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N',N''' -(((ethane-1,2-diylbis(oxy))bis(2,1-phenylene))bis(methaneylylidene))bis(2-hydroxybenzohydrazide)nickel(II), $C_{30}H_{24}N_4NiO_6$

Table 1: Data collection and handling.

Crystal:	Green block
Size:	0.26 × 0.23 × 0.19 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.78 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$\theta_{\max}$, completeness:	27.1°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	25704, 5925, 0.038
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 4465
$N(\text{param})_{\text{refined}}$:	372
Programs:	Bruker [1], Olex2 [2], SHELX [3]

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	0.65144(3)	0.40716(3)	0.26147(3)	0.03935(10)
O1	0.5282(2)	0.8661(2)	0.32221(16)	0.0810(7)
H1	0.510348	0.792199	0.280080	0.121*
O2	0.75370(16)	0.59455(16)	0.36644(14)	0.0463(4)
O3	0.47137(16)	0.22311(16)	0.16405(14)	0.0473(4)
O4	0.57450(16)	0.35071(17)	0.38161(14)	0.0475(4)
O6	0.72072(16)	0.38333(16)	0.14241(14)	0.0455(4)
O7	1.04440(19)	0.2132(2)	0.19092(14)	0.0601(5)
H7	1.010064	0.235157	0.233919	0.090*
N1	0.54941(19)	0.62983(18)	0.26290(15)	0.0397(4)
N2	0.51591(19)	0.49997(18)	0.19744(15)	0.0372(4)
N3	0.79987(19)	0.32613(18)	0.32299(16)	0.0391(4)
N4	0.8562(2)	0.28228(19)	0.25083(16)	0.0417(4)
C1	0.6399(3)	0.8877(2)	0.4163(2)	0.0498(6)
C2	0.7129(2)	0.7957(2)	0.43259(19)	0.0414(5)
C3	0.8235(3)	0.8268(3)	0.5340(2)	0.0628(8)
H3	0.871144	0.765573	0.547160	0.075*
C4	0.8656(3)	0.9446(3)	0.6157(2)	0.0749(9)
H4	0.939717	0.962515	0.683172	0.090*
C5	0.7958(4)	1.0355(3)	0.5959(2)	0.0700(9)
H5	0.825782	1.117425	0.649337	0.084*
C6	0.6838(3)	1.0069(3)	0.4992(2)	0.0650(8)
H6	0.635704	1.068109	0.488185	0.078*
C7	0.6719(2)	0.6657(2)	0.34979(19)	0.0390(5)
C8	0.4142(2)	0.4596(2)	0.1025(2)	0.0426(5)
H8	0.367808	0.519275	0.080946	0.051*
C9	0.3667(2)	0.3255(2)	0.0257(2)	0.0416(5)
C10	0.2876(3)	0.3071(3)	−0.0842(2)	0.0512(6)

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Abstract

$C_{30}H_{24}N_4NiO_6$, triclinic, $P\bar{1}$ (no. 2), $a = 10.5870(2)$ Å, $b = 10.6944(2)$ Å, $c = 13.6830(3)$ Å, $\alpha = 100.700(1)^\circ$, $\beta = 110.090(1)^\circ$, $\gamma = 104.735(1)^\circ$, $Z = 2$, $V = 1342.02(5)$ Å³, $R_{\text{gt}}(F) = 0.0388$, $wR_{\text{ref}}(F^2) = 0.0925$, $T = 298(2)$ 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

Synthesis of the ligand H_2L : All solvents and other reagents were of analytical grade. Synthesis of the ligand was prepared by a similar method reported earlier [4]. 152.15 mg

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Table 2 (continued)

Atom	x	y	z	U_{iso}^*/U_{eq}
H10	0.264931	0.379232	−0.105203	0.061*
C11	0.2422(3)	0.1847(3)	−0.1624(2)	0.0619(7)
H11	0.190675	0.175104	−0.235426	0.074*
C12	0.2736(3)	0.0767(3)	−0.1320(3)	0.0693(8)
H12	0.243963	−0.005851	−0.184647	0.083*
C13	0.3493(3)	0.0911(3)	−0.0229(3)	0.0627(8)
H13	0.369044	0.017672	−0.002346	0.075*
C14	0.3950(2)	0.2135(2)	0.0547(2)	0.0454(6)
C15	0.3800(3)	0.1940(3)	0.2214(2)	0.0643(8)
H15A	0.312554	0.101867	0.187460	0.077*
H15B	0.326532	0.255597	0.219678	0.077*
C16	0.4756(3)	0.2120(3)	0.3358(2)	0.0666(8)
H16A	0.419418	0.194237	0.377920	0.080*
H16B	0.527878	0.149466	0.337235	0.080*
C17	0.6764(2)	0.3773(2)	0.4877(2)	0.0452(6)
C18	0.6421(3)	0.4171(3)	0.5739(2)	0.0609(7)
H18	0.553389	0.426485	0.561002	0.073*
C19	0.7391(3)	0.4432(3)	0.6795(2)	0.0646(8)
H19	0.715809	0.471083	0.737571	0.078*
C20	0.8697(3)	0.4282(3)	0.6994(2)	0.0566(7)
H20	0.934552	0.445163	0.770581	0.068*
C21	0.9041(3)	0.3879(3)	0.6133(2)	0.0496(6)
H21	0.992672	0.377612	0.627217	0.060*
C22	0.8092(2)	0.3622(2)	0.5056(2)	0.0429(5)
C23	0.8559(3)	0.3209(3)	0.4203(2)	0.0471(6)
H23	0.931934	0.288559	0.437900	0.057*
C24	0.8047(2)	0.3146(2)	0.1595(2)	0.0394(5)
C25	0.8485(2)	0.2647(2)	0.07244(19)	0.0400(5)
C26	0.9642(2)	0.2174(2)	0.0917(2)	0.0434(5)
C27	0.9992(3)	0.1726(3)	0.0062(2)	0.0549(7)
H27	1.077156	0.143008	0.019340	0.066*
C28	0.9211(3)	0.1712(3)	−0.0970(2)	0.0640(8)
H28	0.946788	0.141396	−0.153280	0.077*
C29	0.8039(3)	0.2136(3)	−0.1189(2)	0.0653(8)
H29	0.749550	0.210777	−0.189563	0.078*
C30	0.7692(3)	0.2602(3)	−0.0342(2)	0.0535(6)
H30	0.690884	0.289334	−0.048418	0.064*

2-hydroxylbenzohydrazide (0.02 mmol) was dissolved in 10 mL of ethanol. 270.28 mg dialdehyde (0.01 mmol), was dissolved in 10 mL of ethanol, added to the above solution, stirred and refluxed at 55 °C for 6 h. A large amount of white solid appeared in the solution; this was filtered and dried to obtain a ligand. Yield: 73.5%. Synthesis of the nickel(II) complex: A methanol solution (3.0 mL) of nickel(II) acetate (3.73 mg, 0.015 mmol) was added to a chloroform solution (2.0 mL) of H₂L (5.95 mg, 0.010 mmol) at room temperature. After the mixture was stirred for 20 min, the mixture was filtered off. The resulting filtrate was left undisturbed for about several days to form crystals suitable for X-ray crystallographic analysis. Yield: 57.2%. Elemental analysis-Anal. Calcd. for C₃₀H₂₄N₄NiO₆: C, 60.54%; H, 4.06%; N, 9.41%. Found: C, 60.78%; H, 4.04%; N, 9.38%.

Experimental details

Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms.

Comment

Generally, Schiff bases are synthesized through the condensation reaction of aldehydes or ketones with primary amines [4–6]. Schiff base compounds and their metal complexes have been studied for many years [7–10], and successfully applied in many fields. For example, they have been used as antibacterial agents in the medical field. In addition these compounds show antiseptic, anti-tumor and anti-viral biological activity [11, 12]. Schiff base compounds and their derivatives have become indispensable in the development of metal complexes in modern coordination chemistry [13, 14], in classical catalytic processes [15], in the development of compounds with biological activity [16], for photochemical [17, 18] and magnetic materials [19] and supramolecular architectures [20]. X-ray crystallographical analysis reveals that the title compound crystallizes in the triclinic, space group $P\bar{1}$. The crystal structure of the title compound is built up by C₃₀H₂₄N₄NiO₆ molecules, in which all bond lengths and angles are in normal ranges. There are three intramolecular hydrogen bonding interactions (O1–H1···N1, O7–H7···N4 and C3–H3···O2) and one intermolecular hydrogen bonding interaction (C21–H21···O2) that contribute to the stability of the structure.

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