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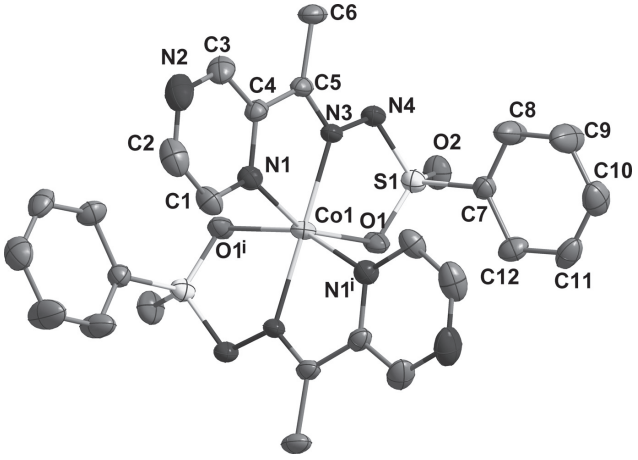
Crystal structure of bis [1-(phenylsulfonyl)-2-(1-(pyrazin-2-yl)ethylidene)hydrazin-1-ido-κ^3*N*,*N*′,*O*] cobalt(II), $C_{24}H_{22}N_8O_4S_2Co$



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Abstract

$C_{24}H_{22}N_8O_4S_2Co$, orthorhombic, *Aba*2 (no. 41), $a = 12.043(5)$ Å, $b = 21.723(9)$ Å, $c = 9.961(4)$ Å, $V = 2605.9(18)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0366$, $wR_{ref}(F^2) = 0.0764$, $T = 296(2)$ 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

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

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Table 1: Data collection and handling.

Crystal:	Black block
Size:	0.24 × 0.23 × 0.18 mm
Wavelength:	Mo Kα radiation (0.71073 Å)
μ:	0.87 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	25.0°, >99%
N(hkl)$_{measured}$, N(hkl)$_{unique}$, R_{int}:	6416, 2220, 0.046
Criterion for I_{obs}, N(hkl)$_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1836
$N(param)_{refined}$:	177
Programs:	SHELX [1], Bruker [2]

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Co1	0.5000	0.5000	0.61170(11)	0.0345(3)
S1	0.60263(10)	0.60434(6)	0.76987(15)	0.0401(3)
N1	0.5729(4)	0.4438(2)	0.4643(4)	0.0422(11)
N2	0.6984(6)	0.3691(2)	0.2973(6)	0.0786(19)
N3	0.6655(3)	0.52172(18)	0.6270(5)	0.0351(10)
N4	0.7042(3)	0.5622(2)	0.7221(5)	0.0408(12)
O1	0.4955(2)	0.57233(17)	0.7585(4)	0.0443(10)
O2	0.6272(4)	0.62934(19)	0.8988(4)	0.0588(12)
C1	0.5258(5)	0.4049(3)	0.3798(7)	0.0529(16)
H1A	0.4488	0.4025	0.3753	0.064*
C2	0.5895(7)	0.3678(3)	0.2982(6)	0.071(2)
H2B	0.5535	0.3405	0.2409	0.086*
C3	0.7450(6)	0.4090(3)	0.3805(7)	0.0605(17)
H3A	0.8220	0.4118	0.3816	0.073*
C4	0.6858(4)	0.4463(3)	0.4652(6)	0.0397(14)
C5	0.7358(4)	0.4898(2)	0.5591(5)	0.0383(14)
C6	0.8589(4)	0.4966(3)	0.5754(6)	0.0531(18)
H6A	0.8961	0.4687	0.5158	0.080*
H6B	0.8802	0.5381	0.5546	0.080*
H6C	0.8791	0.4873	0.6664	0.080*
C7	0.5917(4)	0.6669(3)	0.6563(5)	0.0400(14)
C8	0.6817(5)	0.6861(3)	0.5871(9)	0.077(2)
H8A	0.7492	0.6658	0.5964	0.092*
C9	0.6728(6)	0.7363(4)	0.5024(9)	0.095(3)
H9A	0.7346	0.7496	0.4546	0.114*
C10	0.5747(6)	0.7661(3)	0.4885(8)	0.072(2)
H10A	0.5695	0.8005	0.4332	0.087*

Table 2 (continued)

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C11	0.4848(6)	0.7457(3)	0.5551(6)	0.0609(19)
H11A	0.4171	0.7656	0.5434	0.073*
C12	0.4912(5)	0.6963(3)	0.6396(6)	0.0518(17)
H12A	0.4285	0.6827	0.6853	0.062*

cooled to room temperature. Then cobalt(II) acetate tetrahydrate (0.125 g, 0.5 mmol) was added. After stirring for 1 h, the mixture was 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 [1]. The hydrogen atoms were placed at calculated positions and refined as riding atoms with isotropic displacement parameters.

Comment

Schiff bases, including hydrozones [3], semicarbazones [4] and thiosemicarbazones [5] derived from 2-aceto-pyrazine and their metal complexes have been widely investigated due to their excellent biological activities. However, the sulfonyl hydrazones have been paid much less attention. Our previous work reported the crystal structures of Cu(II) and Ni(II) complexes with *N′*-(1-(pyrazin-2-yl)-ethylidene)benzenesulfonyl hydrazide [6, 7].

The title complex is isostructural with the Cu(II) and Ni(II) ones with the same ligand. In brief, the asymmetric unit contains a half of the complex with Co1 atom lying on the two fold rotational axis. The six-coordinated Co(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 0.0076 Å) and pyrazine (C1—C4/N1,N2, r.m.s. deviation 0.0078 Å) ring is 89.8°. As expected, there exist no classical hydrogen bonds in the crystal.

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