

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BK1254). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

- Balch, A. L., Hart, R. L. & Parkin, S. (1993). *Inorg. Chim. Acta*, **205**, 137–143.
- Balch, A. L., Latos-Grazynski, L., Noll, B. C. & Phillips, S. L. (1993). *Inorg. Chem.* **32**, 1124–1129.
- Boukhris, A., Lecomte, C., Coutsolelos, A. & Guillard, R. (1986). *J. Organomet. Chem.* **303**, 151–165.
- Coutsolelos, A., Guillard, R., Bayeul, D. & Lecomte, C. (1986). *Polyhedron*, **5**, 1157–1164.
- Coutsolelos, A., Guillard, R., Boukhris, A. & Lecomte, C. (1986). *J. Chem. Soc. Dalton Trans.* pp. 1779–1783.
- Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
- Messerschmitt, A. & Pflugrath, J. W. (1987). *J. Appl. Cryst.* **20**, 306–315.
- Scheidt, W. R. & Turowska-Tyrk, I. (1994). *Inorg. Chem.* **33**, 1314–1318.
- Sheldrick, G. M. (1990). *Acta Cryst.* **A46**, 467–473.
- Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.
- Wynne, K. J. (1984). *Inorg. Chem.* **23**, 4658–4663.

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3-Chloro-3,3-bis(triphenylphosphine-P)-1,2-dicarb-3-rhoda-closo-dodecaborane-Dichloromethane (1/1.1)

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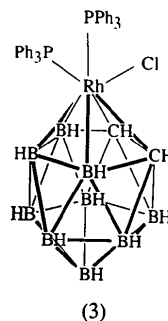
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Abstract

The title compound, [3,3-(PPh₃)₂-3-Cl-3,1,2-RhC₂B₉H₁₁] or [RhCl(C₂H₁₁B₉)(C₁₈H₁₅P)₂].1.1CH₂Cl₂, contains a triangulated icosahedral *closo*-RhC₂B₉ cage. Principal bond distances are Rh—Cl 2.423 (2), Rh—P 2.373 (2) and 2.383 (2), Rh—C 2.190 (6) and 2.191 (6), and Rh—B 2.203 (8)–2.289 (8) Å. The conformation of the Rh(PPh₃)₂Cl group above the C₂B₃ face of the dicarbaborane ligand is such that the Rh—Cl bond is located above the cage C—C vector.

Comment

Rhodium chloride-containing rhodacarbaborane complexes of the type [{Rh(PR₃)_nCl}C₂B₉H₁₁] have been identified as products from a variety of reactions involving [{Rh(PR₃)₂H}C₂B₉H₁₁] reagents. The compounds [3,3-(PR₃)₂-3-Cl-3,1,2-RhC₂B₉H₁₁], with R₃ = Ph₃ (Baker *et al.*, 1984) or MePh₂ (Ferguson *et al.*, 1996), were obtained from the reactions between [3,3-(PPh₃)₂-3-H-3,1,2-RhC₂B₉H₁₁], (1), and aqueous HCl in CHCl₃ (R₃ = Ph₃, 73%), or from [3- $\{\eta^2$ -SC(H)NPh}-3-(PPh₃)-3,1,2-RhC₂B₉H₁₁] and PMePh₂ in CH₂Cl₂ (R₃ = MePh₂, 96%), respectively (Ferguson *et al.*, 1996). The 16-electron species [2-(PPh₃)₂-2-Cl-2,1,7-RhC₂B₉H₁₁], (2), was the unexpected product from the reaction between the 2,1,7-isomer of (1) and aqueous HCl in CHCl₃ (Baker *et al.*, 1984). We now report an alternative route to [3,3-(PPh₃)₂-3-Cl-3,1,2-RhC₂B₉H₁₁], (3), and the structural characterization of (3) using X-ray crystallography.



The overall form of the rhodacarbaborane cage structure in (3) is, as expected, a *closo* 12-atom species (Fig. 1). Considering the bonding to the *exo*-cage ligands first, the Rh—Cl distance [2.423 (2) Å] is very similar to that found in [3,3-(PMePh₂)₂-3-Cl-3,1,2-RhC₂B₉H₁₁] [(4); 2.4205 (8) Å; Ferguson *et al.*, 1996], but the Rh—P distances show some differences between the two structures. The Rh—P distances in (3) are not significantly different from their mean value [2.378 (5) Å], whereas the equivalent bonds in compound (4) are significantly different [Rh—P1 2.3500 (9) and Rh—P2 2.3414 (9) Å]. The Rh—P [2.329 (1) Å] and Rh—Cl [2.299 (1) Å] distances in the 16-electron species [2-(PPh₃)₂-2-Cl-2,1,7-RhC₂B₉H₁₁] [(2); Baker *et al.*, 1984] are significantly shorter than those in the 18-electron compounds (3) and (4), suggesting stronger *exo*-cage bonding in compound (2).

The Rh3—C1 and Rh3—C2 distances in compound (3) (Table 2) are essentially identical and the equivalent distances in (4) [2.208 (3) and 2.205 (3) Å, respectively] are very similar. In contrast, the Rh—C distances in (2) are distinctly different [2.215 (4) and 2.165 (4) Å]. Since the Cl atom in (2) is *trans* with respect to the phosphine ligand, with a P—Rh—Cl angle of 175.2 (1)°, and the

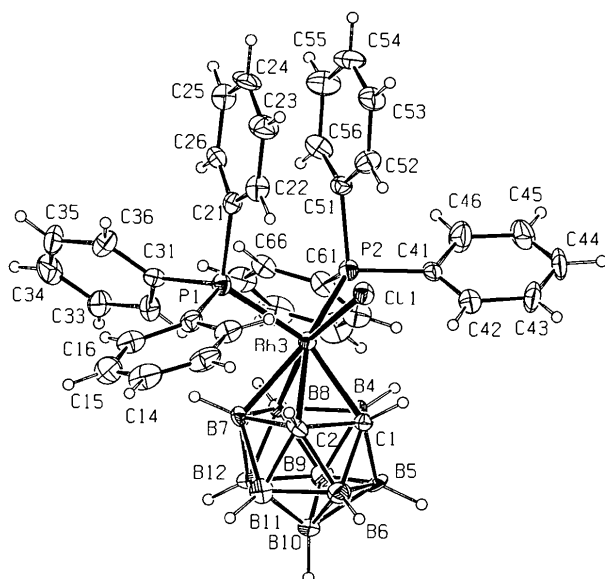


Fig. 1. A view of compound (3) with the numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

C2 atom is *trans* with respect to the chloride ligand, with a Cl—Rh—C7 angle of 172.4 (1)°, it appears that the difference in the Rh—C distances in the rhodacarborane cage of (2) is due to the different *trans* influences exerted by the *exo*-cage ligands.

The bonding patterns in the Rh3—B4—B8—B7 regions of compounds (3) and (4) are essentially the same. In both compounds, the Rh3—B8 distances fall between those of Rh3—B4 and Rh3—B7, while there is a notably wider spread of Rh—B distances in compound (3) [2.203 (8)–2.289 (8) Å] compared with (4) [2.223 (4)–2.254 (4) Å].

The conformation of the P₂ClRh unit above the C₂B₃ face of the carbaborane ligand in (3) is as expected on the basis of extended Hückel molecular-orbital calculations (Mingos, 1977), with the Rh—Cl bond located above the cage C—C vector. The same conformation was observed in compound (4). The Rh atom in compound (3), however, is not exactly symmetrically placed in the (PPh₃)₂Cl(C₂B₃)-ligand coordination sphere and angles subtended at the Rh atom show considerable variation, *viz.* P2—Rh3—B7 137.2 (2) and P1—Rh3—B4 157.7 (2)°, or P2—Rh3—C1 116.3 (2) and P1—Rh3—C2 101.9 (2)°, or Cl1—Rh3—C1 85.4 (2) and Cl1—Rh3—C2 92.6 (2)°.

Related interatomic distances within the C₂B₉ cage sections of compounds (3) and (4) are remarkably similar, as might be expected; the C1—C2 distances are 1.606 (9) and 1.618 (5) Å, the C—B distances 1.670 (11)–1.717 (10) and 1.690 (6)–1.733 (6) Å, and the B—B distances 1.727 (12)–1.827 (10) and 1.748 (7)–1.825 (6) Å in compounds (3) and (4), respectively. There are no unusual intermolecular contacts.

Experimental

closo-[3,3-(PPh₃)₂-3-H-3,1,2-RhC₂B₉H₁₁] [(1); 0.075 g, 0.09 mmol], CH₂Cl₂ (30 ml) and HC≡CCMe₂NH₂ (0.008 g, 0.09 mmol) were introduced into a thick-walled glass microwave reaction vessel (Baghurst & Mingos, 1992). The solution was subjected to microwave irradiation (650 W) for 5 min. Preparative thin-layer chromatography (TLC) afforded the title compound, (3), in 10% yield. Crystals suitable for X-ray analysis were grown by evaporation of a CH₂Cl₂ solution of the compound.

Crystal data

[RhCl(C₂H₁₁B₉)(C₁₈H₁₅P)₂].-

1.1CH₂Cl₂

M_r = 888.8

Triclinic

P $\bar{1}$

a = 11.568 (2) Å

b = 13.156 (3) Å

c = 15.387 (5) Å

α = 78.54 (3)°

β = 80.39 (2)°

γ = 81.39 (2)°

V = 2246.3 (10) Å³

Z = 2

D_x = 1.314 Mg m⁻³

D_m not measured

Mo K α radiation

λ = 0.7107 Å

Cell parameters from 25

reflections

θ = 7.2–13.0°

μ = 0.67 mm⁻¹

T = 294 (1) K

Plate

0.38 × 0.35 × 0.17 mm

Red

Data collection

Enraf–Nonius CAD-4 diffractometer

$\theta/2\theta$ scans

Absorption correction:

ψ scans at 4° steps

(North, Phillips & Mathews, 1968)

T_{min} = 0.78, *T_{max}* = 0.89

7854 measured reflections

7854 independent reflections

3445 observed reflections

[*I* > 2 σ (*I*)]

θ_{\max} = 25°

h = -13 → 13

k = 0 → 15

l = -17 → 18

3 standard reflections

frequency: 120 min

intensity decay: 6.2%

Refinement

Refinement on *F*²

R[*F*² > 2 σ (*F*²)] = 0.0610

wR(*F*²) = 0.1364

S = 0.852

7854 reflections

460 parameters

H atoms riding (C—H 0.93–1.10 and B—H 1.10 Å)

w = 1/[$\sigma^2(F_o^2) + (0.048P)^2$]

where *P* = (*F_o*² + 2*F_c*²)/3

(Δ/σ)_{max} < 0.001

$\Delta\rho_{\max}$ = 0.492 e Å⁻³

$\Delta\rho_{\min}$ = -0.767 e Å⁻³

Extinction correction: none

Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)

	$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$			
	<i>x</i>	<i>y</i>	<i>z</i>	<i>U_{eq}</i>
Rh3	0.28673 (5)	0.20011 (5)	0.19429 (4)	0.0271 (2)
Cl1	0.3292 (2)	0.33781 (14)	0.06752 (11)	0.0396 (5)
P1	0.4373 (2)	0.25555 (15)	0.25608 (12)	0.0341 (5)
P2	0.1213 (2)	0.30962 (15)	0.25154 (12)	0.0322 (5)

C1	0.2569 (6)	0.1123 (5)	0.0948 (4)	0.030 (2)
C2	0.3865 (6)	0.0787 (5)	0.1232 (4)	0.032 (2)
B4	0.1470 (8)	0.0987 (6)	0.1837 (5)	0.037 (2)
B5	0.1816 (8)	0.0070 (6)	0.1127 (5)	0.038 (2)
B6	0.3343 (8)	-0.0027 (7)	0.0702 (6)	0.046 (2)
B7	0.3796 (8)	0.0439 (6)	0.2374 (5)	0.034 (2)
B8	0.2261 (7)	0.0521 (6)	0.2801 (5)	0.029 (2)
B9	0.1628 (8)	-0.0333 (7)	0.2302 (6)	0.043 (2)
B10	0.2783 (8)	-0.0997 (7)	0.1608 (6)	0.043 (2)
B11	0.4074 (8)	-0.0495 (7)	0.1623 (6)	0.043 (2)
B12	0.3070 (8)	-0.0690 (7)	0.2623 (6)	0.044 (2)
C11	0.5826 (6)	0.2057 (6)	0.1998 (5)	0.039 (2)
C12	0.6118 (7)	0.2291 (6)	0.1076 (5)	0.044 (2)
C13	0.7205 (8)	0.1906 (7)	0.0669 (6)	0.060 (3)
C14	0.8009 (7)	0.1313 (8)	0.1161 (8)	0.073 (3)
C15	0.7735 (8)	0.1026 (7)	0.2064 (7)	0.068 (3)
C16	0.6649 (8)	0.1400 (6)	0.2488 (6)	0.055 (2)
C21	0.4466 (6)	0.3970 (6)	0.2452 (5)	0.037 (2)
C22	0.4955 (6)	0.4556 (6)	0.1659 (5)	0.043 (2)
C23	0.5016 (7)	0.5607 (7)	0.1619 (6)	0.063 (3)
C24	0.4590 (7)	0.6096 (6)	0.2333 (7)	0.064 (3)
C25	0.4068 (7)	0.5501 (7)	0.3114 (6)	0.062 (2)
C26	0.4003 (7)	0.4445 (6)	0.3172 (5)	0.045 (2)
C31	0.4493 (6)	0.2105 (5)	0.3745 (5)	0.034 (2)
C32	0.3780 (7)	0.1396 (6)	0.4283 (5)	0.048 (2)
C33	0.3864 (8)	0.1045 (6)	0.5174 (5)	0.054 (2)
C34	0.4707 (9)	0.1367 (7)	0.5562 (5)	0.070 (3)
C35	0.5416 (8)	0.2054 (7)	0.5032 (6)	0.067 (3)
C36	0.5323 (7)	0.2422 (6)	0.4136 (5)	0.054 (2)
C41	0.0088 (6)	0.3528 (5)	0.1759 (5)	0.036 (2)
C42	0.0257 (7)	0.3415 (6)	0.0877 (5)	0.042 (2)
C43	-0.0602 (8)	0.3814 (7)	0.0344 (6)	0.064 (3)
C44	-0.1656 (8)	0.4318 (7)	0.0674 (7)	0.065 (3)
C45	-0.1853 (8)	0.4419 (7)	0.1552 (8)	0.074 (3)
C46	-0.0983 (8)	0.4051 (7)	0.2089 (6)	0.069 (3)
C51	0.1341 (6)	0.4416 (5)	0.2724 (5)	0.034 (2)
C52	0.1742 (7)	0.5133 (6)	0.1985 (5)	0.049 (2)
C53	0.1688 (7)	0.6164 (6)	0.2076 (6)	0.058 (2)
C54	0.1261 (7)	0.6493 (7)	0.2877 (7)	0.062 (3)
C55	0.0865 (8)	0.5784 (7)	0.3606 (6)	0.068 (3)
C56	0.0923 (7)	0.4732 (6)	0.3522 (6)	0.054 (2)
C61	0.0420 (6)	0.2447 (6)	0.3548 (5)	0.041 (2)
C62	-0.0580 (7)	0.1995 (6)	0.3572 (6)	0.051 (2)
C63	-0.1134 (8)	0.1458 (7)	0.4347 (7)	0.073 (3)
C64	-0.0649 (9)	0.1332 (7)	0.5150 (7)	0.077 (3)
C65	0.0326 (8)	0.1785 (6)	0.5137 (5)	0.060 (3)
C66	0.0890 (7)	0.2328 (6)	0.4337 (5)	0.046 (2)

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

Rh3—C11	2.423 (2)	C2—B11	1.670 (11)
Rh3—P1	2.383 (2)	B4—B5	1.745 (11)
Rh3—P2	2.373 (2)	B4—B8	1.827 (10)
Rh3—C1	2.190 (6)	B4—B9	1.736 (11)
Rh3—C2	2.191 (6)	B5—B6	1.773 (12)
Rh3—B4	2.289 (8)	B5—B9	1.766 (11)
Rh3—B7	2.203 (8)	B5—B10	1.779 (12)
Rh3—B8	2.259 (7)	B6—B10	1.797 (12)
P1—C11	1.849 (7)	B6—B11	1.733 (12)
P1—C21	1.852 (7)	B7—B8	1.784 (11)
P1—C31	1.822 (7)	B7—B11	1.808 (12)
P2—C41	1.842 (7)	B7—B12	1.760 (11)
P2—C51	1.858 (7)	B8—B9	1.779 (11)
P2—C61	1.818 (7)	B8—B12	1.770 (11)
C1—C2	1.606 (9)	B9—B10	1.798 (12)
C1—B4	1.706 (10)	B9—B12	1.788 (12)
C1—B5	1.698 (9)	B10—B11	1.727 (12)
C1—B6	1.716 (11)	B10—B12	1.783 (12)
C2—B6	1.698 (11)	B11—B12	1.766 (12)
C2—B7	1.717 (10)		
C11—Rh3—P1	85.85 (7)	P1—Rh3—B4	157.7 (2)
C11—Rh3—P2	90.07 (7)	P1—Rh3—P2	99.43 (7)
C11—Rh3—C1	85.4 (2)	P2—Rh3—C1	116.3 (2)
C11—Rh3—C2	92.6 (2)	P2—Rh3—B7	137.2 (2)
P1—Rh3—C1	143.2 (2)	C1—Rh3—C2	43.0 (2)
P1—Rh3—C2	101.9 (2)		

Molecule (3) crystallized in the triclinic system; space group $P\bar{1}$ was assumed and confirmed by the analysis. The intensities of three standard reflections decayed by 6.2% during the course of the data collection; this was allowed for by appropriate scaling during data reduction. It became obvious from examination of difference maps that there were disordered solvent molecules in two distinct locations in the asymmetric unit. The crystals used for the analysis had been grown from pure dichloromethane, which is thus the only choice, but the geometry pattern of the 'solvent' peaks in the difference-map sections did not correspond with any ordered orientation (or simple disordered orientation) of dichloromethane molecules. Accordingly, the contribution of the density of the dichloromethane molecules was subtracted from the measured structure factors using the *SQUEEZE* (Spek, 1994) option in *PLATON95* (Spek, 1995a). This indicated a total occupancy of 1.1 (1) for the disordered dichloromethane over the two sites in the asymmetric unit. Subsequent refinement converged with lower R factors than when attempts had been made to model various dichloromethane contributions; there were no significant changes to the geometry of the Rh-containing molecule.

Data collection: *CAD-4-PC Software* (Enraf-Nonius, 1992). Cell refinement: *SET4* and *CELDIM* (Enraf-Nonius, 1992). Data reduction: *DATRD2* in *NRCVAX94* (Gabe, Le Page, Charland, Lee & White, 1989). Program(s) used to solve structure: *NRCVAX94* (Patterson heavy-atom method). Program(s) used to refine structure: *NRCVAX94* and *SHELXL93* (Sheldrick, 1993). Molecular graphics: *NRCVAX94*, *ORTEPII* (Johnson, 1976), *PLATON* and *PLUTON* (Spek, 1995b). Software used to prepare material for publication: *NRCVAX94*, *SHELXL93* and *WordPerfect* (macro *PREPCIF*).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1408). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

- Baghurst, D. R. & Mingos, D. M. P. (1992). *J. Chem. Soc. Dalton Trans.* pp. 1151–1155.
- Baker, R. T., Delaney, M. S., King, R. E., Knobler, C. B., Long, J. A., Marder, T. B., Paxson, T. E., Teller, R. G. & Hawthorne, M. F. (1984). *J. Am. Chem. Soc.* **106**, 2965–2978.
- Enraf-Nonius (1992). *CAD-4-PC Software*. Version 1.1. Enraf-Nonius, Delft, The Netherlands.
- Ferguson, G., Pollack, J., McEaney, P. A., O'Connell, D. P., Spalding, T. R., Gallagher, J. F., Macias, R. & Kennedy, J. D. (1996). *J. Chem. Soc. Chem. Commun.* pp. 679–681.
- Gabe, E. J., Le Page, Y., Charland, J.-P., Lee, F. L. & White, P. S. (1989). *J. Appl. Cryst.* **22**, 384–387.
- Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
- Mingos, D. M. P. (1977). *J. Chem. Soc. Dalton Trans.* pp. 602–610.
- North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* **A24**, 351–359.
- Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.

- Spek, A. L. (1994). *Am. Crystallogr. Assoc. Meet. Abstr.* Paper M05.
 Spek, A. L. (1995a). *PLATON. Molecular Geometry Program.*
 University of Utrecht, The Netherlands.
 Spek, A. L. (1995b). *PLUTON. Molecular Graphics Program.*
 University of Utrecht, The Netherlands.

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(4-Nitrophenyl 1,3,5,8,12-pentaaza-cyclotetradec-3-yl ketone-*N*¹,*N*⁵,*N*⁸,*N*¹²)-copper(II) Dinitrate

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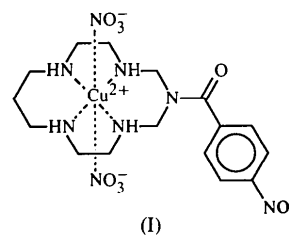
Abstract

In the crystal structure of $[\text{Cu}(\text{C}_{16}\text{H}_{26}\text{N}_6\text{O}_3)](\text{NO}_3)_2$, the metal ion lies in an elongated octahedral coordination. Only the four secondary amine N atoms of the macrocycle are bound to the Cu^{2+} centre which resides 0.080(1) Å out of the N_4 donor plane. The coordination sphere is completed by two nitrate O atoms axially coordinated with different Cu—O distances.

Comment

Pentaaza macrocyclic systems, known as azacyclams, are obtained through one-pot template syntheses in which Cu^{2+} or Ni^{2+} ions behave as templating agents. These reactions involve the closure of an open-chain tetraamine, preoriented through coordination to the metal centre, by a primary amine, amide or sulfonamide group in the presence of excess formaldehyde and base, and offer the opportunity to append a variety of substituent groups onto the aza macrocyclic system (Fabbrizzi *et al.*, 1991; De Blas *et al.*, 1993; Abba *et al.*, 1994). The crystal structure determination of this class of compounds allows the influence of the substituent group on the spectral and electrochemical properties of the complexes to be defined (*e.g.* by comparing the distortions in the square-planar or octahedral geometries). Structures of azacyclam complexes of both Cu^{2+} and Ni^{2+} ions have been determined in recent years (Fabbrizzi *et al.*, 1991; De Blas *et al.*, 1993; Abba *et al.*, 1994), but no crystallographic characterization of a Cu^{2+} –azacyclam complex containing an amido group has been reported previously.

The crystal structure of the title complex, (I), shows copper(II) in an elongated octahedral coordination environment whose deviation from a regular geometry is only partially ascribable to the Jahn–Teller effect (Fig. 1). Only the four secondary amine N atoms of the macrocycle are bound to the metal centre giving a distorted square-planar geometry in which (i) two *trans*-N atoms (N2 and N4) lie up [$\delta(\text{N}2) = 0.036(2)$ and $\delta(\text{N}4) = 0.037(2)$ Å] and the other two (N3 and N5) lie down [$\delta(\text{N}3) = -0.037(2)$ and $\delta(\text{N}5) = -0.037(2)$ Å] with respect to the N_4 mean plane (tetrahedral distortion), and (ii) the Cu^{2+} ion resides 0.080(1) Å out of the N_4 donor plane. The first coordination sphere of the metal ion is completed by two nitrate O atoms (O1 and O6) coordinated axially, with Cu—O distances that are quite different [Cu—O1 2.437(2) and Cu—O6 2.673(2) Å]. This is probably related to the Cu^{2+} displacement from the secondary amine plane towards the nitrate O1 atom. Although the O1—Cu—O6 angle [$168.6(1)^\circ$] does not deviate excessively from linearity, the O—Cu—N angle values, varying in the range $81.9(1)$ – $98.7(1)^\circ$, are indicative of a significant distortion from a regular octahedron.



It is interesting to compare the copper geometry observed here with that found in the structure of (3-methanesulfonyl-1,3,5,8,12-pentaazacyclotetradecane)-dinitratocopper(II) (De Blas *et al.*, 1994). Although the Cu—N bond distances and angles and the average Cu—O distance are very similar, De Blas and co-workers

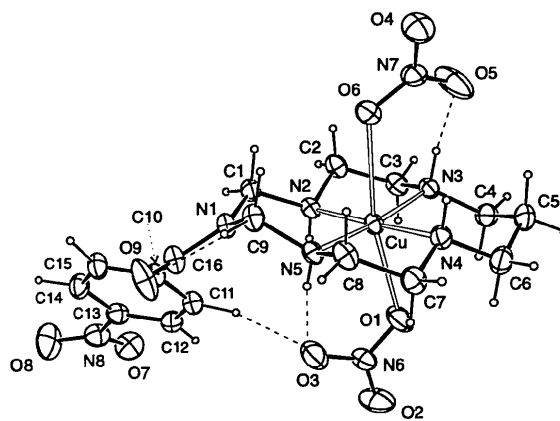


Fig. 1. A perspective view of the title complex shown with 30% probability ellipsoids.