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The crystal structure bis(dimethylsulfoxide- $\kappa^1\text{O}$)-dipyridine- $\kappa^1\text{N}$ -bis(m_2 -(Z)-3-methyl-2-oxido- N -((Z)-oxido(phenyl)methylene)benzohydrazonato- κ^5)trinickel(II) - dimethylsulfoxide (1/2), $\text{C}_{48}\text{H}_{56}\text{N}_6\text{Ni}_3\text{O}_{10}\text{S}_4$

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Abstract

$\text{C}_{48}\text{H}_{56}\text{N}_6\text{Ni}_3\text{O}_{10}\text{S}_4$, monoclinic, $P2_1$ (no. 4), $a = 12.3887(11)$ Å, $b = 15.7405(13)$ Å, $c = 13.4254(11)$ Å, $\beta = 97.364(2)^\circ$, $V = 2596.4(4)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0530$, $wR_{\text{ref}}(F^2) = 0.1316$, $T = 298$ 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:	Orange block
Size:	0.26 × 0.16 × 0.09 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	1.30 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max} , completeness:	25.4°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	31,672, 9473, 0.033
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 8511
$N(\text{param})_{\text{refined}}$:	651
Programs:	Bruker [1], SHELX [2]

Source of material

The ligand was synthesized from the reaction of benzoyl hydrazine (100 mg, 0.60 mmol) and benzoate (50 mg, 0.60 mmol) according to the procedure reported earlier [3]. Yield (0.010 g, 80%).

Experimental details

The carbon-bound H atoms were geometrically placed ($\text{C-H} = 0.95\text{--}0.98$ Å) and refined as riding atoms with $U_{\text{iso}}(\text{H}) = 1.2\text{--}1.5 U_{\text{eq}}(\text{C})$. The N-bound H atoms were located in a difference Fourier map but were refined with a distance

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

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
Ni1	0.76582 (8)	0.49779 (7)	0.75836 (8)	0.0210 (2)
S1	1.3983 (2)	−0.03224 (19)	0.6767 (2)	0.0529 (7)
O1	1.0417 (5)	0.3337 (4)	0.7435 (4)	0.0277 (14)
N1	0.9014 (5)	0.4185 (5)	0.7745 (5)	0.0225 (14)
C1	0.9377 (6)	0.3500 (5)	0.7363 (6)	0.0210 (18)
Ni2	0.41087 (8)	0.58432 (6)	0.68313 (8)	0.0243 (3)
S2	−0.0169 (3)	1.01300 (19)	0.7134 (2)	0.0522 (7)
O2	1.2013 (4)	0.4943 (4)	0.8929 (4)	0.0271 (12)
N2	0.9910 (5)	0.4637 (4)	0.8272 (5)	0.0217 (15)
C2	0.8638 (7)	0.2863 (5)	0.6814 (6)	0.0245 (18)
Ni3	1.12251 (8)	0.41435 (6)	0.81799 (8)	0.0245 (3)
S3	0.6872 (3)	0.43471 (19)	0.9768 (2)	0.0581 (8)
O3	0.8649 (5)	0.5594 (4)	0.8649 (4)	0.0248 (13)
N3	0.5433 (5)	0.5343 (5)	0.6812 (5)	0.0218 (15)
C3	0.7606 (7)	0.2713 (6)	0.7062 (6)	0.0261 (18)
H3	0.734463	0.302795	0.758601	0.031*
S4	0.78961 (19)	0.55382 (16)	0.53825 (17)	0.0334 (5)
O4	0.6904 (5)	0.4270 (4)	0.8664 (4)	0.0297 (13)
N4	0.6310 (5)	0.5768 (5)	0.7384 (5)	0.0243 (15)
C4	0.6952 (8)	0.2100 (6)	0.6542 (7)	0.036 (2)
H4	0.623946	0.200340	0.670808	0.043*
O5	0.8393 (5)	0.5659 (4)	0.6474 (4)	0.0298 (14)
N5	1.2550 (6)	0.3544 (6)	0.8025 (6)	0.0338 (19)
C5	0.7322 (8)	0.1626 (6)	0.5784 (7)	0.034 (2)
H5	0.687117	0.120533	0.543484	0.041*
O6	0.6688 (4)	0.4362 (4)	0.6494 (4)	0.0225 (13)
N6	0.2779 (6)	0.6455 (5)	0.6917 (6)	0.0304 (17)
C6	0.8358 (8)	0.1776 (6)	0.5544 (7)	0.036 (2)
H6	0.861470	0.145683	0.502157	0.043*
O7	0.4907 (5)	0.6657 (4)	0.7597 (5)	0.0277 (14)
C7	0.9027 (7)	0.2382 (6)	0.6049 (7)	0.032 (2)
H7	0.974080	0.247223	0.588338	0.038*
O8	0.3339 (4)	0.5040 (4)	0.6070 (4)	0.0263 (12)
C8	0.9636 (7)	0.5326 (5)	0.8717 (6)	0.0221 (17)
O9	0.0253 (7)	0.9277 (6)	0.7400 (7)	0.067 (2)
C9	1.0511 (7)	0.5817 (6)	0.9304 (6)	0.0243 (17)
O10	1.3235 (6)	0.0176 (5)	0.7314 (6)	0.060 (2)
C10	1.0201 (8)	0.6554 (6)	0.9807 (6)	0.031 (2)
H10	0.945441	0.670737	0.975823	0.037*
C11	1.0972 (9)	0.7051 (7)	1.0366 (8)	0.039 (2)
H11	1.075701	0.753051	1.072185	0.047*
C12	1.2059 (8)	0.6843 (6)	1.0402 (7)	0.037 (2)
H12	1.258494	0.720058	1.076943	0.044*
C13	1.2412 (8)	0.6141 (6)	0.9931 (7)	0.034 (2)
C14	1.1613 (7)	0.5602 (6)	0.9366 (6)	0.0276 (19)
C15	1.3599 (8)	0.5926 (7)	0.9948 (9)	0.048 (3)
H15A	1.403606	0.628943	1.043749	0.071*
H15B	1.381586	0.602024	0.927977	0.071*
H15C	1.371837	0.532909	1.013790	0.071*
C16	0.5718 (6)	0.4629 (5)	0.6369 (6)	0.0200 (17)
C17	0.4854 (6)	0.4156 (6)	0.5736 (6)	0.0229 (17)
C18	0.3734 (7)	0.4378 (6)	0.5640 (6)	0.0253 (19)
C19	0.2986 (7)	0.3857 (6)	0.5029 (7)	0.034 (2)
C20	0.3356 (8)	0.3175 (7)	0.4528 (8)	0.042 (3)
H20	0.284429	0.284046	0.411121	0.051*

Table 2: (continued)

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
C21	0.4461 (8)	0.2957 (7)	0.4611 (7)	0.042 (3)
H21	0.469897	0.247940	0.426462	0.051*
C22	0.5196 (7)	0.3460 (6)	0.5214 (6)	0.028 (2)
H22	0.594974	0.332774	0.527263	0.034*
C23	0.1791 (7)	0.4065 (8)	0.4978 (8)	0.042 (2)
H23A	0.136576	0.365970	0.453335	0.063*
H23B	0.165962	0.464212	0.471605	0.063*
H23C	0.157018	0.402859	0.565246	0.063*
C24	0.5940 (7)	0.6469 (6)	0.7731 (6)	0.0266 (19)
C25	0.6658 (7)	0.7114 (6)	0.8263 (6)	0.0263 (18)
C26	0.7700 (7)	0.7277 (6)	0.8012 (7)	0.029 (2)
H26	0.797981	0.694071	0.751592	0.035*
C27	0.8324 (9)	0.7925 (6)	0.8483 (7)	0.037 (2)
H27	0.903072	0.803427	0.831104	0.044*
C28	0.7911 (9)	0.8417 (7)	0.9212 (7)	0.043 (3)
H28	0.833643	0.886612	0.953021	0.051*
C29	0.6899 (9)	0.8258 (7)	0.9471 (7)	0.041 (2)
H29	0.662653	0.858934	0.997464	0.049*
C30	0.6272 (8)	0.7610 (6)	0.8996 (7)	0.033 (2)
H30	0.556760	0.750329	0.917620	0.039*
C31	1.2559 (9)	0.2717 (7)	0.7887 (9)	0.050 (3)
H31	1.187592	0.243540	0.778434	0.060*
C32	1.3463 (10)	0.2235 (8)	0.7881 (10)	0.053 (3)
H32	1.341797	0.163421	0.782126	0.063*
C33	1.4449 (8)	0.2648 (7)	0.7964 (8)	0.041 (2)
H33	1.510545	0.233657	0.796288	0.049*
C34	1.4471 (8)	0.3504 (8)	0.8047 (7)	0.041 (2)
H34	1.514017	0.380427	0.807654	0.050*
C35	1.3511 (8)	0.3935 (7)	0.8087 (7)	0.037 (2)
H35	1.353340	0.453533	0.816176	0.045*
C36	0.1864 (7)	0.6082 (7)	0.6940 (7)	0.036 (2)
H36	0.185235	0.547923	0.690991	0.044*
C37	0.0872 (8)	0.6506 (8)	0.7008 (8)	0.041 (2)
H37	0.020857	0.620360	0.700999	0.049*
C38	0.0910 (9)	0.7357 (8)	0.7069 (9)	0.046 (3)
H38	0.026706	0.766891	0.714025	0.055*
C39	0.1908 (9)	0.7795 (8)	0.7029 (9)	0.051 (3)
H39	0.194080	0.839769	0.703720	0.061*
C40	0.2804 (9)	0.7320 (7)	0.6978 (9)	0.046 (3)
H40	0.348434	0.760026	0.698385	0.055*
C41	1.4835 (13)	−0.0881 (11)	0.7746 (13)	0.096 (5)
H41A	1.554798	−0.098898	0.752905	0.144*
H41B	1.449232	−0.142284	0.788251	0.144*
H41C	1.492643	−0.053510	0.835784	0.144*
C42	1.4928 (12)	0.0404 (10)	0.6434 (13)	0.091 (5)
H42A	1.564565	0.013329	0.648105	0.136*
H42B	1.496372	0.089243	0.688954	0.136*
H42C	1.470992	0.059595	0.574322	0.136*
C43	0.0669 (10)	1.0866 (8)	0.7899 (8)	0.054 (3)
H43A	0.035366	1.143627	0.781536	0.081*
H43B	0.140155	1.086907	0.769640	0.081*
H43C	0.071198	1.069548	0.860542	0.081*
C44	−0.1352 (10)	1.0264 (8)	0.7740 (9)	0.055 (3)
H44A	−0.164353	1.083730	0.760936	0.083*
H44B	−0.116335	1.018276	0.846545	0.083*
H44C	−0.190186	0.984438	0.747993	0.083*

Table 2: (continued)

Atom	x	y	z	U_{iso}^*/U_{eq}
C45	0.8152 (11)	0.4154 (9)	1.0406 (9)	0.067 (3)
H45A	0.816302	0.431116	1.111402	0.100*
H45B	0.869295	0.449250	1.010875	0.100*
H45C	0.832658	0.354937	1.035827	0.100*
C46	0.6709 (9)	0.5443 (7)	1.0073 (7)	0.040 (2)
H46A	0.673338	0.550411	1.080182	0.060*
H46B	0.600651	0.564782	0.974116	0.060*
H46C	0.729739	0.577617	0.984249	0.060*
C47	0.8575 (10)	0.4620 (7)	0.4985 (8)	0.046 (3)
H47A	0.846252	0.458576	0.425019	0.069*
H47B	0.827842	0.410948	0.526799	0.069*
H47C	0.935544	0.466114	0.521913	0.069*
C48	0.8554 (10)	0.6322 (7)	0.4717 (8)	0.047 (3)
H48A	0.826978	0.629660	0.400167	0.071*
H48B	0.933984	0.621318	0.480079	0.071*
H48C	0.841605	0.688609	0.498090	0.071*

restraint of N–H = 0.88 Å, and with $U_{iso}(H)$ set to $1.2 U_{eq}(N)$ [1, 4].

Comment

Schiff bases ligands have been extensively investigated for many years. Lots of the ligands can form multinuclear metal complexes, but Schiff based ligands are better applicants as they own versatile structures depending on the aldehydes and amines. The structures of their metal complexes with Schiff base ligands are similar with that of biological system. So the Schiff base ligand plays an important part in the development of coordination chemistry. In the following account, we synthesized a new Schiff base ligand. Similar complexes has been reported [5–7].

As shown in the figure, the title complex is composed of three Ni atoms, two Schiff base ligands, two dimethyl sulfoxides and two pyridines. It is the first trinuclear Ni complex with the (*Z*)-3-methyl-2-oxido-*N*-((*Z*)-oxido(phenyl)methylene)benzohydrazonate ligand. The coordination geometry of Ni1 is octahedral and those of Ni2 and Ni3 are square planar. The Ni1 atoms is bound to two oxygens, two hydrazine nitrogens, of two ligands and two sulfur atoms of dimethyl sulfoxides. The bond lengths of Ni1–O are 2.011, 2.018, 2.132 and 2.137 Å, which are similar to the corresponding values [8]. For Ni2 and Ni3, the regular square planar geometry is realized with oxygen, hydrazine nitrogen, phenolic oxygen and pyridine nitrogen atoms. The average

bond distances of Ni–N and Ni–O for Ni2 and Ni3 are 1.874 and 1.833 respectively, which is shorter than the distances of Ni1 atoms. This difference may be attributed to the difference in stereochemistry between the central and terminal Ni atoms. The neighboring Ni–Ni interatomic distances are 4.581 and 4.591 Å, respectively, which are shorter than those in related complexes [9].

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