

Acta Crystallographica Section E

## Structure Reports

Online

ISSN 1600-5368

## Tetraaquabis[2-(2,4-dichlorophenoxy)-acetato]nickel(II)

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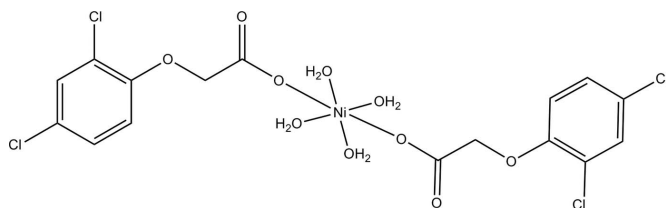
Received 23 August 2009; accepted 24 August 2009

Key indicators: single-crystal X-ray study;  $T = 293$  K; mean  $\sigma(\text{C}-\text{C}) = 0.010$  Å;  $R$  factor = 0.071;  $wR$  factor = 0.214; data-to-parameter ratio = 12.8.

In the title complex,  $[\text{Ni}(\text{C}_8\text{H}_5\text{Cl}_2\text{O}_3)_2(\text{H}_2\text{O})_4]$ , the  $\text{Ni}^{\text{II}}$  atom (site symmetry  $\bar{1}$ ) adopts a slightly distorted  $\text{NiO}_6$  octahedral coordination. An intramolecular  $\text{O}-\text{H}\cdots\text{O}$  hydrogen bond helps to establish the conformation. In the crystal, further  $\text{O}-\text{H}\cdots\text{O}$  hydrogen bonds link the molecules.

## Related literature

For background, see: Cheng *et al.* (2006). For reference structural data, see: Allen *et al.* (1987).



## Experimental

## Crystal data

 $[\text{Ni}(\text{C}_8\text{H}_5\text{Cl}_2\text{O}_3)_2(\text{H}_2\text{O})_4]$  $M_r = 570.81$ Monoclinic,  $P2_1/c$  $a = 16.860$  (3) Å $b = 8.1370$  (16) Å $c = 8.3010$  (17) Å $\beta = 95.87$  (3)° $V = 1132.8$  (4) Å<sup>3</sup> $Z = 2$ Mo  $K\alpha$  radiation $\mu = 1.38$  mm<sup>-1</sup> $T = 293$  K $0.30 \times 0.20 \times 0.10$  mm

## Data collection

Enraf–Nonius CAD-4 diffractometer

Absorption correction:  $\psi$  scan (North *et al.*, 1968) $T_{\text{min}} = 0.683$ ,  $T_{\text{max}} = 0.875$ 

2134 measured reflections

1976 independent reflections  
1596 reflections with  $I > 2\sigma(I)$  $R_{\text{int}} = 0.017$ 

200 standard reflections

every 3 reflections

intensity decay: 1%

## Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.071$  $wR(F^2) = 0.214$  $S = 1.14$ 

1976 reflections

154 parameters

6 restraints

H atoms treated by a mixture of independent and constrained refinement

 $\Delta\rho_{\text{max}} = 0.86$  e Å<sup>-3</sup> $\Delta\rho_{\text{min}} = -1.97$  e Å<sup>-3</sup>

Table 1

Selected bond lengths (Å).

Ni1—O3	2.085 (5)	Ni1—O1	2.130 (4)
Ni1—O4	2.126 (4)		

Table 2

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1—H1A $\cdots$ O2 <sup>i</sup>	0.84 (5)	2.05 (7)	2.723 (7)	136 (8)
O1—H1B $\cdots$ O2	0.84 (3)	1.82 (5)	2.619 (7)	157 (7)
O3—H3A $\cdots$ O1 <sup>i</sup>	0.85 (6)	2.44 (7)	3.217 (7)	153 (7)
O3—H3B $\cdots$ O6 <sup>ii</sup>	0.846 (16)	2.34 (6)	2.980 (7)	133 (8)

Symmetry codes: (i)  $x, -y - \frac{1}{2}, z + \frac{1}{2}$ ; (ii)  $x, -y + \frac{1}{2}, z + \frac{1}{2}$ .

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); 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*.

The project was supported by the Scientific Research Foundation for Returned Overseas Chinese Scholars, State Education Ministry, Educational Commission of Hubei Province (D20091703) and the Natural Science Foundation of Hubei Province (2008CDB038).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5064).

## References

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**supplementary materials**

*Acta Cryst.* (2009). E65, m1148 [ doi:10.1107/S1600536809033662 ]

## Tetraaquabis[2-(2,4-dichlorophenoxy)acetato]nickel(II)

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### Comment

There has been much research interest in acid metal complexes due to their molecular architectures and biological activities (e.g. Cheng *et al.*, 2006). In this work, we report here the crystal structure of the title compound, (I). In (I), all bond lengths are within normal ranges (Allen *et al.*, 1987) (Fig. 1). The Ni<sup>II</sup> atom is six-coordinated by two O atoms from the 2-(2,4-dichlorophenoxy)acetate and four O atoms from the water molecules, forming a slightly distorted octahedral coordination.

### Experimental

A mixture of 2-(2,4-dichlorophenoxy)acetic acid (440 mg, 2 mmol) and NiCl<sub>2</sub>·6H<sub>2</sub>O (1 mmol, 236 mg) in methanol (10 ml) was stirred for 3 h. After keeping the filtrate in air for 7 d, green blocks of (I) were formed.

### Refinement

The water H atoms were located in a difference map and their positions were refined with the restraint O—H = 0.83 (1) Å. The other H atoms were positioned geometrically (C—H = 0.93–0.97 Å) and refined as riding, with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ .

### Figures

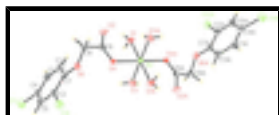


Fig. 1. The molecular structure of (I) showing 30% probability displacement ellipsoids. Atoms with the suffix A are generated by the symmetry operation (1-x, -y, 1-z).

## Tetraaquabis[2-(2,4-dichlorophenoxy)acetato]nickel(II)

### Crystal data

[Ni(C<sub>8</sub>H<sub>5</sub>Cl<sub>2</sub>O<sub>3</sub>)<sub>2</sub>(H<sub>2</sub>O)<sub>4</sub>]

$M_r = 570.81$

Monoclinic,  $P2_1/c$

Hall symbol: -P 2ybc

$a = 16.860$  (3) Å

$b = 8.1370$  (16) Å

$c = 8.3010$  (17) Å

$\beta = 95.87$  (3)°

$V = 1132.8$  (4) Å<sup>3</sup>

$Z = 2$

$F_{000} = 580$

$D_x = 1.673$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 25 reflections

$\theta = 9\text{--}12^\circ$

$\mu = 1.38$  mm<sup>-1</sup>

$T = 293$  K

Block, green

$0.30 \times 0.20 \times 0.10$  mm

## Data collection

Enraf–Nonius CAD-4 diffractometer	$R_{\text{int}} = 0.017$
Radiation source: fine-focus sealed tube	$\theta_{\text{max}} = 25.2^\circ$
Monochromator: graphite	$\theta_{\text{min}} = 1.2^\circ$
$T = 293$ K	$h = -20 \rightarrow 20$
$\omega/2\theta$ scans	$k = -9 \rightarrow 0$
Absorption correction: $\psi$ scan (North <i>et al.</i> , 1968)	$l = 0 \rightarrow 9$
$T_{\text{min}} = 0.683$ , $T_{\text{max}} = 0.875$	200 standard reflections
2134 measured reflections	every 3 reflections
1976 independent reflections	intensity decay: 1%
1596 reflections with $I > 2\sigma(I)$	

## Refinement

Refinement on $F^2$	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.071$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.214$	$w = 1/[\sigma^2(F_o^2) + (0.1181P)^2 + 3.965P]$
$S = 1.14$	where $P = (F_o^2 + 2F_c^2)/3$
1976 reflections	$(\Delta/\sigma)_{\text{max}} = 0.001$
154 parameters	$\Delta\rho_{\text{max}} = 0.86 \text{ e } \text{\AA}^{-3}$
6 restraints	$\Delta\rho_{\text{min}} = -1.97 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: none

## 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 ( $\text{\AA}^2$ )

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.1103 (4)	0.1141 (9)	0.3299 (9)	0.0487 (17)

H1	0.0872	0.0160	0.3585	0.058*
C2	0.1780 (4)	0.4056 (8)	0.2486 (8)	0.0363 (14)
C3	0.1822 (4)	0.1110 (8)	0.2642 (9)	0.0454 (16)
H3	0.2076	0.0113	0.2504	0.054*
C4	0.0724 (4)	0.2598 (9)	0.3534 (9)	0.0458 (16)
C5	0.1072 (4)	0.4079 (8)	0.3131 (8)	0.0431 (15)
H5	0.0821	0.5072	0.3303	0.052*
C6	0.3693 (4)	0.0240 (7)	0.2247 (7)	0.0296 (12)
C7	0.2173 (4)	0.2580 (7)	0.2183 (8)	0.0345 (13)
C8	0.3201 (4)	0.1188 (8)	0.0942 (7)	0.0388 (15)
H8A	0.3533	0.1447	0.0088	0.047*
H8B	0.2770	0.0489	0.0479	0.047*
Cl1	-0.01949 (12)	0.2621 (3)	0.4316 (3)	0.0626 (6)
Cl2	0.22352 (11)	0.5880 (2)	0.1988 (3)	0.0557 (6)
H1A	0.418 (3)	-0.233 (12)	0.523 (6)	0.067*
H3A	0.452 (4)	-0.018 (10)	0.785 (6)	0.067*
H1B	0.436 (4)	-0.226 (11)	0.366 (3)	0.067*
H3B	0.3858 (7)	0.030 (11)	0.687 (9)	0.067*
Ni1	0.5000	0.0000	0.5000	0.0266 (4)
O1	0.4550 (3)	-0.2430 (5)	0.4624 (6)	0.0406 (11)
O2	0.3738 (3)	-0.1289 (5)	0.2003 (5)	0.0433 (11)
O3	0.4362 (3)	0.0347 (7)	0.6996 (6)	0.0503 (12)
O4	0.4045 (2)	0.0994 (5)	0.3429 (5)	0.0319 (9)
O6	0.2866 (3)	0.2687 (6)	0.1489 (6)	0.0420 (11)

Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C1	0.053 (4)	0.039 (4)	0.052 (4)	-0.005 (3)	-0.005 (3)	-0.005 (3)
C2	0.038 (3)	0.029 (3)	0.041 (3)	-0.008 (3)	-0.002 (3)	0.005 (3)
C3	0.048 (4)	0.027 (3)	0.059 (4)	-0.002 (3)	-0.009 (3)	0.004 (3)
C4	0.049 (4)	0.046 (4)	0.040 (4)	-0.006 (3)	-0.006 (3)	0.002 (3)
C5	0.049 (4)	0.032 (3)	0.047 (4)	0.000 (3)	0.000 (3)	0.000 (3)
C6	0.039 (3)	0.018 (3)	0.032 (3)	0.001 (2)	0.006 (2)	-0.002 (2)
C7	0.037 (3)	0.029 (3)	0.036 (3)	0.004 (2)	-0.004 (2)	0.000 (2)
C8	0.046 (4)	0.035 (3)	0.034 (3)	0.006 (3)	-0.001 (3)	-0.009 (3)
Cl1	0.0543 (11)	0.0675 (13)	0.0680 (13)	-0.0061 (9)	0.0147 (9)	-0.0037 (10)
Cl2	0.0552 (11)	0.0276 (8)	0.0841 (14)	0.0013 (7)	0.0062 (9)	0.0059 (8)
Ni1	0.0369 (6)	0.0154 (5)	0.0270 (6)	0.0015 (4)	0.0017 (4)	0.0023 (4)
O1	0.058 (3)	0.028 (2)	0.035 (2)	0.000 (2)	-0.001 (2)	-0.0015 (19)
O2	0.066 (3)	0.031 (2)	0.032 (2)	-0.001 (2)	-0.003 (2)	-0.0056 (18)
O3	0.048 (3)	0.054 (3)	0.050 (3)	0.009 (2)	0.013 (2)	0.007 (2)
O4	0.043 (2)	0.0205 (19)	0.030 (2)	0.0092 (17)	-0.0054 (17)	-0.0048 (17)
O6	0.037 (2)	0.032 (2)	0.056 (3)	0.0010 (18)	-0.002 (2)	0.004 (2)

Geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

C1—C4	1.369 (10)	C7—O6	1.358 (8)
C1—C3	1.381 (11)	C8—O6	1.437 (7)

## supplementary materials

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C1—H1	0.9300	C8—H8A	0.9700
C2—C5	1.358 (10)	C8—H8B	0.9700
C2—C7	1.407 (9)	Ni1—O3	2.085 (5)
C2—C12	1.740 (6)	Ni1—O3 <sup>i</sup>	2.085 (5)
C3—C7	1.403 (9)	Ni1—O4	2.126 (4)
C3—H3	0.9300	Ni1—O4 <sup>i</sup>	2.126 (4)
C4—C5	1.396 (10)	Ni1—O1 <sup>i</sup>	2.130 (4)
C4—C11	1.741 (8)	Ni1—O1	2.130 (4)
C5—H5	0.9300	O1—H1A	0.841 (10)
C6—O4	1.253 (7)	O1—H1B	0.840 (10)
C6—O2	1.265 (7)	O3—H3A	0.844 (10)
C6—C8	1.508 (8)	O3—H3B	0.847 (10)
C4—C1—C3	120.9 (7)	C6—C8—H8B	108.7
C4—C1—H1	119.6	H8A—C8—H8B	107.6
C3—C1—H1	119.6	O3—Ni1—O3 <sup>i</sup>	180.0
C5—C2—C7	122.1 (6)	O3—Ni1—O4	90.93 (19)
C5—C2—C12	120.6 (5)	O3 <sup>i</sup> —Ni1—O4	89.07 (18)
C7—C2—C12	117.3 (5)	O3—Ni1—O4 <sup>i</sup>	89.07 (18)
C1—C3—C7	120.2 (6)	O3 <sup>i</sup> —Ni1—O4 <sup>i</sup>	90.93 (18)
C1—C3—H3	119.9	O4—Ni1—O4 <sup>i</sup>	180.0
C7—C3—H3	119.9	O3—Ni1—O1 <sup>i</sup>	87.9 (2)
C1—C4—C5	120.0 (7)	O3 <sup>i</sup> —Ni1—O1 <sup>i</sup>	92.1 (2)
C1—C4—C11	120.5 (6)	O4—Ni1—O1 <sup>i</sup>	88.46 (16)
C5—C4—C11	119.5 (6)	O4 <sup>i</sup> —Ni1—O1 <sup>i</sup>	91.54 (16)
C2—C5—C4	119.4 (6)	O3—Ni1—O1	92.1 (2)
C2—C5—H5	120.3	O3 <sup>i</sup> —Ni1—O1	87.9 (2)
C4—C5—H5	120.3	O4—Ni1—O1	91.54 (16)
O4—C6—O2	125.2 (5)	O4 <sup>i</sup> —Ni1—O1	88.46 (16)
O4—C6—C8	119.6 (5)	O1 <sup>i</sup> —Ni1—O1	180.0
O2—C6—C8	115.2 (5)	Ni1—O1—H1A	96 (7)
O6—C7—C3	125.1 (6)	Ni1—O1—H1B	94 (6)
O6—C7—C2	117.5 (5)	H1A—O1—H1B	108.9 (18)
C3—C7—C2	117.4 (6)	Ni1—O3—H3A	117 (6)
O6—C8—C6	114.4 (5)	Ni1—O3—H3B	119 (6)
O6—C8—H8A	108.7	H3A—O3—H3B	108.4 (18)
C6—C8—H8A	108.7	C6—O4—Ni1	124.2 (4)
O6—C8—H8B	108.7	C7—O6—C8	117.5 (5)
C4—C1—C3—C7	-0.9 (11)	O4—C6—C8—O6	-28.4 (8)
C3—C1—C4—C5	-1.0 (11)	O2—C6—C8—O6	154.2 (6)
C3—C1—C4—C11	178.4 (5)	O2—C6—O4—Ni1	14.9 (9)
C7—C2—C5—C4	1.0 (10)	C8—C6—O4—Ni1	-162.2 (4)
C12—C2—C5—C4	-179.3 (5)	O3—Ni1—O4—C6	-122.0 (5)
C1—C4—C5—C2	0.9 (10)	O3 <sup>i</sup> —Ni1—O4—C6	58.0 (5)
C11—C4—C5—C2	-178.5 (5)	O4 <sup>i</sup> —Ni1—O4—C6	76 (100)
C1—C3—C7—O6	-178.2 (6)	O1 <sup>i</sup> —Ni1—O4—C6	150.1 (5)

C1—C3—C7—C2	2.7 (10)	O1—Ni1—O4—C6	-29.9 (5)
C5—C2—C7—O6	178.0 (6)	C3—C7—O6—C8	10.6 (9)
C12—C2—C7—O6	-1.6 (8)	C2—C7—O6—C8	-170.3 (5)
C5—C2—C7—C3	-2.8 (10)	C6—C8—O6—C7	-83.3 (7)
C12—C2—C7—C3	177.5 (5)		

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

*Hydrogen-bond geometry* ( $\text{\AA}, ^\circ$ )

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
O1—H1A $\cdots$ O2 <sup>ii</sup>	0.84 (5)	2.05 (7)	2.723 (7)	136 (8)
O1—H1B $\cdots$ O2	0.84 (3)	1.82 (5)	2.619 (7)	157 (7)
O3—H3A $\cdots$ O1 <sup>ii</sup>	0.85 (6)	2.44 (7)	3.217 (7)	153 (7)
O3—H3B $\cdots$ O6 <sup>iii</sup>	0.846 (16)	2.34 (6)	2.980 (7)	133 (8)

Symmetry codes: (ii)  $x, -y-1/2, z+1/2$ ; (iii)  $x, -y+1/2, z+1/2$ .

Fig. 1

