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Bis(dimethyl sulfoxide-κO)bis(1-phenylbutane-1,3-dionato-$\kappa^2 O,O'$)nickel(II)

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Acta Cryst. (2004). E**60**, m367–m369 Ernest Meštrović *et al.* · $[Ni(C_{10}H_9O_2)_2(C_2H_6OS)_2]$

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Bis(dimethyl sulfoxide-*κO*)bis(1-phenylbutane-1,3-dionato-κ^2O,O')nickel(II)

Ernest Meštrović, Ivan Halasz,* Dejan-Krešimir Bučar and Marijana Žgela

Laboratory of General and Inorganic Chemistry, Chemistry Department, Faculty of Science, University of Zagreb, Ul. kralja Zvonimira 8, HR-10002 Zagreb, Croatia

Correspondence e-mail: ihalasz@chem.pmf.hr

In the reaction of bis(1-phenylbutane-1,3-dionato)nickel(II) with dimethyl sulfoxide, the adduct bis(dimethyl sulfoxide)-bis(1-phenylbutane-1,3-dionato)nickel(II), $[Ni(C_{10}H_9O_2)_2(C_2H_6OS)_2]$, is formed, in which two dimethyl sulfoxide (DMSO) molecules are coordinated to the Ni atom through their O atoms in a *trans* arrangement. The compound crystallizes in space group $P2_1/a$ with the Ni atom lying on a center of inversion. The coordination polyhedron around the Ni atom is a distorted octahedron. The chelate ring deviates slightly from planarity. The Ni—O bond distance to the DMSO O atom is slightly longer than that to the 1-phenylbutane-1,3-dionate ligand. In the crystal structure, two-dimensional frameworks are formed by C–H···O interactions.

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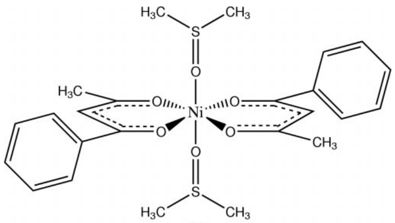
Key indicators

Single-crystal X-ray study
T = 100 K
Mean *σ*(C–C) = 0.003 Å
R factor = 0.035
wR factor = 0.064
Data-to-parameter ratio = 22.8

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

Comment

Metal complexes of *β*-diketones have been extensively studied. Most attention has been given to the simplest *β*-diketone, *viz*. acetylacetone. Appreciable attention has also been given to dibenzoylmethane, while structural studies of complexes of benzoylacetone are still quite rare. Adducts of *β*-diketone complexes with simple electron-donor molecules have proved to be good hosts for the formation of solvates. Since the addend is readily replaceable, the properties of such hosts can easily be varied. By choosing molecules that can form hydrogen bonds, extensive frameworks of intermolecular interactions can be formed.



(I)

The title complex, (I) has two molecules in the unit cell with the asymmetric unit consisting of half of one molecule; the Ni atom lies on an inversion center. Two 1-phenylbutane-1,3-dionate (bzac) anions form two chelate rings with nickel, leaving two coordination sites, which are occupied by O atoms from two dimethyl sulfoxide (DMSO) molecules. The chelate ring deviates slightly from planarity, with a dihedral angle between the Ni/O11/O12 and O11/O12/C12–C14 planes of 4.73 (5)°. The Ni—O(bzac) bond lengths are 1.9978 (12) and 2.0145 (12) Å, while the Ni—O(DMSO) bond length is longer, at 2.1087 (13) Å. In the Cambridge Structural Database (Release 5.25; Allen, 2002), there is only one entry containing

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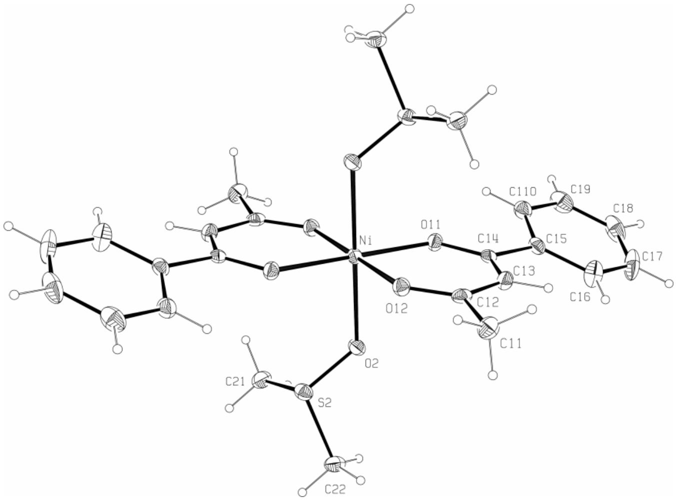

Figure 1
View of of the molecule of (I). Displacement ellipsoids are drawn at the 50% probability level.

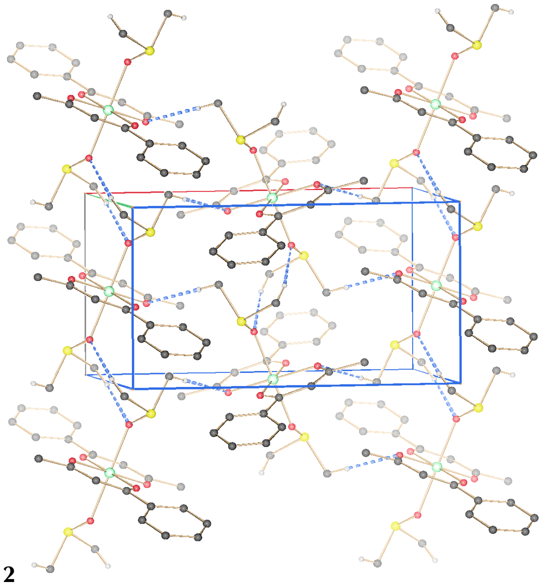

Figure 2
Packing of (I) in the crystal (*SCHAKAL*99; Keller, 1999). The *a* axis is grey, *b* red and *c* green. H atoms other than those involved in hydrogen bonding have been omitted for clarity.

a β-diketonate and oxygen-coordinated dimethyl sulfoxide (Zheng *et al.*, 1992). The reported Ni—O bond distances in the β-diketonate are in the range 1.923–1.941 Å and are shorter than in the title compound, while the Ni—O(DMSO) bond distance is longer, with a value of 2.268 Å. In (I), the dihedral angle between the O11/O12/C12–C14 chelate ring and the phenyl ring is 18.48 (6)°. The bite distance O11···O12 is 2.8431 (17) Å, which is slightly shorter then the value of 2.880 Å reported by Bučar & Meštrović (2003) and Yoshida *et al.* (1999).

Molecules in the crystal structure are linked by C—H···O hydrogen bonds, forming a two-dimensional framework (Table 1). Along the *a* axis, a hydrogen bond is formed through a methyl H atom and the O atom of the DMSO molecule, *viz*. C21—H21*C*···O2($-x$, $-y$, $1-z$). The other hydrogen bond is formed between an O atom of the bzac ligand and an H atom of the other methyl group of the same DMSO molecule, *viz*. C22—H22*A*···O12($-\frac{1}{2}+x$, $\frac{1}{2}-y$, z). The second hydrogen bond links molecules along the *b* axis. There are no interactions linking molecules of the two adjacent two-dimensional frameworks.

Experimental

Bis(1-phenylbutane-1,3-dionato)nickel(II) was prepared according to the method of Bučar & Meštrović (2003). It was dissolved in dimethyl sulfoxide and the resulting solution was filtered and left to evaporate slowly at room temperature. Single crystals appeared after five days.

Crystal data

$[Ni(C_{10}H_9O_2)_2(C_2H_6OS)_2]$
$M_r = 537.31$
Monoclinic, $P2_1/a$
$a = 7.3772$ (17) Å
$b = 13.453$ (2) Å
$c = 12.085$ (2) Å
$\beta = 92.026$ (18)°
$V = 1198.7$ (4) Å^3
$Z = 2$
$D_x = 1.489$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5096 reflections
$\theta = 2.1$–$21.3°$
$\mu = 1.02$ mm^{-1}
$T = 100.0$ (1) K
Block, green
0.30 × 0.14 × 0.10 mm

Data collection

Oxford Diffraction Xcalibur CCD diffractometer
ω scans
Absorption correction: analytical (Alcock, 1970)
$T_{min} = 0.818$, $T_{max} = 0.912$
32 533 measured reflections
3516 independent reflections
2561 reflections with $I > 2\sigma(I)$
$R_{int} = 0.060$
$\theta_{max} = 30.1°$
$h = -10 \rightarrow 10$
$k = -18 \rightarrow 18$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.064$
$S = 0.95$
3516 reflections
154 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0219P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.57$ e Å^{-3}
$\Delta\rho_{min} = -0.32$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ni—O11	1.9978 (12)	C12—C13	1.396 (3)
Ni—O12	2.0145 (13)	C13—C14	1.394 (2)
Ni—O2	2.1087 (13)	C14—C15	1.499 (2)
O12—C12	1.259 (2)	C15—C16	1.385 (3)
O11—C14	1.266 (2)	C15—C110	1.386 (3)
O2—S2	1.5131 (13)	C16—C17	1.381 (3)
S2—C22	1.7688 (18)	C17—C18	1.380 (3)
S2—C21	1.7772 (19)	C18—C19	1.368 (3)
C11—C12	1.510 (3)	C19—C110	1.379 (3)
O11—Ni—O12	90.24 (5)	C14—C13—C12	125.63 (17)
O11—Ni—O2	87.95 (5)	O11—C14—C13	125.34 (16)
O12—Ni—O2	89.00 (5)	O11—C14—C15	114.80 (16)
C12—O12—Ni	126.33 (12)	C13—C14—C15	119.86 (16)
C14—O11—Ni	126.60 (12)	C16—C15—C110	118.23 (18)
S2—O2—Ni	123.12 (7)	C16—C15—C14	123.18 (18)
O2—S2—C22	105.31 (8)	C110—C15—C14	118.52 (16)
O2—S2—C21	105.80 (9)	C17—C16—C15	120.1 (2)
C22—S2—C21	97.72 (9)	C18—C17—C16	120.6 (2)
O12—C12—C13	125.43 (18)	C19—C18—C17	119.9 (2)
O12—C12—C11	116.20 (17)	C18—C19—C110	119.5 (2)
C13—C12—C11	118.37 (17)	C19—C110—C15	121.63 (19)

H atoms were placed at geometrically calculated positions (C—H = 0.96 Å for methyl atoms and 0.93 Å for other H atoms) and were refined as part of a riding model, apart from the methyl H atoms, which were refined as a rigid rotor. The displacement parameters

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were set as follows: $U_{iso}(H) = 1.5U_{eq}(C)$ for methyl H atoms and $1.2U_{eq}(C)$ for H atoms on the phenyl ring.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2003); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2003); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997) and *SCHAKAL*99 (Keller, 1999); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PARST*97 (Nardelli, 1995).

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