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Benzoyl(2,7,12,17-tetraethyl-3,8,13,18-tetramethyl-21*H*,23*H*-porphinato)rhodium(III)

BY R. GRIGG AND J. TROCHA-GRIMSHAW

Department of Chemistry, The Queen's University of Belfast, Belfast BT9 5AG, Northern Ireland

AND K. HENRICK

Department of Chemistry, The Polytechnic of North London, London N7 8DB, England

Abstract. C$_{39}$H$_{41}$N$_4$ORh, M_r = 684·4, triclinic, C$\bar{1}$, a = 12·300 (1), b = 21·621 (3), c = 13·185 (2) Å, α = 90·79 (2), β = 111·13 (1), γ = 91·39 (2)°, U = 3268·9 Å^3, D_c = 1·390 g cm^{-3}, Z = 4, μ(Mo $K\alpha$) = 5·5 cm^{-1}, $F(000)$ = 1424. The final R and R_w values are 0·053 and 0·052 respectively for 3164 observed reflections. The Rh atom lies 0·096 Å out of the plane of the four pyrrole N atoms, away from the benzoyl residue.

Introduction. Oxidative addition of benzaldehyde to the bis[rhodium(I)] complex, [etio-I{Rh(CO)$_2$}$_2$] (etio-I = 2,7,12,17-tetraethyl-3,8,13,18-tetramethyl-21*H*,23*H*-porphine), was shown to give the title compound benzoyl(etio-I)rhodium(III) [(PhCO)Rh(etio-I)] (Abeysekera, Grigg, Trocha-Grimshaw & Viswanatha, 1977). This benzoyl complex showed two ν_{CO} stretching frequencies at 1685 and 1727 cm^{-1} both as a KBr disc and in solution. The crystal structure was investigated to establish whether a conformational process involving two possible orientations of the phenyl ring with respect to the position of the carbonyl group and the macrocycle could be responsible for the observed IR spectrum.

Intensity measurements were made from a crystal of dimensions 0·08 × 0·06 × 0·06 mm with a Nonius CAD-4 diffractometer, graphite monochromator, Mo $K\alpha$ radiation using $\omega/2\theta$ scans (ω-axis/θ-axis scan-speed ratio of 1·333:1). 5722 reflections were measured within the range $3 \le \theta \le 25°$ of which 3164

were considered to be observed [$I \ge 3\sigma(I)$]. Lorentz and polarization corrections were made. No absorption correction was applied. The sample appeared to be homogeneous and unit cells for several crystals were found and all gave the same P$\bar{1}$ cell. The abnormally low values of the P$\bar{1}$ unit-cell angles used in the data collection (a = 12·300, b = 12·293, c = 13·185 Å, α = 68·87, β = 78·89, γ = 61·43°, U = 1631 Å^3, Z = 2) were expected to lead to problems with correlation factors in refinement and consequently the data were transformed to the C$\bar{1}$ cell and the coordinates listed in Table 1 refer to this cell (transformation matrix: 100/$\bar{1}$20/00$\bar{1}$). The structure was solved by the Patterson method and refined by full-matrix least squares with *SHELX* (Sheldrick, 1976). The H-atom positions were estimated geometrically (C–H = 1·08 Å). Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974). In the final cycles of refinement the Rh, N and O atoms were assigned anisotropic thermal parameters. The final R and R_w values were 0·053 and 0·052 with $w = 1/\sigma^2 F$. The maximum residual peak of electron density was 0·31 e Å^{-3}, approximately 0·5 Å from the Rh atom. The refined atomic parameters are given in Table 1.*

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* Lists of structure factors, H-atom coordinates, anisotropic thermal parameters and least-squares-planes data have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36900 (21 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Fractional coordinates and isotropic thermal parameters* $(\text{Å}^2 \times 10^3)$ *for the non-*H *atoms*

	x	y	z	U_{eq}^*/U_{iso}
Rh	0·17253 (6)	0·03548 (3)	0·15019 (5)	35 (1)*
N(1a)	0·0501 (5)	0·0793 (3)	0·0279 (5)	38 (4)*
N(1b)	0·2382 (5)	0·0011 (3)	0·0401 (5)	37 (4)*
N(1c)	0·2845 (6)	−0·0154 (3)	0·2682 (5)	38 (4)*
N(1d)	0·0948 (5)	0·0634 (3)	0·2545 (5)	37 (4)*
C(1a)	−0·0342 (7)	0·1161 (3)	0·0380 (6)	38 (2)
C(2a)	−0·1007 (7)	0·1429 (4)	−0·0661 (6)	44 (2)
C(3a)	−0·0535 (7)	0·1210 (4)	−0·1396 (7)	44 (2)
C(4a)	0·0407 (7)	0·0821 (4)	−0·0792 (6)	47 (2)
C(5a)	0·1140 (7)	0·0516 (3)	−0·1222 (6)	45 (2)
C(6a)	−0·1974 (8)	0·1864 (4)	−0·0852 (7)	60 (3)
C(7a)	−0·0891 (8)	0·1375 (4)	−0·2566 (7)	52 (2)
C(8a)	−0·0305 (9)	0·1960 (5)	−0·2740 (9)	80 (3)
C(1b)	0·2045 (7)	0·0153 (3)	−0·0678 (6)	40 (2)
C(2b)	0·2810 (7)	−0·0145 (3)	−0·1149 (6)	40 (2)
C(3b)	0·3608 (7)	−0·0460 (3)	−0·0342 (6)	40 (2)
C(4b)	0·3342 (7)	−0·0356 (3)	0·0641 (6)	36 (2)
C(5b)	0·3959 (7)	−0·0580 (3)	0·1643 (6)	40 (2)
C(6b)	0·2744 (8)	−0·0073 (4)	−0·2296 (7)	60 (3)
C(7b)	0·4579 (7)	−0·0831 (4)	−0·0419 (7)	50 (2)
C(8b)	0·4236 (8)	−0·1497 (4)	−0·0769 (8)	68 (3)
C(1c)	0·3721 (7)	−0·0494 (3)	0·2599 (6)	38 (2)
C(2c)	0·4393 (7)	−0·0758 (3)	0·3634 (6)	41 (2)
C(3c)	0·3884 (7)	−0·0573 (4)	0·4344 (7)	42 (2)
C(4c)	0·2930 (7)	−0·0190 (3)	0·3764 (6)	43 (2)
C(5c)	0·2214 (7)	0·0119 (3)	0·4184 (6)	45 (2)
C(6c)	0·5433 (8)	−0·1139 (4)	0·3856 (7)	56 (2)
C(7c)	0·4217 (8)	−0·0743 (4)	0·5526 (7)	52 (2)
C(8c)	0·3715 (10)	−0·1365 (5)	0·5658 (9)	81 (3)
C(1d)	0·1306 (7)	0·0506 (3)	0·3637 (7)	42 (2)
C(2d)	0·0580 (7)	0·0834 (4)	0·4114 (7)	48 (2)
C(3d)	−0·0173 (7)	0·1156 (4)	0·3327 (6)	45 (2)
C(4d)	0·0053 (7)	0·1027 (3)	0·2340 (6)	42 (2)
C(5d)	−0·0544 (7)	0·1269 (4)	0·1339 (6)	44 (2)
C(6d)	0·0730 (8)	0·0818 (4)	0·5306 (7)	60 (3)
C(7d)	−0·1095 (3)	0·1586 (4)	0·3407 (8)	61 (3)
C(8d)	−0·0678 (11)	0·2244 (5)	0·3581 (10)	97 (4)
O(1)	0·3882 (5)	0·0963 (2)	0·2003 (5)	59 (4)*
C(9)	0·2872 (7)	0·1047 (3)	0·1855 (6)	39 (2)
C(10)	0·2467 (7)	0·1679 (4)	0·1983 (6)	48 (2)
C(11)	0·2042 (9)	0·2050 (5)	0·1083 (8)	77 (3)
C(12)	0·1757 (10)	0·2668 (5)	0·1224 (11)	98 (4)
C(13)	0·1877 (10)	0·2867 (6)	0·2192 (10)	99 (4)
C(14)	0·2286 (11)	0·2538 (5)	0·3111 (11)	99 (4)
C(15)	0·2590 (9)	0·1911 (5)	0·2989 (8)	74 (3)

$$* U_{eq} = \tfrac{1}{3}(U_{11} + U_{22} + U_{33}).$$

Discussion. [(PhCO)Rh(etio-I)] exists as a discrete five-coordinate approximately square-pyramidal metalloporphyrin with the benzoyl residue as the axial ligand (Fig. 1). The Rh atom lies 0·096 Å out of the plane of the four pyrrole N atoms, away from the benzoyl residue. The macrocycle appears not to block approach of a second *trans*-axial ligand to allow Rh^{III} to exist in its favoured octahedral geometry. The least-squares planes defined by the N atoms, α-C atoms, *meso*-C atoms, and β-C atoms, respectively, are all nearly parallel and are separated by successive distances of 0·023, 0·010 and 0·011 Å. The N_4 plane

lies at a greater distance from the Rh atom than the corresponding β-C plane. The displacement of the Rh atom towards the vacant axial coordination site is unusual in metalloporphyrins. The doming effect found for methyl(5,10,15,20-tetraphenylporphinato)-thallium(III) (Henrick, Matthews & Tasker, 1977) is that the planes as defined above are separated by 0·10, 0·03 and 0·11 Å with the N_4 plane lying closest to the Tl atom. The overall geometry for the $C_{20}N_4$ core in [(PhCO)Rh(etio-I)] can be represented as a superficial ruffled (Hoard, 1971) arrangement (maximum deviation from planarity of 0·06 Å), where [Ni(OEP)] is the exaggerated ruffled prototype (Meyer, 1972) (OEP = 2,3,7,8,12,13,17,18-octaethylporphyrin).

The adjacent pyrrole rings in [(PhCO)Rh(etio-I)] are tilted with respect to each other at angles of *ca* 2·8° giving an average Rh—N distance of 2·032 (7) Å with a $C_t \cdots N$ distance of 2·029 Å (C_t = the centroid of the pyrrole N atoms). This small tilting of the adjacent pyrrole rings may be required to minimize the radial strain in the porphinato core as the $C_t \cdots N$ distance in this complex is greater than the optimum value of 2·01 Å (Collins & Hoard, 1970). The plane of the phenyl ring of the benzoyl ligand lies at an angle of 35·9 (10)° to the N_4 plane (Fig. 2).

Coordinate bond lengths, polyhedral edge lengths, and the angles subtended at the Rh atom are given in Table 2. Distortions from square-pyramidal geometry result from a bending of the Rh—C bond by 3·4 (10)° from the normal to the N plane, giving two pairs of $C \cdots N$ edge lengths of 2·95 (1) and 2·84 (1) Å.

There is good agreement between chemically equivalent bond lengths and angles in the porphinato core (Table 3) with no evidence of bond alternation as found for the unsymmetrically substituted iron(III) protoporphyrin IX dimethyl ester p-nitrobenzenethiolate (Lang *et al.*, 1976). The β-C bond distance [C(2)—C(3)
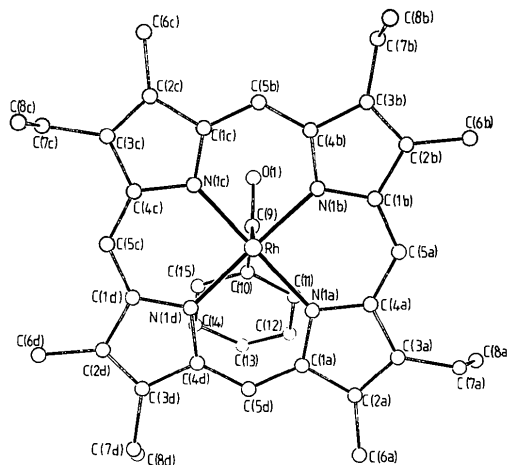

Fig. 1. Perspective view of the [(PhCO)Rh(etio-I)] molecule (*ORTEP*, Johnson, 1965).
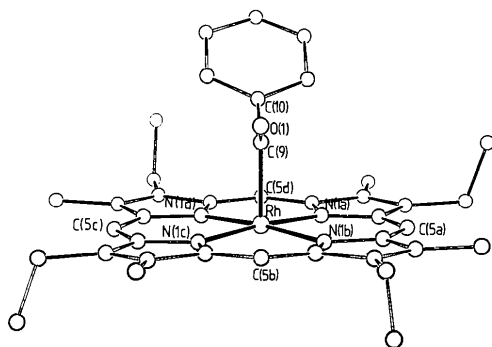
Fig. 2. *ORTEP* drawing of |(PhCO)Rh(etio-I)|: the view is at an angle of 90° from that of (Fig. 1 and 5° from the normal to the axis of the Rh–C bond.

$= 1.360\,\text{Å}|$ is marginally longer than the value of 1·34 Å found for |(Me$_2$NH)$_2$Rh(etio-I)|Cl (Hanson, Gouterman & Hanson, 1973), but is the same as the average reported for several metalloporphyrin structures, 1·358 Å (Hoard, 1971). The remaining bond lengths and angles in the porphinato core are in agreement with the equivalent values found for both the planar six-coordinate complex |(Me$_2$NH)$_2$Rh(etio-I)|Cl and the five-coordinate complex |(pyridine)Zn(OEP)| (Cullen & Meyer, 1976).

The average Rh–N bond length found here (2·032 Å) is similar to that reported in other Rh porphyrins of 2·038 Å (Hanson *et al.*, 1973), 2·031 Å for |MeRh(OEP)| (Takenaka, Syal, Sasada, Omura, Ogoshi & Yoshida, 1976), and 2·03 Å (Fleischer, Dixon & Florian, 1973). The 'structural radii' $C_t\cdots C_a$ and $C_t\cdots C_{meso}$ for |(PhCO)Rh(etio-I)| are 3·055 and 3·413 Å and are intermediate between the same distances for |Cl$_2$Sn(OEP)|, 3·099 and 3·424 Å, (Cullen & Meyer, 1973) and the planar |Ni(OEP)| of 3·006 and 3·381 Å (Cullen & Meyer, 1974) which is consistent with the relative sizes of the central metal atoms.

Table 2. *Dimensions of the coordination group of* |(PhCO)Rh(etio-I)|

(i) Distances (Å)

Rh–C(9)	1·963 (7)	Rh–N(1a)	2·031 (6)
Rh–N(1b)	2·039 (6)	Rh–N(1c)	2·025 (6)
Rh–N(1d)	2·031 (6)		

(ii) Angles (°)

C(9)–Rh–N(1a)	94·8 (4)	N(1a)–Rh–N(1b)	89·2 (2)
C(9)–Rh–N(1b)	90·3 (4)	N(1b)–Rh–N(1c)	90·3 (2)
C(9)–Rh–N(1c)	90·6 (4)	N(1c)–Rh–N(1d)	90·4 (2)
C(9) Rh N(1d)	95·2 (4)	N(1d)–Rh–N(1a)	89·6 (2)
N(1a)–Rh–N(1c)	174·6 (3)	N(1b)–Rh–N(1d)	174·5 (3)

(iii) Polyhedral edge lengths (Å)

C(9)···N(1a)	2·94 (1)	N(1a)···N(1b)	2·86 (1)
C(9)···N(1b)	2·84 (1)	N(1b)···N(1c)	2·88 (1)
C(9)···N(1c)	2·84 (1)	N(1c)···N(1d)	2·88 (1)
C(9)···N(1d)	2·95 (1)	N(1d)···N(1a)	2·86 (1)

Table 3. *Interatomic distances* (Å) *and angles* (°) *in the porphyrin skeleton of* |(PhCO)Rh(etio-I)|

	Subunit			
	a	*b*	*c*	*d*
N(1)–C(1)	1·366 (9)	1·373 (9)	1·354 (9)	1·380 (9)
N(1)–C(4)	1·378 (9)	1·381 (9)	1·396 (9)	1·356 (9)
C(1)–C(2)	1·457 (10)	1·459 (10)	1·450 (10)	1·456 (10)
C(1)·C(5x)*	1·391 (10)	1·360 (10)	1·404 (9)	1·396 (10)
C(3) C(4)	1·444 (11)	1·462 (10)	1·436 (10)	1·451 (10)
C(2)–C(3)	1·381 (10)	1·364 (10)	1·361 (10)	1·334 (10)
C(4)–C(5)	1·399 (10)	1·367 (10)	1·378 (10)	1·377 (10)
C(2)–C(6)	1·486 (11)	1·496 (10)	1·480 (10)	1·516 (11)
C(3)·C(7)	1·494 (11)	1·488 (10)	1·515 (11)	1·515 (11)
C(7)–C(8)	1·503 (11)	1·508 (11)	1·506 (12)	1·487 (12)
C(1)–N(1)–C(4)	106·2 (6)	107·3 (6)	105·9 (6)	106·5 (6)
N(1)–C(1)·C(2)	110·5 (6)	109·3 (7)	111·1 (7)	109·0 (7)
N(1)–C(1) C(5x)*	125·1 (7)	125·4 (7)	124·9 (7)	125·0 (7)
C(2) C(1)··C(5x)*	124·4 (7)	125·3 (7)	124·0 (7)	126·0 (7)
N(1)–C(4)–C(3)	110·8 (7)	109·3 (6)	109·4 (7)	110·2 (7)
N(1)–C(4)·C(5)	124·0 (7)	125·4 (7)	123·5 (7)	124·7 (7)
C(3)–C(4)·C(5)	125·2 (7)	125·2 (7)	127·0 (7)	125·1 (7)
C(1) C(2)–C(3)	106·3 (7)	107·4 (7)	106·0 (7)	107·4 (7)
C(1)··C(2) C(6)	125·4 (7)	124·7 (7)	126·0 (7)	124·3 (7)
C(3)–C(2)–C(6)	128·2 (8)	127·9 (7)	127·9 (8)	128·3 (8)
C(2) C(3)··C(4)	106·2 (7)	106·6 (7)	107·6 (7)	107·0 (7)
C(2)–C(3)· C(7)	126·5 (8)	127·6 (7)	127·5 (8)	128·3 (7)
C(4)–C(3)–C(7)	127·3 (7)	125·8 (7)	124·9 (7)	124·7 (7)
C(3)–C(7)–C(8)	113·3 (7)	113·9 (7)	112·5 (7)	112·6 (8)
C(4)–C(5)–C(1x)*	126·9 (7)	126·6 (7)	127·6 (8)	126·7 (7)

Benzoyl group

C(9)–O(1)	1·205 (8)	C(9)–C(10)	1·493 (10)
C(10)–C(11)	1·388 (12)	C(11)–C(12)	1·417 (13)
C(12)–C(13)	1·296 (15)	C(13)–C(14)	1·354 (14)
C(14)–C(15)	1·436 (14)	C(15) C(10)	1·366 (11)
O(1)–C(9) Rh	121·0 (6)	C(10)–C(9)– Rh	118·0 (6)
O(1)· C(9)–C(10)	120·9 (7)	C(9) C(10) –C(11)	119·5 (7)
C(9)–C(10)· C(15)	120·7 (8)	C(11)–C(10)–C(15)	119·6 (8)
C(10)· C(11)·C(12)	119·4 (10)	C(11)–C(12)–C(13)	118·9 (12)
C(12)· C(13)–C(14)	125·5 (13)	C(13)–C(14)–C(15)	116·5 (12)
C(14)–C(15) C(10)	120·0 (10)		

* *x* refers to an atom in the adjacent subunit of the porphine as in Fig. 1.

The Rh–C(benzoyl) distance (1·963 Å) is shorter than the Rh–C(alkyl) distance of 2·081 Å in the square-pyramidal Rh$^{\text{III}}$ complex |Me(PPh$_3$)$_2$I$_2$Rh| (Troughton & Skapski, 1968) and of 2·031 Å in |MeRh(OEP)| (Takenaka *et al.*, 1976). A similar result was observed for Mo compounds where Mo–acetyl and Mo–alkyl bond lengths of 2·264 and 2·383 Å were found for |(C$_5$H$_5$)(MeCO)(CO)$_2$(PPh$_3$)Mo| (Churchill & Fennessey, 1968) and |(C$_{10}$H$_8$)-Me(CO)$_2$Mo| (Bird & Churchill, 1968) respectively. The shortening of the metal–acyl bond is generally accounted for by invoking d_π–p_π metal to acyl back bonding; however, if back bonding were important in this case the ν_{CO} stretch should differ substantially from normal whereas the ν_{CO} stretch in |(PhCO)Rh(etio-I)| corresponds approximately to that of MeCOPh, and the shorter Rh–C bond length in the acyl case is due to C(sp^2)– as compared to C(sp^3)–Rh bonding.

The only intramolecular contact of any significance close to the Rh atom (apart from the atoms of the C porphinato core) is of 2·783 (5) Å to O(1) and there

are no intermolecular contacts less than 3·5 Å to the Rh atom.

In conclusion we can find no explanation of the double ν_{CO} stretching frequency for this compound from the X-ray analysis.

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Structure du Di-μ-acetato-bis[acetatobis(méthyl-1 imidazole)cuivre(II)] Hexahydraté

PAR Y. BOUKARI

Ecole des Sciences, Laboratoire de Chimie, Université du Bénin, BP 1515, Lome, Togo

ET A. BUSNOT, F. BUSNOT, A. LECLAIRE ET M. A. BERNARD

Laboratoire de Cristallographie et Chimie du Solide, LA 251, Laboratoire de Chimie Minérale B et de Cristallographie–Minéralogie, ISMRA–Université de Caen, 14032 Caen CEDEX, France

Abstract. $[Cu_2(C_4H_6N_2)_4(C_2H_3O_2)_4].6H_2O$, $P\bar{1}$, $a = 9·7257$ (7), $b = 10·0972$ (11), $c = 10·2894$ (8) Å, $\alpha = 85·468$ (8), $\beta = 76·811$ (6), $\gamma = 68·206$ (7)°, $Z = 2$, $V = 913$ Å³, $d_m = 1·45$, $d_c = 1·45$ Mg m⁻³, $\mu = 1·281$ mm⁻¹. The structure was refined to an R of 0·032 and $R_w = 0·035$ with 5285 reflexions [$I > 3\sigma(I)$]. Each Cu atom has a distorted pyramidal environment with a square base. The compound has a binuclear structure with monatomic bridges. There is no water molecule around the copper.

Introduction. Un grand nombre de chercheurs parmi lesquels Baird (1968), Catterick & Thornton (1977), Kato, Jonassen & Fanning (1964), Oldham (1968), se sont intéressés durant ces dernières années aux carboxylates de cuivre en raison de la 'plasticité' de la sphère de coordination du cuivre.

Ce travail prolonge des études effectuées au laboratoire (Borel, Busnot, Busnot, Leclaire & Bernard, 1981a) sur les structures de carboxylates de cuivre(II) diversement solvatés [notamment sur leurs corrélations et sur leurs rapports de filiation: Borel, Busnot, Busnot & Leclaire (1980), Borel, Busnot, Busnot, Leclaire & Bernard (1981b)]. Il nous a semblé intéressant, dans cette optique, d'utiliser comme ligand l'imidazole et ses dérivés. L'un des intérêts de l'imidazole, en tant que fragment de l'histidine tient à ce que l'azote pyridiné du cycle est un site possible de fixation sur le cuivre dans