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
 Sheldrick, G. M. (2001). *SHELXTL*. Version 6. Bruker AXS Inc., Madison, Wisconsin, USA.
 Siebald, H. G. L., Fabre, P.-L., Dartiguenave, M., Dartiguenave, Y., Simard, M. & Beauchamp, A. L. (1996). *Polyhedron*, **15**, 4221–4225.
 Whelan, M., Thiel, R., Fuchs, J., Beck, W. & Fehlhammer, W. P. (2000). *J. Organomet. Chem.* **613**, 159–169.