Table 2. *Selected bond lengths* (Å) *and angles* (°)

Mo(1)—P(11)	2.503 (3)	Mo(1)—P(12)	2.490 (3)
Mo(1)—P(13)	2.506 (4)	Mo(1)—C(11)	1.930 (12)
Mo(1)—C(12)	1.940 (13)	Mo(1)—C(13)	1.935 (13)
P(11)—C(101)	1.796 (12)	P(11)—C(107)	1.822 (12)
P(12)—C(113)	1.820 (12)	P(12)—C(119)	1.824 (11)
P(13)—C(125)	1.815 (15)	P(13)—C(131)	1.820 (14)
O(11)—C(11)	1.176 (15)	O(12)—C(12)	1.152 (16)
O(13)—C(13)	1.176 (17)	Mo(2)—P(21)	2.502 (3)
Mo(2)—P(22)	2.494 (3)	Mo(2)—P(23)	2.516 (4)
Mo(2)—C(21)	1.988 (14)	Mo(2)—C(22)	2.009 (12)
Mo(2)—C(23)	1.982 (12)	P(21)—C(201)	1.829 (12)
P(21)—C(207)	1.844 (11)	P(22)—C(213)	1.829 (11)
P(22)—C(219)	1.843 (11)	P(23)—C(225)	1.843 (12)
P(23)—C(231)	1.833 (11)	O(21)—C(21)	1.112 (17)
O(22)—C(22)	1.140 (14)	O(23)—C(23)	1.135 (15)
P(11)—Mo(1)—P(12)	87.9 (1)	P(11)—Mo(1)—P(13)	85.2 (1)
P(12)—Mo(1)—P(13)	87.1 (1)	P(11)—Mo(1)—C(11)	93.8 (4)
P(12)—Mo(1)—C(11)	178.3 (4)	P(13)—Mo(1)—C(11)	93.1 (4)
P(11)—Mo(1)—C(12)	179.7 (4)	P(12)—Mo(1)—C(12)	92.2 (4)
P(13)—Mo(1)—C(12)	95.2 (4)	C(11)—Mo(1)—C(12)	86.2 (5)
P(11)—Mo(1)—C(13)	92.1 (4)	P(12)—Mo(1)—C(13)	93.0 (4)
P(13)—Mo(1)—C(13)	177.3 (4)	C(11)—Mo(1)—C(13)	86.8 (5)
C(12)—Mo(1)—C(13)	87.6 (6)	Mo(1)—P(11)—C(101)	119.0 (4)
Mo(1)—P(11)—C(107)	118.6 (4)	C(101)—P(11)—C(107)	103.5 (5)
Mo(1)—P(12)—C(113)	119.6 (4)	Mo(1)—P(12)—C(119)	118.1 (4)
C(113)—P(12)—C(119)	102.7 (5)	Mo(1)—P(13)—C(125)	120.4 (5)
Mo(1)—P(13)—C(131)	119.1 (5)	C(125)—P(13)—C(131)	104.2 (6)
Mo(1)—C(11)—O(11)	177.0 (11)	Mo(1)—C(12)—O(12)	173.8 (12)
Mo(1)—C(13)—O(13)	175.8 (10)	P(21)—Mo(2)—P(22)	88.1 (1)
P(21)—Mo(2)—P(23)	88.3 (1)	P(22)—Mo(2)—P(23)	90.2 (1)
P(21)—Mo(2)—C(21)	91.8 (3)	P(22)—Mo(2)—C(21)	88.7 (3)
P(23)—Mo(2)—C(21)	178.9 (3)	P(21)—Mo(2)—C(22)	92.1 (3)
P(22)—Mo(2)—C(22)	175.7 (3)	P(23)—Mo(2)—C(22)	94.1 (3)
C(21)—Mo(2)—C(22)	87.0 (5)	P(21)—Mo(2)—C(23)	176.7 (4)
P(22)—Mo(2)—C(23)	91.7 (3)	P(23)—Mo(2)—C(23)	94.9 (4)
C(21)—Mo(2)—C(23)	84.9 (5)	C(22)—Mo(2)—C(23)	87.8 (5)
Mo(2)—P(21)—C(201)	117.1 (4)	Mo(2)—P(21)—C(207)	121.9 (4)
C(201)—P(21)—C(207)	99.5 (5)	Mo(2)—P(22)—C(213)	118.2 (4)
Mo(2)—P(22)—C(219)	119.1 (4)	C(213)—P(22)—C(219)	101.2 (5)
Mo(2)—P(23)—C(225)	122.0 (4)	Mo(2)—P(23)—C(231)	116.9 (4)
C(225)—P(23)—C(231)	102.4 (5)	Mo(2)—C(21)—O(21)	178.0 (10)
Mo(2)—C(22)—O(22)	174.5 (10)	Mo(2)—C(23)—O(23)	175.9 (10)

The title compound was prepared by direct treatment of $C_7H_8Mo(CO)_3$ (C_7H_8 = cycloheptatriene) (Cotton, McCleverty & White, 1967) with three equivalents of PPh_2H in boiling toluene for 12 h, followed by recrystallization from toluene/hexane. A crystal was mounted in a Lindemann capillary. Data were collected with a scan range of $\pm 0.7°$ (ω) around the $K\alpha_1 - K\alpha_2$ angles, scan speed 2.5–15° (ω) min^{-1}, depending on the intensity of a 2 s pre-scan; backgrounds were measured at each end of the scan for 0.25 of the scan time. The data were processed using profile analysis and then corrected for Lorentz, polarization and absorption effects; crystal bounding faces $\pm(001)$ $d = 0.06$; $\pm(110)$ $d = 0.19$; $\pm(1\bar{1}0)$ $d = 0.17$. Systematic reflection conditions $h0l$, $h + 1 = 2n$, and $0k0$, $k = 2n$, indicate space group $P2_1/n$. The structure was solved by direct methods using *SHELXTL-Plus* (*TREF*) (Sheldrick, 1986) to locate two independent Mo atoms, each with three P atoms. The remaining atoms were then found by *E*-map expansion and successive Fourier syntheses. Anisotropic thermal parameters were used for all non-H atoms. H atoms were given fixed isotropic thermal parameters, $U = 0.08$ Å^2. Those defined by the molecular geometry were inserted at calculated positions and not refined. Fourier syntheses near the end of the refinement showed peaks in appropriate positions for H atoms attached to P atoms. Their coordinates were refined with the bond lengths weakly constrained to 1.3 (1) Å. An area of electron density was attributed to a partly occupied hexane molecule disordered across a centre of inversion. This was modelled as three atoms each with 0.5 occupancy. Final refinement was by least-squares methods. The largest peaks in the final difference Fourier synthesis were in the region of the disordered solvent. All calculations were performed using *SHELXTL-Plus* (Sheldrick, 1986) on a DEC MicroVAX II.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71000 (47 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: LI1041]

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cis-1-Ferrocenyl-2-(2-nitrophenyl)ethylene

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Abstract

The *cis*-ethylene bond environment in (η^5-cyclopentadienyl){1-[2-(2-nitrophenyl)vinyl]-η^5-cyclopentadienyl}-iron is not exactly planar; the corresponding torsion angle around the C=C bond is 8.7 (7)°. The mean planes of the aromatic ring and the ethylene bond form a dihedral angle of 58.9 (1)°; the NO_2-group plane is rotated by 32.8 (2)° out of the benzene ring. A rather short contact between the olefin CH group and an NO_2 oxygen atom [H···O 2.72 (4) Å] may determine the differences in the reactivity of *cis* and *trans* isomers.

Comment

The *cis*/*trans* isomers of 1-ferrocenyl-2-(2-nitrophenyl)-ethylene are rather stable and do not interconvert spon-

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taneously. They have quite different reactivity in the reaction of protophilic isotope hydrogen exchange: the *cis* isomer reacts readily at the expense of the olefin CH group neighbouring the 2-nitrophenyl group, whereas the *trans* isomer is inert under the same conditions (Todres & Ermekov, 1993). In the hope of finding the structural basis for the abovementioned difference in reactivity of the isomers, we have undertaken their structural determination. In the present communication, an X-ray study of the *cis* isomer (I) is reported and its geometrical parameters are compared to those of the related molecules *cis*-2-nitrostilbene (II) (Todres, Gridunova, Dyusengaliev & Struchkov, 1987) and *cis*-1-ferrocenyl-2-(4-nitrophenyl)ethylene (III) (Green, Marder, Thompson, Bandy, Bloor, Kolinski & Jones, 1987).

The geometry of the *ortho*-nitrophenylethylene moiety of molecule (I) is very similar to that observed in (II). The bond lengths C1—C11 1.455 (5), C11=C12 1.334 (5), C12—C13 1.475 (5) Å, bond angles C1—C11—C12 129.3 (3) and C11—C12—C13 125.7 (4)°, as well as the torsion angle C1—C11—C12—C13 8.7 (7)° and dihedral angle C1—C11—C12—C13/C13—C14—C15—C16—C17—C18 58.9 (1)° almost exactly coincide with the corresponding values reported for (II) (1.467, 1.321, 1.483 Å, 130.9, 127.8, 8.5, 58.9°, respectively). The most significant differences involve the dihedral angles C1—C2—C3—C4—C5/C1—C11—C12—C13 [19.0 (3)° in (I) as compared to 27.9° in (II)] and C13—C14—C15—C16—C17—C18/C14—N—O1—O2 [32.8 (2)° in (I) against 22.4° in (II)].

The H12···O1 distance [2.72 (4) Å] seems to be rather long for C12—H12···O1 hydrogen bonding [C12···O1 2.753 (5) Å]. However, the persistence of this contact in molecules of 2-nitrophenylethyleneferrocene and 2-nitrophenylstilbene, both of which show significant *cis*/*trans* isomer reactivity differences, forces one to think of the possible role of this contact in the explanation of chemical behaviour. One should also take into account that the 4-nitrophenylethylene derivative (III), which, naturally, has no short C—H···O distances, does not exhibit any reactivity peculiarities.

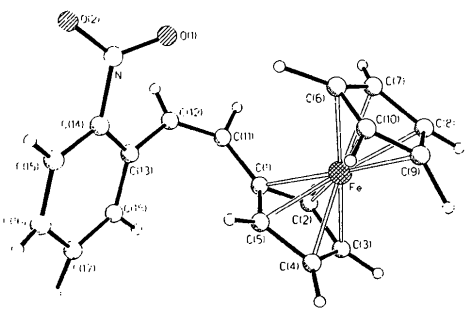

Fig. 1. View of the title compound showing the labelling of non-H atoms.

The X-ray structure of *trans*-1-ferrocenyl-2-(2-nitrophenyl)ethylene and/or force-field calculations, which we plan to carry out in the future, may help to elucidate the abovementioned reactivity issues.

Experimental

Crystal data

$[Fe(C_{13}H_{10}NO_2)(C_5H_5)]$
M_r = 333.16
Monoclinic
$P2_1/c$
a = 18.731 (4) Å
b = 7.789 (2) Å
c = 10.371 (2) Å
β = 96.32 (2)°
V = 1503.9 (6) Å^3
Z = 4
D_x = 1.471 Mg m^{-3}
Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 24 reflections
θ = 13-14°
μ = 1.008 mm^{-1}
T = 293.0 (10) K
Prism
0.3 × 0.2 × 0.4 mm
Dark red

Data collection

Siemens *P3/PC* diffractometer
$\theta/2\theta$ scans
Absorption correction: none
4159 measured reflections
4042 independent reflections
3389 observed reflections [$I > 2\sigma(I)$]
R_{int} = 0.0230
θ_{max} = 30.07°
$h = 0 \rightarrow 25$
$k = 0 \rightarrow 10$
$l = -14 \rightarrow 14$
2 standard reflections monitored every 98 reflections
intensity variation: 2%

Refinement

Refinement on F^2
Final R = 0.0657 for $F > 4\sigma(F)$ data
$wR2$ = 0.1399 for 'observed' F^2 data
S = 1.233
4016 reflections
244 parameters
All H-atom parameters refined
Calculated weights $w = 1/[\sigma^2(F_o^2) + (0.0454P)^2 + 2.0340P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.156
$\Delta\rho_{max}$ = 0.735 e Å^{-3}
$\Delta\rho_{min}$ = −0.521 e Å^{-3}
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

The final refinement was performed on F^2 for all reflections, including those generally believed to be 'unobserved' [$F^2 < 2\sigma(F^2)$] and even those having negative F^2 but larger than $-3\sigma(F^2)$ (no reflections were, in fact, rejected according to the last criterium); 26 reflections obscured by the primary beamstop or affected by shutter failure were omitted. The conventional R factor for the finally refined structure was calculated to be 0.0860 for all reflections and 0.0657 for 3389 'observed' reflections with $F > 4\sigma(F)$. The weighted $wR2$ factor (calculated on F^2) was equal to 0.1597 for all and 0.1399 for 'observed' data. The atoms of the unsubstituted Cp ring exhibited high thermal motion and their anisotropic temperature factors in the final cycles were refined with 'rigid-bond' restraints (Hirshfeld, 1976). Its H atoms were placed in calculated positions and refined with a 'riding-model' approximation with fixed C—H vector directions and variable C—H distances. All other H

atoms in the structure were located in the difference Fourier synthesis and refined isotropically. Program(s) used to solve structure: *SHELXTL-Plus* (Sheldrick, 1989). Program(s) used to refine structure: *SHELXL92* (Sheldrick, 1992).

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$$U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Fe	0.13360 (2)	−0.00692 (6)	0.86752 (4)	0.0371 (2)
O1	0.3867 (2)	0.0660 (5)	0.8688 (4)	0.093 (3)
O2	0.4887 (2)	−0.0475 (6)	0.8479 (4)	0.106 (2)
N	0.4281 (2)	−0.0527 (5)	0.8802 (4)	0.065 (2)
C1	0.1951 (2)	−0.1052 (4)	1.0257 (3)	0.041 (2)
C2	0.1238 (2)	−0.0668 (5)	1.0552 (3)	0.045 (2)
C3	0.0741 (2)	−0.1630 (5)	0.9729 (4)	0.050 (2)
C4	0.1134 (2)	−0.2619 (5)	0.8892 (4)	0.049 (2)
C5	0.1871 (2)	−0.2272 (4)	0.9213 (3)	0.043 (2)
C6	0.1954 (3)	0.1583 (8)	0.7769 (6)	0.087 (2)
C7	0.1479 (4)	0.2485 (6)	0.8439 (5)	0.082 (3)
C8	0.0773 (3)	0.2000 (7)	0.7932 (5)	0.085 (2)
C9	0.0858 (3)	0.0822 (7)	0.6937 (5)	0.080 (2)
C10	0.1567 (3)	0.0611 (7)	0.6873 (5)	0.077 (2)
C11	0.2588 (2)	−0.0186 (5)	1.0870 (3)	0.048 (2)
C12	0.3279 (2)	−0.0569 (5)	1.0825 (4)	0.051 (2)
C13	0.3555 (2)	−0.2141 (5)	1.0259 (3)	0.045 (2)
C14	0.4048 (2)	−0.2157 (5)	0.9339 (4)	0.049 (2)
C15	0.4316 (2)	−0.3644 (7)	0.8863 (5)	0.068 (2)
C16	0.4096 (3)	−0.5201 (7)	0.9296 (7)	0.084 (3)
C17	0.3623 (3)	−0.5249 (6)	1.0212 (7)	0.081 (3)
C18	0.3368 (2)	−0.3741 (6)	1.0702 (5)	0.061 (2)

Table 2. *Geometric parameters* (Å, °)

Fe—C7	2.026 (5)	C2—C3	1.408 (5)
Fe—C6	2.029 (5)	C3—C4	1.424 (5)
Fe—C2	2.029 (3)	C4—C5	1.411 (5)
Fe—C8	2.031 (4)	C6—C10	1.346 (8)
Fe—C5	2.034 (3)	C6—C7	1.379 (8)
Fe—C10	2.034 (4)	C7—C8	1.419 (8)
Fe—C4	2.039 (4)	C8—C9	1.403 (8)
Fe—C9	2.044 (4)	C9—C10	1.346 (7)
Fe—C3	2.045 (4)	C11—C12	1.334 (5)
Fe—C1	2.046 (3)	C12—C13	1.475 (5)
O1—N	1.203 (5)	C13—C18	1.388 (5)
O2—N	1.220 (5)	C13—C14	1.398 (5)
N—C14	1.472 (5)	C14—C15	1.375 (6)
C1—C2	1.433 (5)	C15—C16	1.372 (8)
C1—C5	1.436 (5)	C16—C17	1.368 (9)
C1—C11	1.455 (5)	C17—C18	1.386 (7)
O1—N—O2	123.8 (5)	C9—C10—C6	110.9 (5)
O1—N—C14	119.0 (4)	C12—C11—C1	129.3 (3)
O2—N—C14	117.2 (4)	C11—C12—C13	125.7 (4)
C2—C1—C5	106.1 (3)	C18—C13—C14	115.4 (4)
C2—C1—C11	123.3 (3)	C18—C13—C12	120.1 (4)
C5—C1—C11	130.4 (3)	C14—C13—C12	124.4 (3)
C3—C2—C1	109.2 (3)	C15—C14—C13	123.2 (4)
C2—C3—C4	107.8 (3)	C15—C14—N	117.1 (4)
C5—C4—C3	108.1 (3)	C13—C14—N	119.7 (3)
C4—C5—C1	108.8 (3)	C16—C15—C14	119.5 (5)
C10—C6—C7	107.8 (5)	C17—C16—C15	119.4 (5)
C6—C7—C8	107.7 (5)	C16—C17—C18	120.5 (5)
C9—C8—C7	105.7 (5)	C17—C18—C13	121.9 (5)
C10—C9—C8	108.0 (5)		

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55977 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: VS1004]

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Structure of [(2*S*,4*R*)-γ-Hydroxynorvalinato][(2*R*,4*S*)-γ-hydroxynorvalinato]-copper(II)

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Abstract

The molecule of the title compound, bis(2-amino-4-hydroxypentanoato-*N*,*O*,*O*′)copper(II), possesses crystallographic $\bar{1}$ symmetry. The Cu coordination geometry is distorted square bipyramidal. By virtue of the centre of symmetry, the metal atom and four of the six ligand donor atoms are in an equatorial plane and are rigorously coplanar. The structure consists of discrete molecules linked *via* O—H···O hydrogen bonds and van der Waals attractive forces.

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