

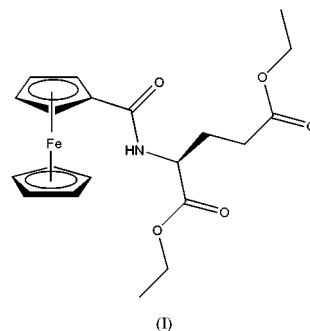
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Key indicators

Single-crystal X-ray study
 $T = 193$ K
Mean $\sigma(\text{C}-\text{C}) = 0.005$ Å
 R factor = 0.038
 wR factor = 0.095
Data-to-parameter ratio = 16.6For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.***N*-Ferrocenoyl-labelled glutamate diethyl ester**

Diethyl *N*-(ferrocenylcarbonyl)glutamate or *N*-ferrocenoyl-glutamate-(OEt)₂, [Fe(C₅H₅)(C₁₅H₂₀O₅N)], (I), is a derivative of *N*-ferrocenoylglutamate-(OBz)₂, the corresponding benzyl ester derivative published previously [Kraatz, Luszyk & Enright (1997). *Inorg. Chem.* **36**, 2400–2405]. These redox-labeled amino acid structures exhibit intermolecular hydrogen bonding that leads to extended networks in the solid state and exhibit typical ferrocene one-electron reversible electrochemical responses. The asymmetric unit of the structure contains two independent molecules, in which the most notable difference is in the conformations of the diethyl ester groups.

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Experimental

The title compound was prepared by the addition of solid EDC [1-ethyl-3-(3-dimethylaminopropyl)carbodiimide hydrochloride] to a stirred mixture of FeCOOH and HOBt (1-hydroxybenzotriazole) in CH₂Cl₂ at room temperature, which caused the orange slurry to change slowly into a clear solution. In a separate flask, H-Glu-OEt-HCl was dissolved in CH₂Cl₂ and Et₃N and then added to the stirred reaction mixture. After stirring overnight, the reaction solution was washed consecutively with aqueous solutions of saturated NaHCO₃, 10% citric acid, saturated NaHCO₃ and finally distilled water. The organic phase was dried by anhydrous Na₂SO₄, filtered, and the solvent removed under reduced pressure, giving the crude orange product. The product was purified by column chromatography ($R_f = 0.5$; hexane/EtOAc/CHCl₃ 1:1:2), giving an orange solid. Suitable crystals of the title compound were obtained by a slow diffusion of layered hexane into a solution of the compound in CH₂Cl₂. Yellow crystals suitable for X-ray crystallography were deposited after a few days.

Crystal data

[Fe(C₅H₅)(C₁₅H₂₀NO₅)]
 $M_r = 415.26$
Trigonal, $P3_1$
 $a = 11.4140$ (3) Å
 $c = 26.7688$ (17) Å
 $V = 3020.2$ (2) Å³
 $Z = 6$
 $D_x = 1.370$ Mg m⁻³Mo $K\alpha$ radiation
Cell parameters from 4477
reflections
 $\theta = 2.2$ – 25.7°
 $\mu = 0.78$ mm⁻¹
 $T = 193$ (2) K
Prism, orange
 $1.00 \times 0.23 \times 0.22$ mm

Data collection

Bruker PLATFORM/SMART 1000
 CCD area-detector
 diffractometer
 ω scans
 Absorption correction: multi-scan
 (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.510$, $T_{\max} = 0.847$
 16 410 measured reflections

8093 independent reflections
 7430 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.025$
 $\theta_{\text{max}} = 26.4^\circ$
 $h = -14 \rightarrow 14$
 $k = -12 \rightarrow 14$
 $l = -33 \rightarrow 32$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.038$
 $wR(F^2) = 0.095$
 $S = 1.04$
 8093 reflections
 487 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0498P)^2 + 0.5792P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.002$
 $\Delta\rho_{\text{max}} = 0.46 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.37 \text{ e } \text{\AA}^{-3}$
 Absolute structure: Flack (1983),
 4131 Friedel pairs
 Flack parameter = 0.010 (12)

Table 1

Hydrogen-bonding geometry (\AA , $^\circ$).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$N1-H1N\cdots O6^i$	0.88	1.97	2.822 (3)	163
$N2-H2N\cdots O1$	0.88	2.01	2.861 (3)	162

Symmetry code: (i) $-x + y, 1 - x, z - \frac{1}{3}$.

All H atoms were placed in calculated positions, with C—H distances ranging from 0.98 to 1.00 \AA , and N—H distances of 0.88 \AA . They were included in the refinement in a riding-model approximation, with $U_{\text{iso}} = 1.2U_{\text{eq}}$ of the carrier atom.

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

References

Bruker (1997). *SMART*, *SAINT* and *SHELXTL*, Bruker AXS Inc., Madison, Wisconsin, USA.
 Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.

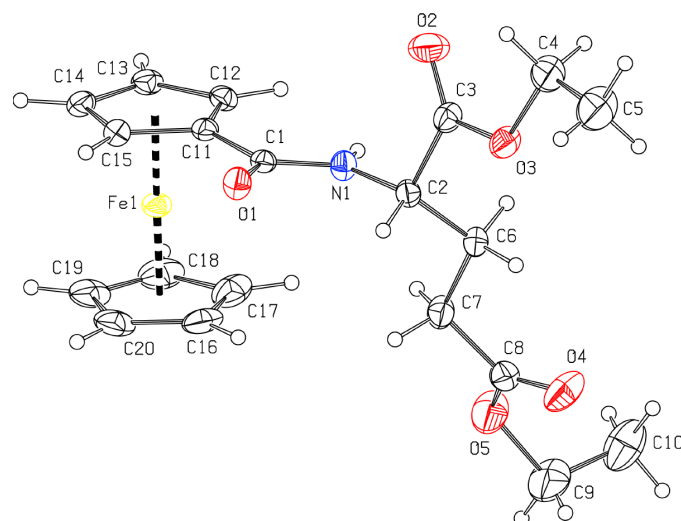


Figure 1

View of molecule 1 of (I), with the crystallographic labeling scheme. Displacement ellipsoids are drawn at the 30% probability level.

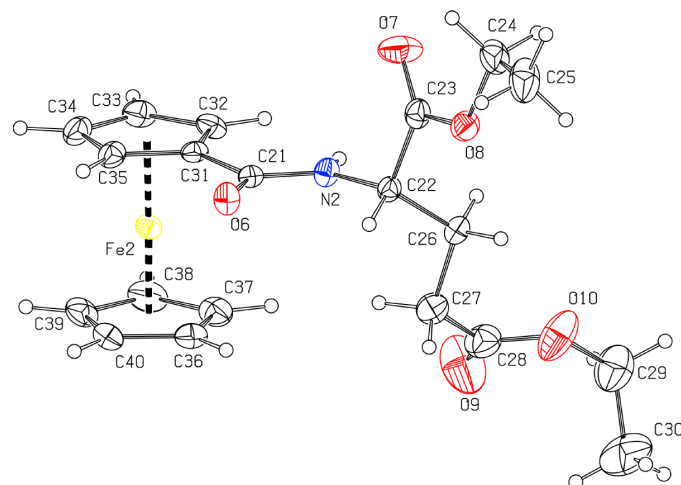


Figure 2

View of molecule 2 of (I), with the crystallographic labeling scheme. Displacement ellipsoids are drawn at the 30% probability level.

Kraatz, H.-B., Luszytyk, J. & Enright, G. D. (1997). *Inorg. Chem.* **36**, 2400–2405.
 Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
 Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.