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The crystal structure of poly[μ_2 -aqua- aqua-(μ_3 -(*E*)-2-(4-((2-carbamothioylhydrazineylidene) methyl)phenoxy)acetato- $\kappa^3 O:S:S$)sodium(I)], $C_{10}H_{14}N_3O_5SNa$

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Abstract

$C_{10}H_{14}N_3O_5SNa$, triclinic, $P\bar{1}$ (no. 2), $a = 6.7015(5)$ Å, $b = 7.5656(5)$ Å, $c = 14.1332(8)$ Å, $\beta = 93.159(5)^\circ$, $V = 654.71(8)$ Å³, $Z = 2$, $R_{gt}(F) = 0.0357$, $wR_{ref}(F^2) = 0.0872$, $T = 273$ 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

4-Formylphenoxyacetic acid (0.5 mmol, 0.0901 g), thiosemicarbazide (0.5 mmol, 0.0456 g) and NaOH (0.5 mmol,

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.11 × 0.10 × 0.08 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.30 mm ⁻¹
Diffractometer, scan mode:	Rigaku SuperNova, ω
θ_{max} , completeness:	25.0°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int} :	4268, 2284, 0.027
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2 \sigma(I_{obs})$, 1969
$N(param)_{refined}$:	205
Programs:	Bruker [1], Olex2 [2], SHELX [3], Diamond [4]

0.020 g) were dissolved into 20 mL of a H₂O/ethanol solution (v:v = 1:1) with stirring at 50 °C. After 20 min, MgCl₂·6H₂O (0.5 mmol, 0.1015 g) was added to the mixture. The mixture was continued to react for 4 h at 80 °C with stirring. The mixture was then cooled to room temperature and continued the stirring for 4 h. Filtering the reaction mixture. After 20 days, the crystals of the title compound were obtained by slowing volatilization at room temperature. **Elemental analysis** (%) calcd. for $C_{10}H_{14}N_3O_5SNa$: C, 38.55; H, 4.50; N, 13.49. Found (%): C, 38.66; H, 4.76; N, 13.17.

Experimental details

The hydrogen atoms were positioned geometrically (C–H = 0.93–0.97 Å and N–H = 0.86 Å). Their U_{iso} values were set to 1.2 U_{eq} of the parent atoms.

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

Atom	x	y	z	U _{iso} */U _{eq}
C1	1.2614 (3)	0.8211 (3)	0.20138 (13)	0.0093 (4)
C2	0.8917 (3)	0.8458 (3)	0.36091 (14)	0.0105 (4)
H2	0.829020	0.911663	0.325921	0.013*
C3	0.8089 (3)	0.7887 (3)	0.45049 (13)	0.0096 (4)
C4	0.6266 (3)	0.8194 (3)	0.47820 (14)	0.0105 (4)
H4	0.560939	0.877113	0.439220	0.013*
C5	0.5405 (3)	0.7665 (3)	0.56205 (14)	0.0105 (4)
H5	0.417806	0.787286	0.578535	0.013*
C6	0.6382 (3)	0.6822 (3)	0.62135 (13)	0.0098 (4)
C7	0.8259 (3)	0.6554 (3)	0.59643 (14)	0.0114 (5)
H7	0.894868	0.602880	0.636993	0.014*
C8	0.9085 (3)	0.7064 (3)	0.51219 (14)	0.0113 (5)
H8	1.031726	0.686224	0.495946	0.014*
C9	0.3739 (3)	0.6464 (3)	0.73119 (14)	0.0103 (4)
H9A	0.259916	0.588714	0.678176	0.012*
H9B	0.403109	0.783253	0.744570	0.012*
C10	0.2982 (3)	0.5538 (3)	0.81928 (13)	0.0099 (5)
H2A	1.047 (4)	0.929 (3)	0.2175 (15)	0.010 (6)*
H4A	−0.330 (4)	0.620 (3)	0.9382 (18)	0.029 (7)*
H4B	−0.289 (4)	0.641 (4)	1.036 (2)	0.039 (8)*
H5A	0.124 (4)	0.828 (4)	0.8493 (18)	0.039 (8)*
H5B	0.259 (4)	1.004 (3)	0.8610 (17)	0.023 (8)*
N1	1.3532 (3)	0.7232 (2)	0.24710 (11)	0.0134 (4)
H1A	1.316859	0.694239	0.302336	0.016*
H1B	1.448956	0.688713	0.221483	0.016*
N2	1.1127 (3)	0.8718 (2)	0.24420 (12)	0.0107 (4)
N3	1.0488 (3)	0.8073 (2)	0.32929 (11)	0.0105 (4)
Na1	0.000000	0.500000	1.000000	0.0124 (3)
Na2	0.000000	1.000000	1.000000	0.0127 (3)
O1	0.5654 (2)	0.62119 (18)	0.70468 (9)	0.0119 (3)
O2	0.3924 (2)	0.45320 (18)	0.85119 (9)	0.0121 (3)
O3	0.1427 (2)	0.58362 (18)	0.85230 (9)	0.0118 (3)
O4	−0.2275 (2)	0.6722 (2)	0.98521 (12)	0.0132 (3)
O5	0.1361 (3)	0.9456 (2)	0.85079 (11)	0.0141 (4)
S1	1.33292 (8)	0.88919 (7)	0.09353 (3)	0.01171 (17)

Comment

Metal coordination polymers show excellent properties in many areas of chemical engineering and materials [5–12]. According to the literature, the studies on synthesis and properties of metal coordination polymer mainly focuses on transition metals. In order to enrich the structure and properties of the metal coordination polymers, our research group is committed to studying the structure and properties of the alkaline earth metal coordination polymers [13–16]. At the same time, we have also studied the catalytic activity of some metal complexes [16]. In order to further investigate the structure and catalytic property of alkali earth metal coordination polymers, in this work, a new Na(I) coordination polymer has been

synthesized by a one-pot method. The title coordination polymer was characterized by EA and single-crystal X-ray diffraction analysis.

As shown in Figure 1, the Na(I) coordination polymer it is made up of one Na(I) ions on inversion centers, one thiosemicarbazide ligand and two coordinated water molecules. Na1 and Na2 atoms adopt a six-coordinated octahedron configuration, where Na1 is coordinated with two O atoms and S atoms of two different L ligands and two O atoms of two water molecules, and Na2 is coordinated with four O atoms of four coordinated water molecules and S atoms of two different L ligands. Thus the title structure contains two different coordinated units: Na(O_L)₂(S_L)₂(O_{water})₂ and Na(O_{water})₄(S_L)₂, which forms a chained structure by the Na–O coordination which is further extended by the Na–S coordination S atoms of L ligands. The Na (I) coordination polymer assembles an extended layered structure. All bond lengths and angles are in the expected ranges [17].

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References

1. Bruker. SAINT and SADABS; Bruker AXS Inc.: Madison, Wisconsin, USA, 2000.
2. Dolomanov O. V., Bourhis L. J., Gildea R. J., Howard J. A. K., Puschmann H. OLEX2: a complete structure solution, refinement and analysis program. *J. Appl. Crystallogr.* 2009, 42, 339–341.
3. Sheldrick G. M. Crystal structure refinement with SHELXL. *Acta Crystallogr.* 2015, C71, 3–8.
4. Brandenburg K. DIAMOND. Visual Crystal Structure Information System (ver. 3.2); Crystal Impact: Bonn, Germany, 2012.
5. Shun K., Ryota M., Shojiro K., Hiroshi N., Tetsuro K. Radical-based coordination polymers as a platform for magnetoluminescence. *J. Am. Chem. Soc.* 2021, 143, 5610–5615.
6. Yasuchika H., Natsumi S., Joe H., Yuichi K., Koji F. Thermo-sensitive Eu^{III} coordination polymers with amorphous networks. *ChemistrySelect* 2021, 6, 2812–2816.
7. Wu M. X., Yang Y. W. A fluorescent pillarene coordination polymer. *Polym. Chem.* 2019, 10, 2980–2985.

8. Fan W. W., Cheng Y., Zheng L. Y., Cao Q. E. Reversible phase transition of porous coordination polymers. *Chem. Eur J.* 2020, 26, 2766–2779.
9. Hu M. L., Abbasi-Azad M., Habibi B., Rouhani F., Moghanni-Bavil-Olyaei H., Liu K. G., Morsali A. Electrochemical applications of ferrocene-based coordination polymers. *ChemPlusChem* 2020, 85, 2397–2418.
10. Zhu W. J., Zhao J. Y., Chen Q., Liu Z. Nanoscale metal-organic frameworks and coordination polymers as theranostic platforms for cancer treatment. *Coord. Chem. Rev.* 2019, 398, 113009.
11. Tian F., Shen N., Huang K. L., Chen S. C., Chen Q., He M. Y. Construction of alkali-metal-based imidazolecarboxylate coordination polymers as efficient catalysts for solvent-free ring-opening polymerization of caprolactone. *Z. Anorg. Chem.* 2021, 647, 704–713.
12. Wang L. H., Kong F. Y., Tai X. S. Crystal structure and catalytic activity of poly[bis(3-bromo-2-hydroxybenzaldehyde)-2-aminopyrimidinemagnesium(II)] for hydrogenation of 1,3-butadiene. *Bull. Chem. React. Eng. Catal.* 2021, 16, 260–266.
13. Tai X. S., Zhao W. H. Synthesis, crystal structure, and antibacterial activity of magnesium(II) coordination polymers formed by hydrogen bonding. *Res. Chem. Intermed.* 2015, 41, 3471–3478.
14. Cao S. H., Tai X. S., Xin C. L. Synthesis, crystal structure and antitumor activity of a Ca(II) coordination polymer based on 4-acetylphenoxyacetate ligands. *Chin. J. Struct. Chem.* 2021, 40, 324–328.
15. Tai X. S., Wang X. Synthesis and crystal structure of a 1D chained coordination polymer constructed from Ca^{2+} and 2-[(E)-(2-furoylhydrazono)methyl]benzenesulfonate. *Crystals* 2015, 5, 458–465.
16. Tai X. S., Wang X. Synthesis, structural characterization and antitumor activity of a Ca(II) coordination polymer based on 4-formyl-1,3-benzenedisulfonate-2-furoic acid hydrazide ligands. *Crystallogr. Rep.* 2017, 62, 242–245.
17. Naveen Tittal R. K., Ghule V. D., Kumar N., Kumar L., Lal K., Kumar A. Design, synthesis, biological activity, molecular docking and computational studies on novel 1,4-disubstituted-1,2,3-triazole-thiosemicarbazone hybrid molecules. *J. Mol. Struct.* 2020, 1209, 127951.