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Di- μ -chloro-bis[(η^5 -cyclopentadienyl)-nitrosylmolybdenum]

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Abstract

The centrosymmetric dimeric molecule of the title compound, [MoCl(NO)(C₅H₅)₂]₂, contains two Mo(Cp)NO units, where Cp is the cyclopentadienyl ring, with two bridging Cl atoms giving a central four-membered Mo₂Cl₂ ring. The mean Mo—Cl distance is 2.407 (1) Å, with the Mo···Mo' distance [2.9098 (3) Å] in the range for a single metal–metal bond.

Comment

The dimer (I) was obtained as a by-product in the synthesis of diene complexes from the reaction of CpMo(NO)I₂ with dienes and sodium amalgam (Christensen, 1989; Christensen, Legzdins, Trotter & Yee, 1991). Similar dimers have been described previously, e.g. Stevenson & Dahl (1967).

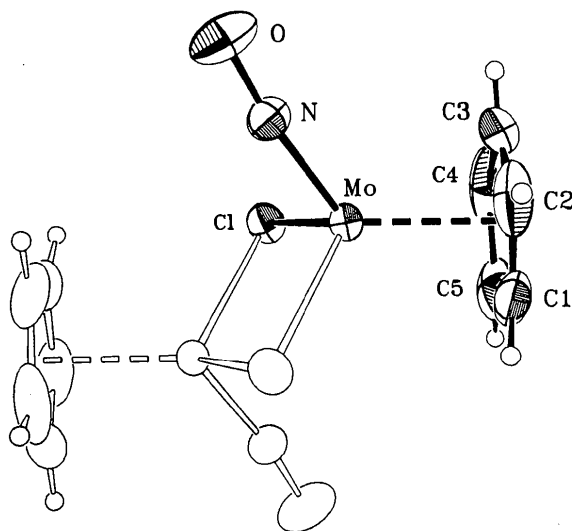
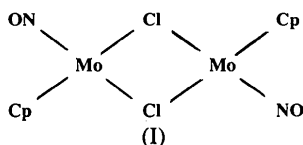


Fig. 1. View of the molecule with 50% probability ellipsoids.

Experimental

Crystal data

[Mo₂Cl₂(NO)₂(C₅H₅)₂]

M_r = 452.98

Monoclinic

*P*2₁/*n*

a = 8.945 (1) Å

b = 11.468 (1) Å

c = 6.628 (1) Å

β = 102.54 (1)°

V = 663.7 (1) Å³

Z = 2

D_x = 2.27 Mg m⁻³

Mo *K*α radiation

λ = 0.7107 Å

Cell parameters from 25 reflections

θ = 28–30°

μ = 2.25 mm⁻¹

T = 294 K

Prism

0.33 × 0.14 × 0.10 mm

Orange

Data collection

Enraf–Nonius CAD-4F

diffractometer

Absorption correction:

analytical

T_{min} = 0.70, *T_{max}* = 0.82

1924 measured reflections

1924 independent reflections

1678 observed reflections

[*I* > 3σ(*I*)]

θ_{\max} = 30°

h = -12 → 12

k = 0 → 16

l = 0 → 9

3 standard reflections

monitored every 150

reflections

intensity variation:

negligible

Refinement

Refinement on *F*

R = 0.020

wR = 0.031

S = 1.33

1678 reflections

103 parameters

All H-atom parameters

refined

w = 1/σ²(*F*)

(Δ/σ)_{max} = 0.06

Δρ_{max} = 0.5 e Å⁻³

Δρ_{min} = -1.1 e Å⁻³

Extinction correction:

Becker & Coppens (1975)

Extinction coefficient:

7.3 (9) × 10⁴

Atomic scattering factors

from *International Tables*

for *X-ray Crystallography*

(1974, Vol. IV)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)

$$U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*a_i \cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U_{eq}</i>
Mo	0.39528 (2)	0.54477 (1)	0.31715 (3)	0.024
Cl	0.43520 (6)	0.61996 (5)	0.66370 (9)	0.032
O	0.5779 (3)	0.7218 (2)	0.1505 (4)	0.056
N	0.5130 (2)	0.6479 (2)	0.2259 (3)	0.032
C1	0.2008 (3)	0.4146 (3)	0.1518 (7)	0.055
C2	0.2119 (4)	0.5040 (5)	0.0199 (5)	0.057
C3	0.1690 (4)	0.6057 (3)	0.1030 (7)	0.060
C4	0.1356 (3)	0.5810 (4)	0.2897 (8)	0.068
C5	0.1546 (4)	0.4573 (4)	0.3223 (7)	0.061
Cp†	0.1744	0.5125	0.1773	0.06

† Cp is the centroid of the cyclopentadienyl ring.

Table 2. Selected geometric parameters (Å, °)

Mo—Mo ⁱ	2.9098 (3)	Mo—C4	2.328 (3)
Mo—Cl	2.406 (1)	Mo—C5	2.382 (4)
Mo—Cl ⁱ	2.408 (1)	N—O	1.196 (3)
Mo—N	1.775 (2)	C1—C2	1.365 (6)
Mo—Cp	2.026 (2)	C2—C3	1.379 (6)
Mo—C1	2.372 (4)	C3—C4	1.364 (7)
Mo—C2	2.321 (4)	C4—C5	1.439 (6)
Mo—C3	2.313 (4)	C5—C1	1.375 (6)

Cl—Mo—Cl ⁱ	105.64 (2)	Mo—N—O	172.8 (2)
Cl—Mo—N	96.3 (1)	C2—C1—C5	109.6 (3)
Cl ⁱ —Mo—N	97.3 (1)	C1—C2—C3	108.3 (3)
Cl—Mo—C _p	115.5 (1)	C2—C3—C4	108.7 (4)
Cl ⁱ —Mo—C _p	114.9 (1)	C3—C4—C5	107.4 (3)
N—Mo—C _p	123.9 (1)	C4—C5—C1	105.9 (3)
Mo—Cl—Mo ⁱ	74.36 (2)		

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

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: local program. Structure solution: Patterson methods (*FORDAP*; Zalkin, UCLA, Los Angeles, USA, unpublished). Program(s) used to refine structure: *ORFLS* (Busing, Martin & Levy, 1962). Molecular graphics: *ORTEPII* (Johnson, 1976).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry, including bond distances and angles involving H atoms, and torsion angles, as well as a packing diagram, have been deposited with the IUCr (Reference: CR1145). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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$[(\eta^6\text{-C}_6\text{H}_3\text{Me}_3)\text{Ru}(\text{L-ala})\text{Cl}]$

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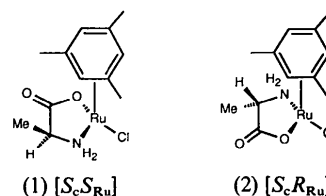
Abstract

The structure of (L-alaninato-*N,O*)chloro(η^6 -1,3,5-trimethylbenzene)ruthenium(II), $[\text{RuCl}(\text{C}_3\text{H}_6\text{NO}_2)(\text{C}_9\text{H}_{12})]$, has been determined. The crystal contains

two diastereoisomers having opposite chiralities at the metal centre. There is evidence of intermolecular hydrogen bonding between the amine protons of one diastereomer and the chloride of the other.

Comment

We have recently embarked on a study of metal complexes of biologically relevant ligands (Carter, Davies, Fawcett & Russell, 1993). As part of this programme we have synthesized some arene-ruthenium-amino acid complexes $[(\text{arene})\text{Ru}(\text{aa})\text{Cl}]$ (aa is an aminoacidate ligand). These half-sandwich complexes are interesting in that they contain a chiral metal centre so that if an L-amino acid ligand is used two diastereoisomers may be formed. The ratio of diastereoisomers varies depending on the amino acid used (Kramer, Polborn, Wanjek, Zahn & Beck, 1990). It has been suggested that intramolecular hydrogen bonding may influence the diastereomer ratio (Sheldrick & Heeb, 1990). In order to investigate further the role of intramolecular hydrogen bonding in such complexes we have determined the structure of $[(\eta^6\text{-C}_6\text{H}_3\text{Me}_3)\text{Ru}(\text{L-ala})\text{Cl}]$. The complex was prepared by refluxing a mixture of L-alanine (L-alaH), sodium methoxide and $[(\eta^6\text{-C}_6\text{H}_3\text{Me}_3)\text{RuCl}_2]_2$ in a 50:50 methanol–water solution for 3 h. Evaporation of the solvent followed by extraction with dichloromethane, filtration to remove NaCl and rotary evaporation of the solvent gave $[(\eta^6\text{-C}_6\text{H}_3\text{Me}_3)\text{Ru}(\text{L-ala})\text{Cl}]$ as a yellow powder. X-ray quality crystals were grown by dissolving the solid in the minimum quantity of methanol and adding mesitylene.



The complex crystallized as a 50:50 mixture of two diastereomers having opposite chiralities at the metal centre (Fig. 1). The absolute configurations of the diastereomers were assigned from the known configuration of the amino acid. It is noteworthy that in D_2O solution the ratio of diastereomers is 60:40 rather than the 50:50 observed in the solid state. Molecule (1) displays the $S_C S_{\text{Ru}}$ configuration while molecule (2) is $S_C R_{\text{Ru}}$. The configurations at the Ru atom were assigned on the basis proposed by Stanley & Baird (1975) and Sloan (1981), and are opposite to those assigned by Sheldrick & Heeb (1990) for the diastereomers of $[(\eta^6\text{-C}_6\text{H}_6)\text{Ru}(\text{L-ala})\text{Cl}]$. The Ru—N, Ru—Cl and Ru—O distances are similar in