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{4,4′-Dibromo-6,6′-dimethoxy-2,2′-[1,2-phenylenebis(nitrilomethanylylidene)]-$\kappa^4O^1,N,N',O^{1'}$}nickel(II)

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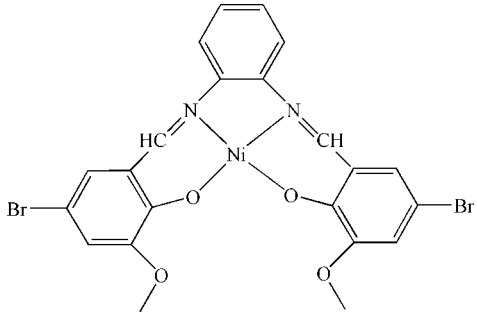
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.009 Å; R factor = 0.065; wR factor = 0.170; data-to-parameter ratio = 12.8.

In the title complex, $[Ni(C_{22}H_{16}Br_2N_2O_4)]$, the Ni^{II} ion is coordinated by two N atoms and two O atoms of a tetradentate Schiff base ligand, forming a slightly distorted square-planar coordination environment. The dihedral angle between the two bromo-substituted benzene rings is 10.1 (3)°.

Related literature

For Schiff base ligands in coordination chemistry, see: Ghosh *et al.* (2006); Nayak *et al.* (2006). For related structures, see: Wang *et al.* (1994); Bhattacharya *et al.* (2011); Yu *et al.* (2009); Kargar *et al.* (2009); Felices *et al.* (2009).



Experimental

Crystal data

$[Ni(C_{22}H_{16}Br_2N_2O_4)]$
M_r = 590.90
Monoclinic, $P2_1/n$
a = 15.288 (7) Å
b = 8.213 (3) Å
c = 16.473 (7) Å
β = 90.171 (8)°
V = 2068.3 (15) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 4.84 mm^{-1}
T = 293 K
0.17 × 0.15 × 0.12 mm

Data collection

Bruker APEXII diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.494, T_{max} = 0.595
9492 measured reflections
3613 independent reflections
2628 reflections with $I > 2\sigma(I)$
R_{int} = 0.088

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.065
$wR(F^2)$ = 0.170
S = 1.02
3613 reflections
282 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.61 e Å^{-3}
$\Delta\rho_{min}$ = −1.16 e Å^{-3}

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5411).

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supplementary materials

Acta Cryst. (2012). E**68**, m282 [doi:10.1107/S1600536812004321]

{4,4′-Dibromo-6,6′-dimethoxy-2,2′-[1,2-phenylenebis(nitrilomethanylylidene)]-$\kappa^4O^1,N,N',O^{1'}$}nickel(II)

Yongling Sun

Comment

Schiff-bases ligands, especially for those which are chelating, play an important role in the development of coordination chemistry as they readily form stable complexes with most transition metals (Ghosh *et al.*, 2006; Nayak *et al.*, 2006). Here, we present the structure of a new Ni(II) complex based on the tetradentate chelating Schiff-base ligand 1,2-diaminobenzene-*N*,*N*′-bis (5-bromo-3-methoxysalicylideneimine).

The molecular structure of the title complex is shown in Figure 1. The coordination of the Ni^{II} ion is slightly distorted square-planar, formed by two N atoms and two O atoms of the Schiff-base ligand. The mean deviation from the plane formed by the two N atoms, two O atoms and the Ni^{II} ion is 0.0426 Å. The Ni—N and Ni—O bond lengths are consistent with the corresponding distances in other nickel(II) complexes containing similar chelating tetradentate schiff-base ligands (Wang *et al.*, 1994; Bhattacharya *et al.*, 2011; Yu *et al.*, 2009; Kargar *et al.*, 2009; Felices *et al.*, 2009).

Experimental

The Schiff-base ligand can be readily synthesized by condensation 1,2-diaminobenzene and 5-bromo-2-hydroxy-3-methoxybenzaldehyde with the ratio 1:2 in ethanol. The preparation of the title complex was carried out by the reaction of $Ni(ClO_4)_2.6H_2O$ and the schiff-base ligand (1:1, molar ratio) in methanol. After the stirring process was continued for about half an hour at room temperature, the mixture was filtered and the filtrate was allowed to slowly evaporate in air for several days to produce crystals suitable for X-ray diffraction with a yield about 56%.

Refinement

H atoms were placed in calculated positions with C—H distances of 0.93 and 0.96 Å, and were allowed for as riding atoms with $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}(C_{methyl})$.

Computing details

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT* (Bruker, 2001; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

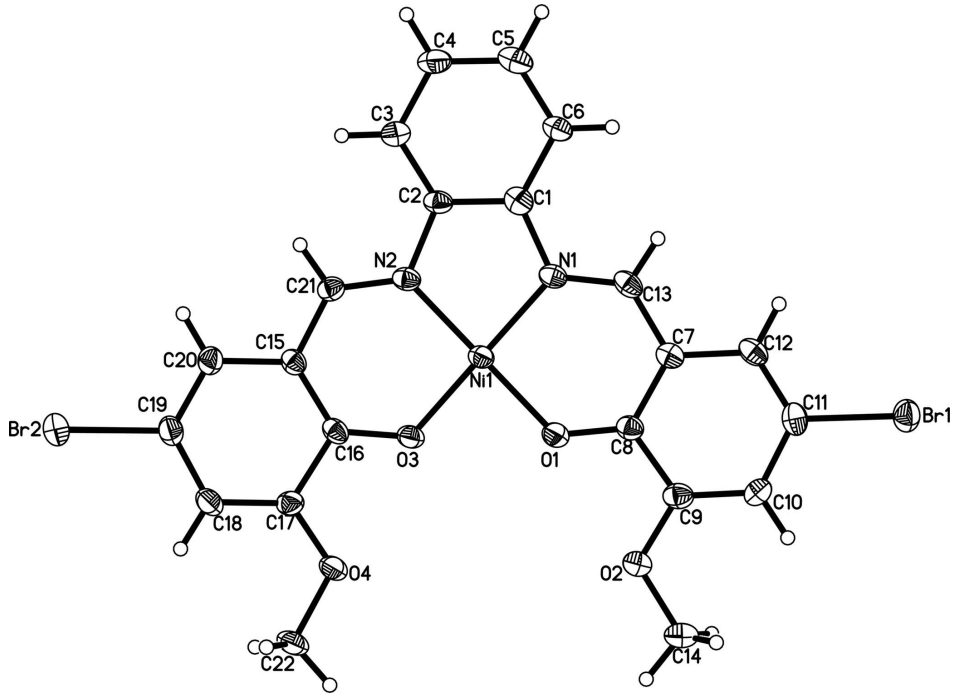

Figure 1

The molecular structure. Displacement ellipsoids are drawn at the 30% probability level.

{4,4′-Dibromo-6,6′-dimethoxy-2,2′-[1,2- phenylenebis(nitrilomethanylylidene)]- κ^4O^1,N,N',O^1'}nickel(II)

Crystal data

$[Ni(C_{22}H_{16}Br_2N_2O_4)]$
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Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
a = 15.288 (7) Å
b = 8.213 (3) Å
c = 16.473 (7) Å
β = 90.171 (8)°
V = 2068.3 (15) Å^3
Z = 4
$F(000)$ = 1168
D_x = 1.898 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2246 reflections
θ = 2.5–25.7°
μ = 4.84 mm^{-1}
T = 293 K
Block, red-brown
0.17 × 0.15 × 0.12 mm

Data collection

Bruker APEXII diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.494, T_{max} = 0.595
9492 measured reflections
3613 independent reflections
2628 reflections with $I > 2\sigma(I)$
R_{int} = 0.088
θ_{max} = 25.0°, θ_{min} = 1.8°
h = −15→18
k = −9→9
l = −19→17

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.065$
$wR(F^2) = 0.170$
$S = 1.02$
3613 reflections
282 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
$w = 1/[\sigma^2(F_o^2) + (0.1048P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 1.61$ e Å^{-3}
$\Delta\rho_{min} = -1.16$ e Å^{-3}

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Ni1	0.03371 (5)	0.81849 (9)	0.06315 (4)	0.0308 (3)
Br1	0.20569 (5)	0.28032 (9)	−0.25514 (4)	0.0469 (3)
Br2	0.01289 (5)	1.43548 (10)	0.38330 (5)	0.0626 (3)
O1	0.1366 (3)	0.7166 (5)	0.0303 (3)	0.0393 (11)
O2	0.2972 (3)	0.6233 (6)	0.0006 (3)	0.0521 (13)
O3	0.1011 (3)	0.9012 (5)	0.1472 (2)	0.0376 (10)
O4	0.2250 (3)	1.0180 (5)	0.2414 (3)	0.0446 (12)
N1	−0.0338 (3)	0.7335 (6)	−0.0210 (3)	0.0325 (12)
N2	−0.0688 (3)	0.9208 (6)	0.0960 (3)	0.0326 (12)
C1	−0.1227 (4)	0.7828 (7)	−0.0186 (4)	0.0368 (15)
C2	−0.1425 (4)	0.8846 (7)	0.0455 (4)	0.0337 (14)
C3	−0.2253 (4)	0.9459 (8)	0.0556 (4)	0.0434 (17)
H3	−0.2377	1.0151	0.0988	0.052*
C4	−0.2910 (5)	0.9029 (10)	0.0002 (4)	0.0535 (19)
H4	−0.3474	0.9436	0.0061	0.064*
C5	−0.2714 (5)	0.7999 (9)	−0.0633 (4)	0.0500 (18)
H5	−0.3151	0.7710	−0.0999	0.060*
C6	−0.1887 (4)	0.7393 (8)	−0.0735 (4)	0.0404 (15)
H6	−0.1765	0.6698	−0.1165	0.048*
C7	0.0816 (4)	0.5844 (7)	−0.0892 (3)	0.0327 (14)
C8	0.1486 (4)	0.6307 (7)	−0.0341 (4)	0.0334 (14)
C9	0.2345 (4)	0.5739 (8)	−0.0532 (4)	0.0394 (16)
C10	0.2505 (4)	0.4733 (8)	−0.1171 (4)	0.0373 (15)
H10	0.3069	0.4355	−0.1268	0.045*
C11	0.1814 (5)	0.4272 (7)	−0.1684 (4)	0.0388 (16)
C12	0.0991 (4)	0.4797 (7)	−0.1560 (4)	0.0368 (15)

H12	0.0541	0.4480	−0.1907	0.044*
C13	−0.0056 (4)	0.6388 (7)	−0.0782 (4)	0.0358 (15)
H13	−0.0468	0.6029	−0.1158	0.043*
C14	0.3853 (5)	0.5783 (12)	−0.0163 (6)	0.079 (3)
H14A	0.3907	0.4619	−0.0148	0.118*
H14B	0.4235	0.6256	0.0236	0.118*
H14C	0.4012	0.6171	−0.0692	0.118*
C15	−0.0078 (4)	1.0701 (7)	0.2093 (4)	0.0317 (14)
C16	0.0768 (4)	1.0105 (7)	0.1998 (4)	0.0332 (14)
C17	0.1444 (4)	1.0825 (7)	0.2515 (4)	0.0363 (15)
C18	0.1245 (5)	1.2049 (8)	0.3053 (4)	0.0429 (17)
H18	0.1681	1.2509	0.3374	0.051*
C19	0.0388 (5)	1.2591 (8)	0.3112 (4)	0.0413 (16)
C20	−0.0283 (4)	1.1942 (7)	0.2665 (4)	0.0371 (15)
H20	−0.0855	1.2302	0.2732	0.045*
C21	−0.0772 (4)	1.0202 (7)	0.1578 (4)	0.0329 (14)
H21	−0.1328	1.0609	0.1686	0.039*
C22	0.2969 (4)	1.1049 (9)	0.2789 (4)	0.0475 (18)
H22A	0.2974	1.2154	0.2598	0.071*
H22B	0.3511	1.0530	0.2649	0.071*
H22C	0.2899	1.1040	0.3367	0.071*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.0287 (5)	0.0273 (5)	0.0365 (5)	−0.0011 (3)	−0.0063 (3)	−0.0006 (3)
Br1	0.0521 (5)	0.0431 (5)	0.0457 (4)	−0.0035 (3)	0.0063 (3)	−0.0091 (3)
Br2	0.0549 (5)	0.0639 (6)	0.0688 (6)	0.0049 (4)	−0.0057 (4)	−0.0335 (4)
O1	0.034 (3)	0.038 (3)	0.045 (3)	−0.0006 (19)	−0.0068 (19)	−0.008 (2)
O2	0.034 (3)	0.063 (3)	0.059 (3)	0.001 (2)	−0.008 (2)	−0.021 (3)
O3	0.031 (2)	0.035 (2)	0.046 (3)	0.0019 (19)	−0.0116 (18)	−0.005 (2)
O4	0.036 (3)	0.038 (3)	0.060 (3)	0.001 (2)	−0.016 (2)	−0.012 (2)
N1	0.030 (3)	0.032 (3)	0.035 (3)	−0.006 (2)	−0.004 (2)	0.003 (2)
N2	0.030 (3)	0.029 (3)	0.039 (3)	−0.003 (2)	−0.006 (2)	0.005 (2)
C1	0.044 (4)	0.027 (3)	0.039 (4)	−0.003 (3)	−0.006 (3)	0.005 (3)
C2	0.030 (3)	0.028 (3)	0.043 (4)	−0.001 (3)	−0.009 (3)	0.005 (3)
C3	0.035 (4)	0.048 (4)	0.047 (4)	0.007 (3)	−0.007 (3)	−0.003 (3)
C4	0.030 (4)	0.074 (5)	0.056 (5)	0.005 (4)	−0.007 (3)	−0.004 (4)
C5	0.045 (4)	0.055 (5)	0.050 (4)	−0.004 (3)	−0.017 (3)	0.003 (3)
C6	0.036 (4)	0.046 (4)	0.039 (4)	−0.003 (3)	−0.011 (3)	−0.001 (3)
C7	0.036 (4)	0.028 (3)	0.035 (3)	−0.002 (3)	−0.006 (3)	0.005 (3)
C8	0.032 (3)	0.031 (3)	0.037 (3)	0.000 (3)	−0.005 (3)	0.002 (3)
C9	0.033 (4)	0.035 (4)	0.049 (4)	−0.007 (3)	−0.001 (3)	0.002 (3)
C10	0.032 (4)	0.036 (4)	0.044 (4)	0.003 (3)	0.006 (3)	0.001 (3)
C11	0.055 (5)	0.027 (3)	0.034 (4)	−0.001 (3)	0.002 (3)	0.000 (3)
C12	0.044 (4)	0.030 (3)	0.037 (4)	−0.007 (3)	−0.010 (3)	0.002 (3)
C13	0.044 (4)	0.025 (3)	0.038 (4)	−0.008 (3)	−0.012 (3)	0.002 (3)
C14	0.032 (4)	0.117 (8)	0.087 (6)	0.003 (5)	−0.011 (4)	−0.041 (6)
C15	0.036 (4)	0.025 (3)	0.034 (3)	−0.004 (3)	−0.003 (3)	0.001 (2)
C16	0.039 (4)	0.024 (3)	0.036 (3)	−0.003 (3)	−0.010 (3)	0.001 (3)

C17	0.030 (4)	0.029 (3)	0.050 (4)	0.002 (3)	−0.005 (3)	0.001 (3)
C18	0.043 (4)	0.040 (4)	0.045 (4)	−0.003 (3)	−0.012 (3)	−0.009 (3)
C19	0.047 (4)	0.039 (4)	0.038 (4)	0.002 (3)	−0.004 (3)	−0.009 (3)
C20	0.041 (4)	0.031 (4)	0.040 (4)	0.003 (3)	−0.001 (3)	−0.002 (3)
C21	0.029 (3)	0.030 (3)	0.040 (4)	−0.001 (3)	0.001 (3)	0.003 (3)
C22	0.036 (4)	0.045 (4)	0.061 (4)	−0.005 (3)	−0.018 (3)	−0.007 (3)

Geometric parameters (Å, º)

Ni1—O3	1.852 (4)	C7—C8	1.418 (8)
Ni1—N2	1.859 (5)	C7—C13	1.418 (9)
Ni1—N1	1.861 (5)	C7—C12	1.423 (8)
Ni1—O1	1.863 (4)	C8—C9	1.429 (9)
Br1—C11	1.908 (6)	C9—C10	1.362 (9)
Br2—C19	1.915 (6)	C10—C11	1.402 (9)
O1—C8	1.288 (7)	C10—H10	0.9300
O2—C9	1.365 (7)	C11—C12	1.347 (9)
O2—C14	1.426 (9)	C12—H12	0.9300
O3—C16	1.302 (7)	C13—H13	0.9300
O4—C17	1.351 (7)	C14—H14A	0.9600
O4—C22	1.448 (7)	C14—H14B	0.9600
N1—C13	1.297 (8)	C14—H14C	0.9600
N1—C1	1.419 (8)	C15—C16	1.392 (9)
N2—C21	1.312 (7)	C15—C21	1.417 (8)
N2—C2	1.430 (7)	C15—C20	1.423 (8)
C1—C2	1.381 (9)	C16—C17	1.462 (8)
C1—C6	1.398 (8)	C17—C18	1.375 (9)
C2—C3	1.374 (9)	C18—C19	1.387 (10)
C3—C4	1.400 (9)	C18—H18	0.9300
C3—H3	0.9300	C19—C20	1.370 (9)
C4—C5	1.379 (10)	C20—H20	0.9300
C4—H4	0.9300	C21—H21	0.9300
C5—C6	1.369 (10)	C22—H22A	0.9600
C5—H5	0.9300	C22—H22B	0.9600
C6—H6	0.9300	C22—H22C	0.9600
O3—Ni1—N2	94.8 (2)	C9—C10—H10	120.2
O3—Ni1—N1	179.5 (2)	C11—C10—H10	120.2
N2—Ni1—N1	85.4 (2)	C12—C11—C10	121.7 (6)
O3—Ni1—O1	85.07 (18)	C12—C11—Br1	120.0 (5)
N2—Ni1—O1	179.8 (2)	C10—C11—Br1	118.3 (5)
N1—Ni1—O1	94.7 (2)	C11—C12—C7	119.2 (5)
C8—O1—Ni1	127.5 (4)	C11—C12—H12	120.4
C9—O2—C14	117.2 (5)	C7—C12—H12	120.4
C16—O3—Ni1	126.3 (4)	N1—C13—C7	126.6 (5)
C17—O4—C22	116.5 (5)	N1—C13—H13	116.7
C13—N1—C1	120.7 (5)	C7—C13—H13	116.7
C13—N1—Ni1	125.6 (4)	O2—C14—H14A	109.5
C1—N1—Ni1	113.7 (4)	O2—C14—H14B	109.5
C21—N2—C2	120.1 (5)	H14A—C14—H14B	109.5

C21—N2—Ni1	126.3 (4)	O2—C14—H14C	109.5
C2—N2—Ni1	113.6 (4)	H14A—C14—H14C	109.5
C2—C1—C6	119.3 (6)	H14B—C14—H14C	109.5
C2—C1—N1	113.9 (5)	C16—C15—C21	121.7 (5)
C6—C1—N1	126.8 (6)	C16—C15—C20	122.2 (5)
C3—C2—C1	121.2 (5)	C21—C15—C20	115.9 (6)
C3—C2—N2	125.4 (6)	O3—C16—C15	125.7 (5)
C1—C2—N2	113.4 (5)	O3—C16—C17	117.7 (5)
C2—C3—C4	119.2 (6)	C15—C16—C17	116.6 (5)
C2—C3—H3	120.4	O4—C17—C18	124.8 (5)
C4—C3—H3	120.4	O4—C17—C16	114.3 (5)
C5—C4—C3	119.5 (7)	C18—C17—C16	120.9 (6)
C5—C4—H4	120.3	C17—C18—C19	119.4 (6)
C3—C4—H4	120.3	C17—C18—H18	120.3
C6—C5—C4	121.3 (6)	C19—C18—H18	120.3
C6—C5—H5	119.4	C20—C19—C18	122.9 (6)
C4—C5—H5	119.4	C20—C19—Br2	118.2 (5)
C5—C6—C1	119.5 (6)	C18—C19—Br2	119.0 (5)
C5—C6—H6	120.2	C19—C20—C15	118.0 (6)
C1—C6—H6	120.2	C19—C20—H20	121.0
C8—C7—C13	120.8 (5)	C15—C20—H20	121.0
C8—C7—C12	121.3 (6)	N2—C21—C15	124.7 (6)
C13—C7—C12	117.9 (5)	N2—C21—H21	117.7
O1—C8—C7	124.7 (5)	C15—C21—H21	117.7
O1—C8—C9	119.6 (5)	O4—C22—H22A	109.5
C7—C8—C9	115.7 (6)	O4—C22—H22B	109.5
C10—C9—O2	123.7 (6)	H22A—C22—H22B	109.5
C10—C9—C8	122.4 (6)	O4—C22—H22C	109.5
O2—C9—C8	113.8 (6)	H22A—C22—H22C	109.5
C9—C10—C11	119.5 (6)	H22B—C22—H22C	109.5