Acta Cryst. (1988). C44, 1663–1664

Structure of μ-[(4,5,8-η; 4′,5′,8′-η)-Bi-1,1′-cyclooct-4-enyl]-bis[(η-pentamethylcyclopentadienyl)nickel]

BY A. L. SPEK*

Vakgroep Algemene Chemie, afdeling Kristal- en Structuurchemie, University of Utrecht, Padualaan 8, 3584 CH Utrecht, The Netherlands

J. L. DE BOER

Material Science Centre, Laboratorium voor Anorganische Chemie, University of Groningen, 9747 AG Groningen, The Netherlands

AND B. FISCHER AND J. BOERSMA

Laboratory of Organic Chemistry, Department of Metal-Mediated Synthesis, Padualaan 8, 3584 CH Utrecht, The Netherlands

(*Received* 22 *March* 1988; *accepted* 18 *May* 1988)

Abstract. $[(C_{10}H_{15}Ni)_2C_{16}H_{24}]$, $M_r = 604{\cdot}20$, monoclinic, $A2/a$, $a = 13{\cdot}025\ (2)$, $b = 9{\cdot}367\ (1)$, $c = 26{\cdot}951\ (3)$ Å, $\beta = 112{\cdot}42\ (2)°$, $V = 3039{\cdot}6\ (7)$ Å^3, $Z = 4$, $D_x = 1{\cdot}320$ (110 K), $D_m = 1{\cdot}29$ g cm^{-3} (295 K), λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 12{\cdot}7$ cm^{-1}, $F(000) = 1304$, $T = 110$ K, $R = 0{\cdot}043$ for 4475 observed reflections with $I > 2{\cdot}5\sigma(I)$. Each Ni atom is pseudo-tetrahedrally coordinated by an Ni—C σ bond and an Ni(C=C) π bond of a C_{16} fragment formed by dimerization of two cod ligands and two bonds of the asymmetric C_5Me_5 ring. The C_5Me_5-ring bond distances range from 1·407 to 1·461 Å.

Experimental. Data were collected (de Boer & Duisenberg, 1984) for a red crystal (0·10 × 0·25 × 0·25 mm) mounted on top of a glass fibre with an Enraf–Nonius CAD-4F diffractometer (110 K) using Mo $K\alpha$ radiation (graphite monochromator). Lattice parameters and their e.s.d.'s were derived from the setting angles of 21 reflections with $22 < \theta < 24°$. Intensity data for 7215 reflections (h 0:20, k 0:15, l −43:40; $\theta < 35°$) were collected in the $\omega/2\theta$ scan mode with $\Delta\omega = 0{\cdot}70 + 0{\cdot}35\tan\theta°$. Three reference reflections ($\bar{1}24$, $1\bar{1}7$, $40\bar{8}$) measured every hour of X-ray exposure time indicated no decay over 82 h of X-ray exposure. The intensity data were corrected for Lp but not for absorption (in view of a variation of less than 8% in the intensity of a 360° ψ scan of the reflection $53\bar{3}$), resulting in 4475 reflections with $I > 2{\cdot}5\sigma(I)$. Variance $\sigma^2(I)$ calculated based on counting statistics plus a term $(PI)^2$, where $P(= 0{\cdot}009)$ is the instability constant as derived from the excess variance in the reference reflections (McCandlish, Stout & Andrews, 1975). The space group was derived from the observed systematic absences. The structure was solved with standard Patterson and Fourier techniques and refined on F by blocked full-matrix least squares with *SHELX*76 (Sheldrick, 1976). H atoms were located from a difference map. Refinement with weights based on $w = 1/\sigma^2(F)$ converged at $R = 0{\cdot}043$ [$wR = 0{\cdot}034$; $S = 2{\cdot}34$; 254 parameters; $(\Delta/\sigma)_{ave} = 0{\cdot}1$, $(\Delta/\sigma)_{max} = 0{\cdot}8$]. The refined parameter set included a scale factor, all coordinates, anisotropic thermal parameters for the non-H atoms and one common isotropic temperature factor for the H atoms. A final difference Fourier map did not show residual peaks outside −0·66 and 0·83 e Å^{-3}. Scattering factors of Cromer & Mann (1968) and anomalous-dispersion terms from Cromer & Liberman (1970) were used. Final parameters are

Table 1. *Final coordinates and equivalent isotropic thermal parameters and their e.s.d.'s in parentheses*

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

	x	y	z	U_{eq}(Å^2)
Ni	0·01701 (2)	0·24202 (3)	0·11785 (1)	0·0117 (1)
C(1)	0·1626 (2)	0·3435 (2)	0·1187 (1)	0·0157 (5)
C(2)	0·1142 (2)	0·2738 (2)	0·0689 (1)	0·0152 (5)
C(3)	0·0040 (2)	0·3288 (2)	0·0423 (1)	0·0150 (5)
C(4)	−0·0132 (2)	0·4395 (2)	0·0747 (1)	0·0154 (5)
C(5)	0·0821 (2)	0·4469 (2)	0·1226 (1)	0·0150 (5)
C(6)	0·2774 (2)	0·3252 (3)	0·1601 (1)	0·0236 (6)
C(7)	0·1649 (2)	0·1592 (3)	0·0466 (1)	0·0257 (7)
C(8)	−0·0769 (2)	0·2849 (3)	−0·0117 (1)	0·0243 (6)
C(9)	−0·1154 (2)	0·5310 (3)	0·0595 (1)	0·0249 (7)
C(10)	0·1052 (2)	0·5512 (2)	0·1677 (1)	0·0225 (6)
C(11)	−0·0372 (2)	0·0424 (2)	0·1071 (1)	0·0227 (6)
C(12)	−0·1251 (2)	0·1397 (2)	0·0990 (1)	0·0200 (6)
C(13)	−0·1892 (2)	0·1698 (3)	0·1344 (1)	0·0217 (6)
C(14)	−0·1299 (2)	0·1593 (3)	0·1952 (1)	0·0194 (6)
C(15)	−0·0244 (1)	0·2501 (3)	0·2184 (1)	0·0144 (4)
C(16)	0·0621 (2)	0·2060 (2)	0·1952 (1)	0·0150 (5)
C(17)	0·0977 (2)	0·0486 (3)	0·2024 (1)	0·0250 (6)
C(18)	0·0197 (2)	−0·0451 (2)	0·1576 (1)	0·0293 (7)

* To whom correspondence should be addressed.

0108-2701/88/091663-02$03.00

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.

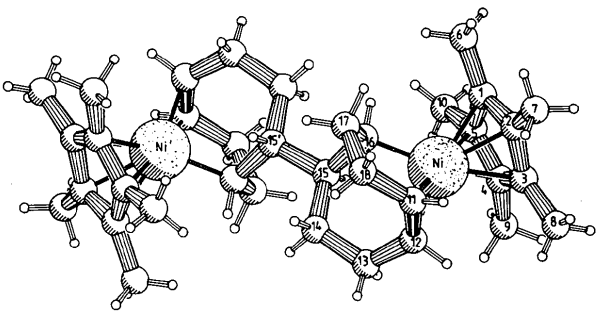

Fig. 1. View of the molecule with numbering scheme.

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).

The investigation was supported by the Netherlands Foundation for Chemical Research (SON) with financial aid from the Netherlands Organization for Scientific Research (NWO).

References

BOER, J. L. DE & DUISENBERG, A. J. M. (1984). *Acta Cryst.* A**40**, C410.

CROMER, D. T. & LIBERMAN, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.

CROMER, D. T. & MANN, J. B. (1968). *Acta Cryst.* A**24**, 321–324.

FISCHER, B., BOERSMA, J., KOJIĆ-PRODIĆ, B. & SPEK, A. L. (1985). *J. Chem. Soc. Chem. Commun.* pp. 1237–1239.

MCCANDLISH, L. E., STOUT, G. H. & ANDREWS, L. C. (1975). *Acta Cryst.* A**31**, 245–249.

SHELDRICK, G. M. (1976). *SHELX*76. Program for crystal structure determination, Univ. of Cambridge, England.

SPEK, A. L. (1982). *The EUCLID Package*. In *Computational Crystallography*, edited by D. SAYRE, p. 528. Oxford: Clarendon Press.

Acta Cryst. (1988). C**44**, 1664–1666

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

BY MARK PENNINGTON, NATHANIEL W. ALCOCK† AND DAVID J. FLANDERS

Department of Chemistry, University of Warwick, Coventry, CV4 7AL, England

(*Received* 1 *March* 1988; *accepted* 4 *May* 1988)

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

* Part 15: Alcock, Flanders, Pennington & Brown (1988).

† To whom correspondence should be addressed.

0108-2701/88/091664-03$03.00