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Structure of [6,13-Dibromo-7,14-bis(dibromomethylene)-5,5,12,12-tetramethyl-1,4,8,11-tetraazacyclotetradeca-3,10-diene-2,9-dionato](pyridine)nickel(II)–Pyridine (1/2)

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Abstract. $[Ni(C_{16}H_{16}Br_6N_4O_2)(C_5H_5N)].2C_5H_5N$, $M_r = 1071{\cdot}85$, monoclinic, $P2_1/c$, $a = 13{\cdot}441\ (4)$, $b = 14{\cdot}934\ (7)$, $c = 18{\cdot}837\ (6)$ Å, $\beta = 96{\cdot}75\ (2)°$, $V = 3754{\cdot}9$ Å^3, $Z = 4$, $D_m = 1{\cdot}90$, $D_x = 1{\cdot}896$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·70926 Å, $\mu = 7{\cdot}51$ mm^{-1}, $F(000) = 2080$, $T = 291$ K, $R = 0{\cdot}038$ for 1849 observed reflexions. The coordination polyhedron about the Ni atom is a rectangular pyramid; the four N atoms of a macrocyclic ligand form the base (the two bearing a formal charge having a shorter bond) while the apex is occupied by a pyridine N atom. The macrocycle contains two deprotonated amide moieties, two imine groups, two bromomethine functions and two dibromoethene groups.

Introduction. Oxidation of coordinated nitrogen-containing ligands has been of interest for many years. The macrocyclic complex (hexamethyl-1,4,8,11-tetraazacyclotetradeca-1,7-diene)nickel(II) has been treated with excess bromine in pyridine solution, followed by hydrolysis, with the hope that portions of the ligand remote from the metal ion could be oxidized under conditions favouring both bromination and dehydrobromination (Samuel, 1977). The product of this reaction is a novel complex, the ligand of which contains bromine, new imine moieties, alkene groups and a carbonyl group in a conjugated amide function. The structure of this new complex (the title compound) was ascertained by a single-crystal X-ray diffraction study.

Experimental. Brown acicular crystals (from pyridine), 0·15 × 0·20 × 0·30 mm, D_m by flotation, Picker FACS-I diffractometer, graphite-monochromatized Mo $K\alpha$ radiation, lattice parameters from setting angles of 11 reflexions with $2\theta > 27°$, absorption and extinction ignored, θ–2θ scan, h 0→11, k 0→12, l −15→16 with $0 < 2\theta < 35°$ (limited by high thermal motion and solvent effects), variations in intensities of two standards <4%, 2390 independent reflexions, 1849 with $I > 2{\cdot}3\sigma(I)$, Lorentz–polarization correction applied; direct methods (symbolic addition procedure), full-matrix refinement with anisotropic temperature factors for Ni, Br, O and uncoordinated pyridine C, N atoms only {one carbon atom [C(28)] of a pyridine molecule had an unreasonably high thermal-motion parameter for U_{22} *viz* 0·47 (8) Å^2, this may involve some disorder of that ring}, H atoms from ΔF synthesis, final $R = 0{\cdot}038$, $wR = 0{\cdot}042$, $w = 1/\sigma^2$, maximum $\Delta/\sigma = 0{\cdot}26$ for final cycle, $\Delta\rho_{max} = |0{\cdot}8|$ e Å^{-3}, scattering factors including anomalous dispersion from *International Tables for X-ray Crystallography* (1974). Computer programs used were those described by Einstein & Jones (1972).

Discussion. The [Ni(1,8-ctBr$_6$)(py)] molecule is illustrated in Fig. 1 and Fig. 2 (I). Final atomic coordinates and isotropic thermal parameters with standard deviations are listed in Table 1.† Bond distances and angles of interest are displayed in Table 2. The results of the X-ray study indicate that the coordination polyhedron of the Ni^{II} ion is a rectangular pyramid and that the Ni—N distances are characteristic of high-spin species. Of the three molecules of pyridine present, only one serves as a ligand. The four N atoms of the macrocycle are near-planar (within 0·06 Å). The Ni atom is 0·39 Å above this plane and the Ni—N(5) axis is 1·3° from the normal to this plane.

Because of the twofold chemical symmetry of the complex, there are 16 pairs of chemically equivalent bond lengths. A comparison of the equivalent sets of bond distances (see Table 2) indicates that the largest difference between members of a pair is 1·6 times the estimated standard deviation of the difference and is therefore not significant. In comparing the 25 pairs of chemically equivalent bond angles involving the Ni and

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† Hydrogen atomic coordinates, anisotropic temperature factors and a list of observed and calculated structure factors have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42851 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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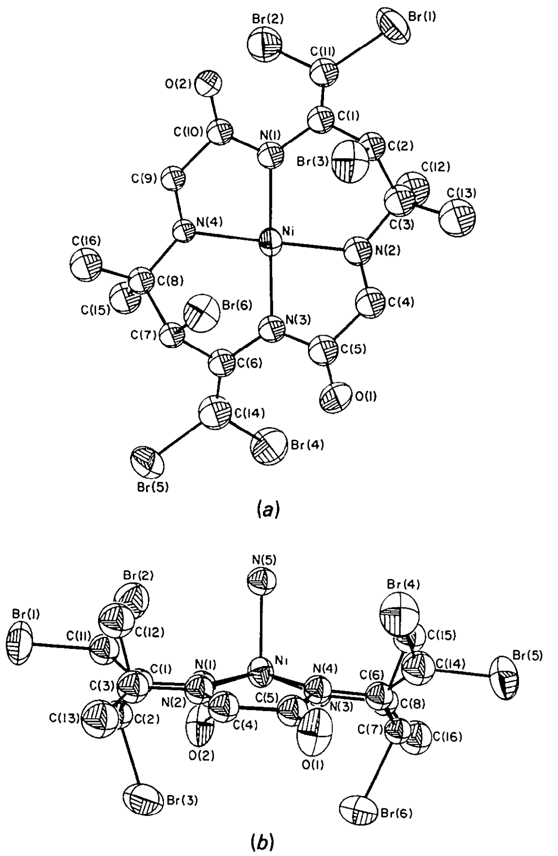
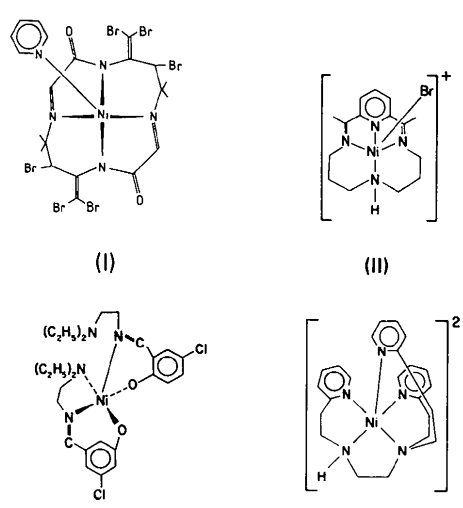


Fig. 1. (*a*) *ORTEP* (Johnson, 1965) drawing of the [Ni(1,8-ctBr$_6$)(py)] molecule excluding the coordinated pyridine molecule, showing crystallographic numbering scheme. (*b*). *ORTEP* (Johnson, 1965) drawing of the [Ni(1,8-ctBr$_6$)(py)] molecule showing only N(5) of the coordinated pyridine molecule.



(I) (II)

(III) (IV)

Fig. 2. Square-pyramidal complexes of Ni^{II} having principally N donor atoms.

macrocyclic non-H atoms, it is observed (Table 2) that the largest difference is 3·2σ, for N(1)–Ni–N(2) *vs* N(3)–Ni–N(4), and only two others are 2·5σ or larger: C(2)–C(3)–C(12) *vs* C(7)–C(8)–C(15), 2·5σ; and N(2)–C(3)–C(13) *vs* N(4)–C(8)–C(16), 2·7σ. A comparison of 18 pairs of torsion angles reveals the greatest deviation from twofold symmetry. The largest difference is 3·8σ [for N(2)–Ni–N(1)–C(1) *vs* N(4)–Ni–N(3)–C(6)] and eight others are 2·5σ or larger.

The topology of the macrocycle, while not folded, is distinctly non-planar. Although the four N atoms form a plane within 0·06 Å, the remainder of the macrocycle assumes a saddle shape. The dibromomethene groups, C(11) and C(14), and the Ni atom are above the mean plane of the N atoms, while the remaining atoms are

Table 1. *Final fractional atomic coordinates* ($\times 10^5$ *for* Ni *and* Br; $\times 10^4$ *for* C, N *and* O) *and isotropic thermal parameters* (Å^2)

For atoms refined anisotropically $B_{eq} = \frac{8}{3}\pi^2 (U_{11} + U_{22} + U_{33})$.

	x	y	z	B/B_{eq}
Ni	31934 (10)	64900 (9)	16653 (7)	3·1
Br(1)	10407 (10)	35910 (9)	27834 (7)	5·7
Br(2)	13848 (12)	54430 (10)	35588 (7)	7·0
Br(3)	11455 (10)	51399 (9)	6231 (7)	5·1
Br(4)	63775 (9)	74012 (9)	10555 (7)	5·4
Br(5)	55783 (11)	93636 (8)	7957 (8)	6·1
Br(6)	24194 (10)	80538 (9)	627 (7)	5·9
O(1)	4794 (5)	6386 (5)	−30 (4)	4·7
O(2)	391 (6)	6655 (5)	2237 (5)	6·2
N(1)	1963 (6)	6076 (6)	2064 (4)	3·2 (2)
N(2)	3455 (6)	5292 (5)	1186 (4)	2·3 (2)
N(3)	4085 (6)	6926 (6)	975 (4)	3·2 (2)
N(4)	2515 (6)	7717 (6)	1741 (4)	3·5 (2)
C(1)	1698 (7)	5173 (7)	2119 (6)	3·1 (2)
C(2)	1879 (7)	4594 (7)	1485 (5)	2·8 (2)
C(3)	2985 (7)	4428 (7)	1353 (5)	3·1 (2)
C(4)	3967 (7)	5395 (7)	677 (5)	3·0 (2)
C(5)	4364 (8)	6306 (7)	510 (6)	3·2 (2)
C(6)	4321 (8)	7837 (7)	872 (5)	3·2 (2)
C(7)	3425 (8)	8438 (7)	841 (6)	3·6 (3)
C(8)	2956 (8)	8579 (8)	1529 (6)	4·1 (2)
C(9)	1656 (8)	7642 (7)	1932 (6)	3·9 (3)
C(10)	1246 (10)	6716 (8)	2108 (6)	4·7 (3)
C(11)	1440 (8)	4823 (8)	2701 (6)	4·6 (3)
C(12)	3553 (8)	4106 (8)	2062 (6)	4·7 (3)
C(13)	3022 (8)	3712 (7)	777 (6)	4·3 (3)
C(14)	5245 (8)	8124 (7)	890 (5)	3·6 (2)
C(15)	3759 (9)	8816 (8)	2138 (6)	5·4 (3)
C(16)	2150 (9)	9329 (8)	1450 (6)	5·6 (3)
N(5)	4190 (6)	6487 (6)	2565 (5)	3·2 (2)
C(17)	5155 (8)	6334 (7)	2541 (6)	3·6 (2)
C(18)	5881 (8)	6392 (8)	3122 (7)	4·7 (3)
C(19)	5598 (9)	6650 (8)	3773 (6)	4·6 (3)
C(20)	4619 (9)	6813 (8)	3803 (6)	5·0 (3)
C(21)	3932 (8)	6713 (7)	3203 (6)	9·1 (3)
N(6)	1679 (15)	1421 (10)	595 (25)	13·7
C(22)	1098 (27)	1651 (24)	1077 (14)	10·6
C(23)	389 (22)	2294 (26)	962 (17)	8·5
C(24)	228 (17)	2711 (15)	299 (30)	10·8
C(25)	756 (25)	2419 (28)	−232 (17)	11·1
C(26)	1400 (36)	1795 (26)	−106 (18)	13·3
N(7)	2634 (28)	541 (20)	3508 (13)	12·9
C(27)	1676 (39)	512 (21)	3283 (15)	12·1
C(28)	1112 (23)	−214 (44)	3348 (17)	16·8
C(29)	1664 (37)	−948 (25)	3693 (14)	14·8
C(30)	2652 (29)	−897 (17)	3915 (11)	9·6
C(31)	3107 (15)	−172 (32)	3821 (10)	8·9

The coordinated pyridine molecule is
N(5)–C(17)–C(18)–C(19)–C(20)–C(21).
The uncoordinated pyridine molecules are
N(6)–C(22)–C(23)–C(24)–C(25)–C(26)
and N(7)–C(27)–C(28)–C(29)–C(30)–C(31).

below this plane. It should be noted that the atoms Br(2)–C(11)–C(1)–N(1)–C(10)–O(2) [also the other (related) atoms] are sterically hindered from being coplanar.

The following salient features arise from an examination of individual bond distances in the macrocyclic portion of the complex. The mean value of the C–O distances is 1·22 (1) Å, which corresponds to a double bond. The sp^3 C–Br bond [mean value 1·968 (7) Å] is considerably longer than the sp^2 C–Br bonds [mean value 1·894 (6) Å]. The mean value of the sp^2 C–Br bonds, 1·894 (6) Å, agrees with the value of 1·897 (2) Å reported by James & Williams (1973) for the mean of 56 aromatic C–Br bond lengths. There are two types of sp^2 C–Br bonds, those *cis* [C(11)–Br(2), C(14)–Br(4)] and those *trans* [C(11)–Br(1), C(14)–Br(5)] to the N atom. The mean values of these two types of bond distances are slightly, but significantly, different: *cis*, 1·866 (8) Å, and *trans*, 1·923 (8) Å.

Table 2. *Bond distances* (Å) *and angles* (°) *and torsion angles* (°) *for* [Ni(1,8-ctBr$_6$)(py)].2(py)

Ni–N(1)	1·993 (8)	Ni–N(3)	1·979 (8)
Ni–N(2)	2·053 (8)	Ni–N(4)	2·059 (9)
N(1)–C(1)	1·402 (14)	N(3)–C(6)	1·416 (14)
N(1)–C(10)	1·367 (15)	N(3)–C(5)	1·357 (14)
N(2)–C(3)	1·486 (13)	N(4)–C(8)	1·491 (15)
N(2)–C(4)	1·254 (13)	N(4)–C(9)	1·254 (14)
C(5)–O(1)	1·233 (14)	C(10)–O(2)	1·206 (16)
C(2)–Br(3)	1·974 (9)	C(7)–Br(6)	1·959 (11)
C(11)–Br(1)	1·928 (12)	C(14)–Br(5)	1·918 (11)
C(11)–Br(2)	1·871 (12)	C(14)–Br(4)	1·862 (11)
C(1)–C(2)	1·517 (15)	C(6)–C(7)	1·498 (15)
C(2)–C(3)	1·556 (14)	C(7)–C(8)	1·521 (16)
C(4)–C(5)	1·508 (15)	C(9)–C(10)	1·539 (16)
C(1)–C(11)	1·298 (16)	C(6)–C(14)	1·310 (15)
C(3)–C(12)	1·536 (14)	C(8)–C(15)	1·522 (15)
C(3)–C(13)	1·528 (15)	C(8)–C(16)	1·553 (17)
Ni–N(5)	2·033 (12)		
N(1)–Ni–N(2)	95·0 (3)	N(3)–Ni–N(4)	93·4 (4)
N(1)–Ni–N(3)	160·5 (4)	N(2)–Ni–N(4)	155·0 (4)
N(1)–Ni–N(4)	81·7 (4)	N(2)–Ni–N(3)	81·3 (3)
N(1)–Ni–N(5)	100·4 (5)	N(3)–Ni–N(5)	99·1 (5)
N(2)–Ni–N(5)	103·6 (5)	N(4)–Ni–N(5)	101·4 (5)
Ni–N(1)–C(1)	123·7 (7)	Ni–N(3)–C(6)	124·7 (7)
Ni–N(1)–C(10)	115·3 (8)	Ni–N(3)–C(5)	115·7 (7)
Ni–N(2)–C(3)	124·4 (6)	Ni–N(4)–C(8)	123·8 (7)
Ni–N(2)–C(4)	111·5 (7)	Ni–N(4)–C(9)	111·7 (7)
N(1)–C(1)–C(2)	115·1 (9)	N(3)–C(6)–C(7)	112·9 (9)
N(1)–C(10)–C(9)	110·0 (10)	N(3)–C(5)–C(4)	110·5 (9)
N(2)–C(3)–C(2)	109·5 (8)	N(4)–C(8)–C(7)	109·0 (9)
N(2)–C(4)–C(5)	120·6 (9)	N(4)–C(9)–C(10)	120·5 (10)
C(1)–C(2)–C(3)	117·5 (8)	C(6)–C(7)–C(8)	117·5 (9)
N(1)–C(10)–O(2)	130·9 (11)	N(3)–C(5)–O(1)	131·1 (10)
C(1)–C(11)–Br(1)	123·8 (9)	C(6)–C(14)–Br(5)	123·0 (8)
C(1)–C(11)–Br(2)	124·9 (9)	C(6)–C(14)–Br(4)	124·6 (8)
N(1)–C(1)–C(11)	123·0 (10)	N(3)–C(6)–C(14)	122·5 (10)
N(2)–C(3)–C(13)	114·0 (8)	N(4)–C(8)–C(16)	110·7 (9)
C(2)–C(3)–C(12)	107·3 (8)	C(7)–C(8)–C(15)	110·3 (9)
Br(1)–C(11)–Br(2)	111·3 (6)	Br(4)–C(14)–Br(5)	112·3 (5)
O(1)–C(5)–C(4)	118·1 (9)	O(2)–C(10)–C(9)	119·0 (11)
C(12)–C(3)–C(13)	110·1 (9)	C(15)–C(8)–C(16)	109·2 (9)
C(1)–C(2)–Br(3)	107·5 (7)	C(6)–C(7)–Br(6)	109·5 (7)
C(3)–C(2)–Br(3)	109·2 (6)	C(8)–C(7)–Br(6)	111·1 (7)
C(2)–C(3)–C(13)	109·7 (8)	C(7)–C(8)–C(16)	111·9 (9)
N(2)–C(3)–C(12)	106·0 (8)	N(4)–C(8)–C(15)	105·5 (9)
C(2)–C(1)–C(11)	121·3 (10)	C(7)–C(6)–C(14)	124·1 (10)
C(1)–N(1)–Ni–N(2)	15·9 (8)	C(6)–N(3)–Ni–N(4)	19·9 (7)
C(10)–N(1)–Ni–N(4)	7·8 (7)	C(5)–N(3)–Ni–N(2)	4·7 (7)
C(3)–N(2)–Ni–N(1)	−15·9 (7)	C(8)–N(4)–Ni–N(3)	−19·1 (7)
C(4)–N(2)–Ni–N(3)	−4·4 (6)	C(9)–N(4)–Ni–N(1)	−5·4 (7)
Ni–N(2)–C(4)–C(5)	3·6 (11)	Ni–N(4)–C(9)–C(10)	2·5 (12)
N(2)–C(4)–C(5)–N(3)	0·1 (10)	N(4)–C(9)–C(10)–N(1)	3·7 (14)
C(4)–C(5)–N(3)–Ni	−4·1 (10)	C(9)–C(10)–N(1)–Ni	−8·2 (11)
O(1)–C(5)–N(3)–Ni	169·3 (9)	O(2)–C(10)–N(1)–Ni	169·6 (11)
O(1)–C(5)–N(3)–C(6)	−1·8 (16)	O(2)–C(10)–N(1)–C(1)	5·6 (17)
C(4)–C(5)–N(3)–C(6)	−175·2 (8)	C(9)–C(10)–N(1)–C(1)	−172·2 (9)
Ni–N(3)–C(6)–C(14)	128·3 (9)	Ni–N(1)–C(1)–C(11)	130·9 (9)
C(5)–N(3)–C(6)–C(14)	−61·4 (13)	C(10)–N(1)–C(1)–C(11)	−66·6 (13)
C(5)–N(3)–C(6)–C(7)	126·3 (10)	C(10)–N(1)–C(1)–C(2)	122·1 (9)
Ni–N(3)–C(6)–C(7)	−44·0 (10)	Ni–N(1)–C(1)–C(2)	−40·5 (10)
C(3)–N(2)–C(4)–C(5)	175·9 (8)	C(8)–N(4)–C(9)–C(10)	177·2 (9)
Ni–N(2)–C(3)–C(2)	38·0 (9)	Ni–N(4)–C(8)–C(7)	40·7 (10)
N(2)–C(3)–C(2)–C(1)	−63·7 (11)	N(4)–C(8)–C(7)–C(6)	−66·4 (11)
N(3)–C(6)–C(7)–C(8)	69·6 (11)	N(1)–C(1)–C(2)–C(3)	67·3 (11)

On the other hand, the four pairs of C–N bonds show a considerable range of distances. The N(2)–C(3) and N(4)–C(8) mean distance of 1·488 (10) Å corresponds to a normal single bond, while the N(2)–C(4) and N(4)–C(9) mean distance of 1·254 (10) Å is that of a normal double bond. However, the two pairs of bonds involving the amide N atoms, N(1) and N(3), are of intermediate length and differ from each other slightly, but significantly. The N(1)–C(1), N(3)–C(6) mean distance is 1·409 (10) Å, while the N(1)–C(10), N(3)–C(5) mean distance is 1·362 (10) Å. Most of the bond distances in the macrocycle correspond fairly well to localized bonds except for the two pairs of C–N bonds involving the amide N atoms. It is reasonable to expect some delocalization of the formal negative charge on these N atoms. Delocalization is apparently limited, however, to the C atoms immediately adjacent.

The bonds from the Ni atom to N(1) and N(3), which bear a formal negative charge, are significantly shorter than those between the Ni atom and the imine N atoms, N(2) and N(4). The mean distances are 1·986 (6) Å for Ni–amide N and 2·056 (6) Å for Ni–imine N, the difference between these being 8·3σ.

The coordination polyhedron of [Ni(1,8-ctBr$_6$)(py)] is best described as a 'rectangular' pyramid in which the *average* Ni–basal N distance and the Ni–apical N distance are the same within their standard deviations. Some square-pyramidal Ni$^{\text{II}}$ complexes having three to five N donor atoms are illustrated in Fig. 2. Complex (II) (Fleischer & Hawkinson, 1968; Karn & Busch, 1966) is diamagnetic and has four N donor atoms and one Br donor atom; as expected, the Ni–Br bond is much longer (2·791 Å) than the Ni–N bonds (1·808–1·932 Å). Complex (III) (Sacconi, Orioli & Di Vaira, 1965; Orioli, Di Vaira & Sacconi, 1966) has three N-atom and two O-atom donors and is high-spin. The Ni–N distances are the same [1·98 (2) to 2·20 (2) Å] within their estimated standard deviations. Two of the N atoms and the two O atoms form a plane within 0·1 Å. The Ni atom is 0·36 Å above this plane but the Ni–apical N is about 5° from the normal to this plane. The structure of complex (IV) (Mazurek, Phillip, Hoskins & Whillans, 1970), which has five N donor atoms and is high-spin, is only slightly distorted from that of a square pyramid. The Ni–apical N distance is somewhat shorter (2·03 Å) than the average Ni–basal N distance (2·10 Å). The Ni atom is 0·32 Å above the basal plane and the Ni–apical N bond is about 2° to the normal of the basal plane.

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μ_3-Benzylidyne-tris(tricarbonylcobalt)(3*Co*–*Co*)

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Abstract. $[Co_3(C_7H_5)(CO)_9]$, $M_r = 518{\cdot}01$, triclinic, $P\bar{1}$, $a = 15{\cdot}771\,(3)$, $b = 15{\cdot}930\,(3)$, $c = 8{\cdot}051\,(3)$ Å, $\alpha = 100{\cdot}97\,(2)$, $\beta = 100{\cdot}02\,(2)$, $\gamma = 101{\cdot}10\,(1)°$, $V = 1901{\cdot}4$ Å^3, $Z = 4$, $D_m = 1{\cdot}83$, $D_x = 1{\cdot}81$ Mg m^{-3}, λ(Mo $K\alpha$) $= 0{\cdot}71069$ Å, $\mu = 2{\cdot}523$ mm^{-1}, $F(000) = 1016$, $T = 293\,(1)$ K, final $R = 0{\cdot}0466$ for 4027 observed $[I > 3\sigma(I)]$ reflections. There are two independent molecules in the asymmetric unit. The three Co atoms form an equilateral triangle, symmetrically capped by the C atom of the μ_3-carbyne ligand, to form a tetrahedral cluster core. The phenyl substituent of the carbyne is displaced slightly from a totally symmetrical orientation towards one of the Co atoms.

Introduction. Clusters of the type $YCCo_3(CO)_9$ (Y = alkyl, aryl, halo *etc.*) are well known (Penfold & Robinson, 1973; Seyferth, 1976; Schmid, 1978) and interest in their chemistry stems from the unusual stability afforded by the clamping effect of the bridging carbyne and the potential of these systems in catalysis (Masters, 1979). The title compound has been the subject of investigation in our laboratories since the discovery that its one-electron reduction leads to the formation of a particularly stable radical anion (Peake, Robinson, Simpson & Watson, 1977; Bond, Peake, Robinson, Simpson & Watson, 1977). This, in turn, provides an effective substrate for electron-transfer-catalyzed substitution (Bezems, Rieger & Visco, 1981; Arewgoda, Robinson & Simpson, 1983) and metal-exchange reactions (Jensen, Robinson & Simpson, 1983). The work reported here was undertaken to determine the detailed structure of the $PhCCo_3(CO)_9$ molecule.

Experimental. Crystals were grown from hexane as brown-black plates. Crystal dimensions 0·94 × 0·38 × 0·08 mm. D_m by flotation (aqueous $ZnBr_2$). Preliminary precession photography (Cu $K\alpha$) indicated a triclinic space group which was confirmed as $P\bar{1}$ by the success of the refinement. Nicolet $P\bar{3}$ diffractometer, Mo $K\alpha$ radiation (graphite monochromator). Cell dimensions determined by least squares from settings of 24 strong reflections in the range $28{\cdot}2 < 2\theta < 30{\cdot}8°$. 5220 reflections measured. Range of hkl: ±17, ±18, 9. $2\theta_{\max} = 45°$. Three standard reflections measured after every 100 reflections; no significant variation. Intensities corrected for Lorentz and polarization effects and empirical absorption corrections applied using *SHELXTL* (Sheldrick, 1980); transmission factors 0·529–0·924. Of 5001 unique reflections ($R_{\text{int}} = 0{\cdot}0172$), 4027 were considered observed with $I > 3\sigma(I)$. Structure determined by direct methods using *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). The two independent molecules were refined in alternating blocked-matrix cycles minimizing $\sum w(|F_o| - |F_c|)^2$, using *SHELX*76 (Sheldrick, 1976). Neutral-atom scattering factors taken from Cromer & Mann (1968); real and imaginary anomalous-dispersion coefficients from Cromer & Liberman (1970). The phenyl substituents

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