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Bis(5-bromo-*N*-cyclohexylsalicylideneaminato)-nickel(II): an orthorhombic polymorph

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.007 Å
R factor = 0.049
wR factor = 0.138
Data-to-parameter ratio = 19.7

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

The title compound, $[Ni(C_{13}H_{15}BrNO)_2]$, crystallizes in the orthorhombic space group *Pbca*, rather than the previously reported monoclinic space group *C*2/*c* [Sun & Ng (2005). *Acta Cryst.* E**61**, m323–m324]. The Ni^{II} atom is coordinated by two N atoms and two O atoms from two Schiff bases, forming a distorted tetrahedral geometry.

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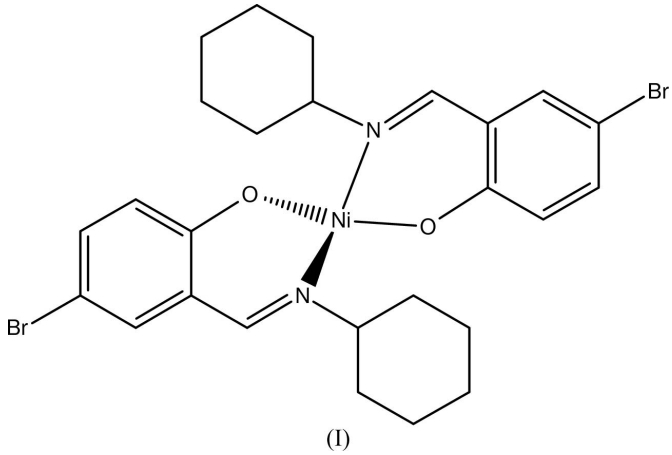
Comment

The crystal structure of the title compound, (I), was first reported in the monoclinic space group *C*2/*c* (Sun & Ng, 2005).



(I)

Here we report the structure of (I) in the orthorhombic space group *Pbca*. The metal atom in (I) shows distorted tetrahedral coordination rather than the previously reported distorted square-planar geometry, which can be seen from the

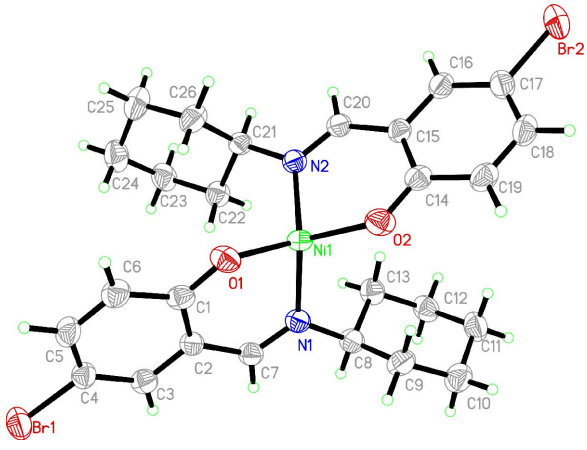


Figure 1
The structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

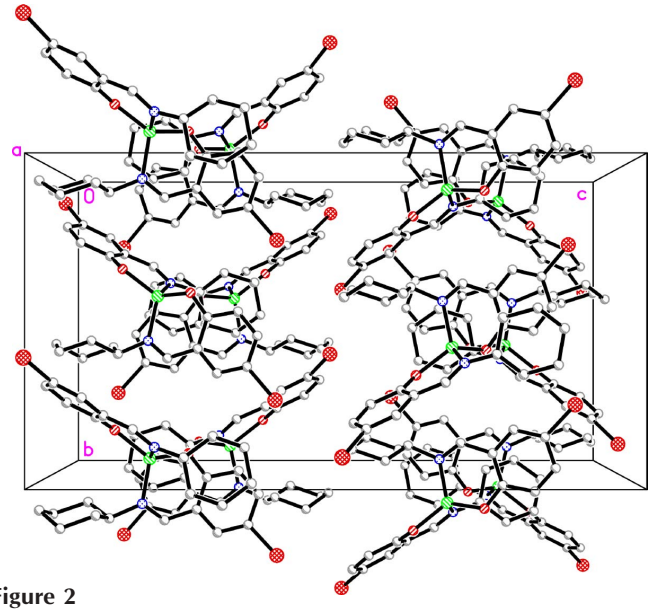


Figure 2
The crystal packing of (I), viewed along the *a* axis. H atoms have been omitted for clarity.

bond angles. The angles subtended at the Ni atom are in the range 93.83 (13)-122.50 (14)° (Table 1). The average Ni—O [1.918 (3) Å] and Ni—N [2.020 (3) Å] bond lengths are a little longer than those in the monoclinic polymorph; this is probably caused by the steric effects of the tetrahedral geometry. There are no short contacts in the crystal structure (Fig. 2).

Experimental

Cyclohexylamine (0.1 mmol, 10.1 mg) and 5-bromosalicylaldehyde (0.1 mmol, 20.2 mg) were dissolved in methanol (10 ml) to give a clear yellow solution after several minutes of stirring. A methanol solution (10 ml) of nickel acetate tetrahydrate (0.1 mmol, 25.4 mg) was then added. Green block-shaped crystals separated from the resulting solution after 11 d.

Crystal data

$[Ni(C_{13}H_{15}BrNO)_2]$
M_r = 621.05
Orthorhombic, *Pbca*
a = 14.979 (3) Å
b = 13.609 (3) Å
c = 25.164 (5) Å
V = 5129.7 (18) Å^3
Z = 8
D_x = 1.608 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4960 reflections
θ = 2.6–20.6°
μ = 3.90 mm^{-1}
T = 298 (2) K
Block, green
0.28 × 0.19 × 0.17 mm

Data collection

Bruker SMART APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.378, T_{max} = 0.515
42110 measured reflections
5884 independent reflections
3526 reflections with $I > 2\sigma(I)$
R_{int} = 0.069
θ_{max} = 27.5°
$h = -19 \rightarrow 19$
$k = -17 \rightarrow 17$
$l = -32 \rightarrow 32$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.138
S = 1.02
5884 reflections
298 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0595P)^2 + 3.6844P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.79 e Å^{-3}
$\Delta\rho_{min}$ = −0.48 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ni1—O1	1.916 (3)	Ni1—N2	2.017 (4)
Ni1—O2	1.919 (3)	Ni1—N1	2.023 (3)
O1—Ni1—O2	119.23 (14)	O1—Ni1—N1	93.83 (13)
O1—Ni1—N2	113.75 (14)	O2—Ni1—N1	113.91 (15)
O2—Ni1—N2	95.61 (14)	N2—Ni1—N1	122.50 (14)

All H atoms were placed in idealized positions and constrained to ride on their parent atoms with C—H distances of 0.93–0.97 Å, and with U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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