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Key indicators

Single-crystal X-ray study
T = 160 K
Mean $\sigma(\text{C}-\text{C}) = 0.011 \text{ \AA}$
R factor = 0.035
wR factor = 0.079
Data-to-parameter ratio = 6.8

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

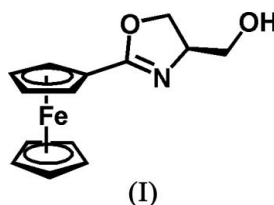
(R)-2-Ferrocenyl-4-hydroxymethyl-4,5-dihydro-1,3-oxazole

The title compound, $[\text{Fe}(\text{C}_5\text{H}_5)(\text{C}_9\text{H}_{10}\text{NO}_2)]$, was prepared from ferrocenecarboxylic acid and serine. In the crystal structure, molecules are arranged in chains with an intermolecular hydrogen bond between hydroxy groups and N atoms.

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Comment

A series of serine-derived oxazoles has been shown to be effective in asymmetric alkylation reactions, and hence their crystal structures are of interest (Jones & Richards, 2004). As representatives of the general amino alcohol class of ligands for these reactions, we have an interest in understanding their non-linear catalytic characteristics. A single-crystal X-ray structure has been reported for a related compound, *viz.* (4*S*)-4-(1-hydroxy-1-methylethyl)-2-ferrocenyl-4,5-dihydro-1,3-oxazole monohydrate (Chesney *et al.*, 1998).



The structure of the title compound, (I) (Fig. 1), reveals that the two cyclopentadienyl rings of ferrocene deviate by only four degrees from a fully eclipsed conformation. The torsion angle $\text{C1}-\text{C1c}-\text{C2c}-\text{C11}$ (where C1c and C2c are the cyclopentadiene ring centroids) is $-3.5 (6)^\circ$. The oxazoline ring is almost coplanar with the cyclopentadienyl ring to which it is attached [interplanar angle = $9.2 (5)^\circ$ and $\text{C2}-\text{C1}-\text{C6}-\text{O1} = -6.3 (10)^\circ$], the O rather than the N atom being slightly closer to iron. The oxazoline hydroxymethyl substituent is oriented away from the iron-cyclopentadienyl group of ferrocene. Significantly, the opposite rotamer, with respect to rotation about the ferrocene-oxazoline $\text{C}-\text{C}$ σ -bond, was observed in the structure reported by Chesney *et al.* (1998). This contains a larger 1-hydroxy-1-methylethoxyoxazoline substituent, and in both cases the hydroxy group is oriented over the oxazoline ring; in the present structure, $\text{N1}-\text{C8}-\text{C9}-\text{O2} = -73.7 (7)^\circ$.

Experimental

LiAlH_4 (68 mg, 1.797 mmol, 1.1 equivalent) was added to a solution of (*S*)-4-carbomethoxy-2-ferrocenyl-1,3-oxazoline (513 mg, 1.634 mmol) in diethyl ether (15 ml) cooled to 273 K. After stirring for 15 min, ethyl acetate (25 ml) was added followed by water (38 ml), and the organic layer was then separated, dried (MgSO_4),

filtered and concentrated *in vacuo* to give the crude alcohol as an orange solid. The crude solid was recrystallized from dichloromethane and hexane (*ca* 1:1) to give the pure oxazoline [434 mg, 93%; m.p. 390–392 K (literature 390–392 K)]. ¹H NMR (CDCl₃): δ 3.59 (1H, *dd*, *J* = 11.4, 3.7 Hz, OCHH) and 3.86 (1H, *dd*, *J* = 11.4, 3.2 Hz OCHH), 4.16 (5H, *s*, Cp), 4.18–4.40 (5H, *m*, OCH₂ + CHN + Cp × 2), 4.72 (2H, *m*, Cp).

Crystal data

[Fe(C₅H₅)(C₉H₁₀NO₂)]
M_r = 285.12
 Monoclinic, *P*₂₁
a = 5.808 (4) Å
b = 7.557 (3) Å
c = 13.716 (8) Å
 β = 92.39 (6)°
V = 601.5 (6) Å³
Z = 2
D_x = 1.574 Mg m⁻³
 Mo *K*α radiation
 Cell parameters from 25 reflections
 θ = 9.9–13.2°
 μ = 1.25 mm⁻¹
T = 160 (2) K
 Prism, orange
 0.30 × 0.13 × 0.08 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
 ω/2θ scans
 Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.706, *T_{max}* = 0.912
 1305 measured reflections
 1137 independent reflections
 877 reflections with *I* > 2σ(*I*)
R_{int} = 0.033
 θ_{max} = 25.0°
h = -1 → 6
k = 0 → 8
l = -16 → 16
 2 standard reflections every 100 reflections
 intensity decay: 5%

Refinement

Refinement on *F*²
R[*F*² > 2σ(*F*²)] = 0.035
wR(*F*²) = 0.079
S = 1.03
 1137 reflections
 168 parameters
 H atoms treated by a mixture of independent and constrained refinement
w = 1/[σ²(*F_o*²) + (0.0336*P*)²]
 where *P* = (*F_o*² + 2*F_c*²)/3
 (Δ/σ)_{max} < 0.001
 Δρ_{max} = 0.54 e Å⁻³
 Δρ_{min} = -0.37 e Å⁻³
 Absolute structure: Flack (1983), 171 Friedel Pairs
 Flack parameter: 0.01 (4)

Table 1

Selected torsion angles (°).

C2–C1–C6–O1	-6.3 (10)	N1–C8–C9–O2	-73.7 (7)
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Table 2

Hydrogen-bond geometry (Å, °).

<i>D</i> –H... <i>A</i>	<i>D</i> –H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> –H... <i>A</i>
O2–H20...N1 ⁱ	0.87 (7)	2.02 (8)	2.877 (7)	165 (7)

Symmetry code: (i) -x + 1, y - 1/2, -z + 1.

H atoms were treated as riding atoms [C–H = 0.95 and 0.99 Å; *U*_{iso}(H) = 1.2*U*_{eq}(C)], except for H20, which was refined freely with an isotropic displacement parameter.

Data collection: *CAD-4/PC* (Enraf–Nonius, 1994); cell refinement: *CAD-4/PC*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *DIRDIF99* (Beurskens *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

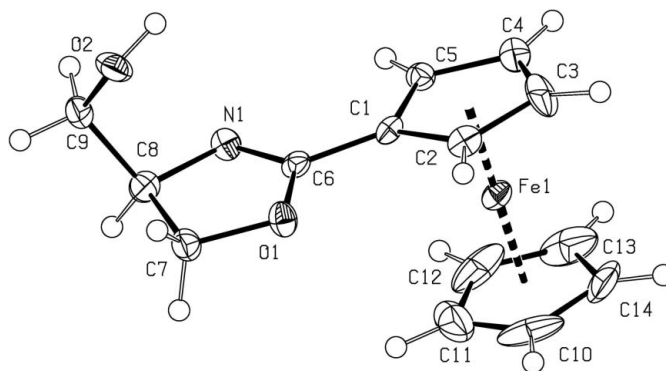


Figure 1

The molecular structure of (I). Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

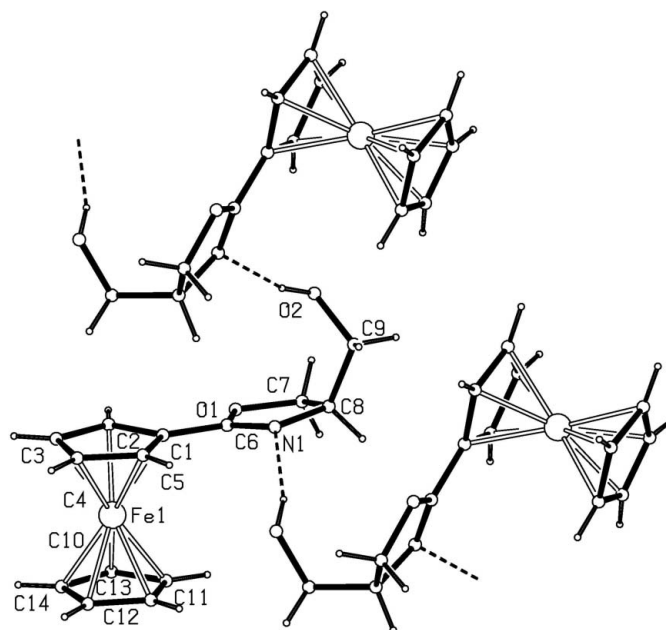


Figure 2

The polymeric assembly of (I), involving hydrogen-bonded (dashed lines) oxazole groups.

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