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Structure of Bis(5,7-dibromo-8-quinolinolato-*N*,*O*)bis(pyridine)nickel(II)–Pyridine (4/5)

By S. García-Granda,*† C. Jansen, Paul T. Beurskens and H. J. J. Behm

Crystallography Laboratory, University of Nijmegen, Toernooiveld, 6525 ED Nijmegen, The Netherlands

and F. Gómez-Beltrán

Departamento de Química-Física, Facultad de Química, Universidad de Oviedo, Calvo Sotelo s/n, 33007 Oviedo, Spain

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Abstract. $[\mathrm{Ni(C_9H_4Br_2NO)_2(C_5H_5N)_2}].1{\cdot}25\mathrm{C_5H_5N}$, $M_r = 919{\cdot}67$, monoclinic, $P2_1/c$, $a = 21{\cdot}261\ (3)$, $b = 38{\cdot}913\ (7)$, $c = 18{\cdot}265\ (2)$ Å, $\beta = 112{\cdot}97\ (1)°$, $V = 13913\ (4)$ Å^3, $Z = 16$, $D_x = 1{\cdot}76$ Mg m^{-3}, Cu $K\alpha$ radiation (graphite monochromator, $\lambda = 1{\cdot}54178$ Å), $\mu(\mathrm{Cu}\ K\alpha) = 65{\cdot}60$ cm^{-1}, $T = 290$ K, $F(000) = 7208$. Final conventional R factor $= 0{\cdot}060$, $wR = 0{\cdot}058$ for 5983 observed reflections and 1467 variables. The structure consists of four Ni complexes, showing a similar geometry, per asymmetric unit. There are also five independent pyridine units stabilizing the molecular packing. The central Ni atom is pseudo-octahedrally coordinated by two 5,7-dibromo-8-quinolinol ligands and two pyridine ligands in *cis* positions. Distances and angles are normal [average Ni—N(quinoline) 2·07 (2), Ni—N(pyridine) 2·11 (2), Ni—O 2·04 (1) Å].

Experimental. A dark yellow crystal approximately 0·50 × 0·10 × 0·10 mm was used during the measurements. Crystals were obtained from recrystallization in pyridine/water mixtures. Throughout the experiment Cu $K\alpha$ radiation was used with a graphite-crystal monochromator on a Nonius CAD-4 single-crystal diffractometer. The unit-cell dimensions were determined from the angular settings of 25 reflections ($18 < \theta < 28°$). The intensity data of 22 689 reflections (one quarter sphere up to $\theta = 65°$), *hkl* range from (0,0,–21) to (24,43,21), were measured, using the ω–2θ scan technique, with a scan angle of 1·50° and a variable scan rate with a maximum scan time of 20 s per reflection. The intensity of the primary beam was checked throughout the data collection by monitoring three standard reflections every 30 min. The final drift correction factors were between 0·98 and 1·09. On all reflections profile analysis was performed (Lehmann & Larsen, 1974; Grant & Gabe, 1978); empirical absorption correction was applied, using ψ scans (North, Phillips & Mathews, 1968), correction factors were in the range 0·61 to 1·00. Some doubly measured reflections were averaged, $R_{\mathrm{int}} = 0{\cdot}068$, resulting in 21 616 unique reflections of which 5983 were observed with $I > 3\sigma(I)$. Lorentz and polarization corrections were applied.

The structure was solved using *DIRDIF* (Beurskens *et al.*, 1983) employing automated vector-search rotation functions, followed by reciprocal-space translation functions (*TRACOR*; Beurskens, Gould, Bruins Slot & Bosman, 1987), on a known part of the assumed structure (one Ni atom with two 5,7-dibromo-8-quinolinol ligands taken from the analogous 5,7-dichloro compound (García-Granda, Beurskens, Behm & Gómez-Beltrán, 1987) which gave all atoms except the atoms of the non-bonded pyridine molecules and the hydrogen atoms.

The structure was refined by least squares using *SHELX* (Sheldrick, 1976); because of the size, a block-matrix refinement was used. Each independent complex was refined in a separate block and the solvent molecules in another.

Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974). Isotropic least-squares refinement converged at $R = 0{\cdot}157$. At this stage an empirical absorption correction was applied (Walker & Stuart, 1983), resulting in a further decrease of R to 0·144.

During the final stages of the refinement the positional parameters and the anisotropic thermal parameters of the atoms of the four Ni complexes were refined. Five non-bonded pyridine rings, of which one is disordered, were found from difference Fourier synthesis and were included in the refinement as rigid groups with ideal geometry. It could not be distinguished which of the ring atoms were the N atoms. Therefore all ring atoms were treated as C atoms. The atoms of the disordered pyridine rings were refined with fixed isotropic temperature factors of 0·13 Å^2. Of the 97 H atoms present, 37 were found from difference Fourier synthesis, and were included in the refinement with ideal geometric bond angles and at a distance of

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* Author to whom correspondence should be addressed.

† Permanent address: Departamento de Química-Física, Facultad de Química, Universidad de Oviedo, Calvo Sotelo s/n, 33007 Oviedo, Spain.

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Table 1. *Fractional positional and equivalent isotropic thermal parameters with e.s.d.'s in parentheses*

$$U_{\rm eq}=\tfrac{1}{3}\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$(Å^2 × 10^3)		x	y	z	$U_{\rm eq}$(Å^2 × 10^3)
Molecule *A*					Molecule *C*				
Ni	0·2282 (2)	0·0035 (1)	0·3351 (2)	45 (2)	Ni	0·6869 (2)	0·2565 (1)	0·2756 (2)	45 (6)
O18	0·1946 (6)	0·0530 (3)	0·3059 (7)	46 (6)	O18	0·6386 (6)	0·2105 (3)	0·2446 (6)	46 (8)
N11	0·2681 (8)	0·0123 (4)	0·2504 (9)	43 (8)	N11	0·5979 (8)	0·2726 (4)	0·1844 (9)	44 (1)
C12	0·3051 (10)	−0·0082 (5)	0·2278 (12)	48 (10)	C12	0·5810 (11)	0·3038 (6)	0·1568 (12)	56 (11)
C13	0·3320 (10)	0·0037 (6)	0·1701 (13)	59 [illegible]	C13	0·5174 (12)	0·3100 (6)	0·0903 (14)	67 (13)
C14	0·3189 (10)	0·0373 (6)	0·1447 (12)	58 (11)	C14	0·4755 (11)	0·2823 (6)	0·0576 (11)	59 (12)
C15	0·2636 (11)	0·0945 (5)	0·1427 (11)	56 (11)	C15	0·4563 (10)	0·2192 (6)	0·0551 (11)	46 (10)
C16	0·2266 (13)	0·1139 (5)	0·1722 (14)	69 (12)	C16	0·4754 (10)	0·1886 (5)	0·0879 (12)	43 (10)
C17	0·2021 (10)	0·1016 (5)	0·2278 (12)	52 (11)	C17	0·5350 (9)	0·1848 (4)	0·1544 (10)	32 (8)
C18	0·2168 (9)	0·0674 (6)	0·2555 (11)	49 (10)	C18	0·5785 (9)	0·2124 (5)	0·1853 (10)	34 (8)
C19	0·2564 (8)	0·0451 (5)	0·2261 (9)	31 (8)	C19	0·5563 (10)	0·2452 (5)	0·1502 (11)	37 (10)
C11	0·2815 (9)	0·0600 (5)	0·1681 (11)	40 (9)	C11	0·4926 (10)	0·2485 (5)	0·0842 (11)	40 (10)
Br15	0·2936 (1)	0·1130 (1)	0·0661 (2)	81 (2)	Br15	0·3732 (1)	0·2226 (1)	−0·0398 (1)	80 (1)
Br17	0·1569 (1)	0·1299 (1)	0·2725 (2)	86 (2)	Br17	0·5613 (1)	0·1415 (1)	0·2011 (1)	62 (1)
O28	0·2752 (6)	−0·0426 (3)	0·3616 (6)	42 (6)	O28	0·7226 (6)	0·3055 (3)	0·3068 (7)	44 (6)
N21	0·3181 (7)	0·0187 (4)	0·4261 (8)	37 (7)	N21	0·6480 (7)	0·2669 (4)	0·3623 (9)	47 (8)
C22	0·3337 (11)	0·0504 (5)	0·4512 (12)	53 (10)	C22	0·6125 (11)	0·2462 (5)	0·3860 (11)	56 (10)
C23	0·3956 (14)	0·0568 (5)	0·5167 (14)	62 (13)	C23	0·5836 (9)	0·2549 (5)	0·4445 (11)	45 (10)
C24	0·4393 (11)	0·0304 (6)	0·5552 (11)	59 (11)	C24	0·5929 (11)	0·2887 (5)	0·4729 (12)	55 (11)
C25	0·4614 (9)	−0·0345 (6)	0·5564 (10)	43 (9)	C25	0·6482 (10)	0·3438 (6)	0·4718 (11)	47 (10)
C26	0·4385 (11)	−0·0653 (6)	0·5204 (12)	61 (12)	C26	0·6905 (11)	0·3639 (5)	0·4430 (11)	54 (9)
C27	0·3779 (11)	−0·0683 (5)	0·4539 (11)	47 (11)	C27	0·7120 (10)	0·3509 (5)	0·3864 (11)	53 (11)
C28	0·3326 (11)	−0·0406 (5)	0·4198 (11)	41 (11)	C28	0·6981 (10)	0·3191 (6)	0·3550 (13)	54 (11)
C29	0·3566 (9)	−0·0091 (5)	0·4587 (11)	36 (9)	C29	0·6587 (9)	0·2997 (5)	0·3912 (13)	46 (10)
C21	0·4218 (10)	−0·0039 (5)	0·5244 (10)	35 (10)	C21	0·6323 (10)	0·3096 (5)	0·4478 (10)	40 (10)
Br25	0·5406 (1)	−0·0310 (1)	0·6505 (1)	71 (1)	Br25	0·6185 (1)	0·3629 (1)	0·5478 (1)	79 (1)
Br27	0·3537 (1)	−0·1124 (1)	0·4074 (1)	63 (1)	Br27	0·7599 (2)	0·3824 (1)	0·3472 (2)	90 (2)
N31	0·1414 (7)	−0·0187 (4)	0·2477 (8)	44 (8)	N31	0·7307 (8)	0·2523 (5)	0·1901 (9)	46 (9)
C31	0·0821 (10)	−0·0021 (5)	0·2160 (12)	54 (10)	C31	0·7456 (13)	0·2794 (6)	0·1600 (14)	81 (16)
C32	0·0214 (10)	−0·0152 (8)	0·1575 (13)	80 (12)	C32	0·7721 (13)	0·2774 (6)	0·0995 (14)	79 (14)
C33	0·0260 (14)	−0·0488 (7)	0·1329 (14)	84 (15)	C33	0·7813 (12)	0·2462 (6)	0·0710 (11)	70 (12)
C34	0·0867 (13)	−0·0683 (6)	0·1669 (14)	88 (14)	C34	0·7639 (9)	0·2168 (5)	0·1016 (10)	41 (8)
C35	0·1437 (11)	−0·0518 (5)	0·2248 (11)	60 (11)	C35	0·7424 (10)	0·2218 (5)	0·1622 (12)	46 (10)
N41	0·1850 (8)	−0·0020 (5)	0·4206 (9)	50 (8)	N41	0·7763 (9)	0·2350 (4)	0·3606 (9)	48 (8)
C41	0·1680 (12)	0·0278 (6)	0·4497 (13)	69 (13)	C41	0·8358 (13)	0·2529 (6)	0·3900 (12)	66 (11)
C42	0·1455 (13)	0·0246 (7)	0·5119 (12)	92 (16)	C42	0·8956 (13)	0·2368 (7)	0·4456 (15)	80 (14)
C43	0·1364 (14)	−0·0087 (10)	0·5376 (14)	99 (17)	C43	0·8880 (13)	0·2033 (9)	0·4678 (15)	95 (15)
C44	0·1520 (12)	−0·0375 (8)	0·5080 (16)	84 (16)	C44	0·8262 (15)	0·1854 (6)	0·4410 (14)	87 (14)
C45	0·1770 (11)	−0·0317 (7)	0·4480 (15)	71 (14)	C45	0·7689 (12)	0·2032 (6)	0·3833 (12)	71 (12)
Molecule *B*					Molecule *D*				
Ni	0·2385 (2)	0·4985 (1)	0·3422 (2)	42 (1)	Ni	0·1473 (2)	0·2540 (1)	−0·0465 (2)	44 (2)
O18	0·2086 (6)	0·5490 (3)	0·3184 (7)	42 (6)	O18	0·1928 (6)	0·3013 (3)	−0·0189 (6)	37 (5)
N11	0·2743 (7)	0·5083 (4)	0·2530 (8)	37 (7)	N11	0·2370 (9)	0·2414 (4)	0·0430 (9)	51 (9)
C12	0·3022 (10)	0·4858 (5)	0·2178 (13)	55 (11)	C12	0·2568 (10)	0·2098 (6)	0·0727 (11)	58 (10)
C13	0·3294 (10)	0·4996 (6)	0·1614 (12)	57 (11)	C13	0·3180 (12)	0·2044 (5)	0·1337 (14)	66 (12)
C14	0·3216 (10)	0·5333 (5)	0·1411 (11)	47 (10)	C14	0·3615 (10)	0·2306 (6)	0·1714 (11)	58 (11)
C15	0·2863 (10)	0·5905 (5)	0·1625 (10)	51 (11)	C15	0·3757 (10)	0·2964 (6)	0·1716 (12)	60 (11)
C16	0·2548 (11)	0·6116 (5)	0·2026 (12)	57 (11)	C16	0·3568 (11)	0·3259 (5)	0·1356 (13)	58 (12)
C17	0·2292 (10)	0·5975 (6)	0·2554 (11)	52 (11)	C17	0·2940 (10)	0·3283 (5)	0·0705 (12)	51 (10)
C18	0·2348 (10)	0·5626 (6)	0·2712 (11)	48 (10)	C18	0·2530 (10)	0·2999 (6)	0·0402 (11)	44 (10)
C19	0·2671 (10)	0·5412 (6)	0·2364 (10)	44 (10)	C19	0·2819 (12)	0·2678 (6)	0·0778 (12)	47 (11)
C11	0·2919 (10)	0·5554 (5)	0·1813 (12)	50 (10)	C11	0·3422 (10)	0·2652 (6)	0·1410 (12)	40 (10)
Br15	0·3144 (2)	0·6111 (1)	0·0863 (1)	83 (2)	Br15	0·4606 (1)	0·2914 (1)	0·2641 (1)	86 (1)
Br17	0·1896 (1)	0·6273 (1)	0·3044 (2)	82 (2)	Br17	0·2663 (1)	0·3714 (1)	0·0244 (1)	70 (1)
O28	0·2812 (6)	0·4506 (3)	0·3671 (6)	43 (6)	O28	0·1170 (6)	0·2043 (3)	−0·0717 (7)	45 (6)
N21	0·3296 (7)	0·5112 (4)	0·4331 (8)	37 (7)	N21	0·1863 (7)	0·2440 (4)	−0·1334 (8)	45 (8)
C22	0·3526 (10)	0·5405 (4)	0·4660 (13)	51 (10)	C22	0·2191 (10)	0·2674 (7)	−0·1607 (12)	71 (12)
C23	0·4139 (13)	0·5454 (5)	0·5298 (13)	64 (12)	C23	0·2443 (11)	0·2525 (6)	−0·2170 (13)	67 (12)
C24	0·4516 (11)	0·5161 (6)	0·5640 (12)	63 (11)	C24	0·2396 (11)	0·2210 (7)	−0·2439 (12)	79 (12)
C25	0·4663 (9)	0·4538 (5)	0·5600 (11)	43 (10)	C25	0·1931 (11)	0·1613 (6)	−0·2265 (11)	52 (11)
C26	0·4412 (10)	0·4243 (5)	0·5241 (12)	42 (9)	C26	0·1602 (13)	0·1411 (5)	−0·1906 (14)	70 (12)
C27	0·3806 (10)	0·4230 (4)	0·4571 (11)	36 (10)	C27	0·1379 (11)	0·1559 (6)	−0·1364 (12)	56 (11)
C28	0·3411 (9)	0·4509 (5)	0·4262 (10)	33 (8)	C28	0·1424 (10)	0·1904 (5)	−0·1194 (12)	52 (10)
C29	0·3666 (10)	0·4829 (5)	0·4638 (11)	40 (9)	C29	0·1818 (10)	0·2096 (6)	−0·1598 (14)	60 (12)
C21	0·4311 (10)	0·4855 (5)	0·5305 (11)	38 (9)	C21	0·2090 (11)	0·1975 (6)	−0·2124 (10)	61 (11)
Br25	0·5468 (1)	0·4545 (1)	0·6551 (1)	78 (1)	Br25	0·2218 (2)	0·1410 (1)	−0·3025 (1)	85 (1)
Br27	0·3502 (1)	0·3792 (1)	0·4092 (1)	59 (1)	Br27	0·0935 (2)	0·1263 (1)	−0·0877 (2)	86 (2)
N31	0·1450 (8)	0·4783 (4)	0·2538 (9)	46 (8)	N31	0·1045 (8)	0·2570 (5)	0·0386 (9)	55 (9)
C31	0·0907 (11)	0·4994 (6)	0·2311 (13)	70 (11)	C31	0·0900 (12)	0·2867 (6)	0·0674 (13)	68 (12)
C32	0·0337 (13)	0·4842 (8)	0·1757 (15)	89 (17)	C32	0·0632 (12)	0·2914 (7)	0·1235 (14)	79 (14)
C33	0·0313 (14)	0·4500 (8)	0·1410 (14)	96 (16)	C33	0·0523 (13)	0·2605 (10)	0·1577 (14)	99 (17)
C34	0·0902 (13)	0·4326 (6)	0·1694 (14)	77 (13)	C34	0·0647 (13)	0·2275 (8)	0·1317 (14)	98 (15)
C35	0·1488 (12)	0·4476 (5)	0·2249 (12)	64 (11)	C35	0·0898 (10)	0·2280 (6)	0·0718 (13)	63 (11)
N41	0·1942 (7)	0·4953 (4)	0·4277 (8)	40 (7)	N41	0·0581 (8)	0·2750 (5)	−0·1327 (10)	52 (8)
C41	0·1794 (10)	0·4655 (6)	0·4496 (11)	49 (10)	C41	0·0576 (12)	0·3075 (6)	−0·1603 (13)	70 (12)
C42	0·1526 (12)	0·4630 (6)	0·5066 (14)	62 (13)	C42	−0·0016 (14)	0·3205 (6)	−0·2221 (14)	88 (15)
C43	0·1403 (12)	0·4913 (9)	0·5388 (12)	79 (14)	C43	−0·0610 (12)	0·2998 (7)	−0·2573 (14)	80 (12)
C44	0·1574 (14)	0·5237 (7)	0·5149 (18)	95 (16)	C44	−0·0593 (14)	0·2671 (7)	−0·2273 (15)	86 (16)
C45	0·1849 (11)	0·5262 (6)	0·4584 (14)	67 (12)	C45	0·0017 (15)	0·2563 (6)	−0·1633 (13)	69 (13)

Table 1 (*cont.*)

	x	y	z	U_{iso}(Å^2 × 10^3)
Uncoordinated pyridine molecules				
C111	0·8938 (10)	0·1246 (3)	0·6107 (7)	80 (7)
C112	0·9538 (10)	0·1417 (3)	0·6569 (7)	106 (9)
C113	0·9583 (10)	0·1589 (3)	0·7257 (7)	110 (9)
C114	0·9027 (10)	0·1591 (3)	0·7482 (7)	105 (9)
C115	0·8426 (10)	0·1420 (3)	0·7020 (7)	88 (8)
C116	0·8382 (10)	0·1247 (3)	0·6332 (7)	98 (8)
C211	0·4453 (8)	0·1330 (4)	0·3181 (8)	78 (7)
C212	0·4641 (8)	0·1676 (4)	0·3279 (8)	99 (8)
C213	0·4287 (8)	0·1906 (4)	0·3564 (8)	84 (8)
C214	0·3746 (8)	0·1791 (4)	0·3751 (8)	77 (7)
C215	0·3559 (8)	0·1445 (4)	0·3652 (8)	114 (9)
C216	0·3912 (8)	0·1215 (4)	0·3367 (8)	118 (10)
C311	−0·0491 (8)	0·1049 (4)	0·4426 (12)	106 (9)
C312	0·0103 (8)	0·1211 (4)	0·4936 (12)	89 (8)
C313	0·0686 (8)	0·1210 (4)	0·4761 (12)	104 (9)
C314	0·0674 (8)	0·1046 (4)	0·4076 (12)	101 (9)
C315	0·0080 (8)	0·0884 (4)	0·3566 (12)	122 (10)
C316	−0·0503 (8)	0·0885 (4)	0·3741 (12)	120 (10)
C411	0·5332 (8)	0·4360 (4)	0·2362 (7)	74 (7)
C412	0·4762 (8)	0·4436 (4)	0·2533 (7)	83 (8)
C413	0·4482 (8)	0·4186 (4)	0·2862 (7)	104 (9)
C414	0·4771 (8)	0·3858 (4)	0·3020 (7)	102 (9)
C415	0·5341 (8)	0·3781 (4)	0·2850 (7)	96 (8)
C416	0·5621 (8)	0·4032 (4)	0·2521 (7)	99 (8)
C511†	0·9664 (22)	0·1296 (7)	0·0334 (17)	130
C512†	1·0272 (22)	0·1110 (7)	0·0647 (17)	130
C513†	1·0346 (22)	0·0849 (7)	0·1199 (17)	130
C514†	0·9813 (22)	0·0774 (7)	0·1437 (17)	130
C515†	0·9205 (22)	0·0960 (7)	0·1124 (17)	130
C516†	0·9130 (22)	0·1221 (7)	0·0572 (17)	130
C611‡	0·8918 (25)	0·1597 (12)	0·0074 (28)	130
C612‡	0·9521 (25)	0·1626 (12)	−0·0053 (28)	130
C613‡	1·0058 (25)	0·1398 (12)	0·0318 (28)	130
C614‡	0·9991 (25)	0·1141 (12)	0·0816 (28)	130
C615‡	0·9387 (25)	0·1112 (12)	0·0943 (28)	130
C616‡	0·8851 (25)	0·1340 (12)	0·0572 (28)	130

† Occupation factor 0·60 (1).
‡ Occupation factor 0·40 (1).

1·0 Å from the atoms to which they are bonded. To these H atoms fixed isotropic temperature factors were assigned, equal to the isotropic equivalents of the parent C.

The final conventional agreement factors were $R = 0{\cdot}060$ and $wR = 0{\cdot}058$ for 5983 'observed' reflections and 1467 variables. The function minimized was $\sum w(F_o - F_c)^2$ with $w = 1/[\sigma^2(F_o) + 0{\cdot}00040\,F_o^2]$ with $\sigma(F_o)$ from counting statistics. The maximum shift-to e.s.d. ratio in the last full-matrix least-squares cycle was less than 0·1. The final difference Fourier map showed a residual electron density between −0·27 and 0·32 e Å^{-3}. Fig. 1, which shows one of the four Ni complexes, was drawn with the *PLUTO* program (Motherwell, 1976). Geometrical parameters and the remaining 35 H atoms, which cannot be included in the refinement because of a lack of storage capacity in the local version of the *SHELX* program, were calculated with *PARST* (Nardelli, 1983). Final positional and thermal parameters are given in Table 1.*

A selection of the molecular geometry data is collected in Table 2, in which the four independent molecules are named *A, B, C* and *D*. The geometry and the crystallographic numbering scheme of one of the complexes is given in Fig. 1. Values for selected torsion angles and angles between least-squares planes and lines have been deposited. No unusual geometric features are present in the molecule.

Related literature. This is the third crystal structure determination in a series of Ni coordination compounds which have 8-quinolinol derivatives as ligands. Previously we reported the crystal structures of Ni$^{\text{II}}$ complexes with 5-chloro- (García-Granda & Gómez-Beltrán, 1986) and 5,7-dichloro-8-quinolinol García-Granda *et al.*, 1987) as ligands. The complex shows a similar geometry to that found in the 5,7-dichloro complex and the coordination distances for nickel also

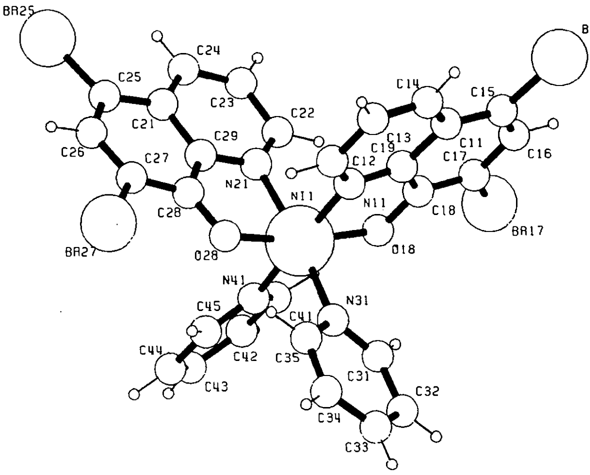

Fig. 1. View of one nickel complex (denoted as *A*) showing the nickel coordination and the atomic numbering. The other three complexes have identical numbering schemes.

Table 2. *Selected bond lengths* (Å) *and angles* (°) (*with e.s.d.'s*)

	A	*B*	*C*	*D*
Ni—O18	2·051 (12)	2·058 (12)	2·030 (11)	2·051 (11)
Ni—O28	2·016 (12)	2·048 (11)	2·048 (12)	2·034 (12)
Ni—N11	2·064 (19)	2·087 (18)	2·071 (14)	2·029 (14)
Ni—N21	2·072 (12)	2·060 (12)	2·092 (19)	2·094 (18)
Ni—N31	2·097 (14)	2·161 (14)	2·114 (20)	2·087 (19)
Ni—N41	2·107 (20)	2·118 (18)	2·103 (15)	2·101 (15)
O18—Ni—N11	81·5 (6)	80·6 (6)	80·6 (6)	79·2 (6)
O18—Ni—N31	94·3 (6)	94·1 (6)	92·6 (6)	93·8 (8)
O18—Ni—N41	94·5 (6)	91·3 (6)	93·9 (6)	92·0 (6)
O28—Ni—O18	170·5 (6)	172·0 (6)	171·7 (6)	171·0 (6)
O28—Ni—N11	91·0 (6)	94·2 (6)	93·8 (6)	93·9 (6)
O28—Ni—N21	81·1 (5)	80·9 (5)	79·7 (6)	80·6 (6)
O28—Ni—N31	91·4 (6)	91·9 (6)	93·3 (6)	91·8 (6)
O28—Ni—N41	93·0 (6)	94·0 (6)	92·0 (6)	95·0 (6)
N11—Ni—N31	89·7 (6)	89·5 (6)	87·5 (6)	87·7 (6)
N11—Ni—N41	176·0 (7)	171·8 (6)	173·4 (6)	170·9 (7)
N21—Ni—O18	93·3 (5)	93·3 (6)	94·3 (6)	93·7 (6)
N21—Ni—N11	91·8 (7)	94·0 (6)	92·4 (6)	92·3 (7)
N21—Ni—N31	172·4 (6)	172·2 (6)	173·0 (7)	172·4 (8)
N21—Ni—N41	88·1 (6)	88·2 (6)	91·6 (6)	90·7 (6)
N31—Ni—N41	90·8 (7)	89·2 (6)	89·0 (7)	90·5 (7)

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, selected torsion angles and the main least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44343 (141 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

compare favourably in the two compounds. The Ni—N distance for the pyridine ligands varies from 2·09 (2) to 2·16 (2) Å against 2·094 (8) and 2·145 (7) Å, average distances for the 5,7-dichloro complex and for the *trans*-5-chloro compound (García-Granda & Gómez-Beltrán, 1986), respectively. The Ni—N distances for the 5,7-dibromo-8-quinolinol ligand are in the range 2·03 (2)–2·09 (2) Å, Ni—O are between 2·01 (1) and 2·06 (1) Å, and the average N—Ni—O quinolinol bite angle is 80·5 (6)°. Recently a crystallographic study on a couple of 8-quinolinol derivatives (Banerjee & Saha, 1986) has been reported.

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Orthorhombic Modification of Dichlorobis(triphenylphosphine)mercury(II)

BY TARLOK S. LOBANA AND MANINDERJIT K. SANDHU

*Department of Chemistry, Guru Nanak Dev University, Amritsar-*143005, *India*

AND MICHAEL R. SNOW AND EDWARD R. T. TIEKINK

Jordan Laboratories, Department of Physical and Inorganic Chemistry, University of Adelaide, South Australia 5001, *Australia*

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Abstract. [$HgCl_2(C_{18}H_{15}P)_2$], $M_r = 796{\cdot}0$, orthorhombic, $Pna2_1$, $a = 17{\cdot}782$ (8), $b = 9{\cdot}9678$ (9), $c = 18{\cdot}283$ (7) Å, $U = 3241$ (4) Å^3, $Z = 4$, D_m (flotation) = 1·64 (1), $D_x = 1{\cdot}631$ Mg m^{-3}, Mo $K\bar{\alpha}$ radiation, $\lambda = 0{\cdot}7107$ Å, $\mu = 4{\cdot}993$ mm^{-1}, $F(000) = 1560$, $T = 295$ (2) K, $R = 0{\cdot}058$ for 2541 observed reflections. The Hg atom in the orthorhombic polymorph of [$HgCl_2\{P(C_6H_5)_3\}_2$] exists in a distorted tetrahedral arrangement of two Cl atoms [Hg—Cl(1) 2·504 (4), Hg—Cl(2) 2·491 (7) Å and Cl(1)—Hg—Cl(2) 106·6 (2)°], which define an approximate mirror plane, and two P atoms [Hg—P(1) 2·503 (6), Hg—P(2) 2·532 (4) Å and P(1)—Hg—P(2) 113·5 (1)°].

Experimental. Well formed crystals were obtained from the symmetrization (Coates & Lauder, 1965) reaction of $(C_6H_5)HgCl.P(C_6H_5)_3$ in acetonitrile solution held at 278–283 K. Enraf–Nonius CAD-4F diffractometer controlled by a PDP8/A computer, graphite-monochromated Mo $K\bar{\alpha}$ radiation; ω:2θ scan technique. Cell parameters on crystal 0·60 × 0·38 × 0·38 mm from least-squares procedure on 25 reflections ($9 \leq \theta \leq 13°$). Analytical absorption correction applied (Sheldrick, 1976); max./min. transmission factors 0·2419 and 0·0588. Space group from analysis. Total of 4286 reflections ($1 \leq \theta \leq 27{\cdot}5°$) measured in the range $-23 \leq h \leq 0$, $-12 \leq k \leq 0$, $-23 \leq l \leq 1$; in addition a number of high-angle Friedel pairs were measured. No significant variation in the net intensities of three reference reflections ($4\bar{7}\bar{4}$, $4\bar{5}\bar{3}$, $2\bar{6}\bar{1}$) measured every 3600 s. 3936 unique reflections; 2541 satisfied $I \geq 2{\cdot}5\sigma(I)$. Structure solved from interpretation of Patterson map; full-matrix least-squares refinement of 118 parameters based on F (Sheldrick, 1976). Anisotropic thermal parameters for Hg, Cl and P; phenyl rings refined as hexagonal rigid groups (Sheldrick, 1976); H atoms included in the model at their calculated positions. At convergence $R = 0{\cdot}058$, wR