(Chaudhuri & Wieghardt, 1987). For small metal ions ($M = Co^{III}$, Ni^{III}) the M—N bonds are stretched as a result of interligand and intraligand H···H repulsions (Thöm, Boeyens, McDougall & Hancock, 1984; Wieghardt *et al.*, 1986). The bond stretching can be substantial; for example, from 1.925 to 1.97 Å for the Co^{III}—N bond. A metal ion with the M—N bond length of 2.08 Å fits ideally between two tacn ligands. For larger metal ions like Cd^{2+} a, possibly small, M—N bond compression is predicted. It results from attractive van der Waals forces and the resistance of N—M—N bond angles to compression (Thöm *et al.*, 1984). For (1) and (2) we observe mean Cd—N bond lengths of 2.36 (1) and 2.36 (6) Å, respectively. The mean N—M—N angles within the chelate rings are 75.7 (4) and 75 (1)°, respectively. Comparison with the other CdN_6-type complexes mentioned in the *Introduction* does not indicate any statistically significant Cd—N bond compression in the case of $[Cd(tacn)_2]^{2+}$.

The N_6 coordination polyhedron of $[Cd(tacn)_2]^{2+}$ can be described as an octahedron strongly elongated along one of its threefold axes of rotation. Due to the position on a centre of inversion, three N—Cd—N angles are exactly 180°. Another consequence of the crystallographically imposed inversion symmetry is the fact that ligands bonded to the same Cd atom must be enantiomeric. Thus, the five-membered CdN_2C_2 chelate rings have λ conformation in one ligand and δ conformation in the adjacent one. The tacn macrocycles have the following average bond lengths (ranges of individual values in parentheses): (1) N—C 1.479 (7) [1.468 (4)–1.490 (4)], C—C 1.522 (7) [1.512 (5)–1.531 (5) Å]; (2) N—C 1.46 (5) [1.32 (2)–1.52 (1)], C—C 1.47 (13) [1.24 (2)–1.64 (3) Å]. The remaining parts of the structures do not show any unusual features.

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Structure of *nido*-$[8,8,8\text{-}(PMe_2Ph)_3\text{-}8,7\text{-}RhSB_9H_{10}]$

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Abstract. 8,8,8-Tris(dimethylphenylphosphine)-7-thia-8-rhoda-9,10-μH-*nido*-undecaborane(10), $C_{24}H_{43}B_9P_3RhS$, $M_r = 656.8$, monoclinic, $P2_1/c$, $a = 15.794$ (2), $b = 12.020$ (3), $c = 17.119$ (2) Å, $\beta = 98.21$ (1)°, $V = 3217$ (2) Å^3, $Z = 4$, $D_x = 1.35$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 7.5$ cm^{-1}, $F(000) = 1352$, $T = 294$ K, $R = 0.022$ for 5421 observed

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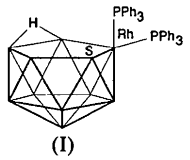
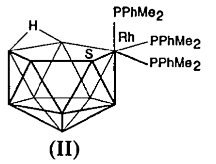
reflections. The title complex has a *nido* $RhSB_9$-cage structure with an open $RhSB_3$ face and Rh adjacent to S. The Rh atom is bonded to three PMe_2Ph ligands. One phosphine is *trans* to the S atom in the open face, a second is *trans* to the B atom bonded to Rh in the open face and the third is perpendicular to both these Rh—P bonds and the open $RhSB_3$ face. The Rh—P distances for these ligands are 2.2979 (5), 2.4538 (6) and 2.3869 (5) Å respectively. The Rh—S distance is 2.3757 (5) Å.

Introduction. The complex $[8,8\text{-}(PPh_3)_2\text{-}8,7\text{-}RhSB_9H_{10}]$ (I) is unusual because it has a Wadian *closo* electron count but a *nido* structure (Ferguson, Jennings, Lough, Coughlan, Spalding, Kennedy, Fontaine & Stibr, 1990). We now show that reaction of (I) with excess PMe_2Ph in dichloromethane affords a compound with a *nido* electron count and *nido* structure $[8,8,8\text{-}(PMe_2Ph)_3\text{-}8,7\text{-}RhSB_9H_{10}]$, (II), in 70% yield. We decided to investigate the structure of (II) and to compare it with (I) to ascertain what, if any, were the effects of the addition of the extra ligand at the Rh atom on the $RhSB_9$ cage.



(I)



(II)

Experimental. An orange plate crystal of (II) which measured 0.33 × 0.38 × 0.18 mm was mounted on a glass fibre with its long axis approximately parallel to the φ axis of the goniometer. The setting angles of 25 reflections with $10 < \theta < 18°$ were measured on a CAD-4 diffractometer and used to determine accurate cell constants and crystal orientation matrix. Intensities of reflections with indices h 0 to 20, k 0 to 15, l −21 to 21 with $2 < 2\theta < 54°$ measured [ω–2θ scans; ω-scan width $(0.6 + 0.35\tan\theta)°$] with graphite-monochromated Mo $K\alpha$ radiation. Lorentz, polarization and absorption corrections (Gaussian integration, transmission coefficients 0.766 to 0.888) were applied. The intensities of three standard reflections measured every 120 min remained constant and no decay correction was required. 7791 reflections were measured, 7003 unique ($R_{\text{int}} = 0.012$), and 5421 had $I > 3\sigma(I)$. These were labelled observed and used in the structure analysis. Space group $P2_1/c$ (systematic absences $h0l$ if $l = 2n + 1$; $0k0$ if $k = 2n + 1$) confirmed by successful least-squares refinement. The structure was solved by the heavy-atom method and remaining non-H atoms found from subsequent difference syntheses. All H atoms were visible in difference maps at intermediate stages of the refinement. The non-bridging H atoms were included in the calculations restrained to ride on the atom to which they were bonded (C—H 0.95 and B—H 1.08 Å). The H atom bridging B9 and B10 was included and refined with an isotropic thermal parameter using as starting coordinates the position found from a difference map. Refinement was by full-matrix least-squares calculations on F, initially with isotropic and finally with anisotropic thermal parameters for all non-H atoms. The final cycle of refinement included 347 variable parameters. $R = 0.022$, $wR = 0.030$, goodness of fit 1.29, $w = 1/[\sigma^2(F_o) + 0.0004(F_o^2)]$. Max. shift/e.s.d. in final cycle < 0.005; density in final difference map ± 0.33 e Å^{-3}. Scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974, Vol. IV). All calculations were performed on a PDP-11/73 computer using *SDP-Plus* (B. A. Frenz & Associates, Inc., 1983). Atomic coordinates and selected dimensions are given in Tables 1, 2 and 3.* Fig. 1 is a view of molecule (II) prepared using *ORTEP*II (Johnson, 1976) showing the numbering scheme.

Discussion. According to Wade's approach to cluster bonding–structure relationships (Wade, 1976), compound (I) should have an 11-atom *closo* structure being analogous with $[B_{11}H_{11}]^{2-}$. Compound (II) would be comparable with $[B_{11}H_{11}]^{4-}$ and have a structure derived from a 12-atom *closo* icosahedral $[B_{12}H_{12}]^{2-}$ cage by the excision of one vertex (BH) unit. Fig. 1 shows the gross $RhSB_9$-cage structure of (II) to be that expected for $[B_{11}H_{11}]^{4-}$ according to Wade's rules.

It is of interest to compare the cage interatomic distances in (I) and (II) since the latter formally has two more electrons in bonding molecular orbitals (Wade, 1976). Table 2 lists the comparable distances around Rh, S and P, the ranges of B—B distances, and the P—Rh—P angles together with the differences in these values. In general most distances in (II) are not significantly different (differences $< 3\sigma$) from the corresponding values in (I) with the following noteworthy exceptions: (*a*) the Rh—B9 distance in (II) [2.254 (2) Å] is considerably longer by 0.108 Å; (*b*) the Rh—B3 [2.274 (2) Å] and S—B3 [2.061 (2) Å] distances in (II) are significantly longer by 0.032 and 0.026 Å respectively. The Rh—S distance in (II) [2.3757 (5) Å] is slightly shorter by 0.0012 Å than the value found for (I) but the difference is only 2σ. Clearly, the most notable effect of the coordination

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* Lists of structure factors, calculated H-atom coordinates, anisotropic thermal parameters and complete bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54617 (65 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: MU0280]

Table 1. *Positional and thermal parameters* ($Å^2$) *with e.s.d.'s in parentheses*

Anisotropically refined atoms are given in the form of the isotropic equivalent thermal parameter defined as: $B_{eq} = (4/3)[a^2B_{11} + b^2B_{22} + c^3B_{33} + ab(\cos\gamma)B_{12} + ac(\cos\beta)B_{13} + bc(\cos\alpha)B_{23}]$. The bridging hydrogen, H, was refined with an individual isotropic thermal parameter.

	x	y	z	B_{eq}
Rh8	0.26094 (1)	0.00863 (1)	0.11257 (1)	1.904 (2)
S7	0.21617 (3)	0.16548 (4)	0.03279 (3)	2.70 (1)
P1	0.14797 (3)	0.05998 (5)	0.19045 (3)	2.48 (1)
P2	0.38020 (3)	0.09573 (5)	0.19026 (3)	2.53 (1)
P3	0.28240 (4)	−0.16399 (4)	0.16964 (3)	2.67 (1)
C1	0.0374 (2)	0.0412 (3)	0.1450 (2)	4.51 (6)
C3	0.1433 (2)	0.2091 (2)	0.2115 (1)	3.66 (5)
C4	0.4825 (2)	0.0222 (2)	0.2037 (2)	3.77 (5)
C5	0.3762 (2)	0.1374 (2)	0.2923 (1)	3.64 (5)
C7	0.3661 (2)	−0.2506 (2)	0.1375 (2)	4.03 (5)
C8	0.3173 (2)	−0.1659 (2)	0.2764 (10	3.70 (5)
C21	0.1443 (1)	−0.0008 (2)	0.2877 (1)	3.04 (4)
C22	0.1084 (2)	−0.1053 (2)	0.2955 (2)	4.70 (5)
C23	0.1123 (2)	−0.1529 (2)	0.3700 (2)	6.72 (7)
C24	0.1503 (2)	−0.0982 (3)	0.4352 (2)	6.67 (8)
C25	0.1834 (2)	0.0051 (3)	0.4287 (2)	5.79 (8)
C26	0.1806 (2)	0.0542 (2)	0.3554 (1)	4.06 (5)
C61	0.4067 (1)	0.2277 (2)	0.1472 (1)	3.11 (4)
C62	0.3554 (2)	0.3203 (2)	0.1535 (2)	4.08 (5)
C63	0.3706 (2)	0.4191 (2)	0.1174 (2)	5.42 (7)
C64	0.4373 (2)	0.4289 (3)	0.0748 (2)	6.50 (8)
C65	0.4883 (2)	0.3385 (3)	0.0681 (2)	6.16 (7)
C66	0.4737 (2)	0.2373 (3)	0.1029 (2)	4.42 (6)
C91	0.1916 (1)	−0.2606 (2)	0.1566 (1)	3.08 (4)
C92	0.1169 (2)	−0.2344 (2)	0.1073 (1)	3.42 (5)
C93	0.0482 (2)	−0.3081 (2)	0.0974 (2)	4.37 (5)
C94	0.0553 (2)	−0.4092 (2)	0.1364 (2)	5.33 (6)
C95	0.1290 (2)	−0.4362 (2)	0.1852 (2)	5.45 (7)
C96	0.1967 (2)	−0.3624 (2)	0.1957 (2)	4.33 (6)
B1	0.2006 (2)	−0.0246 (2)	−0.0893 (1)	2.98 (5)
B2	0.1804 (2)	0.1179 (2)	−0.0789 (1)	3.26 (5)
B3	0.1654 (2)	0.0118 (2)	−0.0002 (1)	2.59 (4)
B4	0.2498 (1)	−0.0878 (2)	−0.0006 (1)	2.51 (4)
B5	0.3131 (2)	−0.0483 (2)	−0.0736 (1)	2.97 (5)
B6	0.2723 (2)	0.0776 (3)	−0.1176 (1)	3.30 (5)
B9	0.3539 (1)	−0.0283 (2)	0.0275 (1)	2.45 (4)
B10	0.3656 (2)	0.0806 (2)	−0.0484 (1)	3.17 (5)
B11	0.2844 (2)	0.1859 (2)	−0.0500 (1)	3.28 (5)
H	0.367 (2)	0.080 (2)	0.021 (1)	4.6 (6)

Table 2. *Comparison of cage interatomic distances* (Å), Rh—P *distances* (Å) *and* P—Rh—P *angles* (°) *in* (I) *and* (II) *with e.s.d.'s in parentheses*

	(II)	(I)	(II) − (I)	[(II) − (I)]/e.s.d.
Rh8—S7	2.3757 (5)	2.3769 (6)	−0.0012	2
Rh8—B3	2.274 (2)	2.242 (4)	0.032	8
Rh8—B4	2.243 (2)	2.236 (3)	0.007	2
Rh8—B9	2.254 (2)	2.146 (3)	0.108	36
S7—B2	1.999 (2)	1.986 (4)	0.013	3
S7—B3	2.061 (2)	2.035 (4)	0.026	7
S7—B11	1.914 (3)	1.908 (4)	0.006	2
B—B range	1.734 (4)–1.896 (4)	1.713 (6)–1.887 (5)		
Rh8—P1	2.4538 (6)	–		
Rh8—P2	2.3869 (5)	2.4197 (5)	−0.0328	66
Rh8—P3	2.2979 (5)	2.2906 (5)	0.0073	15
P1—Rh8—P2	99.44 (2)	–		
P1—Rh8—P3	94.06 (2)	–		
P2—Rh8—P3	95.88 (2)	98.50 (2)		

of the third phosphine in (II) is a localized one, *i.e.* the lengthening of the Rh—B9 distance *trans* to this additional third phosphine, P1 in Fig. 1. Otherwise the architectures of the $RhSB_9$ cages in (I) and (II) are remarkably similar. For example, compare the range of B—B distances in (II) [1.734 (4)–1.896 (4) Å] with that in (I) [1.713 (6)–1.887 (5) Å].

Table 3. *Selected dimensions* (Å, °) *for* (II) *with e.s.d.'s in parentheses*

B1—B2	1.756 (4)	B9—B10	1.872 (4)
B1—B3	1.752 (4)	B9—H	1.320 (25)
B1—B4	1.774 (3)	B10—B11	1.799 (4)
B1—B5	1.781 (4)	B10—H	1.187 (23)
B1—B6	1.784 (4)	S7—B2	1.999 (2)
B2—B3	1.896 (4)	S7—B3	2.061 (2)
B2—B6	1.747 (4)	S7—B11	1.914 (3)
B2—B11	1.838 (4)	P1—C1	1.822 (2)
B3—B4	1.793 (3)	P1—C3	1.832 (2)
B4—B5	1.771 (4)	P1—C21	1.827 (2)
B4—B9	1.794 (3)	P2—C4	1.827 (2)
B5—B6	1.770 (4)	P2—C5	1.826 (2)
B5—B9	1.775 (3)	P2—C61	1.823 (2)
B5—B10	1.780 (4)	P3—C7	1.827 (3)
B6—B10	1.753 (3)	P3—C8	1.833 (2)
B6—B11	1.734 (4)	P3—C91	1.833 (2)

S7—Rh8—P1	86.15 (2)	B2—S7—B3	55.6 (1)
S7—Rh8—P2	96.81 (2)	B2—S7—B11	56.0 (1)
S7—Rh8—P3	167.10 (2)	B3—S7—B11	98.6 (1)
S7—Rh8—B3	52.56 (6)	Rh8—P1—C1	117.61 (9)
S7—Rh8—B4	86.17 (6)	Rh8—P1—C3	113.97 (8)
S7—Rh8—B9	87.31 (6)	Rh8—P1—C21	121.00 (7)
P1—Rh8—B3	90.64 (6)	C1—P1—C3	98.2 (1)
P1—Rh8—B4	128.76 (6)	C1—P1—C21	100.8 (1)
P1—Rh8—B9	172.53 (6)	C3—P1—C21	101.8 (1)
P2—Rh8—B3	147.23 (6)	Rh8—P2—C4	118.11 (8)
P2—Rh8—B4	131.77 (6)	Rh8—P2—C5	121.40 (8)
P2—Rh8—B9	84.92 (6)	Rh8—P2—C61	111.38 (7)
P3—Rh8—B3	114.55 (6)	C4—P2—C5	99.4 (1)
P3—Rh8—B4	83.65 (6)	C4—P2—C61	102.8 (1)
P3—Rh8—B9	91.51 (6)	C5—P2—C61	100.9 (1)
B3—Rh8—B4	46.75 (8)	Rh8—P3—C7	117.10 (9)
B3—Rh8—B9	82.51 (8)	Rh8—P3—C8	116.15 (8)
B4—Rh8—B9	47.01 (8)	Rh8—P3—C91	117.04 (7)
Rh8—S7—B2	110.07 (8)	C7—P3—C8	99.5 (1)
Rh8—S7—B3	61.18 (6)	C7—P3—C91	100.9 (1)
Rh8—S7—B11	111.97 (8)	C8—P3—C91	103.4 (1)

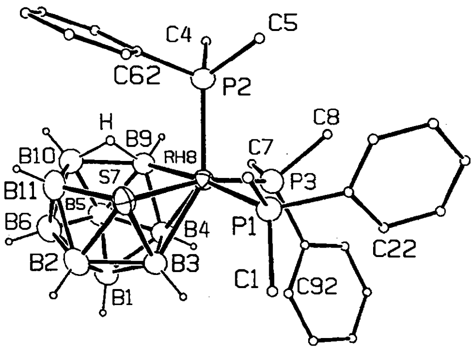

Fig. 1. A view of (II). Atoms are shown as spheres or ellipsoids of an arbitrary size and methyl and phenyl H atoms are omitted.

The Rh—P bond distances in (I) were 2.2906 (5) Å for the P atom *trans* to S and 2.4197 (5) Å for the P atom perpendicular to the $RhSB_3$ face, Table 2. In (II) the comparable distances are 2.2979 (5) and 2.3869 (5) Å. The third Rh—P distance in (II) is 2.4538 (6) Å. The P—Rh—P angles are greater than 90° in both (I) and (II), Table 2. For (II) the values are P1—Rh—P2 99.44 (2), P1—Rh—P3 94.06 (2) and P2—Rh—P3 95.88 (2)°.

All P—C and C—C distances are normal. The B9—H and B10—H distances (Table 3) are 1.32 (3) and 1.19 (2) Å with B9—H—B10 96 (2)°; the corresponding dimensions for (I) were 1.21 (3), 1.30 (3) Å and 96 (2)°. There are no unusual intermolecular contact distances.

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Structure of Bis(salicylato)bis(2-pyridylmethanol)copper(II)

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Abstract. $[\mathrm{Cu}(\mathrm{C_7H_5O_3})_2(\mathrm{C_6H_7NO})_2]$, $M_r = 556.03$, monoclinic, $C2/c$, $a = 15.786$ (1), $b = 13.299$ (2), $c = 12.083$ (1) Å, $\beta = 97.22$ (1)°, $V = 2516.73$ Å^3, $Z = 4$, $D_m = 1.45$, $D_x = 1.46$ g cm^{-3}, $\lambda(\mathrm{Cu}\ K\alpha) = 1.54178$ Å, $\mu = 16.389$ cm^{-1}, $F(000) = 1148$, $T = 293$ K, final $R = 0.046$ for 1300 observed reflections. The copper(II) atom is situated on a centre of symmetry, and has distorted octahedral coordination. It is bonded in a *trans* square-planar arrangement to the nitrogen atoms of two 2-pyridylmethanols [Cu—N = 1.981 (1) Å] and one carboxylate oxygen from each of two salicylate anions [Cu—O = 1.981 (2) Å]. The remaining axial positions are occupied by methanol oxygen atoms of two 2-pyridylmethanol ligands [Cu—O = 2.331 (1) Å].

Introduction. Monomeric Cu(carboxylate)$_2$(substituted pyridine)$_2$ complexes have been reported for a variety of carboxylates including salicylic acid (Doedens, 1976; Melník, 1981; Hanic, Štempelová & Hanicová, 1961) but not 2-pyridylmethanol (2-pyridylcarbinol). We report the crystal structure of bis(salicylato)bis(2-pyridylmethanol)copper(II).

Experimental. The blue crystals of Cu(sal)$_2$(2-pycar)$_2$ (sal = salicylate, 2-pycar = 2-pyridylmethanol) were prepared by treating 2-pyridylmethanol (2.18 g, 0.02 mol) with copper(II) salicylate (4.22 g, 0.01 mol) in hot methanol solution. The solution was left to stand at room temperature. The crude product was recrystallized from methanol to give monocrystals. Prismatic crystal with dimensions 0.25 × 0.72 × 0.81 mm; D_m by flotation in $\mathrm{CHBr_3/CH_3OH}$; Syntex $P2_1$ diffractometer, $\lambda(\mathrm{Cu}\ K\alpha) = 1.54178$ Å, graphite monochromator, $\theta/2\theta$ scan, $2\theta_{\max} = 110°$, time per reflection *ca* 60 s, two standard reflections, variation 2%, 14 reflections with $11.0 \le 2\theta \le 32.1°$ were used for refinement of lattice parameters; an absorption correction was applied, maximum and minimum transmission factors 0.746 and 0.602; index range $-16 \le h \le 15$, $0 \le k \le 13$, $0 \le l \le 12$; 3461 reflections measured, 1549 unique, $R_{\mathrm{int}} = 0.06$, 1300 observed reflections for $F \ge 3\sigma(F)$. Data reduction was performed by program *XP*21 (Pavelčík, 1987). All non-hydrogen atoms were located by direct methods using *SHELXS*86 (Sheldrick, 1990). Refinement by full-matrix least squares based on F values (169

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