

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

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Key indicators

Single-crystal X-ray study

$T = 100$ K

Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å

R factor = 0.035

wR factor = 0.064

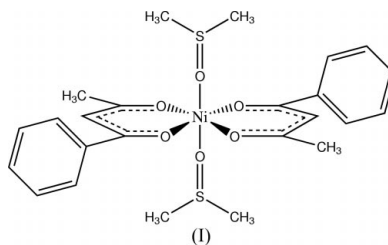
Data-to-parameter ratio = 22.8

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

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), $[\text{Ni}(\text{C}_{10}\text{H}_9\text{O}_2)_2(\text{C}_2\text{H}_6\text{OS})_2]$, 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 \cdots O interactions.

Comment

Metal complexes of β -diketones have been extensively studied. Most attention has been given to the simplest β -diketone, *viz.* acetylacetonone. Appreciable attention has also been given to dibenzoylmethane, while structural studies of complexes of benzoylacetonone 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.



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 $4.73(5)^\circ$. 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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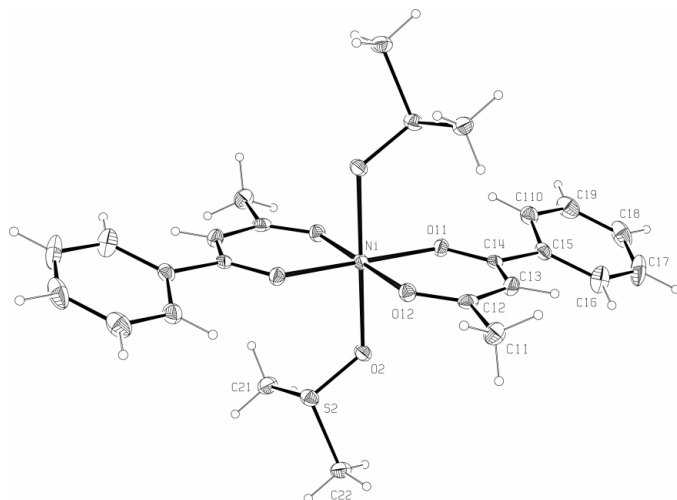


Figure 1
View of the molecule of (I). Displacement ellipsoids are drawn at the 50% probability level.

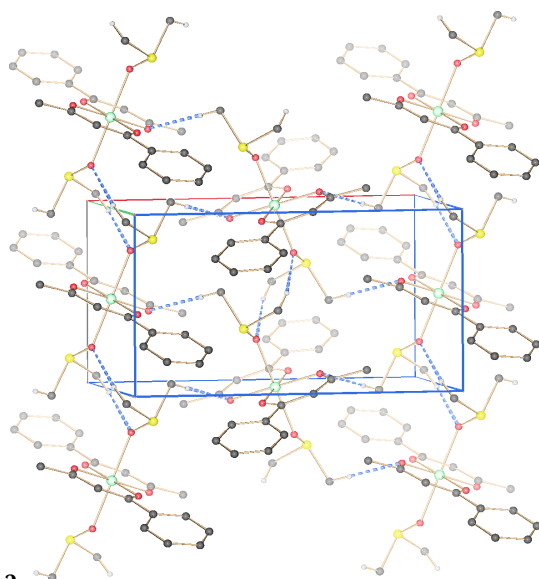


Figure 2
Packing of (I) in the crystal (SCHAKAL99; Keller, 1999). The *a* axis is grey, *b* red and *c* green. H atoms other than those involved in hydrogen bonding have been omitted for clarity.

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 than 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—H21C...O2(−*x*, −*y*, 1 − *z*). The other hydrogen bond is formed between an O atom of the *bz*ac

ligand and an H atom of the other methyl group of the same DMSO molecule, *viz.* C22—H22A...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(C₁₀H₉O₂)₂(C₂H₆OS)₂]
M_r = 537.31
 Monoclinic, *P*2₁/*a*
a = 7.3772 (17) Å
b = 13.453 (2) Å
c = 12.085 (2) Å
 β = 92.026 (18)°
V = 1198.7 (4) Å³
Z = 2

D_x = 1.489 Mg m^{−3}
 Mo *K*α radiation
 Cell parameters from 5096 reflections
 θ = 2.1–21.3°
 μ = 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* > 2σ(*I*)
R_{int} = 0.060
 θ_{\max} = 30.1°
h = −10 → 10
k = −18 → 18
l = −17 → 17

Refinement

Refinement on *F*²
R [*F*² > 2σ(*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 $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.57 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.32 \text{ e } \text{Å}^{-3}$

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—C16—C15 | 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

were set as follows: $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for methyl H atoms and $1.2U_{\text{eq}}(\text{C})$ 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: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *SCHAKAL99* (Keller, 1999); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PARST97* (Nardelli, 1995).

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References

- Alcock, N. W. (1970). *Crystallographic Computing*, edited by F. R. Ahmed, S. R. Hall & C. P. Huber, p. 271. Copenhagen: Munksgaard.
- Allen, F. H. (2002). *Acta Cryst.* **B58**, 380–388.
- Bučar, D.-K. & Meštrović, E. (2003). *Acta Cryst.* **E59**, m985–m987.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
- Keller, E. (1999). *SCHAKAL99*. University of Freiburg, Germany.
- Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
- Oxford Diffraction (2003). *CrysAlis CCD* and *CrysAlis RED*. Version 1.170. Oxford Diffraction Ltd, Abingdon, Oxfordshire, England.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
- Yoshida, T., Suzuki, T., Kanamori, K. & Kaizaki, S. (1999). *Inorg. Chem.* **38**, 1059–1068.
- Zheng, P.-J., Liu, W.-X., Xu, Z., Zhou, J.-Z., You, X.-Z., Yu, Y.-P. & Zhu, D.-L. (1992). *Chin. J. Struct. Chem.* **11**, 211.