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The crystal structure of (2,3-dioxo-5,6:13,14-dibenzo-9,10-benzo-1,4,8,11-7, 11-diene- $\kappa^4 N, N', N'', N'''$)-nickel(II), $\text{Ni}(\text{C}_{22}\text{H}_{14}\text{N}_4\text{O}_2)$

Table 1: Data collection and handling.

Crystal:	Red block
Size:	0.18 × 0.11 × 0.08 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	1.18 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max} , completeness:	26.0°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	11583, 3259, 0.050
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2873
$N(\text{param})_{\text{refined}}$:	262
Programs:	Bruker [1], Olex2 [2], SHELX [3, 4]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Ni1	0.57515(16)	0.51060(4)	0.38413(6)	0.0178(2)
O1	0.4173(14)	0.7128(3)	0.4320(5)	0.0351(12)
O2	0.3905(16)	0.6371(3)	0.6498(6)	0.0401(15)
N1	0.7579(13)	0.4890(3)	0.2400(5)	0.0201(12)
N2	0.6858(13)	0.4172(3)	0.4344(5)	0.0190(11)
N3	0.4068(13)	0.5330(3)	0.5308(5)	0.0202(12)
N4	0.4600(13)	0.6036(3)	0.3310(5)	0.0216(12)
C1	0.3278(16)	0.4857(3)	0.6238(6)	0.0220(14)
C2	0.1479(14)	0.5066(4)	0.7189(5)	0.0213(12)
H2	0.081388	0.554933	0.722775	0.026*
C3	0.0664(18)	0.4586(4)	0.8061(7)	0.0293(16)
H3	−0.058037	0.474269	0.868150	0.035*
C4	0.1626(18)	0.3872(4)	0.8057(7)	0.0283(16)
H4	0.112998	0.354836	0.868285	0.034*
C5	0.3332(17)	0.3651(4)	0.7107(7)	0.0249(15)
H5	0.394083	0.316433	0.706820	0.030*
C6	0.4177(15)	0.4133(4)	0.6202(6)	0.0195(15)
C7	0.5988(16)	0.3831(4)	0.5298(6)	0.0216(14)
H7	0.660130	0.334436	0.540325	0.026*
C8	0.8732(15)	0.3818(3)	0.3522(6)	0.0195(13)
C9	1.0010(16)	0.3130(4)	0.3683(6)	0.0227(14)
H9	0.963245	0.284829	0.438383	0.027*
C10	1.1821(16)	0.2866(4)	0.2814(7)	0.0253(14)
H10	1.269150	0.239849	0.291693	0.030*
C11	1.2392(16)	0.3279(4)	0.1778(7)	0.0237(17)
H11	1.367637	0.309387	0.119407	0.028*
C12	1.1097(15)	0.3956(3)	0.1603(6)	0.0213(13)
H12	1.147348	0.423484	0.089821	0.026*

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Abstract

$\text{C}_{22}\text{H}_{14}\text{N}_4\text{NiO}_2$, monoclinic, $P2_1$ (no. 4), $a = 4.2387(5)$ Å, $b = 18.724(2)$ Å, $c = 10.7345(13)$ Å, $\beta = 97.751(4)^\circ$, $V = 844.17(17)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0435$, $wR_{\text{ref}}(F^2) = 0.1255$, $T = 150$ 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.

Source of material

The title compound was prepared by a method described in the literature [5]: a mixture of 1,1'-oxalylbisatin (1.04 g, 30 mmol), 1,2-diaminobenzene (0.32 g, 30 mmol) and nickel

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Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
C13	0.9227(15)	0.4225(3)	0.2474(6)	0.0198(13)
C14	0.7251(16)	0.5253(4)	0.1357(6)	0.0253(16)
H14	0.825839	0.506768	0.068733	0.030*
C15	0.5526(16)	0.5903(4)	0.1125(6)	0.0216(13)
C16	0.5099(17)	0.6149(3)	−0.0122(7)	0.0244(14)
H16	0.602872	0.589216	−0.074296	0.029*
C17	0.3356(17)	0.6757(4)	−0.0461(7)	0.0281(15)
H17	0.301587	0.691006	−0.131226	0.034*
C18	0.2116(17)	0.7137(4)	0.0459(7)	0.0282(15)
H18	0.094066	0.756076	0.023370	0.034*
C19	0.2544(18)	0.6917(4)	0.1715(7)	0.0249(17)
H19	0.166196	0.719219	0.232638	0.030*
C20	0.4246(16)	0.6299(3)	0.2080(6)	0.0212(13)
C21	0.4304(17)	0.6469(3)	0.4305(6)	0.0243(15)
C22	0.4065(18)	0.6052(4)	0.5520(7)	0.0258(16)

acetate (0.74 g, 30 mmol) in 40 mL H₂O was stirred at room temperature. The pH of the solution was adjusted to 9 with 10% NaOH solution and then heated at 85 °C for 6 h. The mixture was filtered, the red solid residue was added to *N,N*-dimethylformamide (10 mL) and the solution stirred at 120 °C for 2 h. The resulting solution was filtered at room temperature and stored for two weeks to give crystals suitable for single-crystal X-ray diffraction.

Experimental details

All the hydrogen atoms were positioned geometrically and treated as riding, with C—H = 0.94 Å and *U*_{iso}(H) = 1.2 times *U*_{eq} (C) for all H atoms. The Flack parameter refined to −0.006(10) and was determined using 1253 quotients [6].

Comment

The current interest in coordination polymers encompasses the design of heterometallic complexes [5–8]. The metalloligand strategy is a useful and well established approach to obtain such heterometallic compounds [9, 10]. Macrocyclic oxamide metalloligands with two oxygen atoms of the oxamido group are active in chelating metal ions, and extensively employed in the preparation of functional heterobimetallic coordination polymers [11–13].

In the title structure, the central Ni(II) cation is coordinated by four nitrogen atoms which reside in a distorted square-planar environment. The Ni—N bond lengths range from 1.862(5) to 1.876(5) Å. All bond lengths and angles are within the expected ranges. The four N donor atoms (N1–N4) deviate from their corresponding least-squares mean plane

by −0.023 Å, 0.023 Å, −0.023 Å, 0.023 Å, respectively. The distance of the Ni(II) ion to this mean plane is 0.009 Å. The mononuclear metalloligand shows a geometry resembling a flying butterfly. Both carbonyl O-atoms are involved in extensive C—H···O hydrogen bonds.

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