it is likely that its origin lies in crystal-packing effects. Although there are no $H_{cage}\cdots H_{cation}$ contacts $< 2{\cdot}6$ Å, it is clear from the packing diagram (Fig. 3) that the two sides of the $\{B_{10}H_{12}\}$ ligand [that containing B(*a*) and that containing B(*b*)] experience quite different crystal environments. Distances and angles within the $[PhCH_2NMe_3]^+$ cation are quite normal (*e.g.* Mitchell & Welch, 1987; Wynd & Welch, 1989; Macgregor, Yellowlees & Welch, 1990).

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References

BRILL, R., DIETRICH, H. & DIERKS, H. (1971). *Acta Cryst.* B**27**, 2003–2018.

FRITCHIE, C. J. (1967). *Inorg. Chem.* **6**, 1199–1203.

GOULD, R. O. & SMITH, D. E. (1986). *CADABS*. Program for data reduction. Univ. of Edinburgh, Scotland.

GOULD, R. O. & TAYLOR, P. (1986). *CALC*. Program for molecular geometry calculations. Univ. of Edinburgh, Scotland.

GUGGENBERGER, L. J. (1972). *J. Am. Chem. Soc.* **94**, 114–119.

Internatinal Tables for X-ray Crystallograpy (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

KLANBERG, F., WEGNER, P. A., PARSHALL, G. W. & MUETTERTIES, E. L. (1968). *Inorg. Chem.* **7**, 2072–2077.

MACGREGOR, S. A., YELLOWLEES, L. J. & WELCH, A. J. (1990). *Acta Cryst.* C**46**, 551–554.

MALLINSON, P. & MUIR, K. W. (1985). *J. Appl. Cryst.* **18**, 51–53.

MITCHELL, G. F. & WELCH, A. J. (1987). *J. Chem. Soc. Dalton Trans.* pp. 1017–1025.

MOTHERWELL, W. D. S. (1976). *PLUTO*. Program for molecular and crystal structure illustrations. Univ. of Cambridge, England.

SHELDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

WALKER, N. G. & STUART, D. (1983). *Acta Cryst.* A**39**, 158–166.

WYND, A. J. (1988). PhD Thesis, Univ. of Edinburgh, Scotland.

WYND, A. J. & WELCH, A. J. (1987). *J. Chem. Soc. Chem. Commun.* pp. 1174–1176.

WYND, A. J. & WELCH, A. J. (1989). *Acta Cryst.* C**45**, 615–617.

WYND, A. J., WELCH, A. J. & PARISH, R. V. (1990). *J. Chem. Soc. Dalton Trans.* In the press.

Acta Cryst. (1990). C**46**, 1402–1405

A (Dithioformato)rhodadicarbaborane Complex: 3-(Dithioformato-*S*,*S'*)-3-(triphenylphosphine)-1,2-dicarba-3-rhoda-*closo*-dodecaborane(12) Cyclohexane Solvate, $[3\text{-}(S_2CH\text{-}S,S')\text{-}3\text{-}(PPh_3)\text{-}3,1,2\text{-}RhC_2B_9H_{11}].C_6H_{12}$

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Abstract. $C_{21}H_{27}B_9PRhS_2.C_6H_{12}$, $M_r = 658{\cdot}9$, monoclinic, $P2_1/n$, $a = 20{\cdot}085\,(5)$, $b = 16{\cdot}055\,(3)$, $c = 10{\cdot}074\,(2)$ Å, $\beta = 98{\cdot}69\,(2)°$, $V = 3211\,(2)$ Å^3, $Z = 4$, $D_x = 1{\cdot}36$ g cm^{-3}, $\lambda = 0{\cdot}70926$ Å, $\mu(\text{Mo } K\alpha) = 7{\cdot}1$ cm^{-1}, $F(000) = 1352$, $T = 294$ K, $R = 0{\cdot}029$ for 5595 observed reflections. The Rh atom is bonded symmetrically to the bidentate S_2CH ligand [Rh—S 2·352 (1) and 2·356 (1) Å]. Each S atom is *trans* to a C atom in the C_2B_3 face of the C_2B_9-carbaborane ligand which is bonded to the Rh atom [Rh—C 2·195 (3) and 2·201 (3), Rh—B 2·204 (3), 2·205 (3) and 2·239 (3), and C—C 1·631 (3) Å]. The C—Rh—S angles are 164·29 (6) and 165·88 (6)°. The Rh—P distance is 2·374 (1) Å and the PPh_3 ligand is opposite the unique B atom in the C_2B_3 face. The P—Rh—B angle is 175·35 (8)°.

Introduction. A study of the reactions of CS_2 with the rhodaheteroborane complexes $[2,2\text{-}(PPh_3)_2\text{-}2\text{-}(H)\text{-}1,2\text{-}X\text{RhB}_{10}H_{10}]$ [(I) X = Se, Te] has produced several interesting products (Faridoon, Spalding, Ferguson, Kennedy & Fontaine, 1989) including the dithio-

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formato complex [2-(S_2CH-*S*,*S*′)-2-(PPh_3)-1,2-$SeRhB_{10}H_{10}$] (II). The crystal and molecular structure of (II) have been previously reported by us (Ferguson, Faridoon & Spalding, 1988). We have extended our studies to reactions of CS_2 with rhodacarbaboranes including [3,3-$(PPh_3)_2$-3-(H)-3,1,2-$RhC_2B_9H_{11}$] (Callahan & Hawthorne, 1976) which is a Wadian analogue of the rhodaheteroboranes (I). We have isolated the rhodacarbaborane equivalent of (II), *i.e.* [3-(S_2CH-*S*,*S*′)-3-(PPh_3)-3,1,2-$RhC_2B_9H_{11}$)] (III) and find some significant differences in the bonding of the S_2CH-*S*,*S*′ ligand to the Rh atom in (III) and (II).

Table 1. *Positional and thermal parameters and their e.s.d.'s*

Atoms C*H*1 to C*H*6′ are the carbons of the disordered cyclohexane of solvation; atoms C*H*1 and C*H*4 have occupancies of 1·0, the others have 0·5 occupancies.

	x	*y*	*z*	*B*(Å²)*
Rh3	0·12331 (1)	0·12614 (1)	0·12578 (2)	2·980 (3)
S1	0·03436 (3)	0·07451 (5)	0·22943 (7)	4·44 (1)
S2	0·13328 (4)	0·18339 (5)	0·34328 (7)	4·94 (1)
C(*S*)	0·0655 (2)	0·1292 (2)	0·3631 (3)	5·22 (6)
C1	0·1875 (1)	0·1653 (2)	−0·0209 (3)	3·83 (5)
C2	0·1310 (1)	0·0977 (2)	−0·0851 (2)	3·40 (4)
B4	0·1519 (2)	0·2478 (2)	0·0501 (3)	4·34 (6)
B5	0·1801 (2)	0·2554 (2)	−0·1094 (4)	5·06 (7)
B6	0·1677 (2)	0·1595 (2)	−0·1932 (3)	4·41 (6)
B7	0·0521 (1)	0·1285 (2)	−0·0636 (3)	3·56 (5)
B8	0·0614 (1)	0·2289 (2)	0·0196 (3)	4·02 (6)
B9	0·1005 (2)	0·2943 (2)	−0·0879 (4)	5·00 (7)
B10	0·1104 (2)	0·2402 (2)	−0·2378 (3)	4·66 (6)
B11	0·0817 (2)	0·1372 (2)	−0·2204 (3)	3·95 (6)
B12	0·0387 (1)	0·2203 (2)	−0·1567 (3)	4·17 (6)
P	0·19471 (3)	0·01791 (3)	0·22468 (6)	3·05 (1)
C11	0·2017 (1)	−0·0638 (1)	0·1016 (2)	3·18 (4)
C12	0·2621 (1)	−0·0893 (2)	0·0659 (3)	4·74 (6)
C13	0·2633 (2)	−0·1527 (2)	−0·0297 (3)	6·49 (7)
C14	0·2054 (2)	−0·1908 (2)	−0·0860 (3)	5·98 (7)
C15	0·1444 (2)	−0·1641 (2)	−0·0535 (3)	5·07 (6)
C16	0·1427 (1)	−0·0999 (2)	0·0374 (2)	4·05 (5)
C21	0·2801 (1)	0·0511 (2)	0·2855 (2)	3·70 (5)
C22	0·3287 (1)	−0·0066 (2)	0·3421 (3)	4·54 (6)
C23	0·3941 (1)	0·0182 (2)	0·3842 (3)	5·62 (7)
C24	0·4120 (1)	0·1007 (2)	0·3735 (3)	5·78 (7)
C25	0·3645 (1)	0·1586 (2)	0·3216 (3)	5·18 (6)
C26	0·2987 (1)	0·1343 (2)	0·2769 (3)	4·28 (5)
C31	0·1698 (1)	−0·0360 (1)	0·3694 (2)	3·41 (4)
C32	0·1945 (1)	−0·0102 (2)	0·4987 (3)	4·53 (6)
C33	0·1722 (2)	−0·0474 (2)	0·6067 (3)	5·53 (7)
C34	0·1254 (2)	−0·1104 (2)	0·5903 (3)	5·47 (7)
C35	0·1007 (2)	−0·1369 (2)	0·4620 (3)	5·71 (7)
C36	0·1225 (1)	−0·0997 (2)	0·3531 (3)	4·64 (6)
C*H*1	0·3533 (3)	0·0605 (5)	−0·1427 (7)	14·2 (2)
C*H*2	0·3795 (5)	0·1470 (6)	−0·0932 (11)	10·9 (3)
C*H*3	0·4476 (4)	0·1694 (5)	−0·1368 (10)	10·6 (2)
C*H*4	0·4878 (3)	0·1196 (5)	−0·1580 (9)	18·1 (3)
C*H*5	0·4519 (6)	0·0247 (6)	−0·2252 (9)	11·3 (3)
C*H*6	0·3892 (5)	0·0057 (6)	−0·1712 (13)	13·0 (3)
C*H*2′	0·3766 (5)	0·0630 (7)	−0·2660 (8)	9·7 (3)
C*H*3′	0·4451 (5)	0·1073 (8)	−0·2641 (9)	12·4 (3)
C*H*5′	0·4542 (6)	0·1296 (6)	−0·0216 (11)	11·6 (3)
C*H*6′	0·3893 (6)	0·0778 (10)	−0·0280 (10)	16·0 (4)

* Anisotropically refined atoms are given in the form of the isotropic equivalent thermal parameter defined as: $\frac{4}{3}(a^2B_{11} + b^2B_{22} + c^2B_{33} + ab\cos\gamma B_{12} + ac\cos\beta B_{13} + bc\cos\alpha B_{23})$.

Experimental. A yellow flat plate crystal having dimensions 0·25 × 0·49 × 0·59 mm was selected from several similar crystals grown from a cyclohexane–dichloromethane solution. Accurate cell dimensions and crystal orientation matrix were determined on a CAD-4 diffractometer by a least-squares treatment of the setting angles of 25 reflections in the range $8 < \theta < 17°$. Intensities of reflections with indices *h* −24 to 24, *k* 0 to 19, *l* 0 to 12, with $2 < 2\theta < 54°$ measured; ω–2θ scans, ω-scan width $(0·60 + 0·35\tan\theta)°$; graphite-monochromated Mo *Kα* radiation; intensities of three reflections measured every 2 h showed no evidence of crystal decay. 7640 reflections measured, 6994 unique ($R_{\rm int}$ 0·022) and the 5595 with $I > 3\sigma(I)$ labelled observed were used in structure solution and refinement. Data corrected for Lorentz, polarization and absorption effects (max. and min. transmission coefficients 1·171, 0·864). Space group $P2_1/n$ uniquely from the systematic absences (*h*0*l* absent if $h + l = 2n + 1$; 0*k*0 absent if $k = 2n + 1$). The structure was solved by the Patterson heavy-atom method and subsequent difference syntheses. Refinement was by full-matrix least-squares calculations, initially with isotropic and then with anisotropic thermal parameters. At an intermediate stage in the refinement a difference map showed that there was cyclohexane of solvation trapped in the crystal lattice; there is one cyclohexane per molecule of the complex and it is disordered over two orientations in what would have been a void in the crystal lattice.* Subsequent difference maps also showed maxima in positions consistent with the expected locations of the H atoms of the complex; in the final rounds of calculations these H atoms were positioned on geometrical grounds (C—H 0·95, B—H 1·08 Å) and included (as riding atoms) in the structure factor calculations with an overall $B_{\rm iso}$ of 5·0 Å². The final cycle of refinement included 289 variable parameters, $R = 0·029$, $wR = 0·044$, goodness-of-fit 1·79, $w = 1/[\sigma^2(F_o) + 0·055(F_o)^2]$. Max. shift/e.s.d. in final cycle 0·01; density in final difference map ±0·43 e Å^{-3} in the vicinity of the disordered cyclohexane; there were no chemically significant features. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974). All calculations were performed on a PDP11/73 computer using *SDP-Plus* (Frenz, 1983). Atomic coordinates and details of bond lengths are given in Tables 1 and 2. Figs. 1 and 2 are views of the molecule prepared using *ORTEP*II (Johnson, 1976).

Discussion. Fig. 1 shows an overall view of molecule (III) and Fig. 2 is a view of the coordination about

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* An *ORTEP* plot showing the two orientations of the disordered cyclohexane of solvation, a section through a three-dimensional difference map to show the hydrogen of the HCS_2 ligand, full details of molecular geometry, and lists of calculated hydrogen coordinates, thermal parameters, mean-plane data and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52770 (69 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Bond lengths* (Å)

Rh3—S1	2·352 (1)	B4—B9	1·767 (4)
Rh3—S2	2·356 (1)	B5—B6	1·755 (5)
Rh3—C1	2·195 (3)	B5—B9	1·760 (5)
Rh3—C2	2·201 (3)	B5—B10	1·773 (4)
Rh3—B4	2·204 (3)	B6—B10	1·746 (4)
Rh3—B7	2·205 (3)	B6—B11	1·746 (4)
Rh3—B8	2·239 (3)	B7—B8	1·814 (4)
Rh3—P	2·374 (1)	B7—B11	1·775 (4)
S1—C(*S*)	1·650 (3)	B7—B12	1·747 (4)
S2—C(*S*)	1·653 (3)	B8—B9	1·775 (5)
C1—C2	1·631 (3)	B8—B12	1·771 (4)
C1—B4	1·713 (4)	B9—B10	1·779 (5)
C1—B5	1·694 (4)	B9—B12	1·782 (4)
C1—B6	1·723 (4)	B10—B11	1·769 (4)
C2—B6	1·719 (4)	B10—B12	1·789 (5)
C2—B7	1·706 (4)	B11—B12	1·762 (4)
C2—B11	1·683 (3)	P—C11	1·825 (2)
B4—B5	1·787 (5)	P—C21	1·812 (2)
B4—B8	1·822 (4)	P—C31	1·829 (2)

Rh from a viewpoint normal to the plane containing the C_2B_3 face. The bonding about the Rh atom is remarkably symmetrical (Table 2 and Fig. 2), with Rh3—S1 [2·352 (1) Å] and Rh3—S2 [2·356 (1) Å] and equal Rh3—B4 [2·204 (3) Å] and Rh3—B7 [2·205 (3) Å] distances. Likewise the bonding in the S_2CH ligand is symmetrical [S1—CS 1·650 (3), S2—CS 1·653 (3) Å]. The geometry of the [(S_2CH), PPh_3] ligand set above the C_2B_3 face is not unexpected in terms of the postulated molecular orbital interactions that occur between 7,8-$C_2B_9H_{11}$ ligands and Rh(L)L'_2 moieties (Mingos, Forsyth & Welch, 1978; Faridoon, Ni Dhubhghaill, Spalding, Ferguson, Kaitner, Fontaine, Kennedy & Reed, 1988). However the details of the configuration of the *exo*-cage ligands in (III) are not the same as those in (II), see Fig. 3. Compound (II) shows a much less symmetric bonding environment at rhodium [*e.g.* Rh—S1 (*trans* to Se) 2·328 (1) and Rh—S2 (opposite the B3–B4 vector) 2·406 (1) and S1—C1 1·669 (4), S2—C1 1·650 (5) Å] and there is no plane of symmetry bisecting both the S_2CH ligand and the C_2B_9 cage, and containing the Rh–P vector as there is in the present structure (III).

The rhodium-to-C_2B_9-cage bond distances for Rh3—C1, —C2, —B4 and —B7 are not significantly different and lie in the narrow range 2·195 (3) to 2·205 (3) Å. However, the Rh3—B8 distance of 2·239 (3) Å is significantly longer and may reflect the *trans* influence of the PPh_3 ligand opposite B8. These values are not atypical of Rh—C and Rh—B distances which have been reported for 3,1,2-RhC_2B_9 complexes containing a wide variety of *exo*-cage ligand sets including [$(Ph_3P)_2$, H] (Hardy, Callahan, Strouse & Hawthorne, 1976), [$(PPh_3)_2$, (HSO_4)] (Kalb, Teller & Hawthorne, 1979), [PPh_3, $(Br)_2$] (Zheng, Baker, Knobler, Walker & Hawthorne, 1983), [(PPh_3), (NO_3)] (Demidowicz, Teller & Hawthorne, 1979) and {(PPh_3), [C(Ph)C(PPh_3)-CHCPh]} (Kalb, Demidowicz, Speckman, Knobler, Teller & Hawthorne, 1982). Likewise the C—B and B—B distances in the C_2B_9 cage are not unusual (Table 2). The Rh3—P distance [2·374 (1) Å] in (III) is within the range of values reported for the compounds above [2·301 (1) to 2·387 (4) Å].

The NMR properties of (III) were as follows, measured in CD_2Cl_2 solution at 294–297 K, ordered

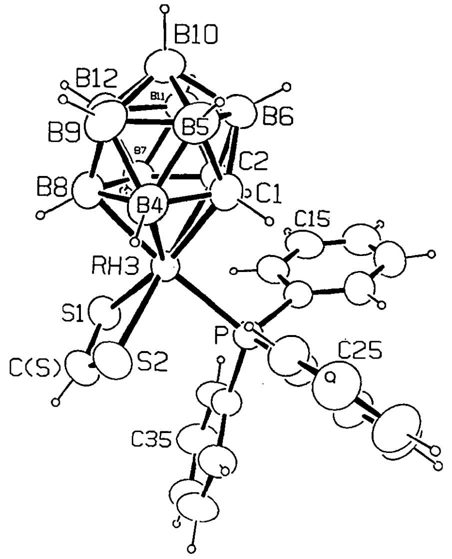

Fig. 1. A general view of [3-(S_2CH-*S*,*S'*)-3-(PPh_3)-3,1,2-$RhC_2B_9H_{11}$] (III) with our numbering scheme. Ellipsoids are at the 50% level. Phenyl-ring carbon atoms are numbered C*i*1–C*i*6, *i* = 1, 2, 3.

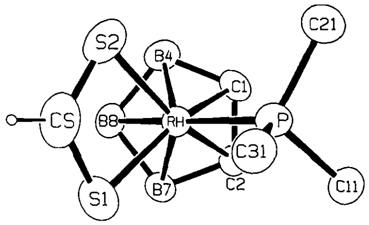

Fig. 2. A view of the Rh coordination in (III) from a viewpoint normal to the C_2B_3 plane showing the orientation of the S_2CH-*S*,*S'* and phosphine ligands on the Rh atom.

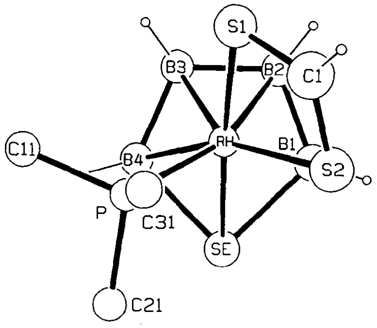

Fig. 3. A similar view to that of Fig. 2 showing the Rh coordination and ligand orientation in [2-(S_2CH-*S*,*S'*)-2-(PPh_3)-1,2-$SeRhB_{10}H_{10}$] (II).

as: relative intensity $\delta(^{11}B)$ (p.p.m.) $\{\delta(^{1}H)$ (p.p.m.) $[^{1}J(^{11}B–^{1}H)/Hz]\}$: 1BH $+6·0$ [$+3·55$ (144)]; 1BH $+3·6$ [$+2·64$ (144)]; 2BH *ca* $-5·3$ [$+2·43$ (not resolved)]; 2BH $-6·1$ [$+2·04$ (139)]; 2BH $-16·7$ [$+1·55$ ($\geq$*ca* 158)]; 1BH $-21·2$ [$+1·60$ (not resolved)]; 2CH $-$[$+3·19$ (—)]. 1HCS$_2$ $-\{+11·42$ [$^{3}J(^{103}Rh–^{1}H)$ and $^{4}J(^{31}P–^{1}H)$ 7·3 and 3·8 Hz]$\}$. These data are entirely consistent with the structure derived from our X-ray study, confirming that the crystal selected for the X-ray analysis was indeed representative of the bulk sample.

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References

CALLAHAN, K. P. & HAWTHORNE, M. F. (1976). *Adv. Organomet. Chem.* **14**, 145–186.

DEMIDOWICZ, Z., TELLER, R. G. & HAWTHORNE, M. F. (1979). *J. Chem. Soc. Chem. Commun.* pp. 831–832.

FARIDOON, NI DHUBHGHAILL, O., SPALDING, T. R., FERGUSON, G., KAITNER, B., FONTAINE, X. L. R., KENNEDY, J. D. & REED, D. (1988). *J. Chem. Soc. Dalton Trans.* pp. 2739–2745.

FARIDOON, SPALDING, T. R., FERGUSON, G., KENNEDY, J. D. & FONTAINE, X. L. R. (1989). *J. Chem. Soc. Chem. Commun.* pp. 906–908.

FERGUSON, G., FARIDOON & SPALDING, T. R. (1988). *Acta Cryst.* C**44**, 1368–1371.

FRENZ, B. A. (1983). *Enraf–Nonius Structure Determination Package*; *SDP Users Guide*. Version of 6 January 1983. Enraf–Nonius, Delft, The Netherlands.

HARDY, G. E., CALLAHAN, K. P., STROUSE, C. E. & HAWTHORNE, M. F. (1976). *Acta Cryst.* B**32**, 264–266.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

JOHNSON, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

KALB, W. C., DEMIDOWICZ, Z., SPECKMAN, D. M., KNOBLER, C., TELLER, R. G. & HAWTHORNE, M. F. (1982). *Inorg. Chem.* **21**, 4027–4036.

KALB, W. C., TELLER, R. G. & HAWTHORNE, M. F. (1979). *J. Am. Chem. Soc.* **101**, 5417–5419.

MINGOS, D. M. P., FORSYTH, M. I. & WELCH, A. J. (1978). *J. Chem. Soc. Dalton Trans.* pp. 1363–1374.

ZHENG, L., BAKER, R. T., KNOBLER, C. B., WALKER, J. A. & HAWTHORNE, M. F. (1983). *Inorg. Chem.* **22**, 3350–3355.

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Tetraaqua-μ_4-(phenylenediaminetetracetato-O^1:O^2:O^3,O^4,N:O^5,O^6,N^1)-dicopper(II) Dihydrate

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Abstract. $[Cu(C_{14}H_{12}N_2O_8)(H_2O)_4].2H_2O$, $M_r = 571·45$, orthorhombic, *Pnab*, $a = 10·020$ (1), $b = 12·065$ (1), $c = 16·589$ (1) Å, $V = 2005·3$ (2) Å^3, $D_x = 1·893$ g cm^{-3}, $Z = 4$, $F(000) = 1168$, $\lambda(Cu\ K\alpha) = 1·54178$ Å, $\mu = 32·92$ cm^{-1}. Room temperature. Final $R = 0·06$ for 1549 unique observed reflections. The Cu ions have a highly distorted octahedral coordination geometry, with the *p*-phenylenediamine-*N*,*N*,*N'*,*N'*-tetraacetic (*p*-phdta) ligand and the O*W*1 water molecule at the vertices of the square and the O*W*2 water molecule and the O11 (through symmetry operation $x + \frac{1}{2}$, $-y$, z) occupying the apical positions. The conformations of the five-membered chelate rings appear to depend on hydrogen bonding and van der Waals interactions.

Introduction. The behaviour of aromatic diamines as coordinating agents depends on the relative positions of N atoms in the aromatic ring. Thus, for derivatives of *o*-phenylenediamine, the two vicinal nitrogens may become coordinated to the same metallic cation, in a single sphere of coordination, as has been established for the complexes of *o*-phenylenediamine-*N*,*N*,*N'*,*N'*-tetraacetic acid with Co^{II} (McCandlish, Michael, Neal, Lingafelter & Rose, 1978), Mn^{II} (Nakasuka, Azuma, Katayama,

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