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Crystal structure of bis(1-(phenylsulfonyl)-2-(1-(pyrazin-2-yl)ethylidene)hydrazin-1-ido- $\kappa^3 N, N', O$) nickel(II), $C_{24}H_{22}N_8O_4S_2Ni$

Table 1: Data collection and handling.

Crystal:	Black block
Size:	0.24 × 0.23 × 0.18 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.97 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω -scans
$\theta_{\max}$, completeness:	25°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	6228, 2194, 0.029
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1928
$N(\text{param})_{\text{refined}}$:	177
Programs:	Bruker programs [1], SHELX [2]

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

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Ni1	0.000000	0.000000	0.88968(9)	0.0314(2)
S1	-0.10160(8)	-0.10123(5)	0.72933(12)	0.0374(3)
N3	-0.1643(2)	-0.02033(15)	0.8758(4)	0.0322(8)
C5	-0.2338(3)	0.01256(18)	0.9431(4)	0.0359(11)
C4	-0.1807(3)	0.0558(2)	1.0346(5)	0.0384(11)
C7	-0.0848(3)	-0.1635(2)	0.8406(5)	0.0401(11)
N1	-0.0652(3)	0.05755(16)	1.0330(3)	0.0376(9)
N2	-0.1885(4)	0.1329(2)	1.2028(5)	0.0729(15)
C6	-0.3589(3)	0.0071(2)	0.9283(5)	0.0510(15)
H6A	-0.395310	0.035658	0.987362	0.076*
H6B	-0.379842	0.015620	0.836603	0.076*
H6C	-0.381939	-0.033559	0.951385	0.076*
C10	-0.0595(6)	-0.2629(3)	1.0045(7)	0.0729(17)
H10A	-0.051394	-0.297267	1.058668	0.087*
C8	-0.1735(4)	-0.1832(2)	0.9153(7)	0.0640(16)
H8A	-0.242438	-0.163077	0.910780	0.077*
C3	-0.2383(5)	0.0936(2)	1.1218(6)	0.0566(13)
H3A	-0.316631	0.091309	1.123558	0.068*
C9	-0.1607(5)	-0.2334(3)	0.9982(7)	0.085(2)
H9A	-0.221279	-0.247063	1.049733	0.101*
C11	0.0286(5)	-0.2421(3)	0.9321(6)	0.0672(16)
H11A	0.097537	-0.262271	0.937097	0.081*
C2	-0.0775(6)	0.1324(2)	1.1998(5)	0.0683(18)
H2B	-0.038842	0.158714	1.257109	0.082*
C12	0.0189(4)	-0.1918(2)	0.8513(5)	0.0531(14)
H12A	0.081055	-0.176966	0.804305	0.064*
N4	-0.2042(3)	-0.06091(16)	0.7826(4)	0.0388(9)
O2	-0.1302(3)	-0.12590(14)	0.6006(3)	0.0541(9)
O1	0.0057(2)	-0.06807(15)	0.7362(3)	0.0426(8)
C1	-0.0154(4)	0.0951(2)	1.1164(5)	0.0476(12)
H1A	0.063035	0.096620	1.119430	0.057*

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Abstract

$C_{24}H_{22}N_8O_4S_2Ni$, orthorhombic, *Aba2* (no. 41), $a = 11.845(6)$ Å, $b = 21.937(11)$ Å, $c = 9.907(5)$ Å, $V = 2574(2)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0285$, $wR_{\text{ref}}(F^2) = 0.0657$, $T = 296(2)$ K.

CCDC no.: 1830030

The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

Benzenesulfonyl hydrazide (0.172 g, 1 mmol) and 2-acetopyrazine (0.122 g, 1 mmol) were dissolved in methanol (20 mL). The reaction mixture was refluxed for 1 h and cooled to room temperature. Then nickel(II) acetate (0.088 g, 0.5 mmol) was added. After stirring for 1 h, the mixture was

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filtered and set aside to crystallize for several days, giving black block crystals.

Experimental details

The structure was solved by direct methods and refined with the SHELX crystallographic software package [2]. The hydrogen atoms were placed at calculated positions and refined as riding atoms with isotropic displacement parameters. The Flack-Parsons parameter was refined to 0.025(11) based on 799 reflections.

Discussion

Schiff bases, including hydrazones [3], semicarbazones [4] and thiosemicarbazones [5] derived from 2-aceto-pyrazine and their metal complexes have been widely investigated due to their potential biological activities. However, sulfonyl hydrazones has been paid much less attention.

In the title structure, the asymmetric unit contains one half of the complex with the Ni1 atom lying on the two fold rotational axis. The six-coordinated Ni(II) ion is in a distorted octahedron geometry with the donor sets of four nitrogen and two oxygen atoms from two independent sulfonyl hydrazone ligands. The dihedral angle between the benzene (C7—C12,

r.m.s. deviation 0.011 Å) and pyrazine (C1—C4/N1,N2, r.m.s. deviation 0.008 Å) ring in each sulfonyl hydrazone is 83.0°. As expected, there exist none classical hydrogen bonds in the crystal. Ni—O, Ni—N bond lengths are all in the expected ranges.

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