Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

trans,trans,trans-Diacetonitriledibromobis(4-fluoroaniline)nickel(II)

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Received 19 May 2005
Accepted 27 May 2005
Online 10 June 2005

Key indicators

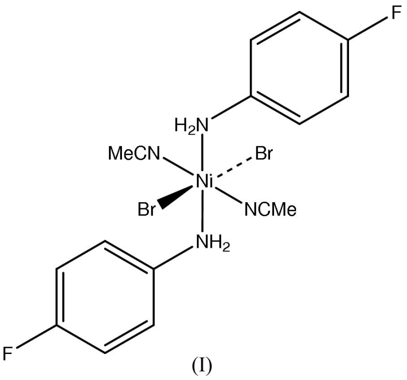
Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.003 Å
R factor = 0.027
wR factor = 0.069
Data-to-parameter ratio = 17.2

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The structure of the centrosymmetric title compound, [(4-F-$C_6H_4NH_2$)$_2$(MeCN)$_2$NiBr_2] or [NiBr_2(C_6H_6FN)$_2$-(C_2H_3N)$_2$], reveals each of the pairs of bromide, acetonitrile and 4-fluoroaniline ligands arranged *trans* to each other with a near octahedral geometry at the Ni atom.

Comment

While fluorinated anilines, $C_6F_xH_yNH_2$ (x = 1 and y = 4; x = 2 and y = 3; x = 5 and y = 0), have been extensively used as precursors to Schiff base ligands, crystallographically characterized examples of transition metal complexes containing the bound aniline itself are rare (Padmanabhan *et al.*, 1985; Visalakshi & Patel, 1994).



(I)

We report here the synthesis and crystal structure of *trans*,*trans*,*trans*-[(4-F-$C_6H_4NH_2$)$_2$(MeCN)$_2$NiBr_2], (I). The Ni atom is located on a centre of symmetry. The geometry at the Ni atom is approximately octahedral, the largest deviation from the ideal bond angles being observed for N1—Ni1—N2 [83.79 (8)°]. The bond distances at nickel are: Ni1—Br1 = 2.5634 (3) Å, Ni1—N1 = 2.0915 (18) Å and Ni1—N2 = 2.0629 (19) Å. Each Br atom is surrounded by H atoms with three intra- and four intermolecular H···Br distances in the range 2.58–3.25 Å. The structure of (I) resembles the *trans* disposition of ligand pairs found in *trans*,*trans*,*trans*-[(H_2O)$_2$(MeCN)$_2$NiCl_2] (Piggot *et al.*, 2004).

Experimental

Under a nitrogen atmosphere, 4-fluoroaniline (0.02 g, 0.18 mmol) was added to a solution of (DME)NiBr_2 (DME = 1,2-dimethoxyethane) (0.05 g, 0.16 mmol) in dichloromethane (20 ml) and the reaction mixture stirred for 12 h at room temperature. The volatiles were removed under reduced pressure and the residue dried overnight. Extraction of the residue into hot acetonitrile and prolonged standing of the solution at room temperature gave pale-green crystals of the title compound suitable for single-crystal X-ray diffraction analysis (0.02 g, 23% yield).

Crystal data

$[NiBr_2(C_6H_6FN)_2(C_2H_3N)_2]$
$M_r = 522.87$
Monoclinic, $P2_1/c$
$a = 11.4533$ (14) Å
$b = 12.9875$ (15) Å
$c = 6.2590$ (7) Å
$\beta = 99.191$ (2)°
$V = 919.07$ (19) Å^3
$Z = 2$

$D_x = 1.889$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4651 reflections
$\theta = 2.4$–28.8°
$\mu = 5.43$ mm^{-1}
$T = 150$ (2) K
Plate, pale green
$0.32 \times 0.19 \times 0.09$ mm

Data collection

Bruker APEX CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.315$, $T_{max} = 0.613$
7593 measured reflections

1995 independent reflections
1823 reflections with $I > 2\sigma(I)$
$R_{int} = 0.052$
$\theta_{max} = 27.0$°
$h = -14 \rightarrow 14$
$k = -16 \rightarrow 16$
$l = -7 \rightarrow 7$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.027$
$wR(F^2) = 0.069$
$S = 1.02$
1995 reflections
116 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0421P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.70$ e Å^{-3}
$\Delta\rho_{min} = -0.57$ e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N1—H1$A\cdots$Br1^{i}	0.92	2.71	3.5498 (19)	152
N1—H1$B\cdots$Br1^{ii}	0.92	2.58	3.4789 (19)	167

Symmetry codes: (i) $-x, y-\frac{1}{2}, -z+\frac{1}{2}$; (ii) $-x, -y+1, -z$.

All H atoms were included in calculated positions and treated as riding, with C—H = 0.95–0.98 and N—H = 0.92 Å. For methyl H atoms, U_{iso}(H) values were set at $1.5U_{eq}$ of the C atom and at $1.2U_{eq}$ for all other H atoms.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXTL*.

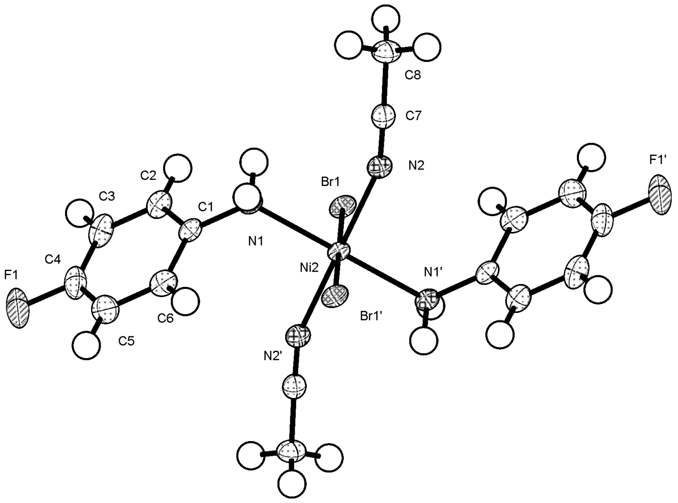


Figure 1
The molecular structure of (I), showing the atom numbering scheme and 50% displacement ellipsoids. The molecule is located on a centre of symmetry [primed atoms are generated by $(-x, 1-y, 1-z)$].

The authors thank the University of Leicester for financial assistance.

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Acta Cryst. (2005). E**61**, m1256–m1257 Fawcett *et al.* • $[NiBr_2(C_6H_6FN)_2(C_2H_3N)_2]$ **m1257**