

Monoclinic

 $P2_1/n$ $a = 8.553 (4) \text{ \AA}$ $b = 12.860 (5) \text{ \AA}$ $c = 15.530 (7) \text{ \AA}$ $\beta = 104.08 (4)^\circ$ $V = 1656.9 (13) \text{ \AA}^3$ $Z = 4$ $D_x = 1.632 \text{ Mg m}^{-3}$ D_m not measured

Data collection

Hilger & Watts Y290 four-circle diffractometer

 $2\theta/\omega$ scans

Absorption correction: none

3819 measured reflections

3819 independent reflections

2805 reflections with

 $I > 2\sigma(I)$

Cell parameters from 25 reflections

 $\theta = 21.2\text{--}26.7^\circ$ $\mu = 0.935 \text{ mm}^{-1}$ $T = 153 (2) \text{ K}$

Block

 $0.3 \times 0.2 \times 0.2 \text{ mm}$

Blue-green

 $R_{\text{int}} = 0.019$ $\theta_{\text{max}} = 27.56^\circ$ $h = -1 \rightarrow 11$ $k = -1 \rightarrow 16$ $l = -20 \rightarrow 19$

3 standard reflections

every 97 reflections

intensity decay: 1%

Refinement

Refinement on F^2 $R[F^2 > 2\sigma(F^2)] = 0.057$ $wR(F^2) = 0.151$ $S = 1.031$

3819 reflections

217 parameters

H atoms: see below

 $w = 1/[\sigma^2(F_o^2) + (0.0713P)^2 + 3.8059P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\text{max}} = 0.001$ $\Delta\rho_{\text{max}} = 0.841 \text{ e \AA}^{-3}$ $\Delta\rho_{\text{min}} = -0.943 \text{ e \AA}^{-3}$

Extinction correction: none

Scattering factors from

International Tables for Crystallography (Vol. C)

The title structure was solved using direct methods. All non-H atoms were refined anisotropically. The H atoms of the pyrazolyl ligand were found explicitly and were refined isotropically. All other H atoms were fixed at ideal positions with common isotropic displacement parameters.

Data collection: Y290 (Abeln & Kopf, 1993). Cell refinement: Y290. Data reduction: Y290. Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997). Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1992). Software used to prepare material for publication: *SHELXL97*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: OS1019). Services for accessing these data are described at the back of the journal.

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Bromotricarbonyl(3,3'-dimethyl-2,2'-bi-quinoline-*N,N'*)rhenium(I)

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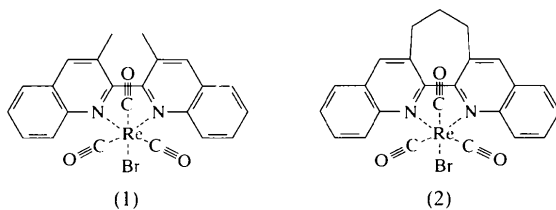
Abstract

In the title compound, [ReBr(C₂₀H₁₆N₂)(CO)₃], the Re atom has a slightly distorted octahedral coordination, with Re—Br = 2.6299 (9), Re—N = 2.217 (4) and Re—C = 1.902 (6)–1.927 (7) Å.

Comment

The coordination chemistry of transition metal complexes containing polypyridinic ligands has led to a significant amount of work over the last few decades, both because this type of compound shows interesting photo-physical and photochemical behaviour and because they are potential candidates as models in photosynthesis processes (Juris *et al.*, 1988; Moya *et al.*, 1994, 1996; Sartori *et al.*, 1992; Ferraudi *et al.*, 1995; Kutal *et al.*, 1987; Kubow *et al.*, 1988, and references therein; Kaliasundaram, 1992; Gratzel, 1983).

We report here the X-ray structure determination of $\text{BrRe}(\text{CO})_3(\text{Me},2\text{N})$, (1) [(Me,2N) = 3,3'-dimethyl-2,2'-biquinoline] and compare the results with those of $\text{BrRe}(\text{CO})_3(3,2\text{N})$ [(3,2N) = 3,3'-trimethylene-2,2'-biquinoline], (2) (Moya *et al.*, 1994). The 3,3'-substitutions on the 2,2'-biquinoline give interesting steric properties to these compounds, which may lead to an improvement of their catalytic and photophysical properties.



The title compound is mononuclear, with a single molecule in the asymmetric unit (Fig. 1). The structure displays a rhenium environment rather similar to that found in other compounds of the series with the same substitution pattern. This is a slightly distorted octahedron with the bromine ion and C1 from one of the carbonyl units occupying the apical sites, and a basal plane defined by C2 and C3 from the remaining carbonyl groups, and by N1 and N2 from the organic ligand. The metal atom is slightly displaced away from the negatively charged Br1 ligand along the apical line [0.113(1) Å], thus giving the base the aspect of a depressed square pyramid. The apical axis is rather linear [Br1—Re1—C1 = 179.2(2)°], with a small departure from the mean plane normal [2.7(1)°]. Analogous considerations of linearity can be made about the Re—carbonyl interactions, with Re—C—O angles in the range 176.7(5)–177.1(6)°.

The organic ligand departs significantly from planarity in order to minimize the otherwise important steric hindrance between the terminal methyl groups. This is mainly achieved through a rotation of the quino-

line groups around the C13—C14 bond, leading to an interplanar angle between them of 37.9(1)° and a C4...C23 distance of 3.05(1) Å, with a closest approach between H atoms of about 2.33 Å.

The need to avoid eventual atomic overlap leads to another structural peculiarity: both in (1), as well as in the previously reported homologue (2), there is a similar striking inclination of the ligand with respect to the equatorial plane of the cation. This configuration is adopted due to the bulky character of the former in order to avoid as much as possible the otherwise important steric hindrance imposed by the equatorial carbonyl groups. In the case of the rather planar molecule (2), an estimate of the effect can be ascertained through inspection of the dihedral angle between the mean plane through the ligand core and the polyhedron equatorial plane (in our present notation, atoms N1, C13, C14, N2 and N1, N2, C2, C3, respectively), which amounts to 32(1)°. Although also significant, the effect can hardly be quantified in (1) due to the existence of a largely deformed non-planar core. In both cases, the ligand is tilted towards the negatively charged side of the coordination sphere.

There are a variety of short Br...H contacts in the structure (calculated values in the range 2.97–3.23 Å) leading to a two-dimensional network parallel to (100). Neighbouring planes are held together by normal van der Waals forces.

Experimental

Treatment of the ligand with a solution of bromopentacarbonylrhenium(I) in a mixture of petroleum benzene (373–413 K) and toluene (inert atmosphere, reflux temperatures, short time) gave a high yield of a deep-red precipitate, which was recrystallized from dichloromethane as small red crystals. The compound obtained proved stable in air, both in the solid state and in solution. The solubility was found to be rather low in the majority of solvents in common use. Conductivity measurements performed in CH_2Cl_2 indicated a non-electrolyte species. IR spectra exhibited three absorption bands in the carbonyl stretching region, in accordance with a *fac*-conformation. The elemental analysis was in good agreement with the formulated structure.

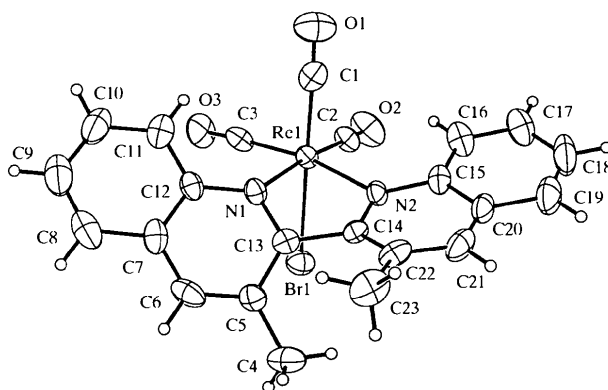


Fig. 1. A molecular diagram of (1) showing the labelling scheme used. Displacement ellipsoids are drawn at the 50% probability level.

Crystal data

[$\text{ReBr}(\text{C}_{20}\text{H}_{16}\text{N}_2)(\text{CO})_3$]

$M_r = 634.49$

Monoclinic

$P2_1/c$

$a = 12.235(3) \text{ \AA}$

$b = 9.645(2) \text{ \AA}$

$c = 18.596(6) \text{ \AA}$

$\beta = 105.97(2)^\circ$

$V = 2109.8(10) \text{ \AA}^3$

$Z = 4$

$D_x = 2.00 \text{ Mg m}^{-3}$

D_m not measured

Mo $K\alpha$ radiation

$\lambda = 0.71073 \text{ \AA}$

Cell parameters from 25 reflections

$\theta = 7.5\text{--}12.5^\circ$

$\mu = 7.68 \text{ mm}^{-1}$

$T = 293(2) \text{ K}$

Prism

$0.38 \times 0.22 \times 0.22 \text{ mm}$

Red

Data collection

Siemens R3M diffractometer $R_{\text{int}} = 0.032$
 $\theta/2\theta$ scans $\theta_{\text{max}} = 25.15^\circ$
 Absorption correction: $h = -14 \rightarrow 13$
 ψ scan (SHELXTLPC; $k = 0 \rightarrow 11$
 Sheldrick, 1994) $l = 0 \rightarrow 22$
 $T_{\text{min}} = 0.127$, $T_{\text{max}} = 0.185$ 2 standard reflections
 3997 measured reflections every 98 reflections
 3785 independent reflections intensity decay: <2%
 2614 reflections with
 $I > 2\sigma(I)$

Refinement

Refinement on F^2 $(\Delta/\sigma)_{\text{max}} = <0.01$
 $R[F^2 > 2\sigma(F^2)] = 0.027$ $\Delta\rho_{\text{max}} = 0.512 \text{ e } \text{\AA}^{-3}$
 $wR(F^2) = 0.055$ $\Delta\rho_{\text{min}} = -0.489 \text{ e } \text{\AA}^{-3}$
 $S = 0.926$ Extinction correction: none
 3785 reflections Scattering factors from
 287 parameters *International Tables for*
 H atoms riding *Crystallography* (Vol. C)
 $w = 1/[\sigma^2(F_o^2) + (0.024P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$

Table 1. Selected bond lengths (Å)

Re—C1	1.927 (7)	Re—N1	2.217 (4)
Re—C2	1.900 (6)	Re—N2	2.217 (4)
Re—C3	1.912 (6)	Re—Br	2.630 (1)

Data collection: P3/P4-PC (Siemens, 1991). Cell refinement: P3/P4-PC. Data reduction: SHELXTLPC (Sheldrick, 1994). Program(s) used to solve structure: SHELXS97 (Sheldrick, 1990). Program(s) used to refine structure: SHELXL97 (Sheldrick, 1997). Molecular graphics: SHELXTLPC. Software used to prepare material for publication: CSD (Allen & Kennard, 1993), SHELXL97 and PARST (Nardelli, 1983).

Data collection was performed at the Universidad de Chile on a single-crystal diffractometer purchased by Fundación Andes.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: FG1459). Services for accessing these data are described at the back of the journal.

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Bis(1-ethyl-3-methylimidazolium) Tetra-chloropalladate(II)

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Abstract

The title compound, (C₆H₁₁N₂)₂[PdCl₄], is a salt that consists of a square-planar tetrachloropalladate(II) anion having interactions, *via* the chlorides, to ring H atoms on two 1-ethyl-3-methylimidazolium (MEI⁺) cations. The Pd atom resides at a center of inversion. Cl[−]⋯H distances range from 2.75 to 3.24 Å, some of which suggest hydrogen bonding. As the title compound was obtained from an acidic room-temperature melt of the chloride salt of the MEI⁺ cation and AlCl₃, a possible use of this type of system as an alternate medium for crystallization is indicated.

Comment

The 1-ethyl-3-methylimidazolium cation (MEI⁺) is an important constituent of the room-temperature molten salt system produced by mixing 1-ethyl-3-methylimidazolium chloride (MEIC) and aluminium chloride. Previous studies (Scordilis-Kelly & Carlin, 1993; Carlin *et al.*, 1994; De Long *et al.*, 1994; Lee *et al.*, 1996) of this unique molten salt system have centered around electrochemical studies of its utility as a room-temperature electrolyte for rechargeable energy-storage batteries, as well as a medium for electrodeposition of metal species solvated in the melt.