Acta Crystallographica Section E
**Structure Reports**
**Online**
ISSN 1600-5368

# Bis(butane-2,3-dione monooxime benzoylhydrazone-$\kappa^3O,N,N'$)nickel(II) sulfate salicylhydroxamic acid

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Received 21 October 2004
Accepted 25 October 2004
Online 6 November 2004

**Key indicators**

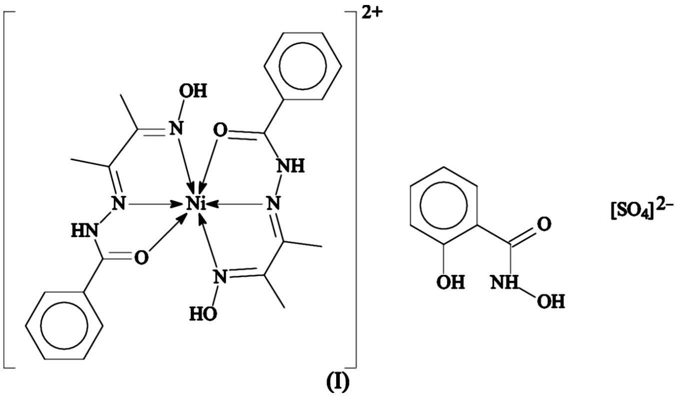
Single-crystal X-ray study
$T$ = 295 K
Mean $\sigma$(C–C) = 0.006 Å
$R$ factor = 0.059
$wR$ factor = 0.152
Data-to-parameter ratio = 16.6

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The Ni atom in the title compound, $[Ni(C_{11}H_{13}N_3O_2)_2]SO_4\cdot C_7H_7NO_3$, is $O,N,N'$-chelated by the two neutral hydrazone ligands in an octahedral environment. The cation interacts with the anion and the salicylhydroxamic acid molecule through hydrogen bonds to form a linear chain running along the $c$ axis.

## Comment

Butane-2,3-dione monooxime benzoylhydrazone in the deprotonated form chelates in a terdentate manner to the metal atom of a nickel(II) monoacetate entity; the oximate OH group is not deprotonated and is involved in hydrogen-bonding interactions in the resulting pyridine adduct (Huo *et al.*, 2004). The pyridine in the formulation arises from an excess of the reagent. In the present study, a similar reaction was attempted with a hydroxylamine-type ligand, *viz*. salicylhydroxamic acid, in place of pyridine, in the hope of synthesizing a nickel complex with two different ligands chelating to nickel. However, the reaction resulted in a bis(butane-2,3-dione monooxime benzoylhydrazone)nickel salt, (I), the salicylhydroxamic acid serving instead as a co-crystal component.



(I)

The Ni atom is $O,N,N'$-chelated by the two ligands in an octahedral geometry (Fig. 1); the cation, sulfate anion and the salicylhydroxamic acid molecule interact through hydrogen bonds (Table 2) to give a chain motif. The literature reports a case of salicylhydroxamic acid co-crystallizing with a coordination compound, the coordination compound being chlorobis(*o*-phenanthroline)copper(II) chloride (O'Brien *et al.*, 2000).

## Experimental

A methanol solution of nickel sulfate hexahydrate (0.26 g, 1 mmol) and salicylhydroxamic acid (0.15 g, 1 mmol) was added to a methanol solution (25 ml) of diacetyl monooxime benzoylhydrazone (0.22 g, 1 mmol). The mixture was heated for several hours. Yellow crystals were isolated from the filtered solution after several days.

### *Crystal data*

$[Ni(C_{11}H_{13}N_3O_2)_2]SO_4 \cdot C_7H_7NO_3$
$M_r = 746.39$
Monoclinic, $P2_1/c$
$a = 7.986$ (2) Å
$b = 23.781$ (5) Å
$c = 17.868$ (4) Å
$\beta = 102.82$ (3)°
$V = 3309$ (1) Å$^3$
$Z = 4$

$D_x = 1.498$ Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 21088 reflections
$\theta = 3.1$–27.4°
$\mu = 0.72$ mm$^{-1}$
$T = 295$ (2) K
Prism, yellow
$0.39 \times 0.27 \times 0.20$ mm

### *Data collection*

Rigaku R-AXIS RAPID diffractometer
$\omega$ scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{\min} = 0.591$, $T_{\max} = 0.870$
29244 measured reflections

7483 independent reflections
4963 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.069$
$\theta_{\max} = 27.5°$
$h = -9 \rightarrow 10$
$k = -30 \rightarrow 30$
$l = -23 \rightarrow 23$

### *Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.059$
$wR(F^2) = 0.152$
$S = 1.02$
7483 reflections
450 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0767P)^2 + 1.6714P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 1.19$ e Å$^{-3}$
$\Delta\rho_{\min} = -0.33$ e Å$^{-3}$

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| Ni1—O2 | 2.108 (2) | Ni1—N4 | 2.078 (3) |
| Ni1—O4 | 2.080 (2) | S1—O8 | 1.476 (3) |
| Ni1—N1 | 2.127 (3) | S1—O9 | 1.473 (3) |
| Ni1—N2 | 1.972 (3) | S1—O10 | 1.474 (3) |
| Ni1—N5 | 1.992 (3) | S1—O11 | 1.462 (3) |
| | | | |
| O2—Ni1—O4 | 90.3 (1) | N1—Ni1—N4 | 97.0 (1) |
| O2—Ni1—N1 | 153.6 (1) | N1—Ni1—N5 | 106.0 (1) |
| O2—Ni1—N2 | 77.5 (1) | N2—Ni1—N4 | 101.9 (1) |
| O2—Ni1—N4 | 93.2 (1) | N2—Ni1—N5 | 177.5 (1) |
| O2—Ni1—N5 | 99.9 (1) | N4—Ni1—N5 | 78.1 (1) |
| O4—Ni1—N1 | 90.6 (1) | O8—S1—O9 | 109.1 (2) |
| O4—Ni1—N2 | 102.8 (1) | O8—S1—O10 | 106.6 (2) |
| O4—Ni1—N4 | 155.3 (1) | O8—S1—O11 | 110.5 (2) |
| O4—Ni1—N5 | 77.2 (1) | O9—S1—O10 | 110.5 (2) |
| N1—Ni1—N2 | 76.6 (1) | O9—S1—O11 | 110.4 (2) |

**Table 2**
Hydrogen-bonding geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O1—H2*o*···O8 | 0.82 | 1.76 | 2.569 (4) | 171 |
| O3—H3*o*···O10$^{\rm i}$ | 0.82 | 1.86 | 2.626 (4) | 156 |
| N3—H3*n*···O11$^{\rm i}$ | 0.86 | 2.06 | 2.707 (4) | 132 |
| N6—H6*n*···O6 | 0.86 | 2.13 | 2.975 (4) | 170 |
| O5—H5*o*···O10 | 0.82 | 2.00 | 2.771 (4) | 157 |
| O7—H7*o*···O9$^{\rm ii}$ | 0.82 | 1.83 | 2.639 (4) | 168 |
| N7—H7*n*···O7 | 0.86 | 1.98 | 2.634 (5) | 132 |

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

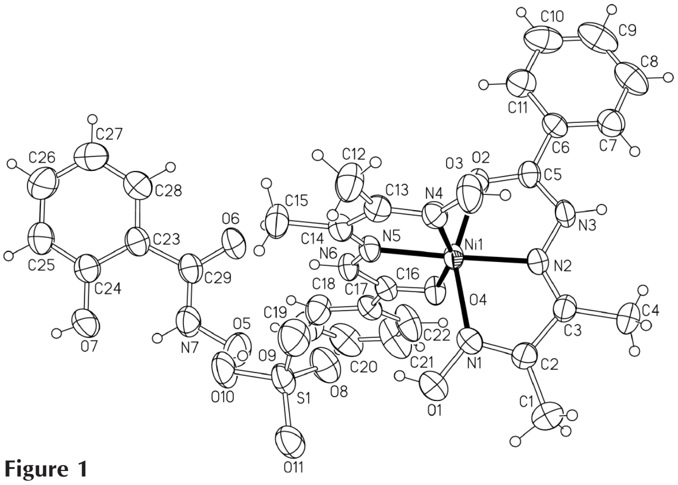


**Figure 1**
*ORTEP*II (Johnson, 1976) plot of (I) with displacement ellipsoids drawn at the 50% probability level. H atoms are drawn as spheres of arbitrary radii.

H atoms were placed in calculated positions [C—H = 0.93 Å, N—H = 0.86 Å and $U_{\rm iso}$(H) = $1.2U_{\rm eq}$(C,N)], whereas the hydroxy and methyl groups were rotated to to fit the electron density [C—H = 0.96 Å, O—H = 0.82 Å and $U_{\rm iso}$(H) = $1.5U_{\rm eq}$(C,O)]. They were included in the refinement in the riding-model approximation. The final difference Fourier map had a peak in a solvent-accessible void 2.8 Å from O11, but attempts to model the electron density led to a worse refinement.

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97.

We thank the National Natural Science Foundation of China (No. 20101003), the Scientific Fund for Remarkable Teachers of Heilongjiang Province (No. 1054G036) and the University of Malaya for supporting this study.

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