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Crystal structure of poly[(m₃-3-carboxyadamantane-1-carboxylato-κ³O:O':O'')-(phenanthroline-κ²N,N')sodium(II)], C₂₄H₂₃N₂NaO₄

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Abstract

C₂₄H₂₃N₂NaO₄, monoclinic, *P*₂/c (no. 14), *a* = 7.5246(5) Å, *b* = 22.554(2) Å, *c* = 11.3358(9) Å, β = 91.881(7)°, *V* = 1922.8(3) Å³, *Z* = 4, *R*_{gt}(*F*) = 0.0496, *wR*_{ref}(*F*²) = 0.1184, *T* = 100K.

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A part of the title coordination polymer 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.

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.12 × 0.11 × 0.09 mm
Wavelength:	Mo Kα radiation (0.71073 Å)
μ:	0.12 mm ⁻¹
Diffractometer, scan mode:	SuperNova, ω
θ _{max} , completeness:	25.0°, >99%
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} , <i>R</i> _{int} :	8172, 3372, 0.046
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 2495
<i>N</i> (<i>param</i>) _{refined} :	281
Programs:	Bruker [1], Olex2 [2], SHELX [3], Diamond [4]

Source of material

The mixture of 0.112 g 1,3-adamantanedicarboxylic acid (0.5 mmol), 0.040 g NaOH (1.0 mmol) were dissolved in 15 mL water-ethanol (v:v = 2:1) at room temperature. After 0.5 h, 0.090 g 1,10-phenanthroline monohydrate (0.5 mmol) was added to the aforementioned solution and stirred for 6 h at 65 °C. Then the reaction mixture was cooled to room temperature and filtered. The colorless crystals of the title compound were obtained from the filtrate in 30 days.

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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_{iso}^*/U_{eq}
C1	0.0022 (3)	0.45503 (11)	0.8129 (2)	0.0165 (6)
H1	−0.031624	0.423859	0.860911	0.020*
C2	−0.0378 (3)	0.51265 (11)	0.8489 (2)	0.0173 (6)
H2	−0.096983	0.519198	0.918494	0.021*
C3	0.0118 (3)	0.55947 (11)	0.7796 (2)	0.0166 (6)
H3	−0.012781	0.598253	0.801698	0.020*
C4	0.1005 (3)	0.54772 (11)	0.6746 (2)	0.0133 (5)
C5	0.1345 (3)	0.48799 (10)	0.6455 (2)	0.0118 (5)
C6	0.2261 (3)	0.47479 (11)	0.5372 (2)	0.0121 (5)
C7	0.2768 (3)	0.52253 (10)	0.4648 (2)	0.0127 (5)
C8	0.2377 (3)	0.58205 (11)	0.4968 (2)	0.0163 (6)
H8	0.271284	0.612867	0.447742	0.020*
C9	0.1524 (3)	0.59471 (11)	0.5975 (2)	0.0148 (6)
H9	0.127540	0.633881	0.616582	0.018*
C10	0.3714 (3)	0.50889 (11)	0.3636 (2)	0.0152 (6)
H10	0.408424	0.539044	0.314099	0.018*
C11	0.4087 (3)	0.45110 (11)	0.3380 (2)	0.0166 (6)
H11	0.472778	0.441376	0.272007	0.020*
C12	0.3484 (3)	0.40693 (11)	0.4133 (2)	0.0174 (6)
H12	0.372202	0.367714	0.393986	0.021*
C13	0.2139 (3)	0.24483 (10)	0.3746 (2)	0.0125 (5)
C14	0.3716 (3)	0.20694 (10)	0.41933 (19)	0.0105 (5)
C15	0.5033 (3)	0.24358 (10)	0.49773 (19)	0.0111 (5)
H15A	0.554629	0.274868	0.451035	0.013*
H15B	0.440048	0.261888	0.561425	0.013*
C16	0.6526 (3)	0.20373 (10)	0.5496 (2)	0.0114 (5)
C17	0.7526 (3)	0.17564 (11)	0.4463 (2)	0.0152 (6)
H17A	0.805742	0.206615	0.399705	0.018*
H17B	0.847213	0.150278	0.477331	0.018*
C18	0.6240 (3)	0.13925 (11)	0.3683 (2)	0.0182 (6)
H18	0.688359	0.121997	0.302838	0.022*
C19	0.5440 (3)	0.08962 (11)	0.4412 (2)	0.0229 (6)
H19A	0.637621	0.063862	0.472153	0.027*
H19B	0.463331	0.066074	0.391885	0.027*
C20	0.4430 (3)	0.11696 (11)	0.5434 (2)	0.0203 (6)
H20	0.391887	0.085130	0.590431	0.024*
C21	0.2930 (3)	0.15709 (10)	0.4952 (2)	0.0154 (6)
H21A	0.229978	0.174279	0.560227	0.019*
H21B	0.209066	0.133866	0.447693	0.019*
C22	0.4731 (3)	0.17870 (11)	0.3190 (2)	0.0158 (6)
H22A	0.522098	0.209571	0.270156	0.019*
H22B	0.392137	0.155096	0.270047	0.019*
C23	0.5706 (3)	0.15382 (11)	0.6225 (2)	0.0155 (6)
H23A	0.664051	0.128552	0.655317	0.019*
H23B	0.506481	0.170723	0.687252	0.019*
C24	0.7908 (3)	0.23927 (11)	0.62139 (19)	0.0124 (5)
N1	0.0857 (2)	0.44171 (9)	0.71437 (17)	0.0141 (5)
N2	0.2594 (2)	0.41705 (9)	0.51083 (17)	0.0136 (5)
Na1	0.10141 (11)	0.33590 (4)	0.61115 (8)	0.0155 (3)
O1	0.15148 (19)	0.28324 (7)	0.43964 (14)	0.0174 (4)
O2	0.1487 (2)	0.23262 (7)	0.27031 (13)	0.0171 (4)
O3	0.81203 (19)	0.29281 (7)	0.60832 (14)	0.0167 (4)
O4	0.8911 (2)	0.20685 (7)	0.69295 (14)	0.0178 (4)
H4	0.975163	0.226742	0.718311	0.027*

Experimental details

The hydrogen atoms were positioned geometrically ($C-H = 0.93-0.97 \text{ \AA}$ and $O-H = 0.82 \text{ \AA}$). Their U_{iso} values were set to $1.2U_{eq}$ or $1.5U_{eq}$ of the parent atoms.

Comment

Carboxylate metal complexes have shown excellent properties in many aspects such as catalytic properties, luminescence and magnetic properties, as well as cytotoxic activities [5–7]. Therefore, it is of great significance to continue to study the structure and properties of carboxylate metal complexes. In our previous work, some carboxylate metal complexes have been synthesized, and their structures have also been characterized [8–11]. In order to study the structures of carboxylate metal complexes, a new Na(I) carboxylate coordination polymer has been synthesized and structurally characterized.

Single-crystal X-ray diffraction reveals that the Na(I) coordination polymer features a 2D framework. The asymmetric unit contains one sodium(I) ion, one phenanthroline (phen) and one 3-carboxyadamantane-1-carboxylate anion (ADA). The Na(I) center is five coordinated with three carboxylate oxygen atoms (O1, O3A, O2B) from three different mono deprotonated ADA ligands and two nitrogen atoms (N1, N2) from one phen ligand. The Na–O and Na–N distances are in the range of $2.3197(18)-2.662(2) \text{ \AA}$, which are comparable to other Na(I) coordination polymers. All ADA ligands possess the same coordination mode, e.g., one deprotonated carboxylate group bridges two different Na(I) ions and the other carboxylate group coordinates with another Na(I) ion [12]. The carboxylic oxygen atoms bridge the Na(I) ions to generate infinite 1D chains. These chains are further linked by the uncoordinated oxygen atoms to form an extended 2D layer. As a result, the 2D layers form a 3D supramolecular framework by the interaction of $\pi-\pi$ stacking interactions between the phen molecules and the weak hydrogen bonding interactions.

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