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Key indicators

Single-crystal X-ray study
 $T = 110$ K
Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
 R factor = 0.023
 wR factor = 0.045
Data-to-parameter ratio = 45.6For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Tetraethylammonium di- μ -methoxo- μ -pyridin-
2-olato-bis[tricarbonylrhenium(I)]

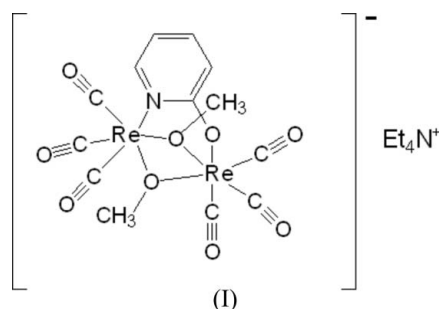
The title compound, $(\text{C}_8\text{H}_{20}\text{N})[\text{Re}_2(\text{C}_5\text{H}_4\text{NO})(\text{CH}_3\text{O})_2(\text{CO})_6]$, was obtained from a ligand-substitution reaction of $(\text{Et}_4\text{N})[\text{Re}_2(\text{OMe})_3(\text{CO})_6]$ with 2-hydroxypyridine in methanol solvent. Both Re centers are in an approximate octahedral geometry.

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Comment

Previously, we have presented the results of ligand exchange of the anion of $(\text{Et}_4\text{N})[\text{Re}_2(\text{CO})_6(\text{OMe})_3]$, with functionalized aryl alcohols (Klausmeyer & Beckles, 2005). In each of these cases, the alcohol contained a distal group that was unable to chelate to the dinuclear Re center. Here we present the results of the reaction with 2-hydroxypyridine which shows, rather than a bridging O as in previous cases, a singly bound O anion with the pyridine nitrogen reaching over to coordinate with the second Re atom.



Both Re centers in the title compound, (I), are in an approximate octahedral geometry including three carbonyls with all Re—C bond lengths in the expected ranges. For Re1, the coordination environment also consists of two O atoms from the bridging methoxides and the N atom of the bridging hydroxypyridine (Fig. 1). The coordination about Re2 is completed by the two bridging methoxides and the non-bridging O from the hydroxypyridine. This complex provides a rather rare example of a terminal alkoxide coordinating to a metal. The non-bonding Re centers are separated by 3.3385 (3) Å.

One tetraethylammonium cation is present to balance the charge of the anion.

Selected geometric parameters are presented in Table 1. All other bond lengths and angles are within expected ranges.

Experimental

Compound (I) was obtained by the ligand-exchange reaction of $(\text{Et}_4\text{N})[\text{Re}_2(\text{OMe})_3(\text{CO})_6]$ with 1 equivalent of 2-hydroxypyridine. In a typical experiment, $(\text{Et}_4\text{N})[\text{Re}_2(\text{OMe})_3(\text{CO})_6]$ (0.100 g) was

dissolved in CH₃OH (20 ml) and 2-hydroxypyridine (1 equivalent) in CH₃OH (15 ml) was added. This solution was stirred overnight at room temperature. The solvent was then removed at reduced pressure to a final volume of about 4 ml and the complex was precipitated from solution by addition of diethyl ether (60 ml), resulting in a yellow solid. Diffraction quality crystals of (I) were obtained by vapor diffusion of diethyl ether into a concentrated acetonitrile solution.

Crystal data

(C₈H₂₀N)[Re₂(C₅H₄NO)-(CH₃O)₂(CO)₆]
M_r = 826.87
 Monoclinic, *P*2₁/*c*
a = 9.3185 (12) Å
b = 25.148 (3) Å
c = 11.7267 (14) Å
 β = 110.288 (3)°
V = 2577.5 (5) Å³
Z = 4
D_x = 2.131 Mg m⁻³
 Mo Kα radiation
 μ = 9.43 mm⁻¹
T = 110 (2) K
 Block, colorless
 0.22 × 0.19 × 0.12 mm

Data collection

Bruker X8 APEX area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
T_{min} = 0.142, *T_{max}* = 0.319
 117327 measured reflections
 14267 independent reflections
 12348 reflections with *I* > 2σ(*I*)
R_{int} = 0.044
 θ_{max} = 38.3°

Refinement

Refinement on *F*²
R [*F*² > 2σ(*F*²)] = 0.023
wR (*F*²) = 0.045
S = 1.07
 14267 reflections
 313 parameters
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.013P)^2 + 2.7693P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 (Δ/σ)_{max} = 0.006
 Δρ_{max} = 1.77 e Å⁻³
 Δρ_{min} = -1.16 e Å⁻³

Table 1

Selected geometric parameters (Å, °).

Re1—O8	2.1195 (13)	Re2—O8	2.1271 (13)
Re1—O7	2.1212 (13)	Re2—O7	2.1350 (13)
Re1—N1	2.2223 (15)	Re2—O9	2.1374 (13)
O8—Re1—O7	76.67 (5)	O8—Re2—O9	77.81 (5)
O8—Re1—N1	82.61 (5)	O7—Re2—O9	81.44 (5)
O7—Re1—N1	81.57 (5)	Re1—O7—Re2	103.33 (5)
O8—Re2—O7	76.21 (5)	Re1—O8—Re2	103.66 (5)

All H atoms were included in calculated positions, with C—H = 0.95–0.99 Å and *U*_{iso}(H) = 1.2*U*_{eq}(C) or 1.5*U*_{eq}(methyl C). The highest residual density peak and the deepest hole are located 0.62 and 0.52 Å, respectively, from atom Re2.

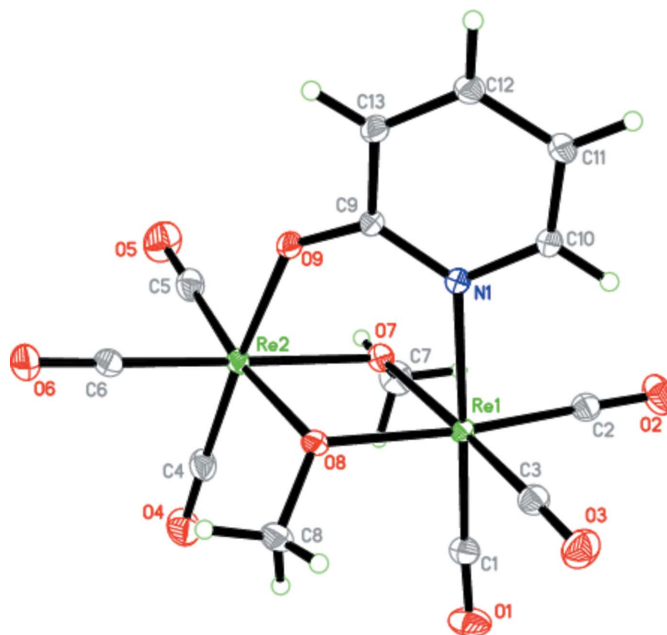


Figure 1
 A view of the complex anion in (I), showing displacement ellipsoids at the 50% probability level.

Data collection: APEX2 (Bruker, 2003); cell refinement: APEX2; data reduction: SAINT-Plus (Bruker, 2003); program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL (Sheldrick, 2000); software used to prepare material for publication: SHELXTL.

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