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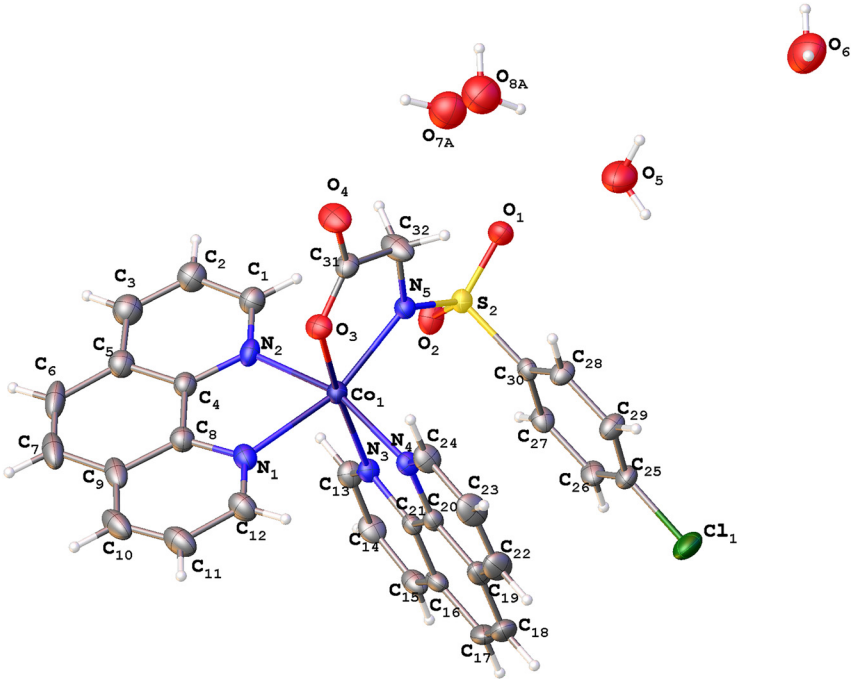
Crystal structure of (((4-chlorophenyl)sulfonyl) glycinato-κ^2 *N*,*O*)bis(1,10-phenanthroline-κ^2 *N*,*N*′) cobalt(II) tetrahydrate, $C_{32}H_{30}ClCoN_5O_8S$



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Abstract

$C_{32}H_{30}ClCoN_5O_8S$, monoclinic, $P2_1/c$ (no. 14), a = 14.5008(9) Å, b = 13.6648(9) Å, c = 17.9741(12) Å, β = 113.086(2)°, V = 3276.4(4) Å^3, Z = 4, $R_{gt}(F)$ = 0.0620, $wR_{ref}(F^2)$ = 0.1696, T = 293(2) K.

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Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

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Table 1: Data collection and handling.

Crystal:	Green block
Size:	0.27 × 0.15 × 0.14 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.73 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	27.5°, 99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	51,167, 7520, 0.057
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 5061
$N(param)_{refined}$:	452
Programs:	Bruker,[1] Olex2,[2] SHELX[3,4]

1 Source of material

In a 250 ml round-bottom flask, a solution was prepared by dissolving glycine (7.51 g, 100 mmol), sodium hydroxide (8.00 g, 200 mmol), and 100 ml of deionized water with thorough stirring to ensure complete solubilization. Subsequently, 4-chlorobenzenesulfonyl chloride (21.11 g, 100 mmol) was introduced into the mixture, which was then stirred continuously at ambient temperature for a duration of 10 h to facilitate the sulfonylation reaction. Upon completion of the

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.3588 (3)	0.2079 (3)	0.4876 (2)	0.0530 (10)
H1	0.347188	0.273553	0.473215	0.064*
C2	0.4032 (4)	0.1843 (4)	0.5698 (3)	0.0656 (12)
H2	0.421054	0.232823	0.609226	0.079*
C3	0.4193 (4)	0.0881 (4)	0.5902 (3)	0.0697 (12)
H3	0.447468	0.070681	0.644581	0.084*
C4	0.3501 (3)	0.0468 (3)	0.4509 (2)	0.0447 (8)
C5	0.3949 (3)	0.0163 (3)	0.5324 (3)	0.0557 (10)
C6	0.4122 (4)	−0.0868 (4)	0.5484 (4)	0.0767 (16)
H6	0.440360	−0.108344	0.601663	0.092*
C7	0.3887 (4)	−0.1526 (4)	0.4883 (4)	0.0828 (18)
H7	0.401893	−0.218381	0.501209	0.099*
C8	0.3244 (3)	−0.0242 (3)	0.3873 (2)	0.0484 (9)
C9	0.3441 (3)	−0.1247 (3)	0.4053 (3)	0.0628 (13)
C10	0.3201 (4)	−0.1894 (3)	0.3400 (4)	0.0759 (16)
H10	0.333129	−0.255861	0.349340	0.091*
C11	0.2778 (4)	−0.1552 (3)	0.2631 (4)	0.0759 (15)
H11	0.261148	−0.197875	0.219481	0.091*
C12	0.2597 (3)	−0.0558 (3)	0.2506 (3)	0.0608 (11)
H12	0.230649	−0.033218	0.197573	0.073*
C13	0.0867 (3)	0.1718 (3)	0.3572 (2)	0.0466 (9)
H13	0.134923	0.180679	0.409218	0.056*
C14	−0.0128 (3)	0.1661 (3)	0.3473 (3)	0.0528 (10)
H14	−0.030089	0.171396	0.391822	0.063*
C15	−0.0846 (3)	0.1526 (3)	0.2727 (3)	0.0521 (10)
H15	−0.151519	0.147618	0.265511	0.063*
C16	−0.0576 (3)	0.1462 (2)	0.2058 (2)	0.0435 (8)
C17	−0.1279 (3)	0.1344 (3)	0.1246 (3)	0.0571 (11)
H17	−0.195831	0.129700	0.114107	0.068*
C18	−0.0978 (3)	0.1300 (3)	0.0629 (3)	0.0615 (12)
H18	−0.145469	0.123127	0.010372	0.074*
C19	0.0055 (3)	0.1358 (3)	0.0762 (2)	0.0499 (9)
C20	0.0776 (3)	0.1473 (2)	0.1556 (2)	0.0366 (7)
C21	0.0448 (2)	0.1526 (2)	0.2215 (2)	0.0346 (7)
C22	0.0426 (4)	0.1304 (4)	0.0150 (3)	0.0660 (12)
H22	−0.001519	0.122160	−0.038534	0.079*
C23	0.1419 (4)	0.1371 (4)	0.0335 (3)	0.0679 (13)
H23	0.166230	0.133373	−0.007216	0.082*
C24	0.2083 (3)	0.1498 (3)	0.1140 (2)	0.0535 (10)
H24	0.276507	0.155173	0.125850	0.064*
C25	−0.0442 (3)	0.3980 (3)	0.0829 (2)	0.0487 (9)
C26	−0.0548 (3)	0.3946 (3)	0.1556 (3)	0.0504 (10)
H26	−0.118299	0.391451	0.156755	0.060*
C27	0.0293 (3)	0.3961 (3)	0.2268 (2)	0.0443 (9)
H27	0.022621	0.393241	0.276169	0.053*
C28	0.1331 (3)	0.4070 (3)	0.1518 (2)	0.0441 (9)
H28	0.196465	0.411718	0.150636	0.053*
C29	0.0493 (3)	0.4053 (3)	0.0803 (2)	0.0509 (10)
H29	0.055847	0.409052	0.030915	0.061*
C30	0.1236 (2)	0.4017 (2)	0.2248 (2)	0.0353 (7)
C31	0.4471 (3)	0.2491 (3)	0.2928 (2)	0.0446 (9)
C32	0.3967 (3)	0.3382 (3)	0.3093 (4)	0.0691 (14)
H32A	0.438877	0.365609	0.361362	0.083*
H32B	0.388943	0.387339	0.268271	0.083*
Cl1	−0.14930 (8)	0.39317 (10)	−0.00707 (8)	0.0761 (4)
Co1	0.26804 (3)	0.16814 (3)	0.30081 (3)	0.03432 (15)
N1	0.2816 (2)	0.0092 (2)	0.3101 (2)	0.0474 (8)
N2	0.3327 (2)	0.1422 (2)	0.42965 (18)	0.0448 (7)
N3	0.1173 (2)	0.1655 (2)	0.29682 (16)	0.0361 (6)
N4	0.1766 (2)	0.1544 (2)	0.17391 (17)	0.0399 (7)
N5	0.2996 (2)	0.3148 (2)	0.30930 (17)	0.0371 (6)
O1	0.28102 (19)	0.49502 (18)	0.31874 (16)	0.0514 (7)
O2	0.1963 (2)	0.3847 (2)	0.37872 (15)	0.0559 (7)
O3	0.40152 (19)	0.16917 (19)	0.28193 (17)	0.0481 (6)
O4	0.5322 (2)	0.2591 (2)	0.2929 (2)	0.0702 (9)
O5	0.3105 (3)	0.6232 (3)	0.1980 (3)	0.0983 (12)
H5A	0.347048	0.666098	0.188612	0.147*
H5B	0.258118	0.613758	0.155492	0.147*
O6	0.3351 (4)	0.8969 (3)	0.0997 (3)	0.1259 (17)
H6A	0.376116	0.937335	0.131724	0.189*
H6B	0.357366	0.877645	0.064874	0.189*
O7A[a]	0.4063 (6)	0.5759 (5)	0.4692 (5)	0.108 (3)
H7AA[a]	0.371085	0.584915	0.496824	0.163*
H7AB[a]	0.454505	0.537395	0.494454	0.163*
O7B[b]	0.4614 (12)	0.5853 (10)	0.4370 (9)	0.146 (6)
H7BA[b]	0.507441	0.607708	0.423668	0.219*
H7BB[b]	0.425621	0.632318	0.441668	0.219*
O8A[a]	0.5302 (6)	0.5306 (7)	0.2911 (6)	0.118 (3)
H8AA[a]	0.579273	0.567926	0.316685	0.176*
H8AB[a]	0.477073	0.564506	0.270094	0.176*
O8B[b]	0.5483 (10)	0.5755 (10)	0.3449 (10)	0.128 (5)
H8BA[b]	0.523108	0.617531	0.366272	0.192*
H8BB[b]	0.565908	0.525171	0.375012	0.192*
S2	0.23263 (7)	0.40128 (6)	0.31590 (5)	0.0367 (2)

[a]Occupancy: 0.597 (10), [b]Occupancy: 0.403 (10).

reaction, the pH of the reaction mixture was carefully adjusted to 1 using a 6 mol/*L* hydrochloric acid solution, prompting the precipitation of a white solid. The precipitate was isolated *via* filtration, rinsed with deionized water to remove any residual impurities, and then dried to yield the desired product, ((4-chlorophenyl)sulfonyl)glycine.

A mixture of ((4-chlorophenyl)sulfonyl)glycine (124.8 mg, 0.5 mmol), $Co(CH_3COO)_2{\cdot}4H_2O$ (124.5 mg, 0.5 mmol), and 1,10-phenanthroline (180.2 mg, 1.0 mmol) was dissolved in a 50 ml volume of a 50 % methanolic solution at ambient temperature. The pH of the resulting solution was meticulously adjusted to neutral (pH 7) using a 2 mol/*L* sodium hydroxide solution. Subsequently, the reaction mixture was subjected to thermal processing at 80 °C for 12 h within a Teflon-lined autoclave and then filtered. The filtrate was then allowed to undergo slow evaporation at room temperature, leading to the crystallization of colorless, block crystals of the title compound over a period of two weeks.

2 Experimental details

Hydrogen atoms were added using riding models. Their U_{iso} values were set to $1.2U_{eq}$ of the parent atoms. The structure was solved with the ShelXT[3] structure solution program and refined with the ShelXL.[4]

3 Comment

The acquisition of single crystals for peptide metal complexes often presents a formidable challenge, prompting the exploration of ligands with structural analogies to peptides to elucidate the intricate interactions between these biomolecules and metal ions.[5–7] Sulfonamide moieties are renowned for their diverse biological activities.[8–10] When amino acids are shielded by sulfonamide groups, they exhibit the versatility to function as monodentate ligands through the oxygen of the carboxyl group, or as bidentate ligands by engaging both the carboxyl oxygen and the amino nitrogen. Intriguingly, the $-SO_2-$ group has been observed to partake in coordination as well.[11–13] Our research group has successfully synthesized a range of ((4-chlorophenyl)sulfonyl)-glycine complexes,[14–17] and this study contributes to our ongoing investigation.

In this paper, the synthesis and crystal structure of a new cobalt(II) complex with ((4-chlorophenyl)sulfonyl)-glycine and 1,10-phenanthroline were reported. Single crystal X-ray structure analysis reveals that the title complex crystallizes in the monoclinic system with space group $P2_1/c$. The asymmetric unit is composed of an independent Co(II) ion, one ((4-chlorophenyl)sulfonyl)glycine ligand, two 1,10-phenanthroline ligands, and four lattice water molecules. The crystallographically unique Co(II) ion of the title complex is six-coordinated by four N atoms (N1, N2, N3, N4) from two 1,10-phenanthroline groups, one O atom (O3) and one N atom (N5) from ((4-chlorophenyl)sulfonyl) glycine group, giving rise to a octahedron geometry. Co–N bond lengths fall in the range 2.0443(18)–2.185(2) Å, and Co–O bond length is 2.0872(16) Å. The bond lengths observed fall within the expected range and align closely with those documented for analogous complexes in prior studies.[16,17] The extended 3D supramolecular network is formed through hydrogen bonds of lattice water molecules.

Author contributions: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

Competing interests: The authors declare no conflicts of interest regarding this article.

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