

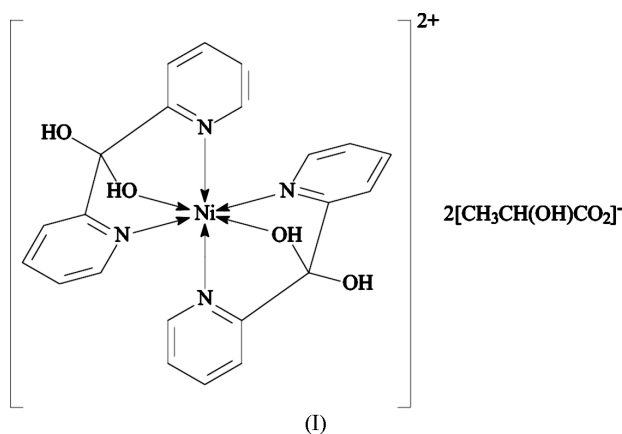
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## Key indicators

Single-crystal X-ray study  
 $T = 293$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.004$  Å  
Disorder in solvent or counterion  
 $R$  factor = 0.045  
 $wR$  factor = 0.112  
Data-to-parameter ratio = 14.6For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.Bis(di-2-pyridylmethanediol- $\kappa^3N,O,N'$ )-nickel(II) dilactateDi-2-pyridyl ketone reacts with nickel(II) dilactate in water to form the title compound,  $[\text{Ni}(\text{C}_{11}\text{H}_{10}\text{N}_2\text{O}_2)_2](\text{C}_3\text{H}_5\text{O}_3)_2$ , whose Ni atom is  $N,O,N'$ -chelated by the ketals in an octahedral environment. The cation interacts with the two anions through extensive hydrogen bonds to furnish a helical chain motif that runs along the  $b$  axis.Received 8 December 2004  
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## Comment

Di-2-pyridyl ketone occasionally undergoes hydrolysis when reacted with metal salts to form the corresponding ketal,  $(\text{C}_5\text{H}_4\text{N})_2\text{C}(\text{OH})_2$ , which itself functions as either a bidentate or a terdentate chelating ligand that binds to the metal atom. A number of complexes of this ligand have been structurally characterized (Tangoulis *et al.*, 1997; Tong *et al.*, 2000, 2002; Serna *et al.*, 2000), but few have a carboxylate ion as the counter-ion. As the metal carboxylate can be synthesized *in situ* by exchanging a halide with a carboxylate anion, the synthesis of the complex with nickel lactate was attempted.

The crystal structure of the title compound, (I), consists of  $[(\text{C}_{11}\text{H}_{10}\text{N}_2\text{O}_2)_2\text{Ni}]^{2+}$  cations and  $[\text{C}_3\text{H}_5\text{O}_3]^-$  anions (Fig. 1). The metal atom is chelated by the two ligands in an all-*trans* octahedral geometry, the ligand chelating in the  $N,O,N'$ -tridentate mode. The bond dimensions involving the metal atom are similar to those found in the corresponding sulfate complex (Wang *et al.*, 1986). The structure is consolidated by strong hydrogen bonds (Table 2); the hydrogen bonds connect the cation and anions into a helical chain that runs along the  $b$  axis.

## Experimental

Di-2-pyridyl ketone (0.192 g, 1.0 mmol) dissolved in water (2 ml) was mixed with an acetonitrile/water solution (4:1 *v/v*) of sodium lactate (0.110 g, 1.0 mmol) and nickel(II) chloride hexahydrate (0.120 g,

0.5 mmol). Green block-shaped crystals separated from the solution after several days.

Crystal data

[Ni(C<sub>11</sub>H<sub>10</sub>N<sub>2</sub>O<sub>2</sub>)<sub>2</sub>](C<sub>3</sub>H<sub>5</sub>O<sub>3</sub>)<sub>2</sub>  
*M<sub>r</sub>* = 641.27  
 Monoclinic, *P*2<sub>1</sub>/*n*  
*a* = 10.8401 (7) Å  
*b* = 17.359 (1) Å  
*c* = 15.611 (1) Å  
 β = 106.534 (1)°  
*V* = 2816.1 (3) Å<sup>3</sup>  
*Z* = 4

*D<sub>x</sub>* = 1.513 Mg m<sup>-3</sup>  
 Mo *K*α radiation  
 Cell parameters from 12650 reflections  
 θ = 1.8–27.5°  
 μ = 0.76 mm<sup>-1</sup>  
*T* = 293 (2) K  
 Block, green  
 0.36 × 0.32 × 0.26 mm

Data collection

Rigaku Mercury CCD area-detector diffractometer  
 ω scans  
 Absorption correction: multi-scan (*CrystalClear*; Rigaku/MS, 2002)  
*T<sub>min</sub>* = 0.764, *T<sub>max</sub>* = 0.823  
 16648 measured reflections

6277 independent reflections  
 5018 reflections with *I* > 2σ(*I*)  
*R<sub>int</sub>* = 0.029  
 θ<sub>max</sub> = 27.5°  
*h* = -14 → 13  
*k* = -19 → 22  
*l* = -13 → 19

Refinement

Refinement on *F*<sup>2</sup>  
*R*[*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.045  
*wR*(*F*<sup>2</sup>) = 0.112  
*S* = 1.01  
 6277 reflections  
 430 parameters  
 H-atom parameters constrained

*w* = 1/[σ<sup>2</sup>(*F<sub>o</sub>*<sup>2</sup>) + (0.0567*P*)<sup>2</sup> + 0.828*P*]  
 where *P* = (*F<sub>o</sub>*<sup>2</sup> + 2*F<sub>c</sub>*<sup>2</sup>)/3  
 (Δ/σ)<sub>max</sub> = 0.001  
 Δρ<sub>max</sub> = 0.40 e Å<sup>-3</sup>  
 Δρ<sub>min</sub> = -0.23 e Å<sup>-3</sup>

Table 1

Selected geometric parameters (Å, °).

Ni1—O1	2.088 (2)	Ni1—N2	2.046 (2)
Ni1—O3	2.093 (2)	Ni1—N3	2.050 (2)
Ni1—N1	2.093 (2)	Ni1—N4	2.092 (2)
O1—Ni1—O3	178.26 (6)	O3—Ni1—N4	77.12 (7)
O1—Ni1—N1	77.54 (7)	N1—Ni1—N2	86.69 (7)
O1—Ni1—N2	79.35 (7)	N1—Ni1—N3	92.74 (7)
O1—Ni1—N3	102.01 (7)	N1—Ni1—N4	178.67 (7)
O1—Ni1—N4	103.04 (7)	N2—Ni1—N3	178.39 (8)
O3—Ni1—N1	102.34 (7)	N2—Ni1—N4	94.59 (8)
O3—Ni1—N2	98.92 (7)	N3—Ni1—N4	85.97 (7)
O3—Ni1—N3	79.73 (6)		

Table 2

Hydrogen-bonding geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O1—H10...O6	0.85	1.67	2.494 (3)	164
O2—H20...O5	0.85	1.77	2.617 (3)	174
O3—H30...O8 <sup>i</sup>	0.85	1.73	2.561 (2)	167
O4—H40...O9 <sup>j</sup>	0.85	1.76	2.605 (3)	174
O7—H70...O8	0.85	2.29	2.999 (3)	141
O10—H100...O5	0.85	2.20	2.967 (3)	150

Symmetry codes: (i)  $\frac{1}{2} - x, y - \frac{1}{2}, \frac{3}{2} - z$ .

The two lactate anions show somewhat large displacement parameters and were satisfactorily modeled as disordered ions sharing a common -CO<sub>2</sub> unit and a hydroxyl O atom. The C—O distances were restrained to 1.25 (1) Å, the C—O<sub>hydroxyl</sub> distances to 1.45 (1) Å and the C—C distances to 1.50 (1) Å. The displacement parameters of the atoms of the minor disorder components [occupancies 0.652 (5) for O7 and 0.789 (5) for O10] were set equal to those of the

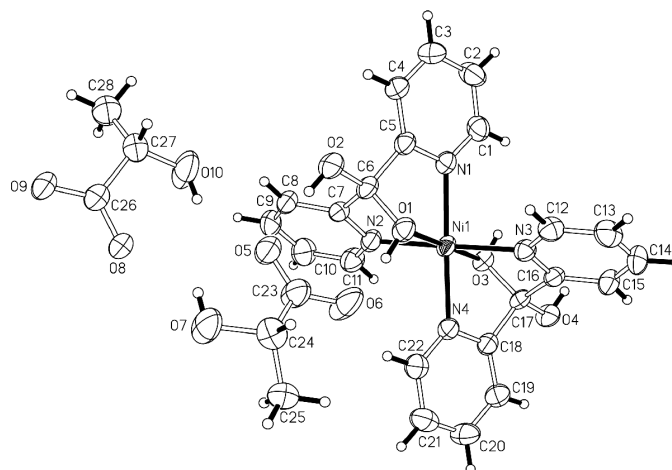


Figure 1

ORTEPII (Johnson, 1976) plot of (I). Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii. The two lactate ions are disordered only in their methyl and methine groups; the minor components are not shown.

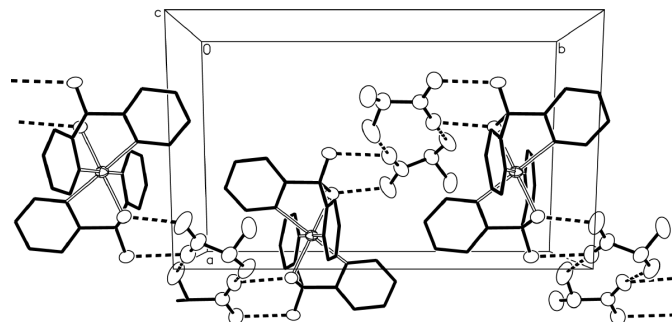


Figure 2

Schematic representation of the hydrogen-bonded (dashed lines) chain structure.

corresponding atoms in the major components [occupancies 0.35 for O7<sup>i</sup> and 0.21 for O10<sup>j</sup>].

H atoms were placed at calculated positions (aromatic C—H = 0.93 Å, methine C—H = 0.98 Å and methyl C—H = 0.96 Å) and were included in the refinement in the riding-model approximation, with *U<sub>iso</sub>*(H) values set at 1.2 or 1.5 times *U<sub>eq</sub>* of the parent atoms. The H atoms of the free ketal group were rotated along the O—H bond to fit the electron density [O—H = 0.85 Å and *U<sub>iso</sub>*(H) = 1.2*U<sub>eq</sub>*(O)] for the major component. Those belonging to the coordinated OH group were generated assuming *sp*<sup>3</sup> hybridization. The H atoms of the hydroxyl groups of the disordered lactate anions were similarly generated to fit the electron density.

Data collection: *CrystalClear* (Rigaku/MS, 2002); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

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