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Bis{2-[(3,5-dibromo-2-oxidophenyl)methylamino]ethanol-κ^3O,N,O'}nickel(II)

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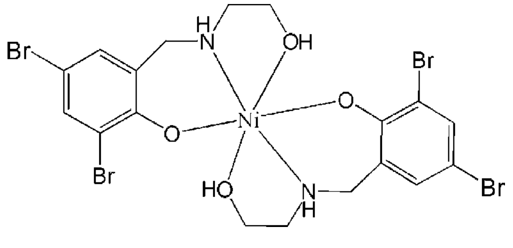
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.017 Å; R factor = 0.064; wR factor = 0.187; data-to-parameter ratio = 14.1.

In the title centrosymmetric complex, [Ni($C_9H_{10}Br_2NO_2$)$_2$], the Ni^{II} ion is chelated by two 2-[(3,5-dibromo-2-oxidophenyl)methylamino]ethanol ligands in a slightly distorted octahedral geometry. In the crystal structure, intermolecular O—H$\cdots$O hydrogen bonds connect molecules into one-dimensional chains, and there are short intermolecular Br$\cdots$Br contacts of 3.592 (1) Å.

Related literature

For a related structure, see: Zhang *et al.* (2007). For related literature, see: Allen *et al.* (1987); Cohen *et al.* (1964); Desiraju (1989); Zordan *et al.* (2005); Sarma & Desiraju (1986); Zaman *et al.* (2004).



Experimental

Crystal data

[Ni($C_9H_{10}Br_2NO_2$)$_2$]
M_r = 706.71
Monoclinic, $P2_1/n$
a = 4.865 (3) Å
b = 10.481 (6) Å
c = 20.103 (11) Å
β = 92.539 (10)°
V = 1024.0 (10) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 8.78 mm^{-1}
T = 293 (2) K
0.26 × 0.23 × 0.23 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.209, T_{max} = 0.237 (expected range = 0.117–0.133)
4304 measured reflections
1880 independent reflections
1136 reflections with $I > 2\sigma(I)$
R_{int} = 0.075

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.065
$wR(F^2)$ = 0.187
S = 1.09
1880 reflections
133 parameters
24 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.19 e Å^{-3}
$\Delta\rho_{min}$ = −1.60 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—Ni1	2.045 (9)	Ni1—O1	2.111 (7)
Ni1—O2	2.071 (8)		
N1^i—Ni1—N1	180	O2—Ni1—O1	90.8 (3)
N1—Ni1—O2^i	98.8 (3)	N1—Ni1—O1^i	90.1 (3)
N1—Ni1—O2	81.2 (3)	O2—Ni1—O1^i	89.2 (3)
O2^i—Ni1—O2	180	O1—Ni1—O1^i	180
N1—Ni1—O1	89.9 (3)		

Symmetry code: (i) $-x+2, -y+1, -z$.

Table 2
Hydrogen-bond geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O2—H2$\cdots$O1^{ii}	0.82	2.35	2.656 (10)	103

Symmetry code: (ii) $-x+3, -y+1, -z$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2001); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2503).

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Zhang, S.-H., Li, G.-Z., Feng, X.-Z. & Liu, Z. (2007). *Acta Cryst.* E**63**, m1319–m1320.
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supplementary materials

Acta Cryst. (2007). E**63**, m2564 [**doi:10.1107/S1600536807044996**]

Bis{2-[(3,5-dibromo-2-oxidophenyl)methylamino]ethanol-κ^3*O*,*N*,*O'*}nickel(II)

S.-H. Zhang, **H.-H. Zou**, **M.-H. Zeng** and **H. Liang**

Comment

Halogens have a ubiquitous presence in both inorganic and organic chemisry, serving as mondentate or bridging ligands for a wide variety of d-block, f-block, and main group metals as well as being common substituents in a large number of organic compounds. Most frequently they lie at the periphery of molecules. The resultant steric accessibility has the potential to make halogenated compounds an attractive target for use in supramolecular chemistry and crystal engineering wherein the halogen atoms are directly involved in forming intermolecular interactions. Indeed interest in packing arrangements of halogenated compounds goes back many years to what was called the "chloro effect", wherein the presence of chloro substituents on aromatic compounds frequently resulted in stacking arrangements with a resultant short(*ca* 4 Å) crystallographic axis (Cohen, *et al.*, 1964, Desiraju, 1989). Herein, we chose *L*H, to construct a new mononuclear nickel coordination complex Ni(L)$_2$ {LH = [(3,5-dibromo-2-oxidophenyl)methyleneamino]ethanol}.

The molecular structure of the tile complex is shown in Fig. 1. The Ni^{II} atom, which lies on a crystallographic inversion center, is coordinated by four O atoms and two N atoms from two difference tridentate L^- ligands, to furnish a slightly distorted octahedral geometry as defined by the bond lengths and angles in Table 1.

All other bond distances and angles are within the normal ranges (Allen *et al.*, 1987). In the crystal structure close Br···Br contacts of 3.592 (1)Å are observed (Fiorenzo, *et al.*, 2005, Zaman, *et al.*, 2004, Jagarlapudi & Gautam, 1986) (Fig.2).

Experimental

A solution of (2 mmol, 0.120 g) 2-Amino-ethanol and (2 mmol, 0.112 g) caustic potash in distilled water was added slowly to a solution of (2 mmol, 0.562 g) 3,5-Dibromo-2-hydroxy-benzaldehyde in methanol. The mixture was stirred for 30 min at room temperature, then added to solid (2 mmol, 0.076 g) sodium borohydride and stirred 2 h; the yellow solution become colourless. Then this mixture was slowly added to a solution of (1 mmol, 0.291 g) nickel nitrate in distilled water. The mixture was stirred for 4 h at room temperature and filtration and the filtrate was left to stand at room temperature. The green block single crystals suitable for X-ray diffration were obtained in a yield of 66%(base on nickel nitrate). analysis found(%):*C*, 30.52; H, 2.96; N, 3.93; $C_{18}H_{20}Br_4N_2NiO_4$ requires (%):*C*, 30.59; H, 2.85; N, 3.96.

Refinement

All hydrogen atoms were positioned geometrically and refined with a riding model, with C—H = 0.97 (CH_2) or 0.93 Å(aromatic ring); U_{iso}(H) = 1.2 U_{eq}(C) and O—H = 0.82 Å; N—H =0.91Å with U_{iso}(H) = 1.5 U_{eq}(O,*N*).

Figures

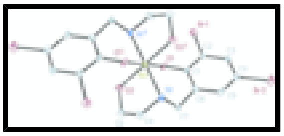

Fig. 1. The molecular structure showing 30% probability displacement ellipsoids for non-H atoms. hydrogen atoms have been omitted. symmetry codes: (i) $2 - x$, $1 - y$, $-z$.

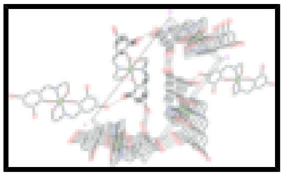

Fig. 2. Part of the crystal structure showing short Br···Br contacts as dashed lines.

Bis{2-[(3,5-Dibromo-2-oxidophenyl)methylamino]ethanol- κ^3*O*,*N*,*O*'}nickel(II)

Crystal data

$[Ni(C_9H_{10}Br_2NO_2)_2]$
$M_r = 706.71$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 4.865$ (3) Å
$b = 10.481$ (6) Å
$c = 20.103$ (11) Å
$\beta = 92.539$ (10)º
$V = 1024.0$ (10) Å^3
$Z = 2$

$F_{000} = 684$
$D_x = 2.292$ Mg m^{-3}
Mo *K*α radiation
$\lambda = 0.71073$ Å
Cell parameters from 4304 reflections
$\theta = 2.2–25.6$º
$\mu = 8.78$ mm^{-1}
$T = 293$ (2) K
Block, green
0.26 × 0.23 × 0.23 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Monochromator: graphite
$T = 293(2)$ K
φ and ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
$T_{min} = 0.209$, $T_{max} = 0.237$
4304 measured reflections

1880 independent reflections
1136 reflections with $I > 2\sigma(I)$
$R_{int} = 0.075$
$\theta_{max} = 25.6$º
$\theta_{min} = 2.2$º
$h = -5 \rightarrow 5$
$k = -12 \rightarrow 12$
$l = -11 \rightarrow 24$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.065$

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained

$wR(F^2) = 0.187$	$w = 1/[\sigma^2(F_o^2) + (0.0856P)^2 + 3.284P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.09$	$(\Delta/\sigma)_{max} < 0.001$
1880 reflections	$\Delta\rho_{max} = 1.19$ e Å^{-3}
133 parameters	$\Delta\rho_{min} = -1.60$ e Å^{-3}
24 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

Special details

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	1.4009 (3)	0.32819 (14)	0.20719 (6)	0.0319 (4)
Br2	0.6079 (3)	−0.05668 (14)	0.16826 (7)	0.0377 (5)
C1	1.144 (2)	0.2727 (11)	0.0800 (6)	0.019 (3)
C2	1.166 (2)	0.2414 (11)	0.1475 (6)	0.019 (3)
C3	1.007 (3)	0.1406 (11)	0.1737 (6)	0.026 (3)
H3	1.0272	0.1185	0.2185	0.031*
C4	0.817 (2)	0.0745 (12)	0.1308 (6)	0.026 (3)
C5	0.804 (2)	0.1025 (12)	0.0650 (7)	0.027 (3)
H5	0.6870	0.0555	0.0366	0.033*
C6	0.963 (3)	0.2010 (13)	0.0384 (6)	0.027 (3)
C7	0.959 (3)	0.2210 (12)	−0.0352 (5)	0.024 (3)
H7A	0.8497	0.1547	−0.0572	0.029*
H7B	1.1451	0.2149	−0.0505	0.029*
C8	0.888 (2)	0.3775 (14)	−0.1243 (6)	0.030 (3)
H8A	0.7656	0.4453	−0.1399	0.036*
H8B	0.8478	0.3022	−0.1511	0.036*
C9	1.198 (2)	0.4193 (14)	−0.1325 (7)	0.033 (3)
H9A	1.3176	0.3453	−0.1315	0.040*
H9B	1.2168	0.4637	−0.1743	0.040*
N1	0.8410 (19)	0.3495 (8)	−0.0538 (5)	0.019 (2)
H1	0.6688	0.3377	−0.0390	0.029*
Ni1	1.0000	0.5000	0.0000	0.0174 (5)
O1	1.2618 (14)	0.3738 (8)	0.0547 (4)	0.0190 (18)
O2	1.2670 (15)	0.5024 (9)	−0.0774 (4)	0.0230 (19)

H2	1.2756	0.5794	−0.0841	0.035*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0260 (7)	0.0401 (9)	0.0286 (7)	−0.0054 (6)	−0.0083 (5)	0.0021 (6)
Br2	0.0267 (8)	0.0384 (9)	0.0481 (9)	−0.0097 (7)	0.0023 (6)	0.0112 (7)
C1	0.014 (6)	0.016 (6)	0.026 (7)	−0.002 (5)	0.000 (5)	−0.006 (5)
C2	0.016 (6)	0.015 (7)	0.027 (7)	0.004 (5)	−0.003 (5)	0.004 (5)
C3	0.028 (7)	0.023 (8)	0.026 (7)	−0.001 (6)	−0.004 (5)	0.002 (6)
C4	0.009 (5)	0.033 (7)	0.036 (6)	−0.001 (5)	0.001 (5)	0.000 (5)
C5	0.019 (7)	0.018 (7)	0.044 (8)	−0.002 (5)	−0.011 (6)	−0.001 (6)
C6	0.019 (7)	0.041 (9)	0.021 (6)	−0.009 (6)	0.001 (5)	−0.003 (6)
C7	0.034 (8)	0.022 (7)	0.017 (6)	−0.009 (6)	−0.002 (5)	0.002 (5)
C8	0.022 (7)	0.046 (9)	0.022 (7)	−0.002 (6)	−0.007 (5)	0.001 (6)
C9	0.015 (6)	0.044 (7)	0.041 (6)	−0.007 (5)	0.003 (5)	−0.009 (6)
N1	0.018 (5)	0.015 (6)	0.025 (5)	0.003 (4)	0.002 (4)	0.007 (4)
Ni1	0.0071 (10)	0.0236 (13)	0.0214 (11)	−0.0016 (9)	−0.0012 (8)	−0.0003 (10)
O1	0.004 (4)	0.016 (4)	0.037 (4)	−0.003 (3)	−0.006 (3)	0.003 (4)
O2	0.005 (4)	0.031 (4)	0.033 (4)	−0.005 (3)	0.001 (3)	−0.009 (4)

Geometric parameters (Å, °)

Br1—C2	1.859 (12)	C8—N1	1.475 (15)
Br2—C4	1.888 (13)	C8—C9	1.586 (17)
C1—O1	1.319 (13)	C8—H8A	0.9700
C1—C2	1.395 (16)	C8—H8B	0.9700
C1—C6	1.403 (16)	C9—O2	1.436 (15)
C2—C3	1.425 (16)	C9—H9A	0.9700
C3—C4	1.415 (17)	C9—H9B	0.9700
C3—H3	0.9300	N1—Ni1	2.045 (9)
C4—C5	1.354 (17)	N1—H1	0.9099
C5—C6	1.409 (18)	Ni1—N1^{i}	2.045 (9)
C5—H5	0.9300	Ni1—O2^{i}	2.071 (8)
C6—C7	1.495 (15)	Ni1—O2	2.071 (8)
C7—N1	1.505 (15)	Ni1—O1	2.111 (7)
C7—H7A	0.9700	Ni1—O1^{i}	2.111 (7)
C7—H7B	0.9700	O2—H2	0.8200
O1—C1—C2	123.2 (10)	O2—C9—H9A	110.5
O1—C1—C6	118.2 (10)	C8—C9—H9A	110.5
C2—C1—C6	118.2 (11)	O2—C9—H9B	110.5
C1—C2—C3	121.0 (11)	C8—C9—H9B	110.5
C1—C2—Br1	122.2 (9)	H9A—C9—H9B	108.7
C3—C2—Br1	116.9 (8)	C8—N1—C7	110.0 (9)
C4—C3—C2	119.1 (11)	C8—N1—Ni1	106.5 (7)
C4—C3—H3	120.4	C7—N1—Ni1	115.3 (7)
C2—C3—H3	120.4	C8—N1—H1	121.7
C5—C4—C3	119.4 (11)	C7—N1—H1	98.3

C5—C4—Br2	123.0 (10)	Ni1—N1—H1	105.3
C3—C4—Br2	117.5 (9)	N1$^{\rm i}$—Ni1—N1	180
C4—C5—C6	121.8 (12)	N1$^{\rm i}$—Ni1—O2$^{\rm i}$	81.2 (3)
C4—C5—H5	119.1	N1—Ni1—O2$^{\rm i}$	98.8 (3)
C6—C5—H5	119.1	N1$^{\rm i}$—Ni1—O2	98.8 (3)
C1—C6—C5	120.4 (11)	N1—Ni1—O2	81.2 (3)
C1—C6—C7	119.6 (11)	O2$^{\rm i}$—Ni1—O2	180
C5—C6—C7	119.7 (11)	N1$^{\rm i}$—Ni1—O1	90.1 (3)
C6—C7—N1	111.1 (10)	N1—Ni1—O1	89.9 (3)
C6—C7—H7A	109.4	O2$^{\rm i}$—Ni1—O1	89.2 (3)
N1—C7—H7A	109.4	O2—Ni1—O1	90.8 (3)
C6—C7—H7B	109.4	N1$^{\rm i}$—Ni1—O1$^{\rm i}$	89.9 (3)
N1—C7—H7B	109.4	N1—Ni1—O1$^{\rm i}$	90.1 (3)
H7A—C7—H7B	108.0	O2$^{\rm i}$—Ni1—O1$^{\rm i}$	90.8 (3)
N1—C8—C9	110.0 (10)	O2—Ni1—O1$^{\rm i}$	89.2 (3)
N1—C8—H8A	109.7	O1—Ni1—O1$^{\rm i}$	180
C9—C8—H8A	109.7	C1—O1—Ni1	116.2 (6)
N1—C8—H8B	109.7	C9—O2—Ni1	116.0 (7)
C9—C8—H8B	109.7	C9—O2—H2	118.8
H8A—C8—H8B	108.2	Ni1—O2—H2	100.0
O2—C9—C8	106.2 (10)		

Symmetry codes: (i) $-x+2, -y+1, -z$.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···O1$^{\rm ii}$	0.82	2.35	2.656 (10)	103

Symmetry codes: (ii) $-x+3, -y+1, -z$.

Fig. 1

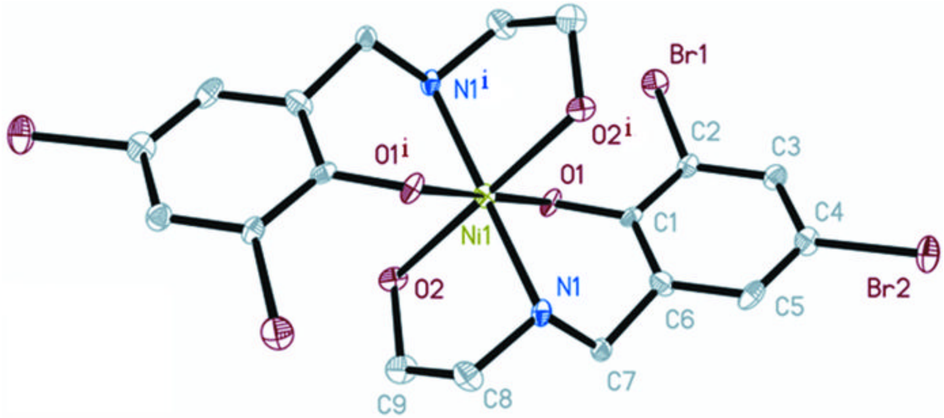

Fig. 2