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# Bis(3-acetyl-6-methyl-2-oxo-2*H*-pyran-4-olato)bis(dimethyl sulfoxide)nickel(II)

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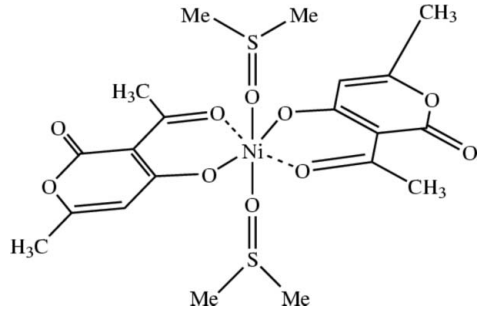
Key indicators: single-crystal X-ray study; $T$ = 100 K; mean $\sigma$(C–C) = 0.004 Å; $R$ factor = 0.037; $wR$ factor = 0.098; data-to-parameter ratio = 16.8.

In the title compound, $[Ni(C_8H_7O_4)_2\{(CH_3)_2SO\}_2]$, the $Ni^{II}$ atom is located on a crystallographic centre of symmetry and has a distorted octahedral coordination geometry of type $MO_6$. The bidentate dehydroacetic acid (DHA) ligands occupy the equatorial plane of the complex in a *trans* configuration, and the dimethyl sulfoxide (DMSO) ligands are weakly coordinated through their O atoms in the axial positions.

## Related literature

3-Acetyl-4-hydroxy-6-methyl-2-oxo-2*H*-pyran (dehydroacetic acid) (Arndt *et al.*, 1936) is a versatile starting material for the synthesis of a wide variety of heterocyclic ring systems (Tan & Ang, 1988). It has been shown to possess modest antifungal properties, see: Rao *et al.* (1978). For natural fungicides possessing structures analogous to 5,6-dihydrodehydroacetic acid, see: Bartels-Keith (1960); Miyakado *et al.* (1982); Ayer *et al.* (1988). The complexes of DHA with copper and with several other transition metal cations are fungistatic, see: Rao *et al.* (1978). For the nickel–DHA complex, see: Casabò *et al.* (1987). The configuration of the complex molecule is similar to that found in $[Zn(DHA)_2{\cdot}2(DMSO)$ and $Cd(DHA)_2{\cdot}2(DMSO)]$ (Zucolotto Chalaça *et al.*, 2002), $[Cu(DHA)_2{\cdot}2(DMSO)]$ (Djedouani *et al.*, 2006) and bis(4,6-dibromo-2-formylphenolato-$\kappa^2O,O'$)-bis(dimethyl sulfoxide)nickel(II) (Zhang *et al.*, 2007). For $Ni-O_{DMSO}$ distances in similar structures, see: Ma *et al.* (2003); Tahir *et al.* (2007); Zhang *et al.* (2007).



## Experimental

### Crystal data

$[Ni(C_8H_7O_4)_2(C_2H_6OS)_2]$
$M_r$ = 549.24
Monoclinic, $P2_1/c$
$a$ = 11.3850 (10) Å
$b$ = 6.2833 (4) Å
$c$ = 19.7434 (15) Å
$\beta$ = 123.525 (6)°
$V$ = 1177.40 (16) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 1.05 mm$^{-1}$
$T$ = 100 K
0.25 × 0.15 × 0.10 mm

### Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.902, $T_{max}$ = 0.902
14084 measured reflections
2628 independent reflections
1962 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.071

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.098
$S$ = 1.11
2628 reflections
156 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.55 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.97 e Å$^{-3}$

**Table 1**
Selected bond lengths (Å).

| | | | |
|---|---|---|---|
| Ni1—O2 | 1.9849 (16) | Ni1—O1 | 2.1255 (18) |
| Ni1—O3 | 2.0159 (15) | | |

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C1—H1$B$···O4$^{ii}$ | 0.96 | 2.55 | 3.384 (4) | 145 |
| C2—H2$B$···O4$^{ii}$ | 0.96 | 2.53 | 3.370 (4) | 146 |
| C2—H2$C$···O2$^{iii}$ | 0.96 | 2.46 | 3.378 (4) | 160 |

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

Data collection: *COLLECT* (Nonius, 2002); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg, 1998) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PARST* (Nardelli, 1995).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2559).

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# supporting information

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## Bis(3-acetyl-6-methyl-2-oxo-2*H*-pyran-4-olato)bis(dimethyl sulfoxide)nickel(II)

**Amel Djedouani, Sihem Boufas, Abderrahmen Bendaas, Magali Allain and Gilles Bouet**

### S1. Comment

3-Acetyl-4-hydroxy-6-methyl-2-oxo-2*H*-pyran (dehydroacetic acid) (Arndt *et al.*, 1936) is a versatile starting material for the synthesis of a wide variety of heterocyclic ring systems (Tan & Ang, 1988). It has been shown to possess modest antifungal properties (Rao *et al.*, 1978). The importance of similar pyrones as potential fungicides is reinforced by the existence of several natural fungicides possessing structures analogous to 5,6-dihydrodehydroacetic acid, such as alternaric acid (Bartels-Keith, 1960), the podoblastins (Miyakado *et al.*, 1982) and lachnelluloic acid (Ayer *et al.*, 1988). Also, it has been shown that the complexes of DHA with copper and with several other transition metal cations are fungistatic (Rao *et al.*, 1978). This has motivated our study of the structural characterization of complexes of dehydroacetic acid. The complex of DHA with nickel was previously reported by Casabò *et al.* (1987), but their characterization of the compound was based only on thermal and elemental analysis, and on IR and NMR spectroscopy.

We present here the crystal structure determination of the title complex, [Ni(DHA)$_2$.2(DMSO)], (I) (DMSO = dimethylsulfoxide). The nature of the title compound, (I), was established by an X-ray structure determination and is shown in Fig. 1

The Ni atom lies on a crystallographic centre of symmetry with the ligands bonded to nickel in an all-*trans* fashion. The configuration of the complex molecule is similar to that found in [Zn(DHA)$_2$. 2(DMSO); Cd(DHA)2.2(DMSO)] (Zucolotto Chalaça *et al.*, 2002), [Cu(DHA)$_2$. 2(DMSO)] (Djedouani *et al.*, 2006), with (DHA: dehydroacetic acid) and Bis(4,6-dibromo-2-formylphenolato-$\kappa^2$ *O*,*O*′)-bis(dimethyl sulfoxide)nickel(II), [Ni($C_7H_3Br_2O_2$)$_2$($C_2H_6OS$)$_2$] (Zhang *et al.*, 2007).

The coordination polyhedron around the Ni atom is a slightly distorted octahedron (Table 1), with the O atoms of the DMSO groups in axial positions; and the Ni—$O_{DMSO}$ distance is in agreement with literature values: [2.1139 (12) Å - 1.9897 (13) Å (Tahir *et al.*, 2007), 1.998 (3) Å - 2.105 (3) Å (Zhang *et al.* 2007), 2.030 (2) Å- 2.057 (2)Å (Ma *et al.*, 2003)].

The orientation of the DMSO molecule can be described by the torsion angles O3—Ni—O1—S [43.32 (4) °] and O2—Ni—O1—S [-137.70 (4) °]. The packing of (I) is stabilized by weak intermolecular C—H···O hydrogen bonds (Table 2) which form a three-dimensional network (Fig. 2).

### S2. Experimental

Compound (I) was prepared by the reaction of dehydroacetic acid with nickel (II) chloride hexahydrate in the presence of sodium acetate (Casabò *et al.* 1987). Crystals of (I) were grown by slow evaporation of a dimethylsulfoxide solution..

### S3. Refinement

H atoms were positioned geometrically and treated as riding, with C—H = 0.93 Å with $U_{iso}$(H) = 1.2Ueq(C). The methyl H atoms were constrained to an ideal geometry (C—H = 0.96 Å) with $U_{iso}$(H) = 1.2$U_{eq}$(C), but were allowed to rotate

freely about the C—C bonds.

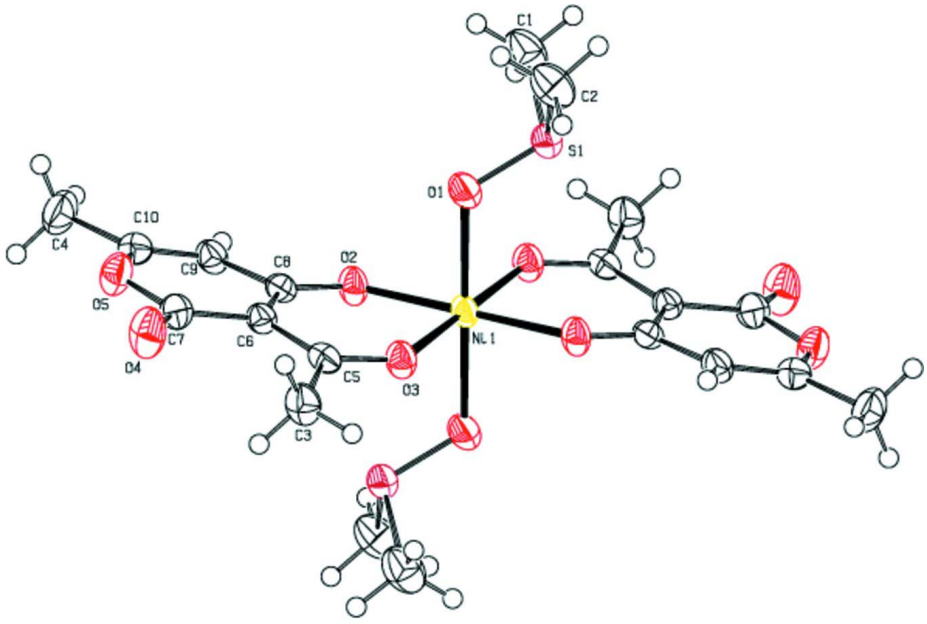


**Figure 1**

The independent components of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

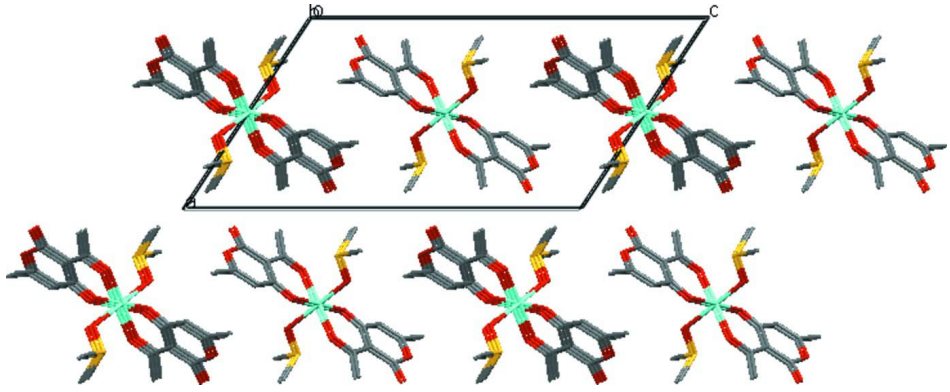


**Figure 2**

The crystal packing of (I); Hydrogen atoms have been omitted for clarity.

**Bis(3-acetyl-6-methyl-2-oxo-2*H*-pyran-4-olato)bis(dimethyl sulfoxide)nickel(II)**

*Crystal data*

$[Ni(C_8H_7O_4)_2(C_2H_6OS)_2]$
$M_r$ = 549.24
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a$ = 11.385 (1) Å
$b$ = 6.2833 (4) Å
$c$ = 19.7434 (15) Å
$\beta$ = 123.525 (6)°
$V$ = 1177.40 (16) Å$^3$
$Z$ = 2
$F$(000) = 572
$D_x$ = 1.549 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 2190 reflections
$\theta$ = 2.8–27.3°
$\mu$ = 1.05 mm$^{-1}$

$T$ = 100 K
Plates, colourless

0.25 × 0.15 × 0.1 mm

*Data collection*

Nonius KappaCCD diffractometer
Radiation source: fine-focus sealed X-ray tube
Graphite monochromator
$\varphi$ scans, and $\omega$ scans with $\kappa$ offsets
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
$T_{min}$ = 0.902, $T_{max}$ = 0.902

14084 measured reflections
2628 independent reflections
1962 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.071
$\theta_{max}$ = 27.5°, $\theta_{min}$ = 3.5°
$h$ = −14→14
$k$ = −8→7
$l$ = −25→25

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.098
$S$ = 1.11
2628 reflections
156 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
$w = 1/[\sigma^2(F_o^2 + 0.3428P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.55 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.97 e Å$^{-3}$
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0084 (19)

*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.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Ni1 | 0.5 | 0.5 | 0.5 | 0.02521 (16) |
| S1 | 0.75777 (6) | 0.76611 (10) | 0.53190 (4) | 0.03283 (19) |
| O5 | 0.22643 (18) | 0.1935 (3) | 0.18093 (9) | 0.0384 (4) |
| O1 | 0.62169 (17) | 0.6626 (3) | 0.46467 (10) | 0.0387 (4) |
| O2 | 0.47029 (16) | 0.2626 (3) | 0.42577 (9) | 0.0307 (4) |
| O3 | 0.32165 (16) | 0.6292 (3) | 0.40600 (9) | 0.0316 (4) |
| O4 | 0.1020 (2) | 0.4798 (3) | 0.15900 (11) | 0.0478 (5) |
| C6 | 0.2779 (2) | 0.4022 (4) | 0.29848 (13) | 0.0252 (5) |
| C3 | 0.1192 (3) | 0.7190 (4) | 0.28219 (15) | 0.0378 (6) |
| H3A | 0.1101 | 0.8133 | 0.3173 | 0.057* |
| H3B | 0.0368 | 0.631 | 0.2527 | 0.057* |
| H3C | 0.1291 | 0.801 | 0.2446 | 0.057* |
| C10 | 0.3256 (3) | 0.0457 (4) | 0.23083 (15) | 0.0317 (6) |
| C8 | 0.3859 (2) | 0.2500 (4) | 0.34964 (14) | 0.0253 (5) |
| C9 | 0.3994 (2) | 0.0651 (4) | 0.31075 (14) | 0.0296 (5) |
| H9 | 0.4616 | −0.0426 | 0.3428 | 0.036* |

| | | | | |
|---|---|---|---|---|
| C5 | 0.2474 (2) | 0.5807 (4) | 0.33237 (13) | 0.0265 (5) |
| C2 | 0.7743 (3) | 1.0036 (4) | 0.48915 (18) | 0.0469 (7) |
| H2A | 0.7626 | 0.9718 | 0.4382 | 0.07* |
| H2B | 0.8659 | 1.0642 | 0.5256 | 0.07* |
| H2C | 0.7034 | 1.1033 | 0.4805 | 0.07* |
| C7 | 0.1958 (2) | 0.3697 (4) | 0.21184 (14) | 0.0312 (6) |
| C4 | 0.3357 (3) | −0.1273 (5) | 0.18287 (17) | 0.0470 (7) |
| H4A | 0.4053 | −0.2289 | 0.2191 | 0.071* |
| H4B | 0.3623 | −0.0679 | 0.1484 | 0.071* |
| H4C | 0.246 | −0.1966 | 0.1501 | 0.071* |
| C1 | 0.8954 (3) | 0.6199 (5) | 0.53594 (19) | 0.0526 (8) |
| H1A | 0.8972 | 0.4776 | 0.5542 | 0.079* |
| H1B | 0.9841 | 0.6879 | 0.573 | 0.079* |
| H1C | 0.8792 | 0.6154 | 0.4828 | 0.079* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Ni1 | 0.0190 (2) | 0.0297 (3) | 0.0197 (2) | 0.00047 (17) | 0.00610 (17) | −0.00322 (16) |
| S1 | 0.0280 (3) | 0.0421 (4) | 0.0247 (3) | −0.0061 (3) | 0.0122 (3) | −0.0037 (3) |
| O5 | 0.0403 (10) | 0.0450 (11) | 0.0241 (9) | 0.0070 (8) | 0.0141 (8) | −0.0024 (7) |
| O1 | 0.0292 (9) | 0.0534 (11) | 0.0275 (9) | −0.0120 (8) | 0.0119 (8) | −0.0051 (8) |
| O2 | 0.0245 (8) | 0.0336 (9) | 0.0212 (8) | 0.0057 (7) | 0.0046 (7) | −0.0021 (7) |
| O3 | 0.0257 (8) | 0.0336 (10) | 0.0260 (9) | 0.0043 (7) | 0.0083 (7) | −0.0040 (7) |
| O4 | 0.0461 (12) | 0.0539 (12) | 0.0237 (9) | 0.0148 (10) | 0.0068 (8) | 0.0052 (8) |
| C6 | 0.0210 (11) | 0.0284 (13) | 0.0226 (11) | −0.0001 (10) | 0.0099 (9) | 0.0003 (9) |
| C3 | 0.0292 (13) | 0.0375 (15) | 0.0335 (13) | 0.0092 (11) | 0.0090 (11) | −0.0011 (11) |
| C10 | 0.0273 (13) | 0.0352 (14) | 0.0342 (13) | −0.0012 (11) | 0.0179 (11) | −0.0038 (10) |
| C8 | 0.0209 (11) | 0.0285 (12) | 0.0255 (11) | −0.0034 (10) | 0.0122 (9) | −0.0008 (9) |
| C9 | 0.0225 (12) | 0.0309 (13) | 0.0286 (13) | 0.0003 (10) | 0.0098 (10) | −0.0023 (10) |
| C5 | 0.0202 (11) | 0.0286 (13) | 0.0266 (12) | −0.0018 (10) | 0.0104 (10) | 0.0025 (9) |
| C2 | 0.0358 (15) | 0.0372 (16) | 0.0524 (17) | 0.0002 (12) | 0.0148 (13) | 0.0047 (12) |
| C7 | 0.0292 (13) | 0.0347 (14) | 0.0264 (12) | 0.0010 (11) | 0.0132 (10) | 0.0013 (10) |
| C4 | 0.0510 (17) | 0.0524 (18) | 0.0395 (15) | 0.0046 (15) | 0.0262 (14) | −0.0127 (13) |
| C1 | 0.0350 (15) | 0.0460 (18) | 0.0587 (19) | 0.0043 (14) | 0.0144 (14) | −0.0056 (14) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Ni1—O2$^{i}$ | 1.9849 (16) | C3—C5 | 1.507 (3) |
| Ni1—O2 | 1.9849 (16) | C3—H3A | 0.96 |
| Ni1—O3 | 2.0159 (15) | C3—H3B | 0.96 |
| Ni1—O3$^{i}$ | 2.0159 (15) | C3—H3C | 0.96 |
| Ni1—O1$^{i}$ | 2.1255 (18) | C10—C9 | 1.321 (3) |
| Ni1—O1 | 2.1255 (18) | C10—C4 | 1.487 (4) |
| S1—O1 | 1.5211 (17) | C8—C9 | 1.447 (3) |
| S1—C2 | 1.775 (3) | C9—H9 | 0.93 |
| S1—C1 | 1.780 (3) | C2—H2A | 0.96 |
| O5—C10 | 1.371 (3) | C2—H2B | 0.96 |

| | | | |
|---|---|---|---|
| O5—C7 | 1.398 (3) | C2—H2C | 0.96 |
| O2—C8 | 1.262 (3) | C4—H4A | 0.96 |
| O3—C5 | 1.250 (3) | C4—H4B | 0.96 |
| O4—C7 | 1.215 (3) | C4—H4C | 0.96 |
| C6—C8 | 1.440 (3) | C1—H1A | 0.96 |
| C6—C7 | 1.441 (3) | C1—H1B | 0.96 |
| C6—C5 | 1.443 (3) | C1—H1C | 0.96 |

| | | | |
|---|---|---|---|
| O2$^{i}$—Ni1—O2 | 180 | C9—C10—C4 | 127.3 (2) |
| O2$^{i}$—Ni1—O3 | 92.94 (6) | O5—C10—C4 | 111.1 (2) |
| O2—Ni1—O3 | 87.06 (6) | O2—C8—C6 | 125.9 (2) |
| O2$^{i}$—Ni1—O3$^{i}$ | 87.06 (6) | O2—C8—C9 | 116.6 (2) |
| O2—Ni1—O3$^{i}$ | 92.94 (6) | C6—C8—C9 | 117.4 (2) |
| O3—Ni1—O3$^{i}$ | 180.0000 (10) | C10—C9—C8 | 121.6 (2) |
| O2$^{i}$—Ni1—O1$^{i}$ | 89.72 (7) | C10—C9—H9 | 119.2 |
| O2—Ni1—O1$^{i}$ | 90.28 (7) | C8—C9—H9 | 119.2 |
| O3—Ni1—O1$^{i}$ | 89.31 (7) | O3—C5—C6 | 123.2 (2) |
| O3$^{i}$—Ni1—O1$^{i}$ | 90.69 (7) | O3—C5—C3 | 114.3 (2) |
| O2$^{i}$—Ni1—O1 | 90.28 (7) | C6—C5—C3 | 122.51 (19) |
| O2—Ni1—O1 | 89.72 (7) | S1—C2—H2A | 109.5 |
| O3—Ni1—O1 | 90.69 (7) | S1—C2—H2B | 109.5 |
| O3$^{i}$—Ni1—O1 | 89.31 (7) | H2A—C2—H2B | 109.5 |
| O1$^{i}$—Ni1—O1 | 180 | S1—C2—H2C | 109.5 |
| O1—S1—C2 | 105.60 (12) | H2A—C2—H2C | 109.5 |
| O1—S1—C1 | 105.41 (12) | H2B—C2—H2C | 109.5 |
| C2—S1—C1 | 97.65 (15) | O4—C7—O5 | 112.9 (2) |
| C10—O5—C7 | 121.85 (18) | O4—C7—C6 | 128.7 (2) |
| S1—O1—Ni1 | 116.97 (9) | O5—C7—C6 | 118.4 (2) |
| C8—O2—Ni1 | 129.32 (15) | C10—C4—H4A | 109.5 |
| C5—O3—Ni1 | 131.49 (15) | C10—C4—H4B | 109.5 |
| C8—C6—C7 | 118.8 (2) | H4A—C4—H4B | 109.5 |
| C8—C6—C5 | 121.45 (19) | C10—C4—H4C | 109.5 |
| C7—C6—C5 | 119.74 (19) | H4A—C4—H4C | 109.5 |
| C5—C3—H3A | 109.5 | H4B—C4—H4C | 109.5 |
| C5—C3—H3B | 109.5 | S1—C1—H1A | 109.5 |
| H3A—C3—H3B | 109.5 | S1—C1—H1B | 109.5 |
| C5—C3—H3C | 109.5 | H1A—C1—H1B | 109.5 |
| H3A—C3—H3C | 109.5 | S1—C1—H1C | 109.5 |
| H3B—C3—H3C | 109.5 | H1A—C1—H1C | 109.5 |
| C9—C10—O5 | 121.6 (2) | H1B—C1—H1C | 109.5 |

Symmetry code: (i) −*x*+1, −*y*+1, −*z*+1.

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C1—H1*B*···O4$^{ii}$ | 0.96 | 2.55 | 3.384 (4) | 145 |

| | | | | |
|---|---|---|---|---|
| C2—H2*B*···O4$^{ii}$ | 0.96 | 2.53 | 3.370 (4) | 146 |
| C2—H2*C*···O2$^{iii}$ | 0.96 | 2.46 | 3.378 (4) | 160 |

Symmetry codes: (ii) $x+1, -y+3/2, z+1/2$; (iii) $x, y+1, z$.