

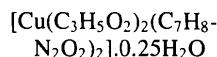
2.653 (2) Å] to complete a distorted octahedral environment around Cu^{II}. The C1—O1 and C1—O2 distances [1.268 (4) and 1.227 (4) Å, respectively] are significantly different.

The propionate chelate angle O1—Cu—O2 is very acute at 54.23 (8)°. When propionate is coordinated as a chelate, the mean Cu—O bond distance of 2.30 Å is about 0.33 Å longer than when coordinated in a *syn-syn* arrangement (Melník, 1982).

Experimental

The title compound was prepared by treating methyl 3-pyridyl-carbamate (3.043 g, 0.02 mol) with copper(II) propionate (2.097 g, 0.01 mol) in hot methanol solution (50 ml). The crude product was recrystallized from methanol to give single crystals.

Crystal data



$M_r = 518.5$

Tetragonal

$P4_2/n$

$a = 20.674 (3) \text{ \AA}$

$c = 5.8040 (10) \text{ \AA}$

$V = 2480.7 (7) \text{ \AA}^3$

$Z = 4$

$D_x = 1.388 \text{ Mg m}^{-3}$

$D_m = 1.40 \text{ Mg m}^{-3}$

D_m measured by flotation in $\text{C}_6\text{H}_6/\text{CHBr}_3$

Cu $K\alpha$ radiation

$\lambda = 1.54180 \text{ \AA}$

Cell parameters from 25 reflections

$\theta = 4.5\text{--}12.9^\circ$

$\mu = 1.648 \text{ mm}^{-1}$

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

Square prism

$0.45 \times 0.32 \times 0.25 \text{ mm}$

Violet

Data collection

Syntex $P2_1$ diffractometer

θ - 2θ scans

Absorption correction: none

2996 measured reflections

2640 independent reflections

1508 reflections with

$I > 2\sigma(I)$

$R_{\text{int}} = 0.079$

$\theta_{\text{max}} = 80.30^\circ$

$h = -19 \rightarrow 26$

$k = 0 \rightarrow 26$

$l = 0 \rightarrow 7$

2 standard reflections

every 100 reflections

intensity decay: 15%

Refinement

Refinement on F^2

$R[F^2 > 2\sigma(F^2)] = 0.043$

$wR(F^2) = 0.165$

$S = 0.789$

2590 reflections

158 parameters

H atoms: riding model

$w = 1/[\sigma^2(F_o^2) + (0.0802P)^2 + 2.2069P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} = 0.001$

$\Delta\rho_{\text{max}} = 0.46 \text{ e \AA}^{-3}$

$\Delta\rho_{\text{min}} = -0.25 \text{ e \AA}^{-3}$

Extinction correction:

$SHELXL93$

Extinction coefficient:

0.0013 (2)

Scattering factors from

International Tables for Crystallography (Vol. C)

O1—Cu—N1 ⁱ	90.61 (8)	C10—O3—C11	115.7 (3)
O1—Cu—N1	89.39 (8)	C4—N1—Cu	121.0 (2)
C1—O1—Cu	107.2 (2)	C8—N1—Cu	119.4 (2)

Symmetry code: (i) $-x, 1 - y, -z$.

The instability of the crystal at room temperature precluded the application of an absorption correction.

Data collection: Syntex $P2_1$ software. Cell refinement: Syntex $P2_1$ software. Data reduction: $XP21$ (Pavelčík, 1993). Program(s) used to solve structure: $SHELXS86$ (Sheldrick, 1990). Program(s) used to refine structure: $SHELXL93$ (Sheldrick, 1993). Molecular graphics: $ORTEP$ (Johnson, 1965). Software used to prepare material for publication: $SHELXL93$.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1193). Services for accessing these data are described at the back of the journal.

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Dichlorobis(quinoline-*N*)zinc(II)

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Abstract

In the structure of the title complex, $[\text{ZnCl}_2(\text{C}_9\text{H}_7\text{N})_2]$, the Zn atom is coordinated by two chloride ligands and by two N atoms from two quinoline ligands. The geometry around the Zn atom is distorted tetrahedral,

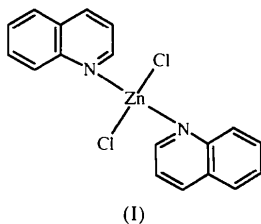
Table 1. Selected geometric parameters (Å, °)

Cu—O1	1.955 (2)	O3—C11	1.439 (4)
Cu—N1	2.025 (2)	N1—C4	1.330 (3)
O1—C1	1.268 (4)	N1—C8	1.342 (3)
O2—C1	1.227 (4)	N2—C10	1.355 (3)
O3—C10	1.337 (4)	N2—C5	1.395 (4)

with Zn—N bond lengths of 2.050 (3) and 2.074 (3) Å, Zn—Cl bond lengths of 2.241 (1) and 2.244 (1) Å, and N—Zn—N and Cl—Zn—Cl bond angles of 109.3 (1) and 115.47 (5)°, respectively.

Comment

In attempting to synthesize a heterometallic zinc(II)–lanthanide(III) complex containing acetate and quinoline ligands, we isolated the title complex, [ZnCl₂(C₉H₇N)₂], (I).



The crystal structure of (I) consists of discrete neutral [ZnCl₂(C₉H₇N)₂] molecules (Fig. 1). The Zn atom is coordinated by two chloride ligands and two N atoms from two quinoline groups in a distorted tetrahedral arrangement, with Zn—N bond lengths of 2.050 (3) and 2.074 (3) Å, Zn—Cl bond lengths of 2.241 (1) and 2.244 (1) Å, and N—Zn—N and Cl—Zn—Cl bond angles of 109.3 (1) and 115.47 (5)°, respectively. The Zn—N and Zn—Cl distances are in accord with the corresponding distances reported for dichlorobis(imidazole)zinc(II) [Zn—N 1.955 (11) and 2.020 (11) Å, and Zn—Cl 2.258 (3) and 2.239 (3) Å; Lundberg, 1966], dichlorobis(1,2-dimethylimidazole)zinc(II) [Zn—N 2.006 (3) and 2.008 (3) Å, and Zn—Cl 2.2509 (8) and 2.2468 (8) Å; Bharadwaj *et al.*, 1991], dichlorobis(purine)zinc(II) [Zn—N 2.027 (3) and 2.033 (4) Å, and Zn—Cl 2.222 (1) and 2.229 (2) Å; Laity & Taylor, 1995] and bis(antazoline)dichlorozinc(II) [Zn—N 2.039 (8) and 2.004 (9) Å, and Zn—Cl 2.243 (3) and 2.259 (3) Å; Parvez & Rusiewicz, 1995].

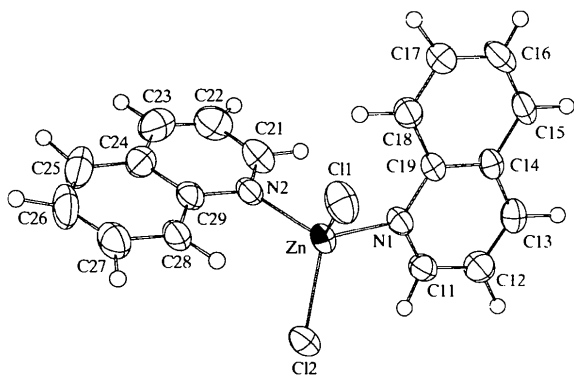


Fig. 1. A view of the molecular structure of (I) showing 50% probability displacement ellipsoids.

The molecular geometry within the quinoline ligands is unexceptional. The dihedral angle formed by the normals to the two quinoline rings is 67.06 (14)°.

Experimental

A mixture of Zn(O₂CCH₃)₂, GdCl₃·6H₂O and quinoline (molar ratio 2:1:1) in water was refluxed for 5 h. The resulting solution was allowed to stand in air and colourless crystals of the title complex were deposited after three weeks.

Crystal data

[ZnCl₂(C₉H₇N)₂]

M_r = 394.58

Triclinic

P $\bar{1}$

a = 8.7690 (1) Å

b = 9.5801 (3) Å

c = 11.0747 (3) Å

α = 80.513 (1)°

β = 72.546 (1)°

γ = 71.890 (2)°

V = 840.93 (4) Å³

Z = 2

D_x = 1.558 Mg m⁻³

D_m not measured

Mo *K*α radiation

λ = 0.71073 Å

Cell parameters from 2638 reflections

θ = 1.93–23.29°

μ = 1.777 mm⁻¹

T = 293 (2) K

Plate

0.30 × 0.10 × 0.04 mm

Colourless

Data collection

Siemens SMART CCD diffractometer

ω scans

Absorption correction: multi-scan (SADABS; Sheldrick, 1996)

T_{min} = 0.542, *T_{max}* = 0.931

3364 measured reflections

2377 independent reflections

2012 reflections with

I > 2σ(*I*)

R_{int} = 0.027

θ_{\max} = 23.29°

h = -9 → 9

k = -7 → 10

l = -12 → 11

Refinement

Refinement on *F*²

R[*F*² > 2σ(*F*²)] = 0.049

wR(*F*²) = 0.131

S = 1.020

2377 reflections

208 parameters

H atoms riding, fixed isotropic *U* = 0.05 Å²

w = 1/[σ²(*F_o*²) + (0.0913*P*)²]

where *P* = (*F_o*² + 2*F_c*²)/3

(Δ/σ)_{max} < 0.001

Δρ_{max} = 0.52 e Å⁻³

Δρ_{min} = -1.02 e Å⁻³

(1.02 Å from Zn)

Extinction correction: none

Scattering factors from

International Tables for Crystallography (Vol. C)

Table 1. Selected geometric parameters (Å, °)

Zn—Cl1	2.244 (1)	Zn—N1	2.074 (3)
Zn—Cl2	2.241 (1)	Zn—N2	2.050 (3)
Cl1—Zn—Cl2	115.47 (5)	Cl2—Zn—N1	106.3 (1)
Cl1—Zn—N1	108.7 (1)	Cl2—Zn—N2	108.3 (1)
Cl1—Zn—N2	108.6 (1)	N1—Zn—N2	109.3 (1)

Data collection: SMART (Siemens, 1995). Cell refinement: SMART and SAINT (Siemens, 1995). Data reduction: SAINT (Siemens, 1995). Program(s) used to solve structure: SHELXS86 (Sheldrick, 1990). Program(s) used to refine structure: SHELXL93 (Sheldrick, 1993). Molecular graphics: SHELXTL/PC (Sheldrick, 1994). Software used to prepare material for publication: SHELXTL/PC.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1252). Services for accessing these data are described at the back of the journal.

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Trichlorooxo(triphenylphosphine)(triphenylphosphine oxide)rhenium(V)

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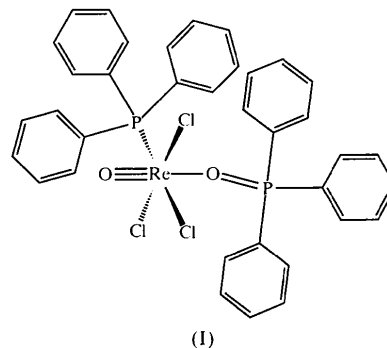
Abstract

The title complex, $[\text{ReCl}_3\text{O}(\text{C}_{18}\text{H}_{15}\text{OP})(\text{C}_{18}\text{H}_{15}\text{P})]$, is produced in a reaction between $[\text{Re}(\text{O})\text{Cl}_3(\text{PPh}_3)_2]$ and ethyl 2-hydroxymethyl sulfoxide. The structure is compared to that of $[\text{Re}(\text{O})\text{Cl}_3(\text{PPhEt}_2)(\text{OPPhEt}_2)]$. The Re—Cl distances are shorter [2.361 (2)–2.384 (2) Å] and the Re—P distance is longer [2.506 (2) Å] in the title complex.

Comment

A variety of interesting and synthetically useful O-atom transfer reactions from sulfoxide substrates catalyzed by the precursor complex $[\text{Re}(\text{O})\text{Cl}_3(\text{PPh}_3)_2]$ have recently been reported (Bryan *et al.*, 1987; Arterburn &

Perry, 1996; Arterburn & Nelson, 1996; Arterburn *et al.*, 1997). This compound was known to react with dimethyl sulfoxide in the presence of hydrochloric acid to form the mixed dimethylsulfide–triphenylphosphine oxide complex $[\text{Re}(\text{O})\text{Cl}_3(\text{SMe}_2)(\text{OPPh}_3)]$. However, no intermediate complexes from the catalytic reactions in organic solvents have yet been structurally identified. The precursor complex was found to react with one equivalent of ethyl 2-hydroxyethyl sulfoxide, $\text{CH}_3\text{CH}_2\text{S}(\text{O})\text{CH}_2\text{CH}_2\text{OH}$, at ambient temperature to give a purple solution. The title complex, (I), and amorphous purple solids were obtained following precipitation with diethyl ether. These results are consistent with catalytic pathways involving coordinated $\text{Re}^{\text{V}}\text{—O}$ complexes as intermediates.



A distorted octahedral coordination geometry is observed around Re (Fig. 1). The major distortion is an increase in the O1—Re—Cl angles, which is commonly observed in octahedral complexes containing a

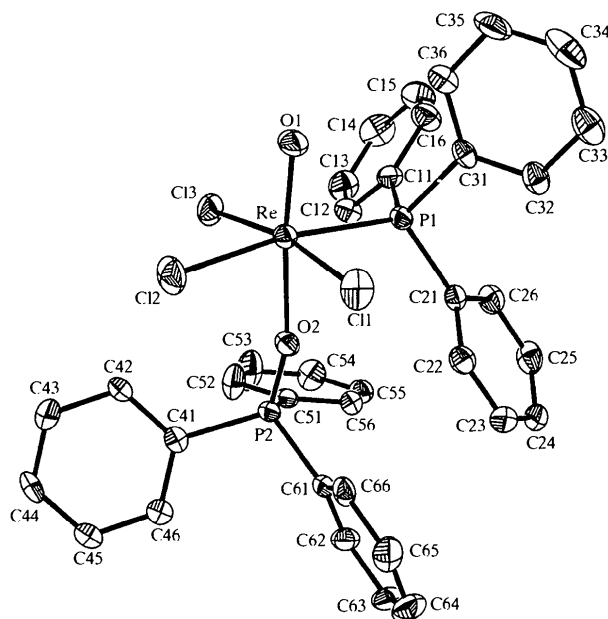


Fig. 1. The molecular structure of (I) showing 50% probability displacement ellipsoids. H atoms have been omitted for clarity.