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Redetermination of *cis*-Bis(4-methylpyridine)bis[4,4,4-trifluoro-1-(2-thienyl)-1,3-butanedionato-*O*,*O*′]nickel(II)

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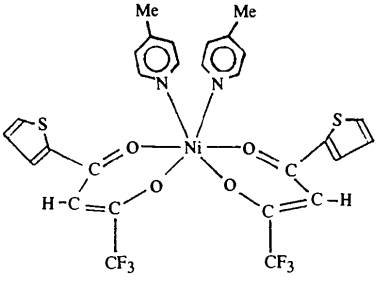
Abstract

The redetermination of *cis*-$[Ni(TTA)_2(pic)_2]$, where TTA = 4,4,4-trifluoro-1-(2-thienyl)-1,3-butanedione ($C_8H_4F_3O_2S$) and pic = 4-methylpyridine (C_6H_7N), corroborates the results of a previous but less accurate study [Pretorius & Boeyens (1978), *J. Cryst. Mol. Struct.* **6**, 169–176]. The Ni—O bond lengths are 2.022 (3) and 2.058 (4) Å and the Ni—N bond lengths are 2.097 (4) Å.

Comment

The structure of $[Ni(TTA)_2(pic)_2]$, (I), was originally determined (Pretorius & Boeyens, 1978) from counter data refined to $R = 0.10$. We are interested in the structure because $M(TTA)_2$ complexes exhibit good tribological behaviour, and neutral nitrogen bases significantly influence the tribological properties of $M(DTP)_2$ if both are present in lubricating oil (Sulek, 1993; Shiomi, Tokashik, Tomizawa & Kuribayashi, 1989). We have redetermined the structure of (I) since, as Pretorius & Boeyens (1978) themselves noted, the precision of the original study was compromised by the poor quality of their crystals.



(I)

Pretorius & Boeyens (1978) report $b = 18.278$, $c = 17.855$ Å [*cf.* our values of 17.599 (6) and 18.111 (3) Å]. Otherwise, our results are in agreement with theirs. The central Ni atom lies on a diad axis and is coordinated by four O atoms from two TTA ligands, and by two N atoms from two 4-methylpyridine ligands. In contrast to (I), $[Ni(TTA)_2(3\text{-mpy})_2]$ (3-mpy = 3-methylpyridine) (Xiong, You, Dong & Huang, 1995) adopts a *trans* configuration, presumably for steric reasons. The Ni—O and Ni—N bond distances in (I) are not significantly different from those in $[Ni(TTA)_2(3\text{-mpy})_2]$ (Xiong *et al.*, 1995).

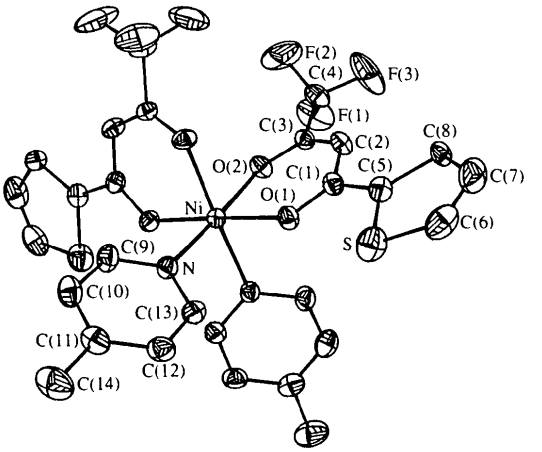


Fig. 1. Molecular structure showing 30% probability displacement ellipsoids. H atoms are omitted for clarity.

Experimental

$Ni(TTA)_2$ was dissolved in ethanol and excess 4-methylpyridine was added until the colour of the solution changed from green to dark green. Dark green prismatic crystals were obtained when the solution was left at room temperature for a few weeks. Recrystallization was from $EtOH/CHCl_3$.

Crystal data

$[Ni(C_8H_4F_3O_2S)_2(C_6H_7N)_2]$
$M_r = 687.30$
Monoclinic
$C2/c$
$a = 9.381$ (3) Å
$b = 17.599$ (6) Å
$c = 18.111$ (3) Å
$\beta = 95.09$ (3)°
$V = 2978$ (1) Å^3
$Z = 4$
$D_x = 1.53$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 13.99$–$14.98°$
$\mu = 0.860$ mm^{-1}
$T = 296$ K
Prism
0.38 × 0.28 × 0.20 mm
Dark green

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
1920 observed reflections [$I > 3\sigma(I)$]
$R_{int} = 0.0115$

Absorption correction: refined from ΔF (*DIFABS*; Walker & Stuart, 1983)	$\theta_{max} = 25°$
$T_{min} = 0.960$, $T_{max} = 0.996$	$h = 0 \rightarrow 11$
2890 measured reflections	$k = -20 \rightarrow 0$
2741 independent reflections	$l = -21 \rightarrow 21$
	3 standard reflections monitored every 300 reflections
	intensity decay: 1.9%

Refinement

Refinement on F	$w = 1/\sigma^2(F)$
$R = 0.064$	$(\Delta/\sigma)_{max} = 0.11$
$wR = 0.074$	$\Delta\rho_{max} = 0.67$ e Å^{-3}
$S = 1.71$	$\Delta\rho_{min} = -0.49$ e Å^{-3}
1920 reflections	Extinction correction: none
195 parameters	Atomic scattering factors from Cromer & Waber (1974)
H-atom parameters not refined	

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

$B_{eq} = (8\pi^2/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{eq}
Ni	1/2	0.23077 (5)	1/4	2.73 (4)
S	0.1432 (2)	0.1871 (1)	0.0482 (1)	5.7 (1)
F(1)	0.2787 (6)	0.4204 (2)	0.3860 (3)	8.4 (3)
F(2)	0.3840 (6)	0.4777 (2)	0.3080 (3)	10.6 (3)
F(3)	0.1609 (6)	0.4640 (3)	0.2943 (3)	10.8 (3)
O(1)	0.3287 (4)	0.2291 (2)	0.1740 (2)	3.4 (1)
O(2)	0.4057 (4)	0.3172 (2)	0.3046 (2)	3.5 (2)
N	0.5988 (4)	0.1501 (2)	0.1863 (2)	3.2 (2)
C(1)	0.2311 (5)	0.2775 (3)	0.1654 (3)	3.2 (2)
C(2)	0.2167 (6)	0.3417 (3)	0.2102 (3)	3.5 (2)
C(3)	0.3047 (6)	0.3557 (3)	0.2739 (3)	3.1 (2)
C(4)	0.2824 (7)	0.4293 (3)	0.3149 (4)	4.4 (3)
C(5)	0.1271 (6)	0.2648 (3)	0.1011 (3)	3.6 (2)
C(6)	0.0043 (9)	0.2123 (5)	−0.0107 (4)	5.9 (4)
C(7)	−0.0589 (8)	0.2750 (5)	0.0062 (4)	6.5 (4)
C(8)	0.0007 (6)	0.3120 (3)	0.0737 (3)	3.5 (2)
C(9)	0.7386 (6)	0.1516 (3)	0.1796 (3)	4.2 (3)
C(10)	0.8070 (7)	0.1001 (4)	0.1379 (4)	4.9 (3)
C(11)	0.7297 (7)	0.0425 (3)	0.1005 (3)	4.4 (3)
C(12)	0.5833 (7)	0.0420 (3)	0.1065 (5)	4.3 (3)
C(13)	0.5230 (6)	0.0951 (3)	0.1492 (3)	3.8 (3)
C(14)	0.797 (1)	−0.0154 (4)	0.0553 (4)	7.1 (4)

Table 2. *Selected geometric parameters* (Å, °)

Ni—O(1)	2.022 (3)	O(1)—C(1)	1.251 (6)
Ni—O(2)	2.058 (4)	O(2)—C(3)	1.253 (6)
Ni—N	2.097 (4)		
O(1)—Ni—O(2)	89.3 (1)	O(1)—Ni—O(1^i)	178.3 (2)
O(1^i)—Ni—O(2)	91.9 (1)	O(2)—Ni—N	174.5 (2)
N^i—Ni—O(1^i)	88.8 (1)	O(2)—Ni—O(2^i)	84.6 (2)
N—Ni—N^i	94.7 (2)	O(2)—Ni—N^i	90.4 (2)
N^i—Ni—O(1)	90.1 (2)		

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

Data were collected using *CONTROL* software (Molecular Structure Corporation, 1988). H atoms were placed in calculated positions (C—H = 0.95 Å) and their parameters refined. The structure was refined by full-matrix least-squares techniques with anisotropic displacement parameters for all non-H atoms. All calculations were performed on a VAX3100 computer using the *TEXSAN* (Molecular Structure Corporation, 1985) program package.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, least-squares-planes data and complete geometry have been deposited with the IUCr (Reference: MU1173). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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The Adduct of Bis(*O*,*O*′-diethyl dithiophosphato)nickel(II) with 3-Aminopyridine

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Abstract

The coordination compound *trans*-bis(3-aminopyridine)-bis(*O*,*O*′-diethyl dithiophosphato-*S*,*S*′)nickel(II), [Ni{$(C_2H_5O)_2PS_2$}$_2$($C_5H_6N_2$)$_2$], displays distorted octahedral geometry around the central Ni atom. In the