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Bis-BBN (9-borabicyclo[3.3.1]nonane) adduct of bis(diethylglyoximato)nickel(II)

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.002 Å
R factor = 0.025
wR factor = 0.031
Data-to-parameter ratio = 16.5

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

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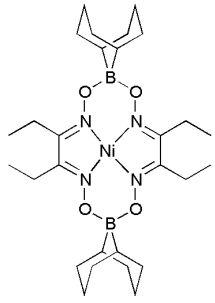
The bis-BBN adduct of bis(diethylglyoximato)nickel(II), [2,9-bis(cyclooctane-1,5-diyl)-4,6,11,13-tetraethyl-1,3,8,10-tetraoxa-4,7,11,14-tetraaza-2,9-diboracyclotetradecane-4,6,11,13-tetraene-κ^4N]nickel(II), [Ni($C_{28}H_{48}B_2N_4O_4$)], crystallizes as a monomer, with no short Ni···Ni contacts. The asymmetric unit contains only half a molecule and the Ni atom lies on an inversion centre.

Comment

In the preceding paper, we reported the structure of a mono-9-borabicyclo[3.3.1]nonane (BBN) adduct of bis-(diethylglyoxato)nickel(II) (Krivokapic *et al.*, 2003). We report here the structure of the bis-adduct, (I). This compound was prepared in good yield by treating bis(diethylglyoximato)nickel(II) with methoxy-9-BBN in toluene at reflux for 2 d.



(I)

The geometry of (I) (Fig. 1 and Table 1) is similar to those of previously reported complexes of this type (Chakravorty, 1974; Krivokapić *et al.*, 2003). Compound (I) adopts a C_i conformation, with approximate C_{2h} symmetry, with the BBN units shifted towards opposite faces of the macrocycle; this conformation evidently prevents stacking. The geometry of the 9-BBN unit leads to a short H91···H132 contact (1.91 Å).

Experimental

Methoxy-9-BBN (1.50 ml, 1.0 *M* in hexanes; 1.50 mmol) was added to a solution of bis(diethylglyoximato)nickel(II) (0.10 g, 0.29 mmol) in toluene (5 ml). After heating to reflux for 2 d, the product was chromatographed (SiO_2, toluene) to yield (I) (0.114 g, 67%) as orange crystals. M.p. 532–534 K; δ_H (400 MHz, $CDCl_3$): 2.61 (8H, *q*), 1.88–1.80 (4H, *m*), 1.75–1.60 (20H, *m*), 1.50–1.43 (4H, *m*), 1.20, (6H, *t*); δ_C (100 MHz, $CDCl_3$) 160.4, 31.6, 24.8, 19.7, 10.2; *m*/*z* (APCI$^+$) 585.56 [*M*+H]$^+$. Crystals of (I) were grown from acetone by evaporation.

Crystal data

$[Ni(C_{28}H_{48}B_2N_4O_4)]$
$M_r = 585.05$
Monoclinic, $P2_1/c$
$a = 7.4047\ (1)$ Å
$b = 19.2467\ (2)$ Å
$c = 10.5508\ (2)$ Å
$\beta = 94.7398\ (5)^\circ$
$V = 1498.52\ (4)$ Å^3
$Z = 2$
$D_x = 1.297$ Mg m^{-3}
Mo Kα radiation
Cell parameters from 3483 reflections
$\theta = 5$–27°
$\mu = 0.69$ mm^{-1}
$T = 150$ K
Block, orange
0.20 × 0.20 × 0.20 mm

Data collection

Enraf–Nonius KappaCCD diffractometer
ω scans
Absorption correction: multi-scan (*DENZO/SCALEPACK*; Otwinowski & Minor, 1997)
$T_{min} = 0.87$, $T_{max} = 0.87$
3497 measured reflections
3386 independent reflections
2934 reflections with $I > 3\sigma(I)$
$R_{int} = 0.03$
$\theta_{max} = 27.4^\circ$
$h = 0 \rightarrow 9$
$k = 0 \rightarrow 24$
$l = -13 \rightarrow 13$

Refinement

Refinement on F
$R = 0.025$
$wR = 0.031$
$S = 1.04$
2934 reflections
178 parameters
H-atom parameters not refined
Weighting scheme: Prince-modified Chebychev polynomial with 3 parameters (Watkin, 1994), $W = [w]\{1 - [\Delta F/6\sigma(F)]^2\}^2$, $w = 2.03$, 0.0469 and 1.40
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.31$ e Å^{-3}
$\Delta\rho_{min} = -0.28$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ni1—N2	1.8576 (9)	O2—N2	1.3508 (12)
Ni1—N2^{i}	1.8576 (9)	O2—B1^{i}	1.5367 (14)
Ni1—N1	1.864 (1)	N1—C1	1.2984 (15)
Ni1—N1^{i}	1.864 (1)	N2—C2	1.2956 (15)
O1—N1	1.3540 (12)	C1—C2	1.4740 (15)
O1—B1	1.5357 (14)		
Ni1—N1—O1	124.50 (7)	N1—C1—C2	112.1 (1)
Ni1—N1—C1	116.26 (8)	N2—Ni1—N2^{i}	179.994
Ni1—N2—O2	124.72 (7)	N2—Ni1—N1	82.71 (4)
Ni1—N2—C2	116.41 (8)	N2^{i}—Ni1—N1	97.29 (4)
O1—N1—C1	118.99 (9)	N2—Ni1—N1^{i}	97.29 (4)
O2—N2—C2	118.76 (9)	N2^{i}—Ni1—N1^{i}	82.71 (4)
O2^{i}—B1—O1	107.68 (9)	N2—O2—B1^{i}	113.43 (8)
N1—Ni1—N1^{i}	179.994	N2—C2—C1	112.46 (9)
N1—O1—B1	113.41 (8)		

Symmetry code: (i) $2 - x, -y, 2 - z$.

H atoms were located in a difference Fourier map and their parameters were not refined.

Data collection: *COLLECT* (Nonius, 1997–2001); cell refinement: *DENZO/SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO/SCALEPACK*; program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994); program(s) used to refine structure: *CRYSTALS* (Watkin *et al.*, 2001); molecular graphics: *CAMERON* (Watkin *et al.*, 1996); software used to prepare material for publication: *CRYSTALS*.

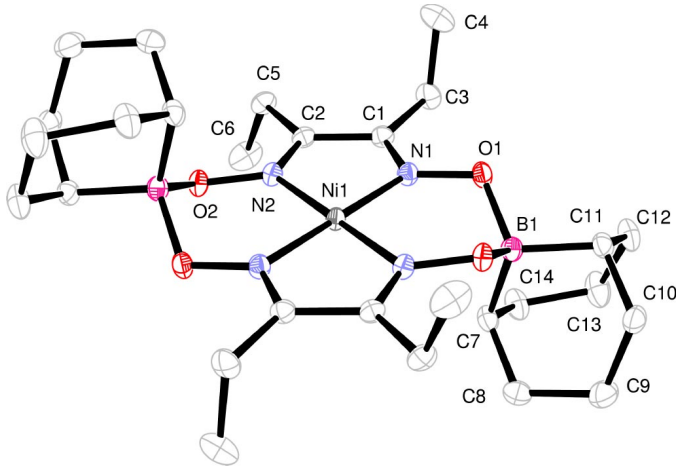


Figure 1
Plot showing the atomic numbering scheme for (I). Displacement ellipsoids are drawn at the 50% probability level for non-H atoms. Only the contents of the asymmetric unit are labelled. The Ni atom lies on an inversion centre.

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