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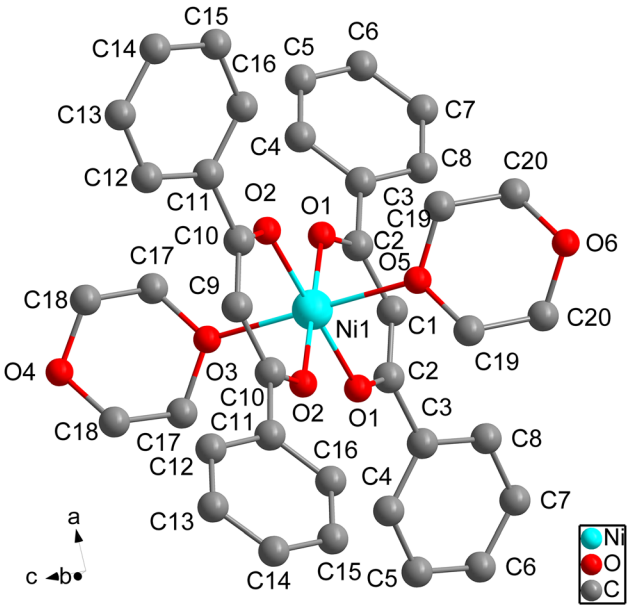
The crystal structure of bis(3-oxo-1,3-diphenylprop-1-en-1-olato-κ^2O:O')-bis(1,4-dioxane-κ^1O)nickel(II), $C_{38}H_{38}O_8Ni$



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Abstract

$C_{38}H_{38}O_8Ni$, orthorhombic, $Cmc2_1$ (no. 36), a = 19.0266(4) Å, b = 8.2409(2) Å, c = 21.4315(5) Å, V = 3,360.38(13) Å^3, Z = 4, $R_{gt}(F)$ = 0.0406, $wR_{ref}(F^2)$ = 0.1058, T = 293 K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colourless needle
Size:	0.16 × 0.06 × 0.05 mm
Wavelength:	Cu $K\alpha$ radiation (1.54184 Å)
μ:	1.27 mm^{-1}
Diffractometer, scan mode:	XtaLAB Synergy, ω
θ_{max}, completeness:	73.8°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	15,013, 3,348, 0.071
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 3,133
$N(param)_{refined}$:	223
Programs:	CrysAlis^{PRO},[1] Olex2,[2] SHELX[3,4]

1 Source of materials

1,3-diphenylpropane-1,3-dione (0.02 mol, 4.485 g) was dissolved in hot ethanol (48 ml) and nickel(II) acetate tetrahydrate (0.01 mol, 2.488 g) were mixed with ethanol (16 ml). Aqueous solution of 1,3-diphenylpropane-1,3-dione solution was fleetingly added to the ethanol mixture of nickel(II) acetate tetrahydrate, and stirred about 48 h at 78 °C. Followed by cooling in an ice bath, the formation of a light green precipitate results. The precipitate was filtered, washed with distilled hot water and ethanol, dried in an oven (90 °C). Subsequently, single crystals of nickel complexes were crystallized by slow evaporation from a mixture of 1,4-dioxane and ethanol.

2 Experimental details

Absorption corrections were performed by using multi-scan program.[1] The structure was solved with Olex2 and SHELX.[2–4] Hydrogen atoms were placed in their geometrically idealized positions. Hydrogen atoms were constrained to ride on their parent atoms.

3 Comment

Nickel compounds are widely used in batteries due to their safety advantages.[5,6] The study of nickel-containing complexes in the field of batteries is challenging but also extremely prospective.[7,8] Nickel-containing complex $Ni(acac)_2$

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	x	y	z	U_{iso}*/U_{eq}
C1	0.500000	0.6348 (5)	0.3927 (2)	0.0429 (10)
H1	0.500000	0.708002	0.359638	0.051*
C2	0.56557 (16)	0.5844 (4)	0.41536 (15)	0.0387 (7)
C3	0.62979 (16)	0.6661 (3)	0.38993 (16)	0.0395 (6)
C4	0.68599 (18)	0.6977 (4)	0.42930 (19)	0.0487 (7)
H4	0.684163	0.664307	0.470718	0.058*
C5	0.74455 (19)	0.7783 (6)	0.4075 (2)	0.0603 (10)
H5	0.781390	0.801260	0.434585	0.072*
C6	0.7490 (2)	0.8251 (5)	0.3459 (3)	0.0619 (12)
H6	0.788757	0.878914	0.331384	0.074*
C7	0.6942 (2)	0.7914 (5)	0.3061 (2)	0.0582 (9)
H7	0.697130	0.821415	0.264319	0.070*
C8	0.63444 (18)	0.7129 (5)	0.32792 (18)	0.0499 (8)
H8	0.597393	0.691593	0.300824	0.060*
C9	0.500000	0.0504 (6)	0.5974 (2)	0.0422 (9)
H9	0.500001	−0.026990	0.628960	0.051*
C10	0.43437 (16)	0.1036 (4)	0.57561 (15)	0.0372 (6)
C11	0.37038 (15)	0.0180 (3)	0.59904 (15)	0.0378 (6)
C12	0.36529 (17)	−0.0382 (5)	0.65994 (17)	0.0468 (7)
H12	0.402406	−0.022596	0.687534	0.056*
C13	0.3050 (2)	−0.1174 (6)	0.67972 (19)	0.0578 (9)
H13	0.301527	−0.153223	0.720755	0.069*
C14	0.2504 (2)	−0.1432 (6)	0.6390 (2)	0.0598 (11)
H14	0.210574	−0.198902	0.652337	0.072*
C15	0.25422 (18)	−0.0871 (5)	0.5785 (2)	0.0574 (9)
H15	0.216916	−0.103690	0.551219	0.069*
C16	0.31385 (16)	−0.0059 (4)	0.55856 (18)	0.0478 (7)
H16	0.316241	0.033071	0.517886	0.057*
C17	0.5619 (3)	0.5603 (6)	0.6056 (3)	0.0713 (12)
H17A	0.563937	0.677699	0.602844	0.086*
H17B	0.603186	0.516165	0.585230	0.086*
C18	0.5606 (4)	0.5095 (11)	0.6723 (3)	0.102 (2)
H18A	0.562238	0.391990	0.674648	0.122*
H18B	0.601921	0.551708	0.693248	0.122*
C19	0.4382 (2)	0.1224 (6)	0.3919 (2)	0.0628 (10)
H19A	0.396838	0.176140	0.408461	0.075*
H19B	0.436073	0.008797	0.403577	0.075*
C20	0.4393 (3)	0.1375 (8)	0.3226 (3)	0.0808 (14)
H20A	0.397283	0.088154	0.305269	0.097*
H20B	0.439436	0.251296	0.311029	0.097*
Ni1	0.500000	0.34709 (8)	0.49657 (4)	0.0366 (2)
O1	0.57604 (11)	0.4791 (3)	0.45726 (11)	0.0449 (5)
O2	0.42402 (11)	0.2140 (3)	0.53588 (11)	0.0435 (5)
O3	0.500000	0.5024 (5)	0.5755 (2)	0.0607 (10)
O4	0.500000	0.5655 (12)	0.7030 (3)	0.128 (3)
O5	0.500000	0.1946 (5)	0.41744 (19)	0.0568 (10)
O6	0.500000	0.0599 (8)	0.2973 (3)	0.0931 (17)

with β-diketone derivatives as ligands are also widely used in battery research.[9] In this paper, a β-diketone derivative nickel complex was successfully synthesized for further study of its application in the field of battery materials.

The title compound is a complex with nickel(II) as the central metal. The coordination sites of the compound was occupied by six oxygen atoms.[10] Four oxygen atoms are derived from the bidentate ligand 1,3-diphenylpropane-1,3-dione, the other oxygen atoms are derived from monodentate ligand 1,4-dioxane.[11]

In the title molecule, the distances of Ni1–O1 and Ni1–O1^1 bonds both are 1.997(2) Å, the distances of Ni1–O2 and Ni1–O2^1 bonds both are 2.001(2) Å, the distance of Ni1–O3 bond is 2.121(4) Å, the distance of Ni1–O3^1 bond is 2.111(4) Å.[12]

In addition, the O2^1–Ni1–O3 angle is 89.72(12)°, the O2–Ni1–O3 angle is 89.72(12)°, the O2–Ni1–O2^1 angle is 92.53(13)°, the O2–Ni1–O5 angle is 90.68(12)°, the O2^1–Ni1–O5 angle is 90.68(12)°, the O1–Ni1–O3 angle is 90.44(12)°, the O1^1–Ni1–O3 angle is 90.44(12)°, the O1–Ni1–O2 angle is 179.77(13)°, the O1–Ni1–O2^1 angle is 87.30(8)°, the O1^1–Ni1–O2 angle is 87.30(8)°, the O1^1–Ni1–O2^1 angle is 179.77(13)°, the O1–Ni1–O^1 angle is 92.86(13)°, the O1–Ni1–O5 angle is 89.16(12)°, the O1^1–Ni1–O5 angle is 89.16(12)°, and the O5–Ni1–O3 angle is 179.4(2)°.[13]

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