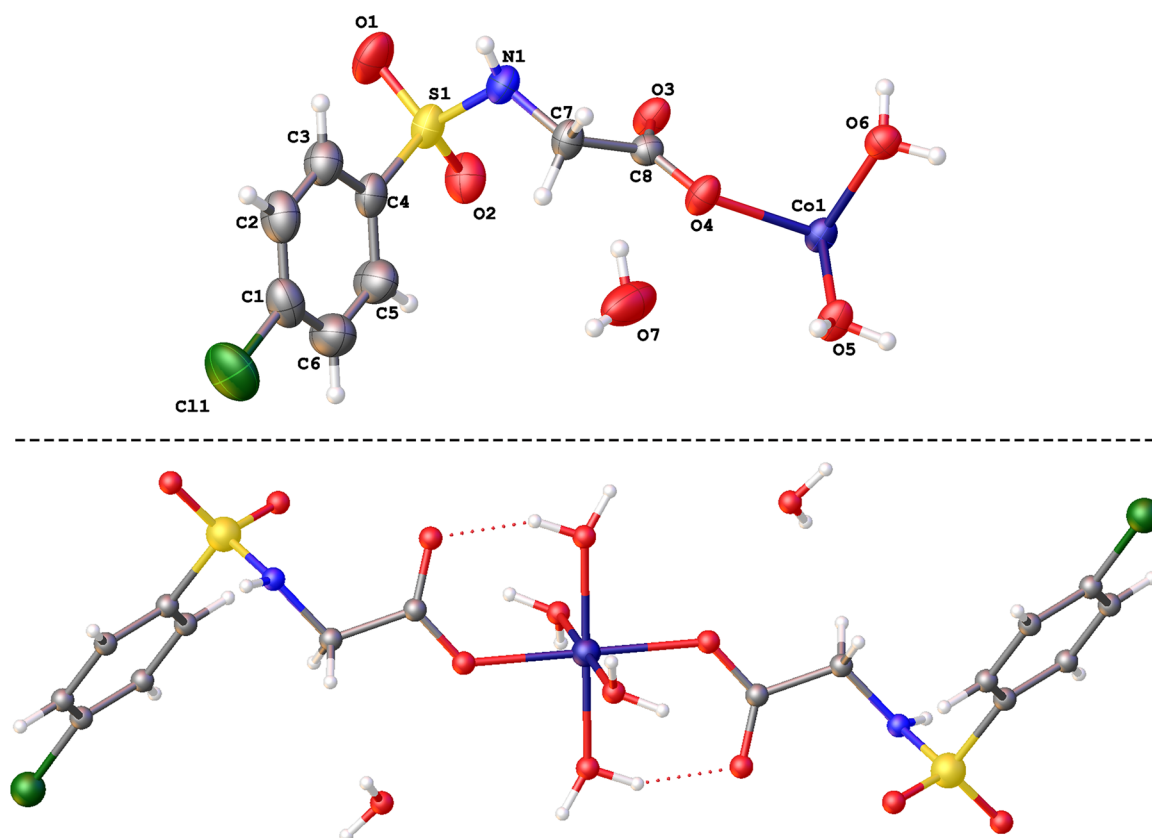


Zheng-Jun Liu*, Xiao-Miao Chen, Qiang Wu, Yu-Tao Zhang, Wu-Chao Zhou, Li Wei and Ying-Xian Zhao

Crystal structure of tetraaqua-bis(((4-chlorophenyl)sulfonyl)glycinato- κO) cobalt(II) dihydrate, $C_{16}H_{26}Cl_2CoN_2O_{14}S_2$



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Abstract

$C_{16}H_{26}Cl_2CoN_2O_{14}S_2$, monoclinic, $P2_1/n$ (no. 14), $a = 5.3261(4)$ Å, $b = 39.755(2)$ Å, $c = 6.2518(4)$ Å, $\beta = 96.880(6)^\circ$, $V = 1314.21(15)$ Å³, $Z = 2$, $R_{gt}(F) = 0.0487$, $wR_{ref}(F^2) = 0.1040$, $T = 153(2)$ K.

***Corresponding author: Zheng-Jun Liu**, Key Laboratory of Agricultural Resources and Environment in High Education Institute of Guizhou Province Anshun University, Anshun, 561000, P.R. China, E-mail: liuzhengjunchem@163.com. <https://orcid.org/0000-0001-6755-7493>
Xiao-Miao Chen, Qiang Wu, Yu-Tao Zhang, Wu-Chao Zhou, Li Wei and Ying-Xian Zhao, Key Laboratory of Agricultural Resources and Environment in High Education Institute of Guizhou Province Anshun University, Anshun, 561000, P.R. China. <https://orcid.org/0000-0001-7641-9084> (X.-M. Chen)

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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:	Pinkish block
Size:	0.50 × 0.43 × 0.18 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	1.08 mm ⁻¹
Diffractionmeter, scan mode:	Bruker SMART CCD 1K, φ and ω
θ_{max} , completeness:	29.4°, 99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int} :	5576, 3001, 0.020
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 2534
$N(param)_{refined}$:	177
Programs:	Bruker [1], Olex2 [2], SHELX [3, 4]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($\text{\AA}^2$)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Co1	0.500000	0.500000	0.000000	0.02721 (15)
Cl1	1.0851 (2)	0.25890 (3)	0.2589 (2)	0.0808 (4)
S1	0.45480 (14)	0.37215 (2)	0.63064 (13)	0.0391 (2)
O1	0.4036 (5)	0.36262 (7)	0.8415 (4)	0.0583 (7)
O2	0.2502 (4)	0.37940 (6)	0.4677 (4)	0.0496 (6)
O3	0.3648 (4)	0.45644 (5)	0.4356 (3)	0.0357 (5)
O4	0.6571 (4)	0.46159 (5)	0.2111 (3)	0.0354 (5)
O5	0.7698 (3)	0.49036 (5)	−0.2059 (3)	0.0356 (5)
H5A	0.925245	0.482193	−0.204072	0.053*
H5B	0.766446	0.506404	−0.304622	0.053*
O6	0.7403 (3)	0.53539 (5)	0.1659 (3)	0.0326 (4)
H6A	0.756214	0.552908	0.082584	0.049*
H6B	0.714259	0.544319	0.290084	0.049*
N1	0.6319 (5)	0.40488 (6)	0.6614 (4)	0.0363 (6)
C1	0.9095 (7)	0.29014 (9)	0.3655 (6)	0.0526 (9)
C2	0.9753 (7)	0.30086 (9)	0.5732 (6)	0.0536 (9)
H2	1.115853	0.291169	0.659335	0.064*
C3	0.8359 (6)	0.32576 (9)	0.6553 (6)	0.0471 (8)
H3	0.879005	0.333362	0.799019	0.057*
C4	0.6320 (6)	0.33972 (8)	0.5274 (5)	0.0391 (7)
C5	0.5675 (7)	0.32881 (10)	0.3193 (6)	0.0558 (9)
H5	0.427153	0.338408	0.232359	0.067*
C6	0.7081 (8)	0.30386 (10)	0.2381 (6)	0.0631 (10)
H6	0.665742	0.296182	0.094445	0.076*
H1	0.723 (8)	0.4037 (11)	0.773 (7)	0.076*
C7	0.7264 (5)	0.41985 (7)	0.4732 (5)	0.0335 (6)
H7A	0.737654	0.402018	0.364239	0.040*
H7B	0.899830	0.428319	0.516677	0.040*
C8	0.5673 (5)	0.44845 (7)	0.3690 (4)	0.0277 (6)
O7	0.9773 (6)	0.58449 (8)	−0.0379 (4)	0.0753 (9)
H7C	0.880553	0.601409	−0.016463	0.113*
H7D	0.961894	0.579674	−0.174688	0.113*

1 Source of material

The mixture of glycine (7.51 g, 100 mmol), sodium hydroxide (8.00 g, 200 mmol) and 100 mL water were stirred in a 250 mL round-bottom flask. Then, 4-chlorobenzenesulfonyl chloride (21.11 g, 0.5 mmol) was added, and the mixture was stirred at room temperature for 10 h. After the reaction is completed, the pH of the reaction mixture was adjusted to 1 with 6 mol/L HCl solution. White precipitates were precipitated from the reaction mixture, filtered, washed with distilled water, and dried to obtain ((4-chlorophenyl)sulfonyl)glycine.

A mixture of ((4-chlorophenyl)sulfonyl)glycine (124.8 mg, 0.5 mmol) and $\text{Co}(\text{CH}_3\text{COO})_2 \cdot 4\text{H}_2\text{O}$ (124.5 mg, 0.5 mmol) was dissolved in 50 mL of 50 % methanolic solution at room temperature. Then the pH was adjusted to 7 with 2 mol/L NaOH solution. The reaction mixture was heated for 12 h at 80 °C in a Teflon-lined reaction vessel and then filtered.

Pinkish block-shaped crystals of the title compound were obtained by slow evaporation at room temperature within two weeks.

2 Experimental details

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

3 Comment

Due to the difficulty in obtaining single crystals of peptide metal complexes, searching for ligands with similar structures to peptides can help understand the interactions between peptides and metals [5–7]. Sulfonamide is a group with various biological activities [8–10]. When amino acids are protected by sulfonamide groups, they can not only act as monodentate ligands through carboxyl O, but also act as bidentate ligands through carboxyl O and amino N. Sometimes $-\text{SO}_2-$ group can also participate in coordination [11–13]. The pH dependent coordination behavior of sulfonylated amino acids is similar to that of many short peptides. Therefore, sulfonylated amino acid complexes can serve as models for studying the interaction between metals and peptides [14–16]. Some ((4-chlorophenyl)sulfonyl)glycine complexes have been successfully synthesized in our group [17, 18]. This contribution constitutes a component of our continuous research focus on complexes of sulfonylated amino acids.

In this paper, the synthesis and crystal structure of a new cobalt(II) complex with ((4-chlorophenyl)sulfonyl)glycine were reported. The asymmetric unit is composed of an independent Co(II) ion, one ((4-chlorophenyl)sulfonyl)glycine ligand, two coordinated water molecules, and one lattice water molecule (upper part of the figure). The crystallographically unique Co(II) ion of the title complex is six-coordinated by two O atoms (O4, O4' which are equivalent) from two ((4-chlorophenyl)sulfonyl)glycine groups, four O atoms (O5, O5', O6, O6', in which O5 and O5' are equivalent, O6 and O6' are equivalent) from four coordinated water molecules, giving rise to an octahedron geometry (lower part of the figure). Co–O bond lengths fall in the range 2.0764(18)–2.1234(18) Å, which are within the normal values [19, 20]. The extended three-dimensional supramolecular network is formed through hydrogen bonds $\text{O} \cdots \text{H} \cdots \text{O}$ ($\text{O5} \cdots \text{O6}^a$: 2.786(3) Å, $\text{O5} \cdots \text{H5A} \cdots \text{O6}^a$ angle

173.6°; O5–O3^b: 2.608(3) Å, O5–H5B...O3 angle 152.5°; $a = 2 - X, 1 - Y, -Z$; $b = 1 - X, 1 - Y, -Z$).

Research ethics: Not applicable.

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

Competing interests: The authors state no conflict of interest.

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Data availability: The raw data can be obtained on request from the corresponding author.

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