Table 3. *Zn—O and Zn—N distances* (Å) *in tetrahedral* Zn *complexes*

	Zn—N	Zn—O	
(2-Amino-3-methylbenzoato)hydroxozinc(II)	2.103 (4)	1.968 (4) 1.933 (4) 1.947 (4)	This work
Zinc dicyanide–2,9-dimethyl-1,10-phenanthroline	2.069		Monge, Martínez-Ripoll & García-Blanco (1977)
Dichlorobis(4-vinylpyridine)zinc(II)	2.040 2.054		Steffen & Palenik (1977)
catena-Dichloro-μ-(9-methyladenine)-zinc(II)	2.040 2.068		McCall & Taylor (1976)
Zinc hydroxide nitrate		1.95	Stählin & Oswald (1970)
Bis(2,6-dimethyl-4*H*-pyran-4-one)-dinitratozinc		1.979	Brown & Lewis (1984)
Dichlorobis(antipyrine)zinc		1.997 (4) 2.019 (6)	Cingi, Guastini, Musatti & Nardelli (1972)
Bis(thiourea)zinc acetate		1.973 (6) 1.954 (8)	Cavalca, Gasparri, Andreetti & Domiano (1967)

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A Monomeric Cyclopentadienyl Indium Complex. Structure of [η^5-(Diphenylphosphino)tetramethylcyclopentadienyl]indium(I)

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Abstract. $C_{21}H_{22}InP$, $M_r = 420.20$, monoclinic, $P2_1/c$, $a = 11.6680$ (8), $b = 10.589$ (1), $c = 15.097$ (3) Å, $\beta = 99.754$ (9)°, $V = 1838.3$ (4) Å^3, $Z = 4$, $D_x = 1.52$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 1.35$ mm^{-1}, $F(000) = 848$, $T = 150$ K. The structure was refined to $R(F) = 0.030$ for 3411 unique observed reflections. The formation of a polymeric structure containing zigzag chains of alternating cyclopentadienyl rings and In atoms is not observed. The In atom is only η^5 bound to the cyclopentadienyl ring [average In—C(ring) = 2.694 (3) Å], and undergoes interactions neither with the lone pair of the P atom [In···P = 3.703 (1) Å] nor with other In atoms [In···In = 5.929 (1) Å].

Introduction. In recent years, ring-substituted cyclopentadienyl complexes of low-valent Group 13 element complexes have been subject to considerable structural (Beachley, Bloom, Churchill, Faegri, Fettinger, Pazik & Vitoriano, 1989; Schumann,

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Janiak, Görlitz, Loebel & Dietrich, 1989; Beachley, Lees, Glassman, Churchill & Buttrey, 1990) and theoretical (Janiak & Hoffmann, 1989, 1990) research. Solid-state structural analyses of indium(I) complexes undertaken so far, have revealed remarkable variations in indium–cyclopentadienyl carbon bond distances and indium(I)–indium(I) interaction patterns. While the variation in bond lengths can be correlated to the degree of covalency of the In—C bond, the reason for the weak indium(I)–indium(I) interactions is not fully understood. The crystallographic elucidation of the title compound can provide a new insight into the structural parameters determining the latter phenomenon.

Experimental. Yellow rectangular crystals were obtained from a saturated tetrahydrofuran solution at 243 K. A crystal of dimensions 0.24 × 0.15 × 0.12 mm was used on an Enraf–Nonius CAD-4 diffractometer with a low-temperature set-up. Intensities were measured at 150 K with an ω–2θ scan technique, the variable scan width was calculated as $\Delta\omega = (0.70 + 0.35\tan\theta)^\circ$. The maximum scan time was 45 s. Accurate unit-cell dimensions were determined from least-squares fit of 40 reflections with $20 < \theta < 24.5^\circ$. A total of 4423 reflections (3821 unique, $R_{\rm int} = 0.0127$) were measured for $0 < \theta < 27^\circ$ and $0 \leq h \leq 15$, $0 \leq k \leq 13$, $-19 \leq l \leq 19$, and 3411 reflections with $F_o > 4\sigma(F_o)$ were used in the refinement. Three intensity-control monitor reflections, measured every 7200 s, showed 0.3% variation. No correction for decay. Absorption correction by ψ scans with minimum 89.53 and maximum 99.95% transmission. A starting structure for the refinement was obtained by using fractional coordinates of all non-H atoms of the isomorphous Tl complex. H atoms were located at idealized positions with C—H = 0.95 Å (Churchill, 1973) and a constant isotropic displacement parameter $U = 0.075$ Å^2. Full-matrix least-squares refinement with 209 variables, minimizing the function $\sum w(\Delta F)^2$, led to $R = 0.030$ and $wR = 0.039$ for $w = 1.89/[\sigma^2(F_o) + 0.00041F_o^2]$, $(\Delta/\sigma) \leq 0.001$, $\Delta\rho_{\rm max} = 0.61$, $\Delta\rho_{\rm min} = -0.61$ e Å^{-3}. Scattering factors for P, C and H were as given in *SHELX76* (Sheldrick, 1976) and for the neutral In atom from Cromer & Mann (1968).

Table 1. *Fractional positional parameters and equivalent isotropic displacement parameters* (Å^2)

$$B_{\rm eq} = (8\pi^2/3)\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	*y*	*z*	$B_{\rm eq}$
In(1)	0.11018 (2)	−0.08225 (2)	−0.15347 (1)	2.31
P(1)	0.1626 (1)	−0.4042 (1)	−0.2736 (1)	2.04
C(1)	0.1860 (2)	−0.2361 (3)	−0.2732 (2)	2.07
C(2)	0.2860 (3)	−0.1601 (3)	−0.2423 (2)	2.30
C(3)	0.2582 (3)	−0.0352 (3)	−0.2698 (2)	2.37
C(4)	0.1432 (3)	−0.0322 (3)	−0.3201 (2)	2.67
C(5)	0.0977 (3)	−0.1560 (3)	−0.3223 (2)	2.28
C(6)	0.4037 (3)	−0.2053 (3)	−0.1961 (2)	2.68
C(7)	0.3394 (3)	0.0774 (3)	−0.2578 (2)	3.07
C(8)	0.0858 (3)	0.0812 (3)	−0.3682 (2)	3.96
C(9)	−0.0187 (3)	−0.1986 (3)	−0.3730 (2)	2.94
C(10)	0.2583 (3)	−0.4663 (3)	−0.1739 (2)	2.18
C(11)	0.2417 (3)	−0.4257 (3)	−0.0888 (2)	2.81
C(12)	0.3026 (3)	−0.4789 (3)	−0.0112 (2)	2.86
C(13)	0.3812 (3)	−0.5765 (3)	−0.0171 (2)	3.14
C(14)	0.3986 (3)	−0.6176 (3)	−0.1004 (2)	3.26
C(15)	0.3376 (3)	−0.5636 (3)	−0.1785 (2)	2.83
C(16)	0.2348 (3)	−0.4590 (3)	−0.3653 (2)	2.33
C(17)	0.2126 (3)	−0.5815 (3)	−0.3973 (2)	2.90
C(18)	0.2643 (3)	−0.6290 (4)	−0.4661 (2)	3.16
C(19)	0.3387 (3)	−0.5540 (4)	−0.5055 (2)	3.77
C(20)	0.3598 (3)	−0.4303 (4)	−0.4759 (2)	3.69
C(21)	0.3084 (3)	−0.3821 (3)	−0.4066 (2)	2.73

Table 2. *Bond lengths* (Å) *and bond angles* (°)

In(1)—C(1)	2.692 (3)	C(4)—C(8)	1.501 (4)
In(1)—C(2)	2.760 (3)	C(5)—C(9)	1.510 (4)
In(1)—C(3)	2.710 (3)	C(10)—C(11)	1.390 (4)
In(1)—C(4)	2.661 (3)	C(10)—C(15)	1.394 (4)
In(1)—C(5)	2.646 (3)	C(11)—C(12)	1.385 (5)
P(1)—C(1)	1.801 (3)	C(12)—C(13)	1.394 (5)
P(1)—C(10)	1.837 (3)	C(13)—C(14)	1.378 (5)
P(1)—C(16)	1.834 (3)	C(14)—C(15)	1.393 (5)
C(1)—C(2)	1.429 (4)	C(16)—C(17)	1.393 (4)
C(1)—C(5)	1.439 (4)	C(16)—C(21)	1.404 (4)
C(2)—C(3)	1.407 (4)	C(17)—C(18)	1.381 (5)
C(2)—C(6)	1.509 (4)	C(18)—C(19)	1.383 (6)
C(3)—C(4)	1.426 (4)	C(19)—C(20)	1.393 (5)
C(3)—C(7)	1.515 (4)	C(20)—C(21)	1.388 (5)
C(4)—C(5)	1.412 (4)		
C(1)—C(2)—C(3)	107.4 (3)	P(1)—C(1)—C(5)	119.1 (2)
C(2)—C(3)—C(4)	109.1 (3)	P(1)—C(10)—C(11)	118.9 (2)
C(3)—C(4)—C(5)	107.9 (3)	P(1)—C(10)—C(15)	122.7 (2)
C(1)—C(5)—C(4)	107.5 (3)	C(11)—C(10)—C(15)	118.0 (3)
C(2)—C(1)—C(5)	108.1 (3)	C(10)—C(11)—C(12)	121.4 (3)
C(1)—C(2)—C(6)	126.9 (3)	C(11)—C(12)—C(13)	119.8 (3)
C(3)—C(2)—C(6)	125.5 (3)	C(12)—C(13)—C(14)	119.5 (3)
C(2)—C(3)—C(7)	126.6 (3)	C(13)—C(14)—C(15)	120.6 (3)
C(4)—C(3)—C(7)	124.0 (3)	C(10)—C(15)—C(14)	120.6 (3)
C(3)—C(4)—C(8)	125.3 (3)	P(1)—C(16)—C(17)	118.3 (2)
C(5)—C(4)—C(8)	126.6 (3)	P(1)—C(16)—C(21)	123.1 (2)
C(1)—C(5)—C(9)	126.0 (3)	C(17)—C(16)—C(21)	118.6 (3)
C(4)—C(5)—C(9)	126.3 (3)	C(16)—C(17)—C(18)	121.4 (3)
C(1)—P(1)—C(10)	106.3 (1)	C(17)—C(18)—C(19)	120.0 (3)
C(1)—P(1)—C(16)	103.1 (1)	C(18)—C(19)—C(20)	119.5 (3)
C(10)—P(1)—C(16)	102.3 (1)	C(19)—C(20)—C(21)	120.8 (3)
P(1)—C(1)—C(2)	132.3 (2)	C(16)—C(21)—C(20)	119.8 (3)

Discussion. Final fractional coordinates are given in Table 1.* Important bond lengths and angles are compiled in Table 2. The title compound is made up of well separated monomeric molecules. The molecular structure, along with the atom-numbering scheme is shown in Fig. 1 and a stereographic drawing of the molecular packing is presented in Fig. 2. The In atom is bound to the cyclopentadienyl ring [C(1)–C(5)] in an η^5 fashion. Further stabilization by formation of a polymeric chain structure, as was found for CpIn (Cp = C_5H_5), MeCpIn (MeCp = $CH_3C_5H_4$) (Beachley, Pazik, Glassman, Churchill, Fettinger & Bloom, 1988) and $Me_3SiCpIn$ (Beachley *et al.* 1990) or by oligomerization *via* weak indium(I)–indium(I) interactions like in the hexamer $(Cp^*In)_6$ [Cp* =

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* Lists of structure factors, anisotropic thermal parameters, bond angles and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54638 (25 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AB0237]

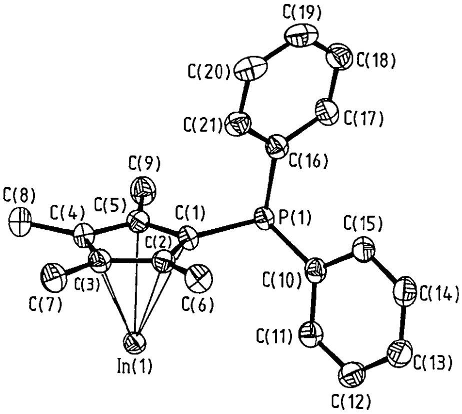

Fig. 1. Molecular structure of the title compound along with the atom-numbering scheme. H atoms are omitted. Thermal ellipsoids (*ORTEP*II; Johnson, 1976) are drawn at 50% probability level.

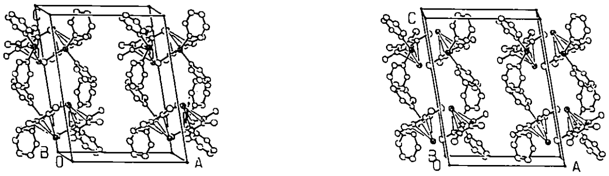

Fig. 2. Stereoscopic view of the molecular packing of the title compound.

$C_5(CH_3)_5$] (Beachley *et al.*, 1989) or in the 'quasi-dimer' $(C_6H_5CH_2)_5C_5In$ (Schumann, Janiak, Görlitz, Loebel & Dietrich, 1989) is not observed. The shortest In···In distance is as long as 5.929 (1) Å which is substantially longer than 3.986 (1), 3.986 (1), 5.428, 3.963 (1) and 3.631 (2) Å in the related structures of CpIn, MeCpIn, $Me_3SiCpIn$, $(Cp^*In)_6$ and $(C_6H_5CH_2)_5C_5In$, respectively. Furthermore the shortest In···P $(-x,\ 0.5+y,\ -0.5-z)$ distance of 3.703 (1) Å is not significantly shorter than the van der Waals distance of In and P of 3.73 Å (Bondi, 1964). The averaged In—C bond and the In—Cp(centroid) distances of 2.694 (3) and 2.4073 (15) Å are in good agreement with 2.671 and 2.382 (2) Å in the structurally closely related $(C_6H_5CH_2)_5C_5In$ complex. Internal bond parameters of the ligand exhibit no unusual magnitudes.

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Structure of Perchlorato[(2*S*,6*R*,9*S*,13*R*)-2,6,9,13-tetraazatetradecane]copper(II) Perchlorate

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Abstract. $[Cu(ClO_4)(C_{10}H_{26}N_4)]ClO_4$, $M_r = 464.79$, monoclinic, $P2_1/n$, $a = 7.440$ (1), $b = 14.678$ (3), $c = 16.903$ (3) Å, $\beta = 90.845$ (1)°, $V = 1845.7$ Å^3, $Z = 4$, $D_x = 1.670$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.7093 Å, $\mu = 1.52$ mm^{-1}, $F(000) = 963.80$, $T = 298$ (4) K, $R = 0.049$, $wR = 0.074$ for 2275 significant reflections. The coordination about Cu^{II} is distorted square pyramidal with the tetraamine equatorial and the O atom of a perchlorate ion axial. The Cu atom is slightly (0.014 Å) out of the square coordination

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