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*E.K. Trunova, A.M. Mishchenko, A.A. Rogovtsov, T.A. Makotryk***CRYSTAL STRUCTURE OF A NOVEL COBALT(II) COMPLEX WITH ETHYLENEDIAMINEDISUCCINIC ACID****V.I. Vernadsky Institute of General and Inorganic Chemistry of the NAS of Ukraine, Kyiv**

Due to their valuable properties, coordination compounds of 3d transition metals with aminocarboxylic acids are perspective bioactive compounds. In the present work, potassium (ethylenediamine-*N,N'*-disuccinato)cobaltate(II) dihydrate was prepared and structurally characterized by single-crystal X-ray diffraction analysis. Structure determination was performed by a least-squares fit of 6669 reflections of highest intensity and refined with the full-matrix least squares procedure. Present compound is a closed-type binuclear Co(II) complex of ethylenediaminedisuccinic acid composed of $[(C_{10}H_{12}N_2O_8)_2Co_2]$ moieties bridged via potassium atoms in a zig-zag fashion and possesses the monoclinic $P2_1/c$ space group. Each Co(II) ion has a distorted octahedral environment formed by sets of three 5-membered and one 6-membered chelate cycles. One of the positions in the $[CoO_4N_2]$ octahedron is occupied by oxygen atom of the neighboring ligand molecule. The acetate branches of ligand are situated in the axial positions of the distorted Co(II) octahedron on the contrary to previously studied sodium-containing complex of analogous composition. Each potassium atom has a seven-fold coordination involving three oxygen atoms of water molecules, two oxygen atoms from the nearest carboxyl group and two additional oxygen atoms from two adjacent ligand molecules. The complex under study also displays a developed system of inter-ligand $N-H\cdots O$, $C-H\cdots O$ and hydration $O-H\cdots O$ bonds.

Keywords: cobalt complex, ethylenediaminedisuccinic acid, X-ray structure analysis, chelate cycle, hydrogen bond.

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Introduction

The coordination compounds of 3d metals with aminocarboxylic acids are perspective bioactive compounds due to their high stability, water solubility and, in consequence, high assimilability by living organisms. Ethylenediaminedisuccinic acid (EDDS, commonly denoted as H_4edds to emphasize its tetrabasic character) is a biodegradable chelating agent that can be used as a substitute for ethylenediaminetetraacetic acid in different applications, e.g. for the extraction of heavy metals from aqueous solutions, soils and phytoextraction [1–3], in the pulp and paper-making industry and for radionuclide decontamination [4]. The complexes of biogenic metals (Mn^{II} , Fe^{III} , Co^{II} , Cu^{II} , Zn^{II}) with EDDS have attracted much interest due to their potential and practical applications in biochemistry [5,6]. Since its molecule contains two natural fragments (the residues of succinic and aspartic acids), not only does EDDS transport microelement

but it also acts as a biostimulating component.

Among other microelements, cobalt is responsible for accumulation of sugars and lipids in plants and favors the synthesis of chlorophyll. As far as we know, only a few Co-based monometallic complexes with EDDS were structurally characterized previously [7–9]. Recently we have reported the structure of polymeric complex in which dimeric $[Co_2(edds)_2]^{4-}$ units are connected via outer-sphere Na^+ counterions [10]. Each Co(II) ion has a distorted octahedral $[CoO_4N_2]$ environment formed by sets of three 5-membered and one 6-membered chelate cycles. Sodium ions have a distorted trigonal-bipyramidal coordination environment defined by three equatorial oxygen atoms of α -carboxylic groups and two coordinated water molecules in the axial position. To study the effect of the alkaline metal ion substitution on the spatial arrangement of chelate cycles, we have synthesized another Co(II) complex with EDDS containing potassium as counterion. In

the present work its structure was determined by single-crystal X-ray diffraction analysis and compared with sodium-containing complex.

Experimental

Materials and methods

EDDS was prepared by condensation reaction of maleic acid with ethylenediamine [11]. All other chemicals were analytical grade and used as received.

Elemental analysis for carbon, hydrogen and nitrogen was performed by the combustion method with a Perkin-Elmer CHN 2400 analyzer. The metal contents were determined by atomic absorption using a Philips Pye Unicam 8000 atomic absorption spectrophotometer. IR spectrum was recorded on a Specord M80 spectrophotometer (Carl Zeiss Jena) as KBr pellet in the 400–4000 cm^{-1} range.

Preparation

A mixture of $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ (2.38 g, 10 mmol) and EDDS (2.92 g, 10 mmol) was dissolved in distilled water (10 ml). Then pH was adjusted to 5.0 by concentrated (40%) solution of KOH. Reaction mixture was refluxed under constant stirring for 24 h. After cooling to room temperature, diethyl ether was added into the solution giving a powder crude product. Precipitate was filtered off and washed with methanol for several times (yield 83%). Anal. Calcd for $\text{K}_2[\text{Co}(\text{edss})] \cdot 2\text{H}_2\text{O}$ (%): K 16.9; Co 12.8; C 26.0; H 3.5; N 6.1. Found: K 16.6; Co 13.0; C 26.1; H 3.4; N 6.3. IR (cm^{-1}): 3460, 3224 ($\nu_{\text{H}_2\text{O}}$); 2912, 2364 ($\nu_{\text{C-H}}$); 1650, 1583 ($\nu_{\text{as COO}^-}$); 1492, 1390, 1319 ($\nu_{\text{s COO}^-}$); 1060, 1040, 877 ($\nu_{\text{C-N}}$); 964, 924 ($\nu_{\text{C-C}}$); 708 (δ_{OCO}); 626, 603, 537 ($\nu_{\text{Co-O}}$); 449 ($\nu_{\text{Co-N}}$).

To obtain crystals suitable for X-ray diffraction, the complex was dissolved in water and was stored in a dark place for slow evaporation. After 8 days violet prismatic crystals were formed.

Crystallography

Diffraction data were collected at 293(2) K by the φ - and ω -scan technique up to $2\theta=60.5^\circ$, on a Bruker Smart Apex II with graphite-monochromatized MoK α radiation ($\lambda=0.71073 \text{ \AA}$). The data were corrected for Lorentz-polarization and absorption effects. Accurate unit-cell parameters were determined by a least-squares fit of 6669 reflections of highest intensity, chosen from the whole experiment. The structures were solved with DIRDIF-2008 program system and refined with the full-matrix least squares procedure on F^2 by SHELXL97 [12]. Scattering factors incorporated in SHELXL97 were used. The function $R_w(F_o^2 - F_c^2)^2$ was minimized with $w^{-1} = [\sigma^2(F_o^2) + (0.0209P)^2 + 0.6157P]$ (where $P = [\max(F_o^2, 0) + 2F_c^2]/3$). All non-hydrogen atoms were refined anisotropically, hydrogen atoms were put in the idealized positions

Table 1
Crystal data, data collection and structure refinement

Empirical formula	$\text{C}_{10}\text{H}_{16}\text{CoN}_2\text{K}_2\text{O}_{10}$
Formula weight (g mol^{-1})	461.41
Temperature (K)	293
Wavelength (\AA)	0.71073
Crystal system	Monoclinic
Space group	$\text{P}2_1/\text{c}$
a (\AA)	7.8016(1)
b (\AA)	10.1712(2)
c (\AA)	18.5122(4)
β ($^\circ$)	95.349(1)
V (\AA^3)	1462.58(5)
Z	4
D_{Calcd} (g cm^{-3})	1.918
μ (mm^{-1})	1.52
F (000)	864
Crystal size (mm)	0.1 \times 0.1 \times 0.1
θ range ($^\circ$)	2.287–30.563
Index ranges	$-9 \leq h \leq 9$ $0 \leq k \leq 12$ $0 \leq l \leq 22$
Reflections:	
Collected	6669
Unique (R_{int})	2875 (0.03)
With $I > 2\sigma(I)$	2572
Number of parameters	217
Final R_1/wR_2 [$I > 2\sigma(I)$]	0.0219/0.0567
R_1/wR_2 (all data)	0.0258/0.0612
Max/min, $e/\text{\AA}^3$	0.37/–0.21
Goodness-of-fit on F^2	1.05

(those from water molecules and imines groups was found in the difference Fourier map), and refined as riding model. Their isotropic thermal parameters were set 1.2 times larger compared with the appropriate carrier atom. Relevant crystal data are listed in Table 1, together with refinement details. Table 2 contains atomic coordinates and displacement parameters.

Results and discussion

In contrast to previously reported sodium-containing orthorhombic Co(II)–EDDS complex [10], the obtained compound crystallizes in the monoclinic $\text{P}2_1/\text{c}$ space group. Its asymmetric unit is shown in Fig. 1. Bond lengths and angles of slightly distorted $[\text{CoO}_4\text{N}_2]$ octahedron (Table 3) fall in the ranges typical for octahedral cobalt complexes [9, 13].

Each cobalt atom is coordinated by N1, N2 atoms from ethylenediamine bridge and O1, O3, O5

Table 2

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (E²)

Atom	x	y	z	U _{iso} */U _{eq}
Co1	0.05715(3)	0.04342(2)	0.362730(10)	0.00930(10)
K1	0.24325(5)	0.45653(4)	0.45811(2)	0.01440(10)
O1	0.06654(14)	0.20908(11)	0.41257(6)	0.0124(3)
O2	-0.09642(15)	0.34518(12)	0.47055(7)	0.0181(4)
O3	-0.06631(14)	0.12167(11)	0.28128(6)	0.0128(3)
O4	-0.11860(16)	0.08758(12)	0.16260(6)	0.0180(4)
O5	0.26779(14)	0.07690(11)	0.31920(6)	0.0133(3)
O6	0.45301(15)	0.05250(12)	0.23687(7)	0.0187(4)
O7	-0.16937(14)	0.03242(11)	0.55161(6)	0.0132(3)
O8	-0.34768(15)	0.17135(12)	0.60109(6)	0.0165(4)
O9	0.54576(16)	0.60697(12)	0.41856(7)	0.0202(4)
O10	0.40815(17)	0.33412(13)	0.35125(7)	0.0254(4)
N1	-0.15643(17)	0.01962(13)	0.40389(7)	0.0109(4)
N2	0.01538(17)	-0.12104(13)	0.31358(7)	0.0116(4)
C1	-0.0774(2)	0.24386(16)	0.43601(9)	0.0128(5)
C2	-0.2300(2)	0.15161(17)	0.41609(9)	0.0120(5)
C3	-0.2654(2)	-0.07085(17)	0.35624(9)	0.0149(5)
C4	-0.1471(2)	-0.17954(17)	0.33614(9)	0.0150(5)
C5	0.0151(2)	-0.08531(17)	0.23585(9)	0.0132(5)
C6	-0.0669(2)	0.04957(16)	0.22327(9)	0.0125(5)
C7	0.2026(2)	-0.07714(17)	0.21884(9)	0.0152(5)
C8	0.3152(2)	0.02428(16)	0.26106(9)	0.0138(5)
C9	-0.3627(2)	0.15381(17)	0.47154(9)	0.0134(5)
C10	-0.2902(2)	0.11981(16)	0.54829(9)	0.0122(5)
H2	0.25468	-0.16295	0.22758	0.0182
H3	0.20398	-0.0584	0.16752	0.0182
H4	-0.04722	-0.1514	0.20512	0.0158
H5	0.1016	-0.17834	0.32417	0.0139
H6	-0.1209	-0.23731	0.37738	0.018
H7	-0.20315	-0.23107	0.29664	0.018
H8	-0.31451	-0.0252	0.31317	0.0179
H9	-0.35836	-0.10605	0.38176	0.0179
H10	-0.13275	-0.01371	0.44716	0.0131
H11	-0.41372	0.24075	0.47175	0.0161
H12	-0.45367	0.09196	0.45642	0.0161
H13	-0.28747	0.18173	0.36972	0.0143
H14	0.53778	0.57518	0.38224	0.0303
H15	0.48712	0.68169	0.41084	0.0303
H16	0.38043	0.25671	0.33273	0.0381
H17	0.44406	0.37579	0.31431	0.0381

atoms of three carboxylate groups and forms three 5-membered rings: Co1–O3–C6–C5–N2 ($\varphi=333.6(2)^\circ$, twisted conformation), glycine cycle Co1–O1–C1–C2–N1 ($\varphi=322.3(2)^\circ$, envelope conformation), ethylenediamine cycle Co1–N1–C3–C4–N2 ($\varphi=71.94(17)^\circ$, envelope conformation), and one 6-membered β -alanine ring Co1–O5–C8–C7–C5–N2 ($\tau=131.16(13)^\circ$, $\varphi=102.58(16)^\circ$) [14]. The sixth position in the [CoO₄N₂] octahedron is

occupied by oxygen atom of the neighboring EDDS molecule [$d(O7\dots O7^i)=3.4658(16)$ Å] producing a binuclear moiety (Fig. 2). The fourth carboxylate group of EDDS is non-coordinated and possess outward direction coplanar with (110) plane. The glycine and ethylenediamine rings are situated in the equatorial plane of the [CoO₄N₂] octahedron forming a conjugated system, while the β -alanine ring is located perpendicularly.

Table 3
Geometrical parameters of the $[\text{CoO}_4\text{N}_2]$ polyhedron

Bond	d, Å	Angles	$\theta, ^\circ$
Co1–O1	1.9192(11)	O1–Co1–O3	90.44(5)
Co1–O3	1.8878(11)	O3–Co1–N2	86.41(5)
Co1–O5	1.9269(11)	O1–Co1–N1	85.13(5)
Co1–O7	1.9019(11)	O1–Co1–N2	172.40(5)
Co1–N1	1.9111(13)	O1–Co1–O5	92.87(5)
Co1–N2	1.9175(13)	N1–Co1–N2	87.87(6)

Apart from metal–ligand bonds, a number of intramolecular hydrogen bonds ranging from 2.7385(17) to 3.0920(18) Å are presented in the complex (Table 4). Water molecules interact with N2 donor atom resulting in extended N–H...O hydrogen bond network, while the sequence of N1–H10...O7 bonds displays chain-like arrangement (Fig. 3).

K1 atom coordinates three oxygen atoms O9, O10, O9^{iv} of water molecules [$d(\text{K1}\cdots\text{O9})=2.9613(13)$ Å, $d(\text{K1}\cdots\text{O10})=2.7555(14)$ Å, $d(\text{K1}\cdots\text{O9}^{\text{iv}})=2.7638(13)$ Å], two oxygen atoms O1, O2 from the same carboxyl group [$d(\text{K1}\cdots\text{O1})=2.9545(12)$ Å, $d(\text{K1}\cdots\text{O2})=2.9106(12)$ Å] and two additional oxygens O2ⁱⁱⁱ and O4ⁱⁱ from two adjacent EDDS molecules [$d(\text{K1}\cdots\text{O2}^{\text{iii}})=2.7214(13)$ Å, $d(\text{K1}\cdots\text{O4}^{\text{ii}})=2.7038(12)$ Å] (Fig. 1). Two nearest potassium atoms share common edge of their polyhedrons along the O2...O2ⁱ direction forming a singular cell comprised of four $[(\text{C}_{10}\text{H}_{12}\text{N}_2\text{O}_8)_2\text{Co}_2]$ moieties with a volume of ca. $12.6 \times 17.3 \times 30.4$ Å³. Potassium bipolyhedral assemblies extend along the

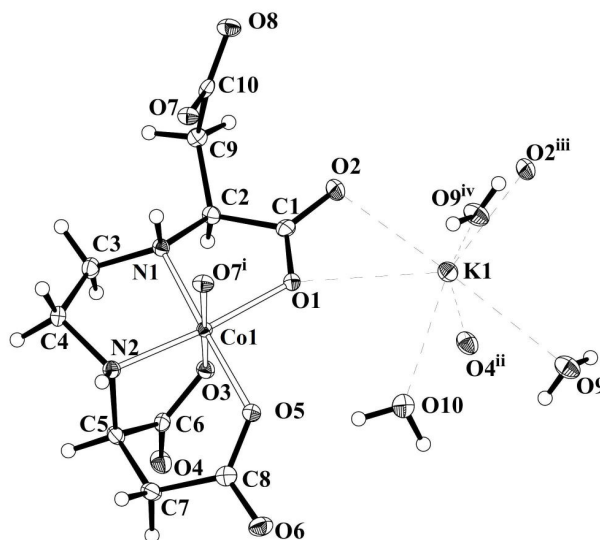


Fig. 1. Anisotropic ellipsoid representation of the complex asymmetric unit together with atom labeling scheme. The ellipsoids are drawn at 50% probability level, hydrogen atoms are depicted as spheres with arbitrary radii, K...O contacts are shown as the dashed lines. Symmetry codes:
(i) $-x, -y, -z+1$; (ii) $-x, y+S, -z+S$;
(iii) $-x, -y+1, -z+1$; (iv) $-x+1, -y+1, -z+1$

[001] direction in a zig-zag fashion with a period of translation equal to the lattice constant $a=7.8016(1)$ Å.

Conclusions

To sum up, single-crystal X-ray diffraction analysis revealed a structural rearrangement which takes place in a Co(II)–EDDS monometallic complex when the Na^+ counterion is replaced with K^+ . In the potassium-containing complex the acetate

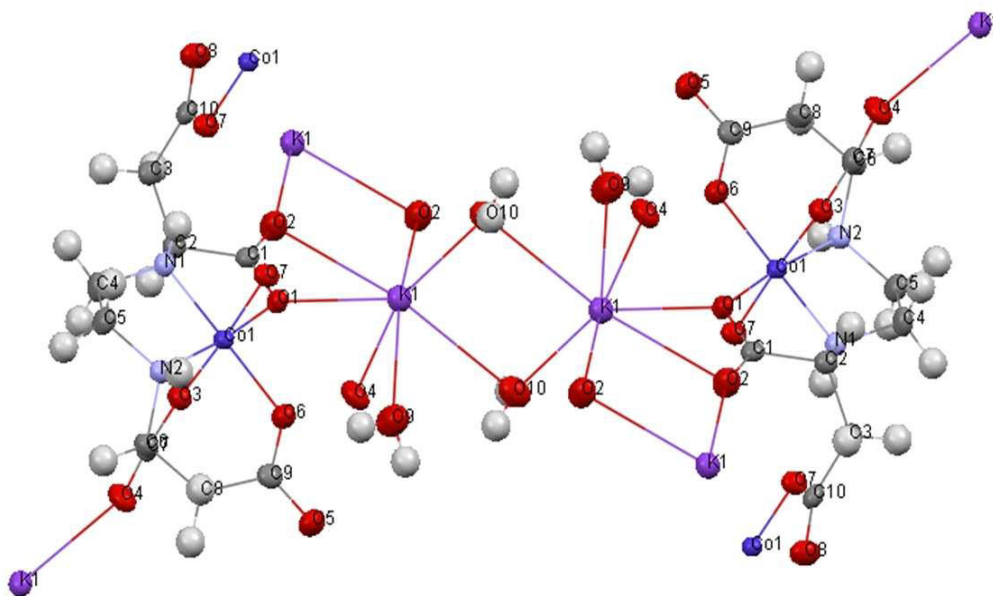


Fig. 2. Molecular structure of the complex under study

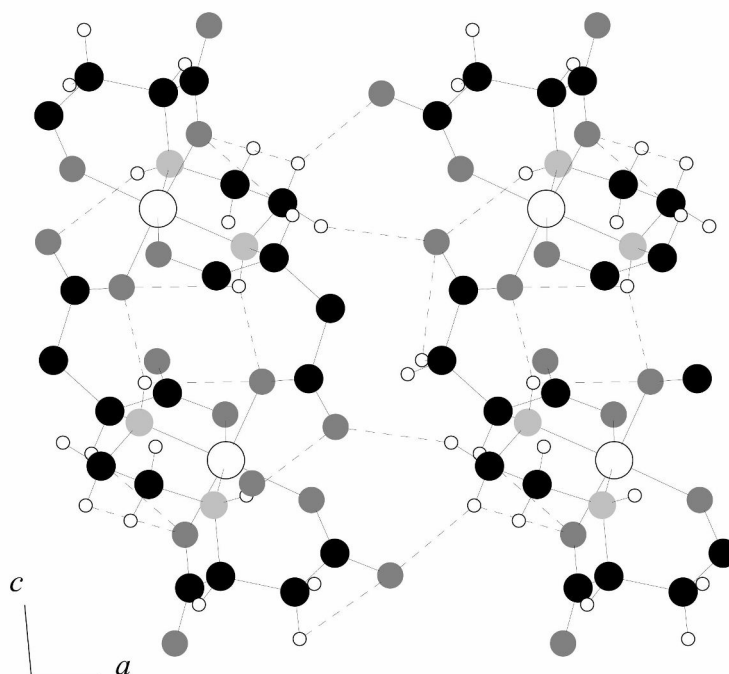


Fig. 3. Hydrogen bonding of two adjacent $[(C_{10}H_{12}N_2O_8)_2Co_2]$ moieties viewed along the $[010]$ direction. Color codes: light grey – nitrogen, dark grey – oxygen, black-filled spheres are carbon atoms. Empty spheres indicate hydrogen, cobalt and potassium atoms are of sizes proportional to their covalent radii

Table 4

Geometric parameters of hydrogen bonds

D–H...A	d(D–H), Å	d(H...A), Å	d(D...A), Å	$\theta(D-H...A)$, °
N2–H5...O4 ^{vii}	0.90	2.40	3.0920(18)	134
N2–H5...O8 ⁱ	0.90	2.26	2.9520(18)	134
N1–H10...O7	0.87	2.04	2.7490(17)	138
O9–H14...O6 ^{vi}	0.74	2.22	2.9311(18)	159
O9–H15...O8 ⁱⁱⁱ	0.89	1.85	2.7385(17)	174
O10–H16...O5	0.88	2.03	2.8766(17)	160
O10–H17...O6 ^x	0.87	2.22	3.0162(18)	152
C5–H4...O1 ^{xi}	0.98	2.59	3.464(2)	148
C5–H4...O3 ^{viii}	0.98	2.48	3.028(2)	115
C3–H8...O3	0.97	2.56	2.928(2)	103
C3–H8...O6 ^{xii}	0.97	2.33	3.222(2)	152
C3–H9...O8 ^v	0.97	2.44	3.350(2)	157
C2–H13...O3	0.98	2.56	2.923(2)	102

branches of ligand are replaced in the axial position of the distorted Co(II) octahedron. The combination of ethylenediamine (with angle N–Co–N < 90°) and glycine (with angle N–Co–O ≥ 90°) cycles decreases the distortion of cobalt octahedron in the equatorial plane compared to the sodium-containing complex.

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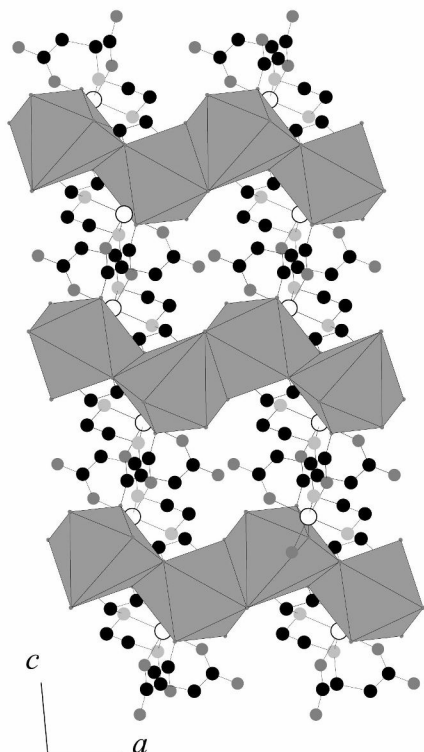


Fig. 4. Extension of the $[KO_7]$ polyhedrons (grey-filled on the picture) along the $[001]$ direction

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КРИСТАЛІЧНА БУДОВА НОВОГО КОМПЛЕКСУ КОБАЛЬТУ(II) З ЕТИЛЕНДИАМІНДИ ЯНТАРНОЮ КИСЛОТОЮ

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Завдяки своїм цінним властивостям координаційні сполуки 3d перехідних металів з амінокарбоновими кислотами є перспективними біологічно активними сполуками. В даній роботі синтезовано дигідрат (етилендіамін-N,N'-дисукцинато)-кобальтату(II) калію і досліджено його будову методом рентгеноструктурного аналізу. Визначення структури виконано шляхом аналізу найбільш інтенсивних 6669 відбиттів, структуру уточнено методом найменших квадратів. Дана сполука являє собою біядерний комплекс $Co(II)$ з етилендіаміндибуриштинною кислотою закритого типу, що складається з фрагментів $[(C_{10}H_{12}N_2O_8)_2Co_2]$, сполучених між собою атомами калію з утворенням зигзагоподібної структури, і належить до просторової групи $P2_1/c$. Кожен з іонів $Co(II)$ знаходиться у викривленому октаедричному оточенні, утвореному трьома 5-членними та одним 6-членним хелатними циклами. Одну з позицій в октаедрі $[CoO_4N_2]$ займає атом кисню сусідньої молекули ліганду. При цьому ацетатні гілки лігандів розташовані в аксіальних позиціях октаедру атома $Co(II)$, на відміну від раніше дослідженого натрієвмісного комплексу аналогічного складу. Координаційна сфера атома калію утворена трьома атомами кисню молекул води, двома атомами кисню сусідньої карбоксильної групи, а також двома атомами кисню двох сусідніх молекул ліганду. Досліджений комплекс також демонструє розгалужену систему міжлігандних $N-H \cdots O$, $C-H \cdots O$ та гідратних $O-H \cdots O$ водневих зв'язків.

Ключові слова: комплекс кобальту, етилендіаміндибуриштинна кислота, рентгеноструктурний аналіз, хелатний цикл, водневий зв'язок.

CRYSTAL STRUCTURE OF A NOVEL COBALT(II) COMPLEX WITH ETHYLENEDIAMINEDISUCCINIC ACID

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*Due to their valuable properties, coordination compounds of 3d transition metals with aminocarboxylic acids are perspective bioactive compounds. In the present work, potassium (ethylenediamine-*N,N'*-disuccinato)cobaltate(II) dihydrate was prepared and structurally characterized by single-crystal X-ray diffraction analysis. Structure determination was performed by a least-squares fit of 6669 reflections of highest intensity and refined with the full-matrix least squares procedure. Present compound is a closed-type binuclear Co(II) complex of ethylenediaminedisuccinic acid composed of $[(C_{10}H_{12}N_2O_8)_2Co_2]$ moieties bridged via potassium atoms in a zig-zag fashion and possesses the monoclinic $P2_1/c$ space group. Each Co(II) ion has a distorted octahedral environment formed by sets of three 5-membered and one 6-membered chelate cycles. One of the positions in the $[CoO_4N_2]$ octahedron is occupied by oxygen atom of the neighboring ligand molecule. The acetate branches of ligand are situated in the axial positions of the distorted Co(II) octahedron on the contrary to previously studied sodium-containing complex of analogous composition. Each potassium atom has a seven-fold coordination involving three oxygen atoms of water molecules, two oxygen atoms from the nearest carboxyl group and two additional oxygen atoms from two adjacent ligand molecules. The complex under study also displays a developed system of inter-ligand $N-H\cdots O$, $C-H\cdots O$ and hydration $O-H\cdots O$ bonds.*

Keywords: cobalt complex; ethylenediaminedisuccinic acid; X-ray structure analysis; chelate cycle; hydrogen bond.

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