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Key indicators

Single-crystal X-ray study
 $T = 150$ K
Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
 R factor = 0.027
 wR factor = 0.069
Data-to-parameter ratio = 17.2For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.*trans,trans,trans*-Diacetonitriledibromo-
bis(4-fluoroaniline)nickel(II)

The structure of the centrosymmetric title compound, $[(4\text{-F-C}_6\text{H}_4\text{NH}_2)_2(\text{MeCN})_2\text{NiBr}_2]$ or $[\text{NiBr}_2(\text{C}_6\text{H}_4\text{FN})_2(\text{C}_2\text{H}_3\text{N})_2]$, 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.

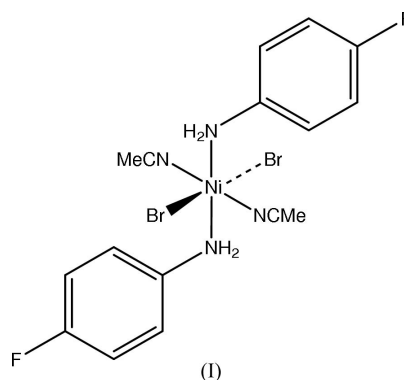
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Comment

While fluorinated anilines, $\text{C}_6\text{F}_x\text{H}_y\text{NH}_2$ ($x = 1$ 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).



We report here the synthesis and crystal structure of *trans,trans,trans*- $[(4\text{-F-C}_6\text{H}_4\text{NH}_2)_2(\text{MeCN})_2\text{NiBr}_2]$, (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 $\text{N1}-\text{Ni1}-\text{N2}$ [$83.79(8)^\circ$]. The bond distances at nickel are: $\text{Ni1}-\text{Br1} = 2.5634(3)$ Å, $\text{Ni1}-\text{N1} = 2.0915(18)$ Å and $\text{Ni1}-\text{N2} = 2.0629(19)$ Å. Each Br atom is surrounded by H atoms with three intra- and four intermolecular $\text{H}\cdots\text{Br}$ 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*- $[(\text{H}_2\text{O})_2(\text{MeCN})_2\text{NiCl}_2]$ (Piggot *et al.*, 2004).

Experimental

Under a nitrogen atmosphere, 4-fluoroaniline (0.02 g, 0.18 mmol) was added to a solution of $(\text{DME})\text{NiBr}_2$ (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

[NiBr₂(C₆H₆FN)₂(C₂H₃N)₂]
M_r = 522.87
 Monoclinic, *P*2₁/*c*
a = 11.4533 (14) Å
b = 12.9875 (15) Å
c = 6.2590 (7) Å
 β = 99.191 (2)°
V = 919.07 (19) Å³
Z = 2

D_x = 1.889 Mg m⁻³
 Mo *K*α radiation
 Cell parameters from 4651 reflections
 θ = 2.4–28.8°
 μ = 5.43 mm⁻¹
T = 150 (2) K
 Plate, pale green
 0.32 × 0.19 × 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* > 2σ(*I*)
R_{int} = 0.052
 θ_{\max} = 27.0°
h = -14 → 14
k = -16 → 16
l = -7 → 7

Refinement

Refinement on *F*²
R[*F*² > 2σ(*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 $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.70 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.57 \text{ e \AA}^{-3}$

Table 1

Hydrogen-bond geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N1—H1A...Br1 ⁱ	0.92	2.71	3.5498 (19)	152
N1—H1B...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 1.5*U_{eq}* of the C atom and at 1.2*U_{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

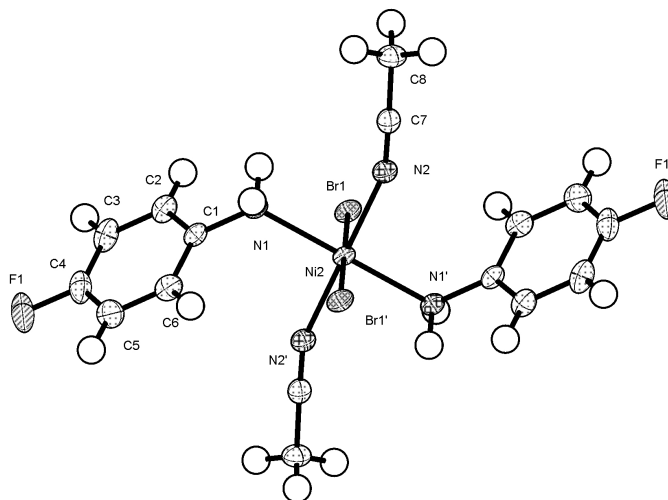


Figure 1

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

structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXTL*.

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References

- Bruker (1997). *SMART*. Version 5.622. Bruker AXS Inc., Madison, Wisconsin, USA.
 Bruker (1999). *SAINT*. Version 6.02. Bruker AXS Inc., Madison, Wisconsin, USA.
 Bruker (2000). *SHELXTL*. Version 6.10. Bruker AXS Inc., Madison, Wisconsin, USA.
 Padmanabhan, V. M., Patel, R. P. & Ranganathan, T. N. (1985). *Acta Cryst. C* **41**, 1305–1309.
 Piggot, P. M. T., Hall, L. A., White, A. J. P., Williams, D. J. & Thompson, L. K. (2004). *Inorg. Chem.* **43**, 1167–1174.
 Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
 Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
 Visalakshi, R. & Patel, R. P. (1994). *Synth. React. Inorg. Met.-Org. Chem.* **24**, 1043–1053.