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Structure of Bis(hexafluoroacetylacetonato)bis(tetrahydrofuran)nickel(II)

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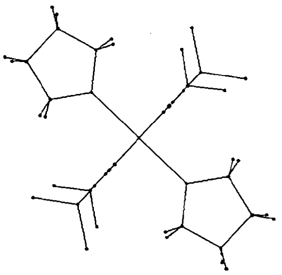
Abstract. Bis(hexafluoroacetylacetonato)bis(tetrahydrofuran)nickel(II),[$Ni(C_5HF_6O_2)_2(C_4H_8O)_2$],$M_r$ = 617·0, tetragonal, $P4_2/mnm$, $a = 8{\cdot}524\,(2)$, $c = 17{\cdot}452\,(4)$ Å, $V = 1267{\cdot}9\,(5)$ Å^3, $D_x = 1{\cdot}616$ Mg m^{-3}, $Z = 2$, $\mu = 0{\cdot}882$ mm^{-1}, λ(Mo $K\alpha$) = 0·71073 Å, $F(000) = 620$, $T = 298$ K, $R = 0{\cdot}065$ and $wR = 0{\cdot}0935$ for 312 averaged reflections with $I > 3\sigma(I)$. The X-ray crystal structure of an octahedral Ni$^{\text{II}}$ complex containing hexafluoroacetylacetonato (hfa) ligands is reported. The Ni atom resides at the cell origin and forms bonds to two equatorial hfa ligands and two molecules of THF that are coordinated axially in a *trans* configuration. The complex is distorted tetragonally and possesses D_{2h} point group symmetry.

Experimental. Bis(hexafluoroacetylacetonato)bis(tetrahydrofuran)nickel(II) was prepared by refluxing anhydrous bis(hexafluoroacetylacetonato)nickel(II) in THF. Crystals suitable for analysis were obtained by slow evaporation from THF. A small, regular-shaped green crystal 0·1 × 0·1 × 0·15 mm was selected and mounted on the end of glass fiber in a random orientation. Tetragonal symmetry suggested on the basis of the unit-cell constants and axial photographs which all exhibited m symmetry. Refined cell parameters obtained from the setting angles of 25 reflections with $15 < 2\theta < 30°$. Data collection was carried out using a Nicolet *R3m/V* diffractometer (graphite-monochromated Mo $K\alpha$ radiation) using the ω-scanning technique in bisecting geometry. Scan rate variable, 3–15° min^{-1}; scan range, 1·5° in ω. Intensities were measured for 3648 reflections ($-2 < h < 9$, $-9 < k < 9$, $-18 < l < 18$) with $3{\cdot}5 < 2\theta < 45°$ and averaged in the $4/mmm$ Laue class to give 477 unique reflections ($R_{\text{int}} = 0{\cdot}0241$) with $(\sin\theta/\lambda_{\text{max}}) = 0{\cdot}582$ Å^{-1}. Three standards ($\bar{2}\bar{2}\bar{5}$, $\bar{2}2\bar{5}$, $\bar{2}25$) measured every 100 data showed no significant variation over the period of data collection. Data were corrected for absorption, Lorentz and polarization effects. Absorption corrections applied empirically on the basis of azimuthal scans of seven strong reflections spanning a range of 2θ values (min. and max. transmission, 0·5737 and 0·6239, respectively). Structure solution and refinement carried out using *SHELXTL* (Sheldrick, 1986). Intensity statistics favored a centrosymmetric cell and satisfactory refinement was obtained using the $P4_2/mnm$ space group. Coordinates of most of the atoms were obtained using direct methods; remaining non-H



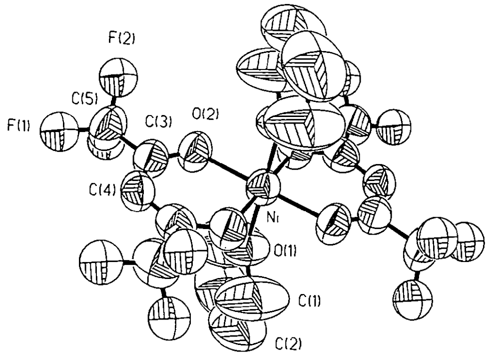


Fig. 1. A perspective view of the bis(hexafluoroacetylacetonato)-bis(tetrahydrofuran)nickel(II) adduct illustrating the atomic number scheme. Thermal ellipsoids have been drawn at the 50% probability level. H atoms have been omitted for clarity.

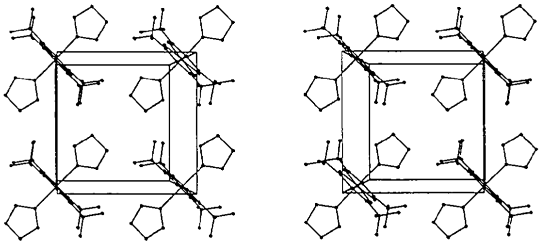


Fig. 2. A stereoview packing diagram projected down the crystallographic c axis.

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Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic displacement coefficients* (Å$^2 \times 10^3$)

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Ni	0	0	5000	82 (1)
O(1)	1693 (10)	1693	5000	121 (5)
O(2)	−1197 (6)	1197	4197 (4)	98 (3)
C(1)	3204 (20)	1474 (25)	5000	250 (18)
C(2)	4038 (22)	2865 (25)	5000	241 (17)
C(3)	−995 (10)	995	3488 (8)	94 (4)
C(4)	0	0	3120 (9)	88 (4)
C(5)	−2042 (14)	2042	3026 (9)	123 (5)
F(1)	−1937 (9)	1937	2294 (5)	187 (5)
F(2)	−3500 (9)	1810 (14)	3183 (5)	247 (5)

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

Ni—O(1)	2·041 (12)	Ni—O(2)	2·011 (5)
Ni—O(1*A*)	2·041 (12)	Ni—O(2*A*)	2·011 (5)
Ni—O(2*B*)	2·012 (5)	Ni—O(2*C*)	2·012 (5)
O(1)—C(1)	1·301 (19)	O(1)—C(1*A*)	1·301 (19)
O(2)—C(3)	1·262 (15)	C(1)—C(2)	1·383 (29)
C(2)—C(2*A*)	1·415 (41)	C(3)—C(4)	1·360 (10)
C(3)—C(5)	1·497 (12)	C(4)—C(3*A*)	1·360 (10)
C(5)—F(1)	1·283 (19)	C(5)—F(2)	1·288 (15)
C(5)—F(2*A*)	1·288 (15)		

O(1)—Ni—O(2)	90·0 (2)	O(1)—Ni—O(1*A*)	180·0 (1)
O(2)—Ni—O(1*A*)	90·0 (2)	O(1)—Ni—O(2*A*)	90·0 (2)
O(2)—Ni—O(2*A*)	180·0 (1)	O(1*A*)—Ni—O(2*A*)	90·0 (2)
O(1)—Ni—O(2*B*)	90·0 (2)	O(2)—Ni—O(2*B*)	91·7 (3)
O(1*A*)—Ni—O(2*B*)	90·0 (2)	O(2*A*)—Ni—O(2*B*)	88·3 (3)
O(1)—Ni—O(2*C*)	90·0 (2)	O(2)—Ni—O(2*C*)	88·3 (3)
O(1*A*)—Ni—O(2*C*)	90·0 (2)	O(2*A*)—Ni—O(2*C*)	91·7 (3)
O(2*B*)—Ni—O(2*C*)	180·0 (1)	Ni—O(1)—C(1)	126·7 (10)
Ni—O(1)—C(1*A*)	126·7 (10)	C(1)—O(1)—C(1*A*)	106·5 (20)
Ni—O(2)—C(3)	123·0 (1)	O(1)—C(1)—C(2)	112·7 (17)
C(1)—C(2)—C(2*A*)	104·0 (11)	O(2)—C(3)—C(4)	129·3 (8)
O(2)—C(3)—C(5)	111·4 (6)	C(4)—C(3)—C(5)	119·3 (12)
C(3)—C(4)—C(3*A*)	123·7 (15)	C(3)—C(5)—F(1)	116·9 (6)
C(3)—C(5)—F(2)	111·7 (12)	F(1)—C(5)—F(2)	105·6 (12)
C(3)—C(5)—F(2*A*)	111·6 (12)	F(1)—C(5)—F(2*A*)	105·6 (12)
F(2)—C(5)—F(2*A*)	104·5 (11)		

atoms located on difference Fourier maps. H atoms were placed in idealized positions with fixed isotropic *U*. All non-H atoms were refined anisotropically using scattering factors, including terms for anomalous dispersion, taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Refinement based on *F* using weights of the form $w^{-1} = [\sigma^2(F) + 0{\cdot}0022(F^2)]$. Convergence to conventional *R* values of $R = 0{\cdot}0645$ and $wR = 0{\cdot}0935$ obtained using 58 variable parameters and 312 observed reflections with $I > 3\sigma(I)$. No reflections had intensities beyond the range for valid coincidence correction. For final cycle, max. shift/σ = 0·000 with min. and max. residual electron densities of −0·30 and 0·28 e Å^{-3}, respectively. A perspective drawing of the structure illustrating the atomic numbering scheme is given in Fig. 1; Fig. 2 is a stereoview packing diagram of the complex. Atomic positional parameters and equivalent isotropic thermal parameters for all non-H atoms are presented in Table 1.* Bond distances and angles are summarized in Table 2.

Related literature. A number of transition metal complexes containing hfa ligands have been characterized crystallographically and the coordination chemistry of β-diketones has been reviewed (Fackler, 1965; Kawaguchi, 1986). The structure reported here is similar to a recently reported Ni(hfa)$_2$ complex containing nitroxyl radical ligands (Porter, Dickman & Doedens, 1988).

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52662 (4 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Tetrakis(μ-acetato)bis(2-pyridone)dicopper

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Abstract. $[Cu_2(C_5H_5NO)_2(C_2H_3O_2)_4]$, $M_r = 553{\cdot}4$, monoclinic, $C2/c$, $a = 13{\cdot}569\ (3)$, $b = 8{\cdot}6013\ (13)$, $c = 18{\cdot}564\ (4)$ Å, $\beta = 93{\cdot}541\ (11)$°, $V = 2162$ Å^3, $Z = 4$ (implying that each molecule lies on a twofold special position), $D_x = 1{\cdot}700$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71073$ Å, $\mu = 2{\cdot}027$ mm^{-1}, $F(000) = 1128$, $T =$

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