$[(C_{10}H_{15}Ni)_2C_{16}H_{24}]$

Table 2. *Bond distances (Å) and bond angles (°) for the non-H atoms*

Ni—C(1)	2·114 (3)	C(3)—C(8)	1·491 (4)
Ni—C(2)	2·169 (3)	C(4)—C(5)	1·411 (4)
Ni—C(3)	2·139 (2)	C(4)—C(9)	1·503 (4)
Ni—C(4)	2·140 (2)	C(5)—C(10)	1·496 (3)
Ni—C(5)	2·082 (2)	C(11)—C(12)	1·414 (3)
Ni—C(11)	1·981 (2)	C(11)—C(18)	1·517 (3)
Ni—C(12)	1·971 (3)	C(12)—C(13)	1·513 (4)
Ni—C(16)	1·966 (3)	C(13)—C(14)	1·525 (4)
C(1)—C(2)	1·407 (3)	C(14)—C(15)	1·532 (4)
C(1)—C(5)	1·461 (3)	C(15)—C(15^i)	1·577 (4)
C(1)—C(6)	1·496 (4)	C(15)—C(16)	1·538 (3)
C(2)—C(3)	1·434 (4)	C(16)—C(17)	1·536 (3)
C(2)—C(7)	1·500 (4)	C(17)—C(18)	1·525 (4)
C(3)—C(4)	1·429 (3)		

C(2)—C(1)—C(5)	107·7 (2)	C(4)—C(5)—C(10)	127·5 (2)
C(2)—C(1)—C(6)	128·0 (2)	C(12)—C(11)—C(18)	125·2 (2)
C(5)—C(1)—C(6)	124·2 (2)	C(11)—C(12)—C(13)	129·2 (2)
C(1)—C(2)—C(3)	108·0 (2)	C(12)—C(13)—C(14)	119·3 (2)
C(1)—C(2)—C(7)	127·5 (2)	C(13)—C(14)—C(15)	114·2 (2)
C(3)—C(2)—C(7)	124·5 (2)	C(14)—C(15)—C(15^i)	111·7 (2)
C(2)—C(3)—C(4)	108·4 (2)	C(14)—C(15)—C(16)	111·5 (2)
C(2)—C(3)—C(8)	126·1 (2)	C(15^i)—C(15)—C(16)	112·6 (2)
C(4)—C(3)—C(8)	125·4 (2)	Ni—C(16)—C(15)	114·6 (2)
C(3)—C(4)—C(5)	107·9 (2)	Ni—C(16)—C(17)	104·6 (2)
C(3)—C(4)—C(9)	125·2 (2)	C(15)—C(16)—C(17)	116·0 (2)
C(5)—C(4)—C(9)	126·9 (2)	C(16)—C(17)—C(18)	112·0 (2)
C(1)—C(5)—C(4)	107·8 (2)	C(11)—C(18)—C(17)	110·4 (2)
C(1)—C(5)—C(10)	124·4 (2)		

Symmetry code: (i) $-x, \frac{1}{2}-y, \frac{1}{2}-z$.

given in Table 1* with bond lengths and angles in Table 2. The molecule is shown in Fig. 1. The programs *PLATON* and *PLUTON* (Spek, 1982) were used for the calculation of the geometrical data and the plot respectively.

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and geometrical data concerning H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51048 (52 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
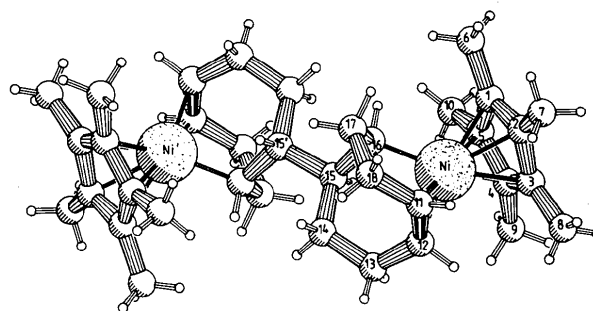

Fig. 1. View of the molecule with numbering scheme.

Related literature. For the preparation of the compound and a discussion of the results based on room-temperature X-ray data see Fischer, Boersma, Kojić-Prodić & Spek (1985).

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Actinide Structural Studies. 16.* Dinitratodioxobis(pyridine)uranium(VI)

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Abstract. $[UO_2(C_5H_5N)_2(NO_3)_2]$, $M_r = 552·24$, monoclinic, $P2_1/a$, $a = 16·456$ (3), $b = 7·861$ (3), $c = 5·719$ (1) Å, $\beta = 95·12$ (2)°, $U = 736·9$ (4) Å³, $Z = 2$, $D_x = 2·49$ g cm⁻³, $T = 290$ K, $\mu(\text{Mo } K\alpha) = 104·9$ cm⁻¹, $F(000) = 508$, $\lambda(\text{Mo } K\alpha) = 0·71069$ Å, $R = 0·075$ for 826 observed $[I/\sigma(I) \geq 3·0]$ reflections. The complex is centrosymmetric with hexagonal bipyramidal geometry about U. U—O(uranyl) bond length 1·751 (15), mean U—O(NO₃) 2·487 (14), U—N 2·543 (15) Å. The

* Part 15: Alcock, Flanders, Pennington & Brown (1988).
† To whom correspondence should be addressed.

Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic temperature factors* ($\text{Å}^2 \times 10^3$) *with e.s.d.'s in parentheses*

	x	y	z	U_{eq}*
U(1)	5000	5000	0	42 (1)
O(1)	5270 (8)	5952 (22)	−2587 (25)	59 (5)
O(2)	5774 (7)	7368 (18)	2034 (24)	55 (5)
O(3)	5261 (15)	9853 (20)	2731 (39)	75 (7)
O(4)	4478 (7)	7770 (19)	1344 (24)	62 (5)
N(1)	5190 (10)	8380 (24)	2095 (26)	52 (6)
N(2)	3522 (9)	5634 (26)	−1416 (33)	52 (6)
C(1)	2936 (15)	5109 (23)	−183 (42)	53 (7)
C(2)	2110 (17)	5250 (23)	−854 (57)	65 (10)
C(3)	1904 (11)	6027 (36)	−2954 (40)	64 (9)
C(4)	2487 (12)	6598 (34)	−4329 (35)	66 (8)
C(5)	3316 (11)	6418 (29)	- 3449 (34)	56 (7)

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

Table 2. *Bond lengths* (Å) *and angles* (°) *around the* U *atom with e.s.d.'s in parentheses*

U(1)–O(1)	1·751 (15)	U(1)–O(2)	2·486 (13)
U(1)–O(4)	2·488 (15)	U(1)–N(2)	1·543 (15)

O(1)–U(1)–O(2)	85·2 (6)	O(1)–U(1)–O(4)	90·4 (6)
O(2)–U(1)–O(4)	51·4 (4)	O(1)–U(1)–N(2)	87·4 (6)
O(2)–U(1)–N(2)	116·0 (5)	O(4)–U(1)–N(2) '	65·2 (5)

dimensions of the pyridine ring and nitrate group agree with standard values.

Experimental. The complex was prepared by the method described by Barr & Horton (1952). The bright yellow crystals were washed with ether and dried. Freshly prepared crystals encapsulated in a Lindemann capillary in a pyridine atmosphere were used because of a rapid loss of pyridine when exposed to air. Data were collected with a Syntex $P2_1$ automatic four-circle diffractometer. Maximum 2θ was 50°, scan range $\pm 1 \cdot 1°$ (2θ) around $K\alpha_1$–$K\alpha_2$, scan speed 5–29·3° min^{-1} depending on the intensity of a 2 s prescan. Background intensities were measured at each end of the scan for 0·25 of the scan time. Three standard reflections, monitored every 200 reflections, showed a 6% reduction during data collection; data were rescaled to correct for this; $h = 0$ to 20, $k = 0$ to 10, $l = -6$ to 6. Unit-cell dimensions and standard deviations were obtained by least-squares fit to 15 high-angle reflections ($25 < 2\theta < 29°$). The crystal was recentred every 100 reflections because of movement in the capillary. Of the 1414 reflections collected (1260 unique, $R_{int} = 0\cdot08$), 826 were considered observed [$I/\sigma(I) \geq 3\cdot0$] and corrected for Lorentz, polarization and absorption effects, the last by the Gaussian method. Because the U atom lies in a special position, only about 120 of the observed reflections have ($h + k$) odd. The crystal dimensions were 0·41 × 0·49 ×

0·57 mm, giving rise to maximum and minimum transmission factors of 0·112 and 0·031.

Systematic absences $h0l$: $h \neq 2n$ and $0k0$: $k \neq 2n$ gave the space group as $P2_1/a$. The U atom was placed at 0·5, 0·5, 0·0, and lighter atoms were found by successive Fourier syntheses. Anisotropic temperature factors were used for all except the H atoms which were inserted at calculated positions with fixed isotropic temperature factors ($U = 0\cdot07 \text{ Å}^2$) and not refined. Final refinement on F was by cascaded least-squares methods for 106 parameters, to a final shift/e.s.d. ratio of 0·01. A weighting scheme of the form $w = 1/[\sigma^2(F) + g(F^2)]$ was applied with $g = 0\cdot00090$. Calculations performed on a Data General DG30 using the *SHELXTL* system (Sheldrick, 1984). Final $R = 0\cdot075$, $wR = 0\cdot086$. The relatively high R value is certainly due to the magnitude of the absorption effect. Largest positive and negative peaks on a final difference Fourier synthesis were at heights of 4·26 and $-3\cdot69$ e Å^{-3}, with all of the relatively large residuals close to the U atom. Scattering factors in the analytical form and anomalous-dispersion factors were taken from *International Tables for X-ray Crystallography* (1974). Atomic coordinates are given in Table 1. Bond lengths and angles around the U atom are listed
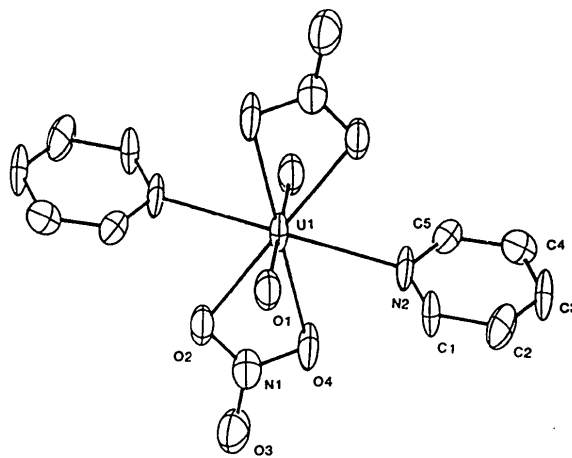

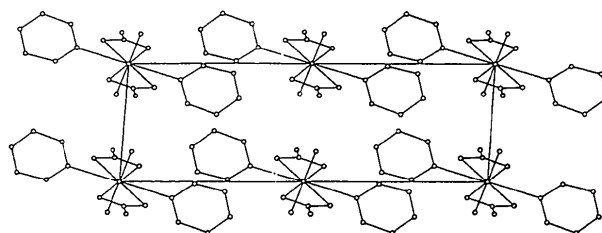Fig. 1. View of a molecule of the title compound.

Fig. 2. Unit-cell contents viewed down **b**.

in Table 2.* Fig. 1 shows a molecular diagram and Fig. 2 the unit-cell contents.

Related literature. The structures of relatively few uranyl complexes which contain a U—N bond are known. They include the following pyridine diketonate complexes: $UO_2(CH_3COCHCOCH_3)_2C_5H_5N$ (Alcock, Flanders & Brown, 1984a,b), UO_2-$(C_8H_{12}N)(CH_3COCHCH_3)_2$ (Alcock & Flanders, 1987), $UO_2(PhCOCHCOPh)_2(C_5H_5N)$ and UO_2-$(CH_3COCHCOOBu^t)_2(C_5H_5N)$ (Alcock, Flanders, Pennington & Brown, 1987), and have a range of U—N distances of 2·47 (1)–2·595 (2) Å. These distances are slightly shorter than in the bis-tropolonato uranyl

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* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, full bond lengths and angles, and details of mean planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51005 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

complex described by Degetto (U—N 2·62 Å; Degetto, Marangoni, Bombieri, Forsellini, Baracco & Gaziani, 1974).

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Structure of Benzoylferrocene

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Abstract. $(C_5H_5)Fe(C_5H_4COC_6H_5)$, $C_{17}H_{14}FeO$, $M_r = 290·15$, monoclinic, $P2_1/n$, $a = 6·1969$ (4), $b = 15·2293$ (3), $c = 13·9109$ (7) Å, $\beta = 99·180$ (3)°, $V = 1296·0$ (1) Å^3, $Z = 4$, $D_x = 1·487$ g cm^{-3}, $\lambda(Mo\,K\alpha_1) = 0·70930$ Å, $\mu = 11·3$ cm^{-1}, $F(000) = 600$, $T = 295$ K, $R = 0·028$ for 2310 reflections. The ferrocene group is 6° from an eclipsed orientation, with Fe—C = 2·035–2·053 (2), mean 2·043 Å (libration corrections increase all bond distances by ~0·01 Å). The C—C bonds in the substituted cyclopentadienyl (Cp) ring show evidence of some double-bond localization, with the bonds at the substituted C atom being 1·438 and 1·442 (3) Å, with the other three bonds 1·405, 1·421 and 1·411 (3) Å; in the other Cp ring, the C—C bonds are all in the range 1·405–1·411 (4) Å. The effect of this bond localization is also noted in the short Cp—CO distance of 1·467 (3) Å, relative to Ph—CO of 1·496 (3) Å. The carbonyl group is slightly non-planar [with the C atom 0·009 (2) Å out of the plane of the three bonded atoms and 0·040 (2) Å out of the Cp plane], and rotated 14·2° out of the Cp plane; the phenyl group is rotated out of the carbonyl plane by a further 26·2°, these deviations from coplanarity being a result of steric interaction between Cp and Ph hydrogen atoms.

Experimental. Reaction of bis(benzonitrile)dichloropalladium with lithioferrocene gave a product which crystallized from dichloromethane/hexane as red wedges, {100}, (010), (001), (0$\bar{1}$2) developed, 0·18 × 0·25 × 0·27 mm. Enraf–Nonius CAD-4F diffractometer, lattice parameters from 25 reflections with $\theta = 20$–24°. Intensities for $\theta \leq 30·0°$, hkl: 0 to 8, 0 to 21, −19 to 19, ω–2θ scan, ω scan width (0·60 + 0·35 tanθ)° at 1·3–10° min^{-1}, extended 25% on each side for background measurement, three standard reflections showed negligible variations in intensity, Lp and absorption corrections (numerical integration, transmission factors 0·664–0·836), 3764 independent reflections measured, 2310 with $I \geq 3\sigma(I)$, where $\sigma^2(I) = S + 4(B_1 + B_2) + (0·04I)^2$, $S =$ scan, B_1 and $B_2 =$ background counts. Structure by Patterson and