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Mono-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) = 0.003 Å
R factor = 0.030
wR factor = 0.038
Data-to-parameter ratio = 13.7

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

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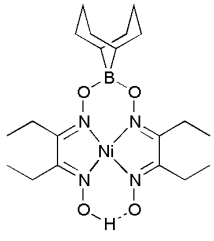
The mono-BBN adduct of bis(diethylglyoximato)nickel(II), [7-(cyclooctane-1,5-diyl)-2,3,11,12-tetraethyl-6,8-dioxa-5,9-diaza-7-boratridecane-1,13-dicarbaldehyde dioximato-κ^4N]-nickel(II), [Ni($C_{20}H_{35}BN_4O_4$)], crystallizes with two molecules in the asymmetric unit and with a short Ni···Ni distance [3.5042 (3) Å].

Comment

Square-planar metal bis-glyoxime complexes have long attracted interest because of their ability to bind axial ligands (Chakavorty, 1974; Stephens & Vagg, 1980). We are interested in exploring the scope of these units for supramolecular self-assembly. These complexes can often be stabilized and solubilized by boronylation (Schrauzer, 1962; Bakac *et al.*, 1986). We present here the synthesis and crystal structure of a bis-(diethylglyoximato)nickel(II) complex, with one 9-borabicyclo[3.3.1]nonane (BBN) unit chelated to the O atoms, (I). To the best of our knowledge, BBN adducts of nickel glyoxime complexes have not been reported previously, although some related iron(II) complexes are known (Harshani de Silva *et al.*, 1995). Compound (I) was prepared in good yield by treating bis(diethylglyoximato)nickel(II) with methoxy-9-BBN in toluene.



(I)

The geometry of (I) is similar to those of previously reported complexes of this type (Chakravorty, 1974) (Fig. 1 and Table 1). The asymmetric unit contains two molecules, and there is a short Ni···Ni distance of 3.5042 (3) Å (Fig. 1). The geometry of the 9BBN unit leads to short contacts between H151···H192 (1.91 Å) and H352···H391 (1.91 Å).

Experimental

Methoxy-9-BBN (0.61 ml, 1.0 *M* in hexanes; 0.61 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 h, the product was chromatographed (SiO_2, 5:1 hexane/ethyl acetate) to yield (I) (0.105 g, 78%) as red crystals. M.p.: 464–466 K; δ_H (400 MHz, $CDCl_3$):

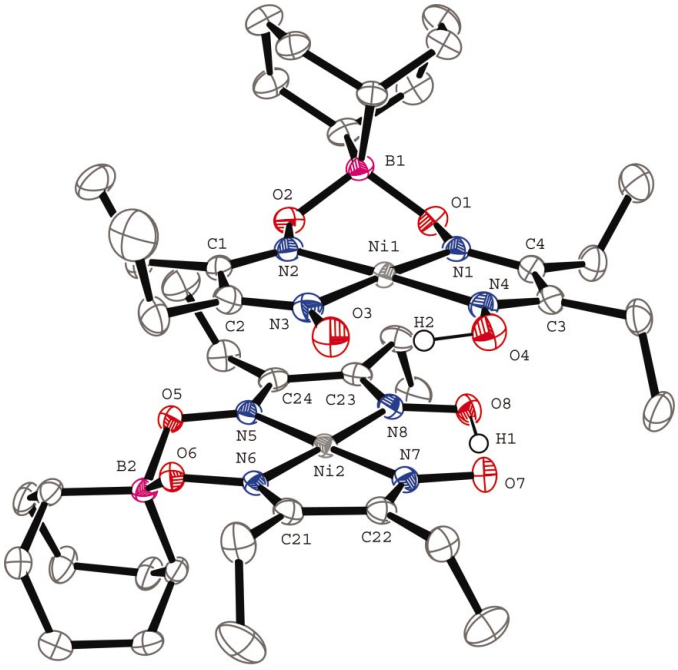


Figure 1
The asymmetric unit of (I). Displacement ellipsoids are drawn at the 50% probability level for non-H atoms.

17.77 (1H, *s*), 2.64 (4H, *q*), 2.58 (4H, *q*), 1.84–1.76 (2H, *m*), 1.71–1.62 (10H, *m*), 1.78–1.43 (2H, *m*), 1.23 (6H, *t*), 1.18 (6H, *t*); δ_C (100 MHz, $CDCl_3$): 161.7, 155.7, 31.6, 24.8, 19.3, 19.0, 10.3, 9.9; *m*/*z* (APCI$^+$) 465.18 $[M+H]^+$. Crystals of (I) were grown from acetone by evaporation.

Crystal data

$[Ni(C_{20}H_{35}BN_4O_4)]$
M_r = 465.04
Triclinic, $P\overline{1}$
a = 11.5210 (2) Å
b = 14.8794 (2) Å
c = 14.9455 (3) Å
α = 79.4462 (6)°
β = 70.0605 (7)°
γ = 69.4812 (11)°
V = 2249.76 (7) Å^3
Z = 4
D_x = 1.373 Mg m^{-3}
Mo Kα radiation
Cell parameters from 8108 reflections
θ = 4–27°
μ = 0.90 mm^{-1}
T = 150 K
Block, red
0.30 × 0.20 × 0.19 mm

Data collection

Enraf–Nonius KappaCCD diffractometer
ω scans
Absorption correction: multi-scan (*DENZO/SCALEPACK*; Otwinowski & Minor, 1997)
T_{min} = 0.84, T_{max} = 0.84
16 886 measured reflections
9767 independent reflections
7437 reflections with $I > 3\sigma(I)$
R_{int} = 0.01
θ_{max} = 27.4°
$h = -14 \rightarrow 14$
$k = -19 \rightarrow 19$
$l = -19 \rightarrow 19$

Refinement

Refinement on F
R = 0.030
wR = 0.038
S = 1.04
7437 reflections
541 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 = 0.901, 0.532 and 0.614
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ni1—N1	1.8651 (15)	O6—N6	1.3601 (18)
Ni1—N2	1.8612 (14)	O6—B2	1.533 (2)
Ni1—N3	1.8612 (15)	O7—N7	1.3318 (19)
Ni1—N4	1.8598 (15)	O8—N8	1.3405 (19)
Ni2—N5	1.8626 (14)	N1—C4	1.297 (2)
Ni2—N6	1.8570 (14)	N2—C1	1.295 (2)
Ni2—N7	1.8630 (14)	N3—C2	1.300 (2)
Ni2—N8	1.8666 (15)	N4—C3	1.291 (2)
O1—N1	1.3630 (18)	N5—C24	1.291 (2)
O1—B1	1.539 (2)	N6—C21	1.296 (2)
O2—N2	1.3571 (19)	N7—C22	1.305 (2)
O2—B1	1.532 (2)	N8—C23	1.299 (2)
O3—N3	1.338 (2)	C1—C2	1.473 (3)
O4—N4	1.3468 (19)	C3—C4	1.482 (3)
O5—N5	1.3539 (19)	C21—C22	1.473 (2)
O5—B2	1.530 (2)	C24—C23	1.469 (3)
Ni1—N1—O1	124.48 (11)	N1—Ni1—N3	177.37 (6)
Ni1—N1—C4	116.06 (12)	N1—Ni1—N4	82.60 (6)
Ni1—N2—O2	123.71 (11)	N1—O1—B1	111.77 (12)
Ni1—N2—C1	116.45 (13)	N1—C4—C3	112.00 (16)
Ni1—N3—O3	123.62 (12)	N2—Ni1—N1	97.62 (6)
Ni1—N3—C2	116.31 (13)	N2—Ni1—N3	82.54 (7)
Ni1—N4—O4	123.67 (12)	N2—Ni1—N4	178.05 (6)
Ni1—N4—C3	116.57 (12)	N2—O2—B1	112.95 (12)
Ni2—N5—O5	125.35 (11)	N2—C1—C2	112.15 (15)
Ni2—N5—C24	116.65 (12)	N2—C1—C5	124.35 (17)
Ni2—N6—O6	123.6 (1)	N3—C2—C1	112.22 (16)
Ni2—N6—C21	116.79 (12)	N4—Ni1—N3	97.14 (7)
Ni2—N7—O7	123.22 (11)	N4—C3—C4	112.23 (15)
Ni2—N7—C22	116.26 (12)	N5—Ni2—N6	97.40 (6)
Ni2—N8—O8	123.44 (11)	N5—Ni2—N8	82.29 (6)
Ni2—N8—C23	116.23 (12)	N5—O5—B2	113.58 (12)
O1—N1—C4	119.01 (15)	N5—C24—C23	112.42 (15)
O2—N2—C1	119.53 (15)	N6—Ni2—N8	177.77 (6)
O2—B1—O1	106.97 (13)	N6—O6—B2	112.79 (12)
O3—N3—C2	120.04 (16)	N6—C21—C22	112.16 (15)
O4—N4—C3	119.72 (15)	N7—Ni2—N5	179.21 (6)
O5—N5—C24	117.97 (14)	N7—Ni2—N6	82.56 (6)
O6—N6—C21	119.36 (14)	N7—Ni2—N8	97.72 (6)
O6—B2—O5	107.78 (13)	N7—C22—C21	112.14 (15)
O7—N7—C22	120.52 (14)	N8—C23—C24	112.19 (15)
O8—N8—C23	120.28 (15)		

H atoms were located in a difference Fourier map and their positional and isotropic displacement parameters were not refined. Owing to the poor quality of the crystal, the data are only 95.3% complete to θ_{max} of 27.4°.

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*.

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References

Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435.
Bakac, A., Brynildson, M. E. & Espenson, J. H. (1986). *Inorg. Chem.* **25**, 4108–4114.
Chakravorty, A. (1974). *Coord. Chem. Rev.* **13**, 1–46.
Harshani de Silva, D. G. A., Leznoff, D. B., Impey, G., Vernik, I., Jin, Z. & Stynes, D. V. (1995). *Inorg. Chem.* **34**, 4015–4025.

Nonius (1997–2001). *COLLECT*. Nonius BV, Delft, The Netherlands.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr and R. M. Sweet, pp. 307–326. New York: Academic Press.
Schrauzer, G. N. (1962). *Chem. Ber.* **95**, 1438–1445.
Stephens, F. S. & Vagg, R. S. (1980). *Inorg. Chim. Acta*, **42**, 139–143.
Watkin, D. J. (1994). *Acta Cryst.* A**50**, 411–437.
Watkin, D. J., Prout, C. K., Carruthers, J. R., Betteridge, P. W. & Cooper R. I. (2001). *CRYSTALS*. Issue 11. Chemical Crystallography Laboratory, Oxford, England.
Watkin, D. J., Prout, C. K. & Pearce, L. J. (1996). *CAMERON*. Chemical Crystallography Laboratory, Oxford, England.