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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.

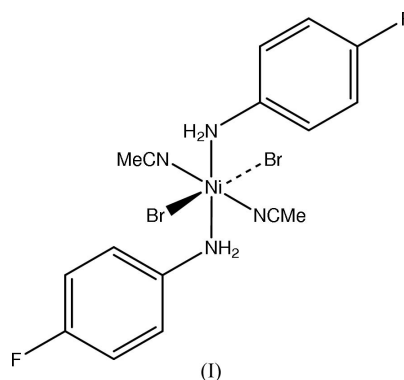
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## Comment

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

[NiBr<sub>2</sub>(C<sub>6</sub>H<sub>6</sub>FN)<sub>2</sub>(C<sub>2</sub>H<sub>3</sub>N)<sub>2</sub>]  
*M<sub>r</sub>* = 522.87  
 Monoclinic, *P*2<sub>1</sub>/*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<sup>3</sup>  
*Z* = 2

*D<sub>x</sub>* = 1.889 Mg m<sup>-3</sup>  
 Mo *K*α radiation  
 Cell parameters from 4651 reflections  
 $\theta$  = 2.4–28.8°  
 $\mu$  = 5.43 mm<sup>-1</sup>  
*T* = 150 (2) K  
 Plate, pale green  
 0.32 × 0.19 × 0.09 mm

#### Data collection

Bruker APEX CCD area-detector diffractometer  
 $\varphi$  and  $\omega$  scans  
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)  
*T<sub>min</sub>* = 0.315, *T<sub>max</sub>* = 0.613  
 7593 measured reflections

1995 independent reflections  
 1823 reflections with *I* > 2σ(*I*)  
*R<sub>int</sub>* = 0.052  
 $\theta_{\max}$  = 27.0°  
*h* = -14 → 14  
*k* = -16 → 16  
*l* = -7 → 7

#### Refinement

Refinement on *F*<sup>2</sup>  
*R*[*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.027  
*wR*(*F*<sup>2</sup>) = 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**

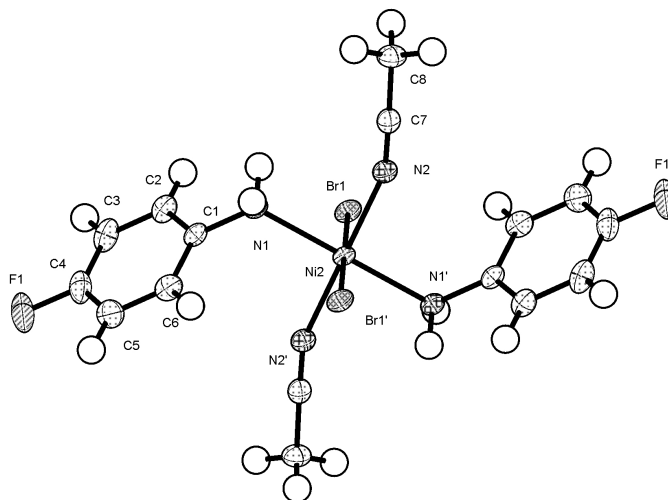
Hydrogen-bond geometry (Å, °).

<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 <sup>i</sup>	0.92	2.71	3.5498 (19)	152
N1—H1B...Br1 <sup>ii</sup>	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*<sub>iso</sub>(H) values were set at 1.5*U*<sub>eq</sub> of the C atom and at 1.2*U*<sub>eq</sub> 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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## supporting information

*Acta Cryst.* (2005). E61, m1256–m1257 [https://doi.org/10.1107/S1600536805016995]

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

**John Fawcett, Fabrizio Sicilia and Gregory A Solan**

***trans-trans-trans*-Dibromobis(4-fluoroaniline)bis(acetonitrile)nickel(II)**

*Crystal data*

[NiBr<sub>2</sub>(C<sub>6</sub>H<sub>4</sub>FN)<sub>2</sub>(C<sub>2</sub>H<sub>3</sub>N)<sub>2</sub>]

*M<sub>r</sub>* = 522.87

Monoclinic, *P*2<sub>1</sub>/*c*

Hall symbol: -*P* 2ybc

*a* = 11.4533 (14) Å

*b* = 12.9875 (15) Å

*c* = 6.2590 (7) Å

$\beta$  = 99.191 (2)°

*V* = 919.07 (19) Å<sup>3</sup>

*Z* = 2

*F*(000) = 516

*D<sub>x</sub>* = 1.889 Mg m<sup>-3</sup>

Mo *K*α radiation,  $\lambda$  = 0.71073 Å

Cell parameters from 4651 reflections

$\theta$  = 2.4–28.8°

$\mu$  = 5.43 mm<sup>-1</sup>

*T* = 150 K

Plate, pale green

0.32 × 0.19 × 0.09 mm

*Data collection*

Bruker APEX CCD area-detector  
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

$\varphi$  and  $\omega$  scans

Absorption correction: multi-scan  
(SADABS; Sheldrick, 1996)

*T<sub>min</sub>* = 0.315, *T<sub>max</sub>* = 0.613

7593 measured reflections

1995 independent reflections

1823 reflections with *I* > 2σ(*I*)

*R<sub>int</sub>* = 0.052

$\theta_{\max}$  = 27.0°,  $\theta_{\min}$  = 1.8°

*h* = -14→14

*k* = -16→16

*l* = -7→7

*Refinement*

Refinement on *F*<sup>2</sup>

Least-squares matrix: full

*R*[*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.027

*wR*(*F*<sup>2</sup>) = 0.069

*S* = 1.03

1995 reflections

116 parameters

0 restraints

Primary atom site location: structure-invariant  
direct methods

Secondary atom site location: difference Fourier  
map

Hydrogen site location: inferred from  
neighbouring sites

H-atom parameters constrained

*w* = 1/[σ<sup>2</sup>(*F<sub>o</sub>*<sup>2</sup>) + (0.0421*P*)<sup>2</sup>]

where *P* = (*F<sub>o</sub>*<sup>2</sup> + 2*F<sub>c</sub>*<sup>2</sup>)/3

(Δ/σ)<sub>max</sub> < 0.001

Δρ<sub>max</sub> = 0.70 e Å<sup>-3</sup>

Δρ<sub>min</sub> = -0.57 e Å<sup>-3</sup>

*Special details*

**Experimental.** absorption correction based on 5283 reflections (*SADABS*);  $R_{\text{int}}$  0.126 before correction and 0.031 after correction.

**Geometry.** All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

**Refinement.** Refinement of  $F^2$  against ALL reflections. The weighted  $R$ -factor  $wR$  and goodness of fit  $S$  are based on  $F^2$ , conventional  $R$ -factors  $R$  are based on  $F$ , with  $F$  set to zero for negative  $F^2$ . The threshold expression of  $F^2 > \sigma(F^2)$  is used only for calculating  $R$ -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement.  $R$ -factors based on  $F^2$  are statistically about twice as large as those based on  $F$ , and  $R$ -factors based on ALL data will be even larger.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	0.02345 (2)	0.632904 (16)	0.20705 (3)	0.02041 (10)
Ni1	0.0000	0.5000	0.5000	0.01528 (12)
N2	-0.15001 (18)	0.44011 (14)	0.3156 (3)	0.0198 (4)
C1	0.2126 (2)	0.38636 (17)	0.3677 (4)	0.0196 (5)
C7	-0.2080 (2)	0.38921 (18)	0.1966 (4)	0.0207 (5)
F1	0.57305 (15)	0.37955 (12)	0.4412 (3)	0.0459 (5)
N1	0.08695 (17)	0.38864 (14)	0.3421 (3)	0.0184 (4)
H1A	0.0627	0.3253	0.3842	0.022*
H1B	0.0593	0.3947	0.1964	0.022*
C2	0.2695 (2)	0.42016 (18)	0.2041 (4)	0.0246 (5)
H2	0.2246	0.4446	0.0730	0.030*
C6	0.2778 (2)	0.35105 (18)	0.5588 (4)	0.0258 (6)
H6	0.2387	0.3283	0.6732	0.031*
C8	-0.2798 (2)	0.3224 (2)	0.0440 (4)	0.0267 (5)
H8A	-0.2854	0.3512	-0.1020	0.040*
H8B	-0.3592	0.3169	0.0827	0.040*
H8C	-0.2435	0.2540	0.0479	0.040*
C3	0.3910 (2)	0.4190 (2)	0.2280 (4)	0.0309 (6)
H3	0.4307	0.4428	0.1153	0.037*
C4	0.4534 (2)	0.38288 (19)	0.4180 (5)	0.0313 (6)
C5	0.3993 (3)	0.34882 (19)	0.5833 (4)	0.0312 (6)
H5	0.4447	0.3239	0.7134	0.037*

*Atomic displacement parameters ( $\text{\AA}^2$ )*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Br1	0.02936 (17)	0.01745 (14)	0.01536 (14)	-0.00154 (8)	0.00643 (10)	0.00117 (8)
Ni1	0.0186 (2)	0.0147 (2)	0.0128 (2)	-0.00185 (15)	0.00356 (15)	-0.00117 (14)
N2	0.0219 (10)	0.0195 (10)	0.0184 (9)	-0.0021 (8)	0.0045 (8)	-0.0004 (8)
C1	0.0247 (13)	0.0146 (10)	0.0204 (11)	0.0007 (9)	0.0061 (9)	-0.0030 (9)
C7	0.0218 (12)	0.0229 (11)	0.0183 (11)	0.0014 (10)	0.0061 (10)	0.0039 (10)
F1	0.0261 (9)	0.0440 (10)	0.0688 (13)	0.0103 (7)	0.0109 (9)	-0.0030 (8)
N1	0.0226 (10)	0.0172 (9)	0.0160 (9)	-0.0012 (8)	0.0051 (8)	-0.0006 (7)
C2	0.0313 (14)	0.0200 (12)	0.0239 (12)	0.0035 (10)	0.0088 (10)	0.0028 (10)

C6	0.0340 (15)	0.0209 (12)	0.0230 (12)	0.0019 (10)	0.0057 (11)	0.0018 (10)
C8	0.0269 (14)	0.0285 (13)	0.0241 (12)	-0.0079 (11)	0.0022 (10)	-0.0053 (11)
C3	0.0332 (15)	0.0264 (13)	0.0374 (15)	0.0024 (11)	0.0184 (12)	0.0011 (11)
C4	0.0239 (14)	0.0234 (13)	0.0474 (17)	0.0040 (10)	0.0086 (12)	-0.0043 (11)
C5	0.0372 (16)	0.0240 (13)	0.0305 (14)	0.0102 (11)	-0.0005 (12)	0.0025 (10)

*Geometric parameters (Å, °)*

Br1—Ni1	2.5634 (3)	N1—H1B	0.9200
Ni1—N2	2.0629 (19)	C2—C3	1.376 (4)
Ni1—N2 <sup>i</sup>	2.063 (2)	C2—H2	0.9500
Ni1—N1 <sup>i</sup>	2.0915 (18)	C6—C5	1.376 (4)
Ni1—N1	2.0915 (18)	C6—H6	0.9500
Ni1—Br1 <sup>i</sup>	2.5634 (3)	C8—H8A	0.9800
N2—C7	1.129 (3)	C8—H8B	0.9800
C1—C2	1.371 (3)	C8—H8C	0.9800
C1—C6	1.383 (3)	C3—C4	1.369 (4)
C1—N1	1.422 (3)	C3—H3	0.9500
C7—C8	1.445 (3)	C4—C5	1.362 (4)
F1—C4	1.356 (3)	C5—H5	0.9500
N1—H1A	0.9200		
N2—Ni1—N2 <sup>i</sup>	180.00 (10)	Ni1—N1—H1B	107.1
N2—Ni1—N1 <sup>i</sup>	96.21 (8)	H1A—N1—H1B	106.8
N2 <sup>i</sup> —Ni1—N1 <sup>i</sup>	83.79 (8)	C1—C2—C3	120.6 (2)
N2—Ni1—N1	83.79 (8)	C1—C2—H2	119.7
N2 <sup>i</sup> —Ni1—N1	96.21 (8)	C3—C2—H2	119.7
N1 <sup>i</sup> —Ni1—N1	180.00 (9)	C5—C6—C1	120.0 (3)
N2—Ni1—Br1 <sup>i</sup>	88.46 (5)	C5—C6—H6	120.0
N2 <sup>i</sup> —Ni1—Br1 <sup>i</sup>	91.54 (5)	C1—C6—H6	120.0
N1 <sup>i</sup> —Ni1—Br1 <sup>i</sup>	90.88 (5)	C7—C8—H8A	109.5
N1—Ni1—Br1 <sup>i</sup>	89.12 (5)	C7—C8—H8B	109.5
N2—Ni1—Br1	91.54 (5)	H8A—C8—H8B	109.5
N2 <sup>i</sup> —Ni1—Br1	88.46 (5)	C7—C8—H8C	109.5
N1 <sup>i</sup> —Ni1—Br1	89.12 (5)	H8A—C8—H8C	109.5
N1—Ni1—Br1	90.88 (5)	H8B—C8—H8C	109.5
Br1 <sup>i</sup> —Ni1—Br1	180.0	C4—C3—C2	118.5 (2)
C7—N2—Ni1	160.0 (2)	C4—C3—H3	120.8
C2—C1—C6	119.8 (2)	C2—C3—H3	120.8
C2—C1—N1	120.1 (2)	F1—C4—C5	118.9 (3)
C6—C1—N1	120.1 (2)	F1—C4—C3	118.8 (3)
N2—C7—C8	178.6 (3)	C5—C4—C3	122.2 (3)
C1—N1—Ni1	120.71 (14)	C4—C5—C6	118.9 (2)
C1—N1—H1A	107.1	C4—C5—H5	120.6
Ni1—N1—H1A	107.1	C6—C5—H5	120.6
C1—N1—H1B	107.1		
N1—Ni1—N2—C7	1.8 (5)	N1—C1—C2—C3	179.5 (2)

Br1 <sup>i</sup> —Ni1—N2—C7	−87.5 (5)	C2—C1—C6—C5	−0.7 (4)
Br1—Ni1—N2—C7	92.5 (5)	N1—C1—C6—C5	180.0 (2)
C2—C1—N1—Ni1	−105.1 (2)	C1—C2—C3—C4	0.4 (4)
C6—C1—N1—Ni1	74.2 (2)	C2—C3—C4—F1	178.7 (2)
N2—Ni1—N1—C1	172.55 (17)	C2—C3—C4—C5	−0.4 (4)
N2 <sup>i</sup> —Ni1—N1—C1	−7.45 (17)	F1—C4—C5—C6	−179.2 (2)
Br1 <sup>i</sup> —Ni1—N1—C1	−98.90 (16)	C3—C4—C5—C6	−0.2 (4)
Br1—Ni1—N1—C1	81.10 (16)	C1—C6—C5—C4	0.7 (4)
C6—C1—C2—C3	0.2 (4)		

Symmetry code: (i)  $-x, -y+1, -z+1$ .

*Hydrogen-bond geometry (Å, °)*

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
N1—H1A $\cdots$ Br1 <sup>ii</sup>	0.92	2.71	3.5498 (19)	152
N1—H1B $\cdots$ Br1 <sup>iii</sup>	0.92	2.58	3.4789 (19)	167

Symmetry codes: (ii)  $-x, y-1/2, -z+1/2$ ; (iii)  $-x, -y+1, -z$ .