

Table 1. Atomic coordinates ( $\times 10^4$ ) and isotropic thermal parameters ( $\text{\AA}^2 \times 10^3$ ) for [Os(bpy)<sub>3</sub>](PF<sub>6</sub>)<sub>2</sub>

	x	y	z	U*
Os	0	0	2500	41 (1)
N	822 (4)	1897 (5)	3138 (3)	48 (2)
C(1)	433 (5)	2848 (5)	2874 (3)	50 (2)
C(2)	834 (6)	4101 (6)	3304 (3)	63 (3)
C(3)	1650 (8)	4398 (7)	3993 (4)	71 (3)
C(4)	2067 (7)	3444 (7)	4246 (4)	66 (3)
C(5)	1655 (7)	2229 (6)	3809 (3)	59 (3)
P(1)	6667	3333	3698 (2)	76 (1)
F(1)	5414 (7)	3195 (9)	3157 (4)	139 (4)
F(2)	7929 (8)	3512 (9)	4258 (4)	163 (5)

\* The equivalent isotropic  $U$  is defined as one third of the trace of the orthogonalized  $U_{ij}$  tensor.

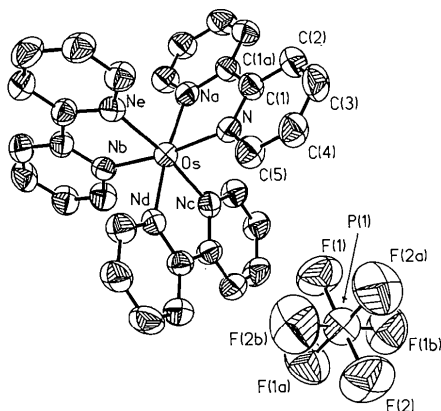


Fig. 1. A view of the title compound showing thermal ellipsoids.

photocatalysts (Lumpkin, Kober, Worl, Murtaza & Meyer, 1990), features which make them desirable for incorporation into solar-energy conversion schemes. The title compound is isostructural with [Ru(bpy)<sub>3</sub>](PF<sub>6</sub>)<sub>2</sub>, the structure of which has been reported previously (Rillema & Jones, 1979).

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## Structure of a Ferrocenylalkyl–Aminomalonamide Derivative

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**Abstract.** *N*-Benzoyl-*N*-(1-ferrocenyl-2,2-dimethyl-1-propyl)amino-*N*',*N*'-di(1,1,3,3-tetramethyl-1-butyl)-malonamide, C<sub>41</sub>H<sub>61</sub>FeN<sub>3</sub>O<sub>3</sub>,  $M_r = 699.81$ , triclinic,

0108-2701/91/112444-03\$03.00

Table 2. Bond distances ( $\text{\AA}$ ) and angles ( $^\circ$ ) for [Os(bpy)<sub>3</sub>](PF<sub>6</sub>)<sub>2</sub>

Atom labels are defined in the figure.			
Os—N	2.062 (4)	C(2)—C(3)	1.367 (9)
N—C(1)	1.359 (9)	C(3)—C(4)	1.377 (13)
N—C(5)	1.349 (7)	C(4)—C(5)	1.359 (9)
C(1)—C(2)	1.387 (8)	P(1)—F(1)	1.560 (8)
C(1)—C(1a)	1.470 (9)	P(1)—F(2)	1.572 (9)
N—Os—N(a)	96.6 (1)	C(2)—C(3)—C(4)	119.1 (6)
N—Os—N(c)	89.3 (2)	C(3)—C(4)—C(5)	119.4 (6)
N(b)—Os—N(c)	77.9 (2)	N—C(5)—C(4)	122.6 (7)
N—Os—N(d)	172.4 (2)	F(1)—P(1)—F(2)	178.2 (5)
Os—N—C(1)	116.6 (3)	F(1)—P(1)—F(1a)	91.0 (3)
Os—N—C(5)	125.1 (5)	F(2)—P(1)—F(1a)	90.9 (5)
C(1)—N—C(5)	118.3 (5)	F(2)—P(1)—F(1b)	88.3 (3)
Os—N—C(1)	120.8 (5)	F(1)—P(1)—F(2a)	88.8 (5)
N—C(1)—C(1a)	114.3 (4)	F(2)—P(1)—F(2a)	89.4 (4)
C(2)—C(1)—C(1a)	124.9 (5)	F(1)—P(1)—F(2b)	90.8 (3)
C(1)—C(2)—C(3)	119.8 (7)		

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$P\bar{1}$ ,  $a = 8.564$  (1),  $b = 12.000$  (2),  $c = 20.111$  (2)  $\text{\AA}$ ,  $\alpha = 79.35$  (1),  $\beta = 78.93$  (1),  $\gamma = 77.64$  (1) $^\circ$ ,  $V = 1958.9$   $\text{\AA}^3$ ,  $Z = 2$ ,  $D_x = 1.186$  g cm<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) =$

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0.71069 Å,  $\mu = 4.2 \text{ cm}^{-1}$ ,  $F(000) = 756$ ,  $T = 296 (2) \text{ K}$ ,  $R = 0.062$ ,  $wR = 0.058$ ,  $w = 1/\sigma^2(F_o)$  for 472 refined parameters and 5346 unique reflexions. The ferrocenyl substituted C atom C(1), bearing one H atom, a *tert*-butyl group and a large amide residue  $\text{N}[\text{C}(\text{O})\text{C}_6\text{H}_5]\text{CH}[\text{C}(\text{O})\text{NHC}(\text{CH}_3)_2\text{CH}_2\text{C}(\text{CH}_3)_3]_2$  as further substituents, is only approximately tetrahedral. The angle included between the ferrocenyl substituent and the central C atom C(11) of the *tert*-butyl group [117.9 (3)°] deviates most from the tetrahedral standard due to the almost eclipsed conformation of C(11) with the cyclopentadienyl ring plane. This, in turn, is certainly caused by the steric requirements of the amide residue. The malonamide part of the molecule is in a rather extended conformation which is stabilized by two short intramolecular hydrogen bonds between the NH groups N(2)/N(3) of the malonamide part and the carbonyl groups C(2)O(2)/C(4)O(4), respectively, while the third carbonyl group C(5)O(5) does not participate in any hydrogen bonding. The cyclopentadienyl rings show no appreciable disorder. They are in an eclipsed conformation with respect to each other and virtually parallel, the ring tilt being only 1.4 (5)°. The iron–ring distances are identical.

**Experimental.** A suitable single crystal was grown from chloroform. Enraf–Nonius CAD-4 diffractometer. Exact cell parameters by least-squares procedures on the Bragg angles of 25 selected reflections ( $5.5 \leq \theta \leq 19^\circ$ ) automatically centered on the diffractometer. Reduced-cell calculations (Zimmermann & Burzlaff, 1985) did not indicate symmetry higher than triclinic. The integrated intensities of 6829 independent reflexions were recorded up to  $(\sin\theta/\lambda)_{\text{max}} = 0.594 \text{ \AA}^{-1}$  [ $hkl$  range +11,  $\pm 15$ ,  $\pm 24$ ;  $\theta/2\theta$  scans,  $\Delta\omega = (0.9 + 0.35\tan\theta)^\circ$ , scan speed 1.0–10.0°min<sup>-1</sup>]. Isotropic intensity fluctuations ( $\pm 1\%$ ) in the net intensities of three reference reflexions (412, 029, 062), remeasured every 50 reflexions, were corrected for. Due to the unfavorable crystal dimensions (0.7 × 0.25 × 0.2 mm) an empirical absorption correction, based on  $\psi$  scans in 10° intervals around the diffraction vectors of seven selected reflexions near  $\chi = 90^\circ$ , was applied (relative transmission = 0.87–1.00). 5352 reflexions satisfied the criterion  $F_o \geq 4.0\sigma(F_o)$  and were used for all further calculations. The structure was solved by Patterson methods (SHELXS86; Sheldrick, 1985) and completed by difference Fourier synthesis techniques. After anisotropic refinement of the non-H atoms, 57 H atoms could be located in difference maps; the remainder were calculated at idealized geometrical positions. They were kept constant in final refinement cycles with the exception of the methyl H atoms which were treated together with their respective C atoms as rigid groups ( $U_{\text{iso}} = 0.05 \text{ \AA}^2$  constant for all H

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters for the non-H atoms

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

	x	y	z	$U_{\text{eq}}$ (Å <sup>2</sup> )
Fe	0.49571 (7)	0.22661 (5)	0.15114 (3)	0.034
Cp11	0.6109 (4)	0.0974 (3)	0.2172 (2)	0.029
Cp12	0.7017 (4)	0.1048 (3)	0.1496 (2)	0.032
Cp13	0.6028 (6)	0.0877 (3)	0.1036 (2)	0.043
Cp14	0.4517 (5)	0.0690 (3)	0.1431 (2)	0.044
Cp15	0.4558 (5)	0.0749 (3)	0.2120 (2)	0.037
Cp21	0.4818 (6)	0.3802 (3)	0.1833 (2)	0.046
Cp22	0.3250 (5)	0.3560 (4)	0.1881 (2)	0.046
Cp23	0.3031 (6)	0.3452 (4)	0.1215 (3)	0.051
Cp24	0.4457 (6)	0.3626 (4)	0.0760 (2)	0.054
Cp25	0.5561 (6)	0.3841 (4)	0.1141 (3)	0.054
C1	0.6766 (4)	0.1098 (3)	0.2800 (2)	0.027
C11	0.5738 (5)	0.0814 (3)	0.3518 (2)	0.035
C111	0.5313 (5)	-0.0388 (4)	0.3670 (2)	0.048
C112	0.6653 (5)	0.0959 (4)	0.4074 (2)	0.052
C113	0.4167 (5)	0.1731 (4)	0.3558 (2)	0.051
N1	0.8488 (3)	0.0453 (2)	0.2768 (1)	0.046
C2	0.9744 (5)	0.0940 (3)	0.2862 (2)	0.031
O2	1.1019 (3)	0.0366 (2)	0.2993 (1)	0.020
C21	0.9455 (4)	0.2243 (3)	0.2814 (2)	0.031
C22	0.8981 (5)	0.2994 (3)	0.2241 (2)	0.040
C23	0.8777 (5)	0.4181 (4)	0.2227 (3)	0.052
C24	0.9033 (6)	0.4603 (4)	0.2781 (3)	0.057
C25	0.9543 (6)	0.3859 (4)	0.3339 (3)	0.054
C26	0.9769 (5)	0.2679 (4)	0.3354 (2)	0.042
C3	0.8868 (4)	-0.0733 (3)	0.2609 (2)	0.026
C4	0.9182 (5)	-0.1781 (4)	0.3175 (2)	0.033
O4	0.8730 (4)	-0.2654 (2)	0.3150 (1)	0.024
N2	0.9972 (4)	-0.1615 (3)	0.3656 (2)	0.059
C41	1.0367 (6)	-0.2467 (4)	0.4273 (2)	0.045
C411	1.1035 (7)	-0.1819 (4)	0.4700 (2)	0.064
C412	0.8776 (6)	-0.2793 (5)	0.4674 (3)	0.068
C42	1.1511 (6)	-0.3580 (4)	0.4081 (2)	0.052
C43	1.3283 (6)	-0.3648 (4)	0.3767 (3)	0.058
C431	1.3852 (8)	-0.4850 (5)	0.3566 (4)	0.105
C432	1.3524 (6)	-0.2759 (5)	0.3125 (3)	0.072
C433	1.4375 (7)	-0.3525 (5)	0.4251 (3)	0.089
C5	1.0184 (4)	-0.0808 (3)	0.1948 (2)	0.030
O5	1.0625 (3)	0.0014 (2)	0.1595 (1)	0.019
N3	1.0652 (4)	-0.1928 (3)	0.1826 (2)	0.054
C51	1.1627 (5)	-0.2309 (3)	0.1188 (2)	0.035
C511	1.2145 (6)	-0.3605 (3)	0.1361 (2)	0.049
C512	1.3151 (5)	-0.1769 (4)	0.0997 (2)	0.048
C52	1.0653 (5)	-0.1929 (3)	0.0591 (2)	0.039
C53	0.9167 (5)	-0.2448 (4)	0.0543 (2)	0.043
C531	0.8335 (7)	-0.1645 (4)	-0.0019 (2)	0.062
C532	0.7958 (5)	-0.2510 (4)	0.1196 (2)	0.053
C533	0.9686 (7)	-0.3643 (4)	0.0324 (3)	0.065

atoms). All non-H atoms were refined with anisotropic displacement parameters by full-matrix least-squares techniques based on  $F$  (472 refined parameters on 5346 structure factors; six structure factors were suppressed due to background problems). At convergence (max. ratio of shift/e.s.d. = 0.033)  $R = 0.062$ ,  $wR = 0.058$ ,  $w = 1/\sigma^2(F_o)$ , function minimized:  $\sum w(|F_o| - |F_c|)^2$ , no extinction correction (SHELX76; Sheldrick, 1976). The final difference map was qualitatively featureless with  $\Delta\rho(\text{max./min.}) = 0.58, -0.55 \text{ e \AA}^{-3}$ . Scattering factors for neutral, isolated atoms (except for H) from Cromer & Waber (1965), those for H atoms based on a bonded spherical atom model as given by Stewart, Davidson & Simpson (1965), corrections for  $f'$  and  $f''$  (Mo  $K\alpha$ ) applied for all except H atoms from *International Tables for X-ray Crystallography* (1974, Vol. IV). Table 1 contains the atomic parameters of the non-H

Table 2. Bond lengths (Å), angles (°), and some torsion angles (°) with e.s.d.'s in parentheses

Fe—Cp11	2.057 (4)	Fe—Cp21	2.036 (4)
Fe—Cp12	2.035 (4)	Fe—Cp22	2.034 (4)
Fe—Cp13	2.033 (4)	Fe—Cp23	2.040 (4)
Fe—Cp14	2.046 (4)	Fe—Cp24	2.042 (4)
Fe—Cp15	2.052 (4)	Fe—Cp25	2.036 (4)
Fe—D1 <sup>a</sup>	1.65	Fe—D2 <sup>a</sup>	1.65
Cp11—C1	1.519 (5)	C1—C11	1.560 (5)
C1—N1	1.506 (4)	N1—C2	1.386 (4)
C2—O2	1.207 (40)	C2—C21	1.518 (5)
N1—C3	1.471 (4)		
C3—C4	1.548 (5)	C3—C5	1.571 (5)
C4—O4	1.204 (4)	C5—O5	1.191 (4)
C4—N2	1.347 (5)	C5—N3	1.372 (4)
N2—C41	1.497 (5)	N3—C51	1.482 (4)
N2...O2 <sup>b</sup>	2.741 (5)	N3...O4 <sup>b</sup>	2.937 (6)
Cp11—C1—C11	117.9 (3)	Cp11—C1—N1	109.3 (3)
C1—C1—N1	111.8 (3)	C1—N1—C2	123.0 (3)
C1—N1—C3	118.8 (3)	C2—N1—C3	118.3 (3)
N1—C2—C21	122.5 (3)	N1—C2—O2	122.5 (3)
C21—C2—O2	120.0 (3)	N1—C3—C4	121.0 (3)
N1—C3—C5	109.6 (3)	C4—C3—C5	112.7 (3)
C3—C4—N2	114.9 (3)	C3—C5—N3	110.3 (3)
C3—C4—O4	119.0 (3)	C3—C5—O5	123.4 (3)
N2—C4—O4	126.1 (4)	N3—C5—O5	126.2 (3)
C4—N2—C41	125.7 (3)	C5—N3—C51	125.7 (3)
Cp12—Cp11—C1—C11	-171.0 (8)	Cp12—Cp11—C1—N1	-41.9 (8)
C1—N1—C2—O2	162.5 (8)	N1—C2—C21—C22	-56.1 (8)
C1—N1—C3—C4	-104.8 (8)	C1—N1—C3—C5	121.4 (8)
N1—C3—C4—O4	144.4 (8)	N1—C3—C5—O5	-8.6 (8)
N1—C3—C4—N2	-36.3 (8)	N1—C3—C5—N3	174.9 (8)
C3—C4—N2—C41	177.3 (8)	C3—C5—N3—C51	167.8 (8)
N2—C41—C42—C43	70.0 (8)	N3—C51—C52—C53	-69.4 (8)

(a) Centroid of the Cp ring.  
(b) Hydrogen bond.

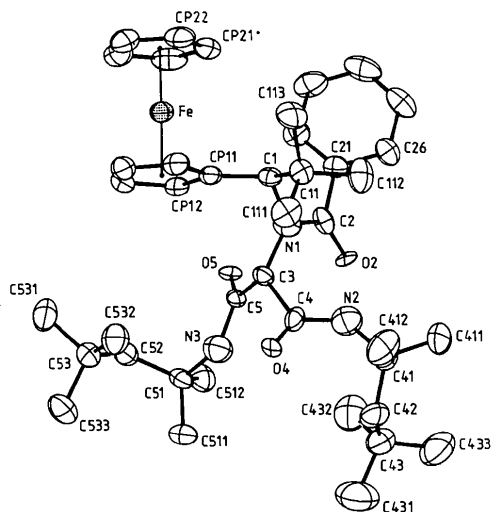
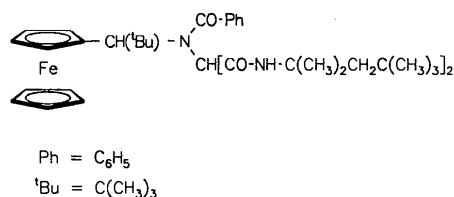


Fig. 1. Molecular structure and crystallographic numbering scheme adopted (ORTEP, Johnson, 1976). Displacement ellipsoids are drawn at the 50% probability level; the H atoms are omitted for clarity. The intramolecular hydrogen bonds are between the amide groups N(2)/N(3) and the carbonyl groups C(2)O(2)/C(4)O(4), respectively.

atoms, Table 2\* bond distances, angles and torsion angles. The molecular structure is shown in Fig. 1 together with the atom numbering adopted.



**Related literature.** The title compound is formed as a by-product in the four-component condensation of benzoic acid, isobutyraldehyde,  $\alpha$ -ferrocenyl neopentylamine and isooctyl isocyanide (Ugi, Lohberger & Karl, 1991). It is extremely acid labile. Under protic conditions it reacts under substitution of the ferrocenyl alkyl part (C<sub>5</sub>H<sub>5</sub>)Fe(C<sub>5</sub>H<sub>4</sub>—CH—*tert*-Bu) at nitrogen with hydrogen. The mechanism of its formation was investigated by <sup>13</sup>C-labeling experiments and computer-assisted generation of a network of conceivable mechanistic pathways (Lohberger, Fontain, Ugi, Müller & Lachmann, 1991).

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\* Lists of structure factors, anisotropic displacement parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54179 (41 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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