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# {4,4′,6,6′-Tetraiodo-2,2′-[propane-1,3-diylbis(nitrilomethanylylidene)]-diphenolato-$\kappa^4$*O*,*N*,*N*′,*O*′}nickel(II)

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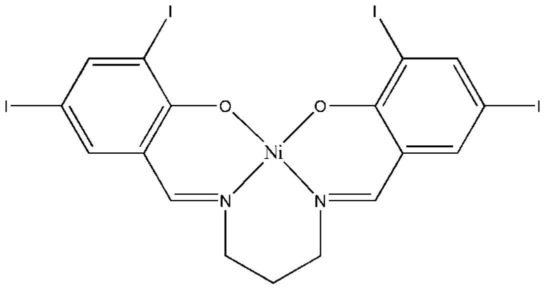
Key indicators: single-crystal X-ray study; *T* = 291 K; mean σ(C–C) = 0.006 Å; *R* factor = 0.031; *wR* factor = 0.055; data-to-parameter ratio = 18.2.

The asymmetric unit of the title compound, [Ni($C_{17}H_{12}I_4N_2O_2$)], comprises half of a Schiff base complex. The $Ni^{II}$ and central C atom of the propyl chain are located on a twofold rotation axis. The geometry around the $Ni^{II}$ atom is square planar, supported by the $N_2O_2$ donor atoms of the coordinated ligand. In the crystal, there are no significant intermolecular interactions present. The crystal studied was a non-merohedral twin with a refined twin component ratio of 0.944 (1):0.056 (1).

## Related literature

For standard bond lengths, see: Allen *et al.* (1987). For applications of Schiff bases in coordination chemistry, see, for example: Granovski *et al.* (1993); Blower *et al.* (1998). For the structure of the Schiff base ligand, see: Kargar *et al.* (2012*a*). For related structures, see, for example: Kargar *et al.* (2012*b*,*c*,*d*,*e*).



## Experimental

### *Crystal data*

| | |
|---|---|
| [Ni($C_{17}H_{12}I_4N_2O_2$)] | $V$ = 2010.8 (2) Å$^3$ |
| $M_r$ = 842.60 | $Z$ = 4 |
| Monoclinic, $C2/c$ | Mo $K\alpha$ radiation |
| $a$ = 26.1229 (18) Å | $\mu$ = 7.12 mm$^{-1}$ |
| $b$ = 10.7409 (7) Å | $T$ = 291 K |
| $c$ = 7.2387 (5) Å | 0.22 × 0.12 × 0.08 mm |
| $\beta$ = 98.107 (3)° | |

### *Data collection*

| | |
|---|---|
| Bruker SMART APEXII CCD area-detector diffractometer | 7468 measured reflections |
| Absorption correction: multi-scan (*TWINABS*; Bruker, 2005) | 2188 independent reflections |
| $T_{min}$ = 0.303, $T_{max}$ = 0.600 | 1755 reflections with $I > 2\sigma(I)$ |
| | $R_{int}$ = 0.033 |

### *Refinement*

| | |
|---|---|
| $R[F^2 > 2\sigma(F^2)]$ = 0.031 | 120 parameters |
| $wR(F^2)$ = 0.055 | H-atom parameters constrained |
| $S$ = 1.04 | $\Delta\rho_{max}$ = 0.70 e Å$^{-3}$ |
| 2188 reflections | $\Delta\rho_{min}$ = −0.55 e Å$^{-3}$ |

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); 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* and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SU2472).

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# supporting information

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## {4,4′,6,6′-Tetraiodo-2,2′-[propane-1,3-diylbis(nitrilomethanylylidene)]diphenolato-$\kappa^4O,N,N',O'$}nickel(II)

**Hadi Kargar, Reza Kia, Amir Adabi Ardakani and Muhammad Nawaz Tahir**

### S1. Comment

Schiff base complexes are one of the most important stereochemical models in transition metal coordination chemistry, with their ease of preparation and structural variations (Granovski *et al.*, 1993; Blower *et al.*, 1998). In continuation of our work on the crystal structure of Schiff base metal complexes (Kargar *et al.*, 2012*b,c,d,e*), we determined the X-ray structure of the title compound.

The asymmetric unit of the title compound, Fig. 1, comprises a Schiff base complex. The bond lengths (Allen *et al.*, 1987) and angles are within the normal ranges and are comparable to the bond lengths and angles of the related ligand (Kargar *et al.*, 2012*a*) and related Ni-complexes (Kargar *et al.*, 2012*b,c,d,e*). The $Ni^{II}$ and C9 atom of the propyl segment are located on a two-fold rotation axis. The geometry around $Ni^{II}$ atom is square-planar which is supported by the $N_2O_2$ donor atoms of the coordinated ligand.

There are no significant intermolecular interactions in the crystal structure.

The crystal used was a non-merohedral twin with refined twin components ratio of 0.944 (1)/0.056 (1).

### S2. Experimental

The title compound was synthesized by adding 3,5-diiodo-salicylaldehyde-1,3-propanediamine (2 mmol) to a solution of $NiCl_2$. $6H_2O$ (2.1 mmol) in ethanol (30 ml). The mixture was refluxed with stirring for 1h. The resultant solution was filtered. Red single crystals of the title compound suitable for *X*-ray structure determination were recrystallized from ethanol by slow evaporation of the solvents at room temperature over several days.

### S3. Refinement

The H-atoms were included in calculated positions and treated as riding atoms: C—H = 0.93 and 0.97 Å for CH and $CH_2$ H-atoms, respectively, with $U_{iso}$ (H) = 1.2 $U_{eq}$(C).

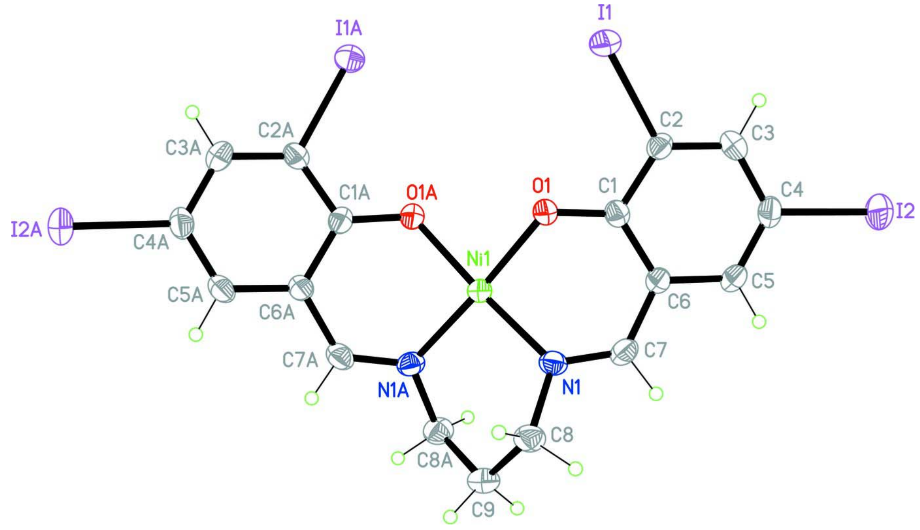

**Figure 1**

A view of the molecular structure of the title molecule, showing 50% probability displacement ellipsoids and the atomic numbering [symmetry code for suffix A = -x, y, -z + 1/2].

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### *Crystal data*

$[Ni(C_{17}H_{12}I_4N_2O_2)]$
$M_r = 842.60$
Monoclinic, $C2/c$
Hall symbol: -C 2yc
$a = 26.1229$ (18) Å
$b = 10.7409$ (7) Å
$c = 7.2387$ (5) Å
$\beta = 98.107$ (3)°
$V = 2010.8$ (2) Å$^3$
$Z = 4$

$F(000) = 1536$
$D_x = 2.783$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 526 reflections
$\theta = 2.5$–27.5°
$\mu = 7.12$ mm$^{-1}$
$T = 291$ K
Block, red
0.22 × 0.12 × 0.08 mm

### *Data collection*

Bruker SMART APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega$ scans
Absorption correction: multi-scan (*TWINABS*; Bruker, 2005)
$T_{min} = 0.303$, $T_{max} = 0.600$

7463 measured reflections
2188 independent reflections
1755 reflections with $I > 2\sigma(I)$
$R_{int} = 0.033$
$\theta_{max} = 27.1°$, $\theta_{min} = 1.6°$
$h = -33 \rightarrow 33$
$k = -13 \rightarrow 13$
$l = -8 \rightarrow 9$

### *Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.031$
$wR(F^2) = 0.055$
$S = 1.04$
2188 reflections
120 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

$w = 1/[\sigma^2(F_o^2) + (0.0177P)^2 + 2.2669P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.70$ e Å$^{-3}$
$\Delta\rho_{min} = -0.55$ e Å$^{-3}$

*Special details*

**Geometry**. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2\text{sigma}(F^2)$ 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$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| Ni1 | 0.0000 | 0.48944 (7) | 0.2500 | 0.02132 (18) | |
| I1 | 0.090843 (12) | 0.89044 (3) | 0.36252 (5) | 0.03652 (10) | |
| I2 | 0.279631 (12) | 0.60955 (3) | 0.20407 (5) | 0.04421 (11) | |
| N1 | 0.05062 (13) | 0.3679 (3) | 0.3138 (5) | 0.0242 (8) | |
| O1 | 0.04733 (11) | 0.6174 (2) | 0.3090 (4) | 0.0269 (7) | |
| C1 | 0.09612 (15) | 0.6129 (4) | 0.2932 (6) | 0.0224 (9) | |
| C2 | 0.12636 (16) | 0.7229 (4) | 0.3024 (6) | 0.0240 (9) | |
| C3 | 0.17729 (16) | 0.7234 (4) | 0.2769 (6) | 0.0291 (10) | |
| H3 | 0.1954 | 0.7980 | 0.2804 | 0.035* | |
| C4 | 0.20183 (16) | 0.6119 (4) | 0.2457 (6) | 0.0291 (10) | |
| C5 | 0.17586 (16) | 0.5018 (4) | 0.2474 (6) | 0.0276 (10) | |
| H5 | 0.1930 | 0.4273 | 0.2327 | 0.033* | |
| C6 | 0.12328 (15) | 0.5005 (4) | 0.2715 (6) | 0.0233 (10) | |
| C7 | 0.09902 (16) | 0.3837 (4) | 0.3042 (6) | 0.0271 (10) | |
| H7 | 0.1201 | 0.3136 | 0.3198 | 0.033* | |
| C8 | 0.03503 (17) | 0.2492 (4) | 0.3903 (6) | 0.0292 (10) | |
| H8A | 0.0173 | 0.2661 | 0.4967 | 0.035* | |
| H8B | 0.0658 | 0.2012 | 0.4344 | 0.035* | |
| C9 | 0.0000 | 0.1721 (5) | 0.2500 | 0.0298 (14) | |
| H9A | −0.0212 | 0.1188 | 0.3165 | 0.036* | 0.50 |
| H9B | 0.0212 | 0.1188 | 0.1835 | 0.036* | 0.50 |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Ni1 | 0.0199 (4) | 0.0179 (4) | 0.0259 (4) | 0.000 | 0.0026 (3) | 0.000 |
| I1 | 0.03286 (18) | 0.02414 (16) | 0.0539 (2) | 0.00044 (13) | 0.01095 (15) | −0.00832 (15) |
| I2 | 0.02416 (17) | 0.0449 (2) | 0.0661 (3) | 0.00048 (15) | 0.01512 (16) | 0.00269 (17) |
| N1 | 0.0261 (19) | 0.0191 (19) | 0.026 (2) | −0.0019 (15) | 0.0007 (16) | 0.0025 (15) |
| O1 | 0.0204 (15) | 0.0220 (15) | 0.0392 (19) | −0.0010 (12) | 0.0072 (13) | −0.0029 (13) |
| C1 | 0.021 (2) | 0.024 (2) | 0.022 (2) | −0.0018 (18) | 0.0026 (18) | 0.0021 (18) |
| C2 | 0.023 (2) | 0.024 (2) | 0.025 (2) | 0.0037 (18) | 0.0038 (19) | −0.0022 (18) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C3 | 0.025 (2) | 0.029 (2) | 0.033 (3) | −0.007 (2) | 0.003 (2) | −0.002 (2) |
| C4 | 0.019 (2) | 0.034 (3) | 0.034 (3) | 0.002 (2) | 0.0038 (19) | 0.000 (2) |
| C5 | 0.024 (2) | 0.025 (2) | 0.033 (3) | 0.0070 (19) | 0.004 (2) | −0.0007 (19) |
| C6 | 0.021 (2) | 0.022 (2) | 0.027 (2) | 0.0014 (18) | 0.0023 (19) | −0.0017 (18) |
| C7 | 0.025 (2) | 0.024 (2) | 0.031 (3) | 0.0064 (19) | 0.001 (2) | 0.0020 (19) |
| C8 | 0.032 (2) | 0.025 (2) | 0.030 (3) | 0.000 (2) | 0.000 (2) | 0.0072 (19) |
| C9 | 0.036 (4) | 0.021 (3) | 0.033 (4) | 0.000 | 0.005 (3) | 0.000 |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Ni1—O1$^{i}$ | 1.858 (3) | C3—H3 | 0.9300 |
| Ni1—O1 | 1.858 (3) | C4—C5 | 1.365 (6) |
| Ni1—N1$^{i}$ | 1.869 (3) | C5—C6 | 1.409 (6) |
| Ni1—N1 | 1.869 (3) | C5—H5 | 0.9300 |
| I1—C2 | 2.098 (4) | C6—C7 | 1.440 (5) |
| I2—C4 | 2.096 (4) | C7—H7 | 0.9300 |
| N1—C7 | 1.287 (5) | C8—C9 | 1.514 (5) |
| N1—C8 | 1.471 (5) | C8—H8A | 0.9700 |
| O1—C1 | 1.296 (5) | C8—H8B | 0.9700 |
| C1—C2 | 1.418 (5) | C9—C8$^{i}$ | 1.514 (5) |
| C1—C6 | 1.421 (5) | C9—H9A | 0.9700 |
| C2—C3 | 1.369 (5) | C9—H9B | 0.9700 |
| C3—C4 | 1.391 (6) | | |
| | | | |
| O1$^{i}$—Ni1—O1 | 84.57 (17) | C4—C5—C6 | 120.3 (4) |
| O1$^{i}$—Ni1—N1$^{i}$ | 92.01 (13) | C4—C5—H5 | 119.8 |
| O1—Ni1—N1$^{i}$ | 176.57 (13) | C6—C5—H5 | 119.8 |
| O1$^{i}$—Ni1—N1 | 176.57 (13) | C5—C6—C1 | 121.1 (4) |
| O1—Ni1—N1 | 92.01 (13) | C5—C6—C7 | 119.3 (4) |
| N1$^{i}$—Ni1—N1 | 91.4 (2) | C1—C6—C7 | 118.9 (4) |
| C7—N1—C8 | 117.4 (3) | N1—C7—C6 | 125.6 (4) |
| C7—N1—Ni1 | 124.1 (3) | N1—C7—H7 | 117.2 |
| C8—N1—Ni1 | 118.3 (3) | C6—C7—H7 | 117.2 |
| C1—O1—Ni1 | 125.6 (3) | N1—C8—C9 | 113.2 (4) |
| O1—C1—C2 | 120.9 (4) | N1—C8—H8A | 108.9 |
| O1—C1—C6 | 123.7 (4) | C9—C8—H8A | 108.9 |
| C2—C1—C6 | 115.5 (4) | N1—C8—H8B | 108.9 |
| C3—C2—C1 | 122.9 (4) | C9—C8—H8B | 108.9 |
| C3—C2—I1 | 119.4 (3) | H8A—C8—H8B | 107.7 |
| C1—C2—I1 | 117.7 (3) | C8—C9—C8$^{i}$ | 113.7 (5) |
| C2—C3—C4 | 119.8 (4) | C8—C9—H9A | 108.8 |
| C2—C3—H3 | 120.1 | C8$^{i}$—C9—H9A | 108.8 |
| C4—C3—H3 | 120.1 | C8—C9—H9B | 108.8 |
| C5—C4—C3 | 120.2 (4) | C8$^{i}$—C9—H9B | 108.8 |
| C5—C4—I2 | 118.9 (3) | H9A—C9—H9B | 107.7 |
| C3—C4—I2 | 120.8 (3) | | |
| | | | |
| N1$^{i}$—Ni1—N1—C7 | 152.0 (4) | I2—C4—C5—C6 | −178.8 (3) |

| | | | |
|---|---|---|---|
| N1$^{i}$—Ni1—N1—C8 | −31.8 (2) | C4—C5—C6—C1 | 0.4 (6) |
| O1$^{i}$—Ni1—O1—C1 | −148.9 (4) | C4—C5—C6—C7 | −169.6 (4) |
| N1—Ni1—O1—C1 | 31.2 (3) | O1—C1—C6—C5 | 177.4 (4) |
| Ni1—O1—C1—C2 | 166.0 (3) | C2—C1—C6—C5 | −4.4 (6) |
| Ni1—O1—C1—C6 | −15.9 (6) | O1—C1—C6—C7 | −12.5 (6) |
| O1—C1—C2—C3 | −176.6 (4) | C2—C1—C6—C7 | 165.7 (4) |
| C6—C1—C2—C3 | 5.2 (6) | C8—N1—C7—C6 | −166.2 (4) |
| O1—C1—C2—I1 | 4.6 (5) | Ni1—N1—C7—C6 | 10.0 (6) |
| C6—C1—C2—I1 | −173.7 (3) | C5—C6—C7—N1 | −174.2 (4) |
| C1—C2—C3—C4 | −1.9 (7) | C1—C6—C7—N1 | 15.6 (7) |
| I1—C2—C3—C4 | 177.0 (3) | C7—N1—C8—C9 | −116.1 (4) |
| C2—C3—C4—C5 | −2.4 (7) | Ni1—N1—C8—C9 | 67.4 (4) |
| C2—C3—C4—I2 | 179.5 (3) | N1—C8—C9—C8$^{i}$ | −32.1 (2) |
| C3—C4—C5—C6 | 3.2 (7) | | |

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