

STRUCTURAL FEATURES AND HIRSHFELD SURFACE ANALYSIS OF A THREE-DIMENSIONAL CO(II)-FORMATE FRAMEWORK**K.B. Kholduraev**Department of Chemistry, Termez State University, 43 Barkamol Avlod Street, Termez-190111,
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Abstract: A novel cobalt(II)-based coordination polymer, formulated as $[\text{Co}(\text{HCO}_2)_3]_n \cdot n(\text{C}_3)_2\text{N}$, was successfully synthesized and comprehensively characterized by single-crystal X-ray diffraction, polyhedral geometry analysis, and Hirshfeld surface analysis. Single-crystal X-ray diffraction reveals that the compound crystallizes in the trigonal crystal system with the centrosymmetric space group $R\bar{3}c$, forming a highly symmetric three-dimensional coordination framework in which each Co(II) center exhibits an almost ideal octahedral coordination geometry. The cobalt ions are coordinated exclusively by six oxygen atoms from six symmetry-related μ_2 -bridging formate ligands, with all Co-O bond lengths being crystallographically equivalent, resulting in infinite catena-type chains that interconnect into an extended polymeric network. Polyhedral analysis confirms the regularity of the CoO_6 octahedra, with an effective coordination number of 6.000, a zero distortion index, near-unity quadratic elongation, and minimal bond-angle variance. Collectively, these results highlight the highly symmetric coordination environment, robust polymeric architecture, and well-defined intermolecular interactions of the title cobalt(II) formate coordination polymer.

Key words: coordination polymer, crystal structure, Hirshfeld surface.

Introduction

Coordination polymers based on simple carboxylate ligands have attracted considerable attention due to their versatile structural architectures and potential applications in catalysis, gas storage, magnetism, and adsorption [1-3]. Among these ligands, formate (HCOO^-) is particularly

interesting because of its small size and flexible coordination modes, which allow the formation of extended networks ranging from one-dimensional chains to three-dimensional frameworks. In particular, μ_2 -formate bridges enable the assembly of robust polymeric chains with short metal-metal separations, making $[\text{Co}(\text{HCOO})_3]_n$ an excellent model system for studying low-dimensional coordination polymers [4-6].

Transition-metal formate coordination polymers with the general formula $[\text{M}(\text{HCOO})_3]_n$ ($\text{M} = \text{Mn}, \text{Fe}, \text{Co}, \text{Ni}$) have attracted considerable attention owing to their rich structural diversity and multifunctional properties. These materials are known to exhibit a wide range of architectures, including one-, two-, and three-dimensional frameworks, which are highly dependent on the nature of the metal centre, coordination preferences, and synthetic conditions such as solvent, temperature, and pH [7-9]. In particular, manganese and iron formate polymers have been extensively studied and are reported to display perovskite-like networks, layered structures, or infinite chain motifs stabilized by μ_2 - or μ_3 -formate bridges. Among this family, cobalt(II) formate polymers are especially noteworthy due to the remarkable flexibility of the Co^{2+} ion, which can adopt various coordination geometries, including octahedral, distorted octahedral, and occasionally tetrahedral environments. This flexibility enables the formation of robust three-dimensional catena-type coordination polymers constructed exclusively from μ_2 -formate bridges, in which each formate ligand acts as a bidentate linker connecting adjacent cobalt centres [10-12]. The structural integrity and dimensionality of $[\text{Co-L}]_n$ are primarily dictated by the μ -formate Co-O-Co connectivity, which forms continuous one-dimensional chains along a specific crystallographic axis. Each Co(II) centre is coordinated octahedrally by six oxygen atoms from six formate ligands, with each formate adopting a μ -bridging mode that links two adjacent cobalt ions. This bridging arrangement not only maintains the linear propagation of the polymeric chain but also stabilizes the overall framework by distributing the coordination interactions uniformly along the chain. The resulting Co-O-Co linkages produce relatively short metal-metal separations, which contribute to the rigidity and mechanical stability of the polymer, while also influencing the packing of chains in the crystal lattice. Such connectivity is a defining feature of catena-type structures and serves as a fundamental factor in determining the dimensionality and robustness of the coordination polymer [13-15].

In this work, we report the solvothermal synthesis, crystal structure, and comprehensive characterization of a new three-dimensional cobalt(II) formate polymer, $[\text{Co}(\text{HCOO})_3]_n$. Single-crystal X-ray diffraction analysis reveals that each Co(II) centre is octahedrally coordinated by six oxygen atoms from μ_2 -formate ligands, which bridge adjacent cobalt ions to form extended catena-type chains. The geometrical parameters of the CoO_6 octahedra and Co-O-Co bond angles were examined in detail, providing insight into the topology, rigidity, and stability of the polymeric framework. To further understand the intermolecular interactions within the crystal lattice, Hirshfeld surface analysis was performed, highlighting the contributions of various contacts, particularly $\text{O}\cdots\text{H}/\text{H}\cdots\text{O}$ interactions from the formate ligands, to the overall packing and structural stabilization. Fingerprint plots derived from these surfaces allowed for a quantitative assessment of the relative contributions of different contacts, offering a deeper understanding of how the local coordination environment and long-range packing effects collectively govern the formation, organization, and properties of this μ_2 -formate-bridged cobalt(II) polymer.

Synthesis of the complex

All starting materials were commercially available analytical-grade reagents and were used without further purification. The coordination polymer catena-(dimethylammonium tris(μ_2 -formato)-cobalt(II)) was obtained by solvothermal synthesis under experimental conditions similar to those previously reported for related systems.

A mixture of 10 mL of DMF, 0.23 mL of formic acid (6 mmol), and 0.87 g of $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (3 mmol) was placed in a 20 mL scintillation vial. The vial was capped and immersed in a silicone oil bath maintained at a constant temperature of 110 °C for 40 h. During this period, high-quality pink crystalline blocks of compound 1 were gradually deposited (0.36 g, 71% yield). The crystals were isolated by filtration in air and dried at room temperature.

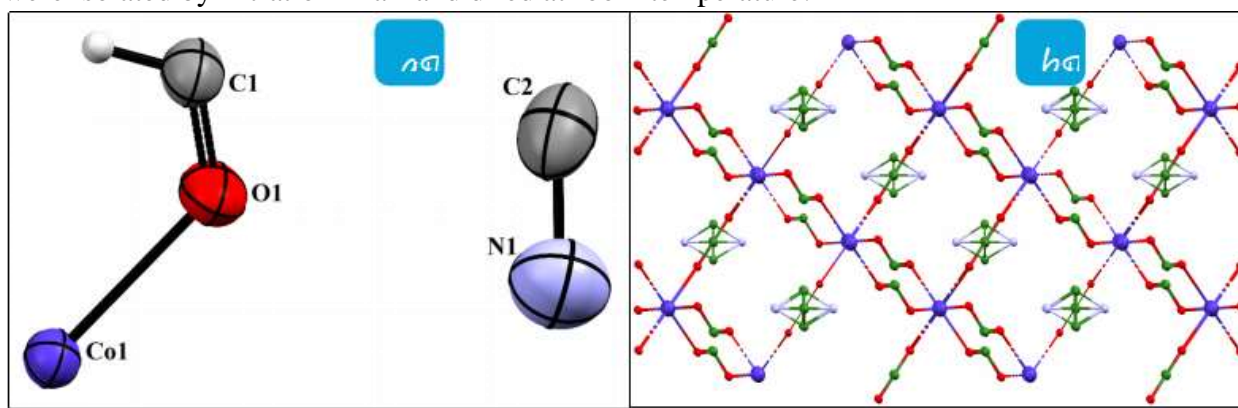


Figure 1. (a) Asymmetric unit of $[\text{Co}(\text{HCO}_2)_3]_n \cdot n(\text{C}_3)_2\text{N}$ (b) three-dimensional polymeric network.

Single-Crystal X-ray Diffraction Analysis

The experimental conditions for single-crystal X-ray diffraction data collection and subsequent structure refinement are summarized in Table 2. The coordination polymer $[\text{Co}(\text{HCO}_2)_3]_n \cdot n(\text{C}_3)_2\text{N}$ crystallizes in the trigonal crystal system with the centrosymmetric space group $R\bar{3}c$ (No. 167), indicative of a high-symmetry crystal environment and a well-defined coordination geometry around the cobalt(II) centres. The molecular structure is depicted in Fig. 1, with displacement ellipsoids drawn at the 50% probability level and atoms labelled according to the standard crystallographic numbering scheme.

Single-crystal X-ray diffraction analysis reveals that each cobalt(II) centre exhibits a coordination number of six, being coordinated exclusively by six oxygen atoms from six symmetry-related formate ligands. This coordination arrangement results in a highly regular octahedral geometry. Notably, all six Co-O bond lengths are crystallographically equivalent, reflecting the high local symmetry imposed by the space group and indicating the absence of significant octahedral distortion around the Co^{2+} ion.

Each formate ligand adopts a μ_2 -bridging coordination mode, linking two adjacent cobalt(II) centres through its oxygen atoms. Consequently, each Co^{2+} ion is connected to six neighboring cobalt centres via six equivalent μ_2 -formato bridges, leading to the formation of an extended coordination polymer. The equivalence of the Co-O bond distances arises directly from the symmetric bridging mode of the formate ligands in combination with the crystallographic symmetry of the lattice. The μ_2 -formato bridges propagate along multiple crystallographic

directions, generating infinite catena-type chains that are further interconnected to produce a robust three-dimensional coordination framework. The absence of terminally coordinated formate ligands or additional donor groups confirms the purely polymeric nature of the structure. The high degree of structural regularity within the coordination sphere is further enforced by the crystallographic symmetry, which mandates equivalence among all Co-O bonds.

A detailed polyhedral analysis was performed to quantitatively assess the coordination environment of the cobalt(II) centre. As summarized in Table 1, the Co(1) atom is coordinated by six oxygen atoms, all exhibiting identical Co-O bond lengths of 2.187(3) Å, with an average Co-O distance of 2.1871 Å. These values are in excellent agreement with those reported for octahedrally coordinated high-spin Co(II) centres in formate-based coordination polymers, thereby validating the structural model.

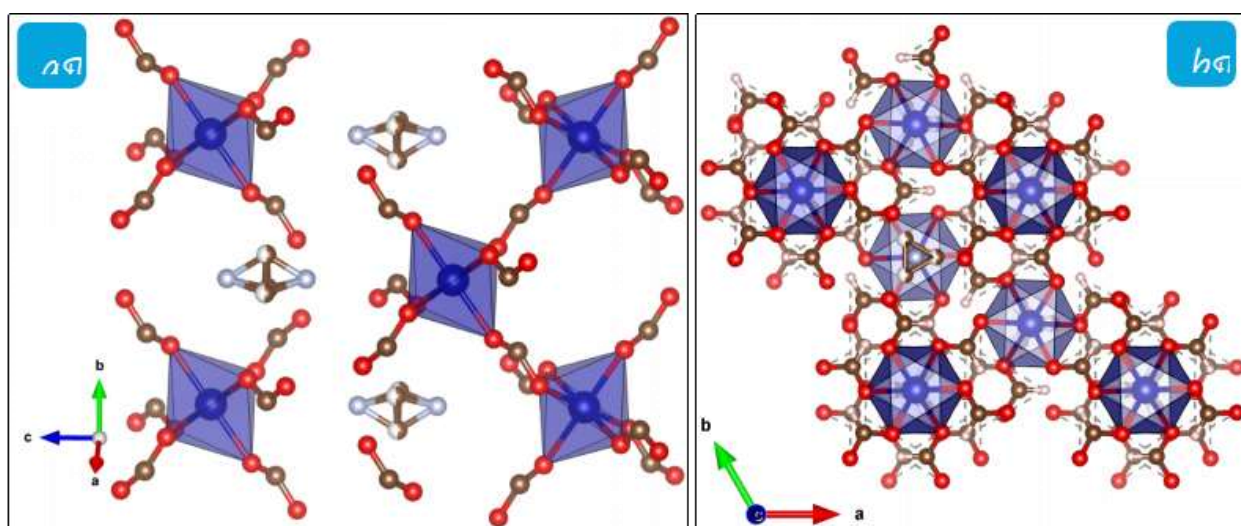


Figure 2. Polyhedral view of the Co(II) center in $[\text{Co}(\text{HCO}_2)_3]_n \cdot n(\text{C}_3)_2\text{N}$ showing the octahedral coordination geometry.

The effective coordination number (ECoN) of 6.0000 unequivocally confirms an ideal six-coordinate environment around the cobalt centre. The distortion index derived from the bond lengths is exactly zero, indicating complete equivalence of all Co-O distances and the absence of bond-length distortion within the coordination polyhedron. The polyhedral volume of 13.9461 Å³ is characteristic of a regular CoO₆ octahedron and reflects an optimal spatial arrangement of the coordinating oxygen atoms around the metal centre. Moreover, the quadratic elongation value of 1.0001 is extremely close to the ideal value of unity, signifying a negligible deviation from ideal octahedral geometry. In addition, the bond angle variance of 0.4685°² indicates only minimal angular distortion from the ideal O-Co-O angles of 90° and 180°.

Table 1. Polyhedral parameters for $[\text{Co}(\text{HCO}_2)_3]_n \cdot n(\text{C}_3)_2\text{N}$

Bond	Distance (Å)
Co(1)-O(1)	2.187(3)
Co(1)-O(1)	2.187(3)
Co(1)-O(1)	2.187(3)

Co(1)-O(1)	2.187(3)
Co(1)-O(1)	2.187(3)
Co(1)-O(1)	2.187(3)
Average bond length (Å)	2.1871
Polyhedral volume (Å ³)	13.9461
Distortion index (bond length)	0.00000
Quadratic elongation	1.0001
Bond angle variance (° ²)	0.4685
Effective coordination number	6.0000

Collectively, these polyhedral parameters demonstrate that the Co(II) centre in the title compound resides in an almost ideal octahedral coordination environment. The exceptionally low distortion indices and near-ideal quadratic elongation arise from the high crystallographic symmetry of the *R*-3*c* space group and the uniform μ_2 -formato bridging mode. Such a highly symmetric coordination environment is expected to play a significant role in determining the physical properties of the material, particularly its magnetic anisotropy and thermal stability, which are known to be sensitive to deviations from ideal octahedral symmetry.

Hirshfeld Surface Analysis of the Polymeric Coordination Compound

Hirshfeld surface (HS) analysis was employed to investigate the intermolecular interactions and packing features present in the polymeric coordination compound. Unlike discrete molecular systems, polymeric coordination compounds extend infinitely in one, two, or three dimensions; therefore, Hirshfeld surface analysis provides valuable insight into the non-covalent interactions operating between adjacent polymeric chains, layers, or frameworks within the crystal lattice.

The Hirshfeld surfaces were generated by partitioning the crystal space based on the Hirshfeld ratio, which defines the surface as the region where the electron density contribution from the reference polymeric fragment equals that of the surrounding fragments. A promolecule constructed from spherical atomic electron densities was used, with the surface defined at an electron density ratio of 0.5. This approach enables the visualization and quantitative assessment of intermolecular contacts between neighboring polymeric units.

$$d_{norm} = \frac{d_i - r_i^{vdW}}{r_i^{vdW}} + \frac{d_e - r_e^{vdW}}{r_e^{vdW}}$$

(1)

A key parameter derived from Hirshfeld surface analysis is the normalized contact distance (d_{norm}), which incorporates both the internal (d_i) and external (d_e) distances from the Hirshfeld surface to the nearest nuclei, normalized with respect to the van der Waals radii (r^{vdW}) of the interacting atoms. The d_{norm} function effectively highlights regions of significant intermolecular interactions within the polymeric crystal structure.

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CrystalData	
Formula	$\text{C}_{2.233}\text{H}_{2.233}\text{Co}_{0.744}\text{O}_{4.465,0.744}(\text{C}_{1.5}\text{N}_2)$
Formula Weight	178.62
Crystal System	trigonal
Space group	R-3c(No.167)
a, b, c [Å]	8.3313(5)8.3313(5)22.918(2)
α, β, γ [°]	9090120
V [Å ³]	1377.6(2)
Z	8
D(calc) [g/cm ³]	1.736
Mu (CuK α) [mm ⁻¹]	14.798
F(000)	714
Crystal Size [mm]	0.10x0.15x0.20
DataCollection	
Temperature (K)	293
Radiation [Å]	CuK α 1.54184
Theta Min-Max [°]	7.3,76.3
Dataset	-10:9;-10:10;-28:22
Tot., Uniq. Data, R(int)	2688, 323, 0.081
Observed Data [I > 0.0 sigma(I)]	296
Refinement	
N _{ref} , N _{par}	323, 25
R, wR2, S	0.0496, 0.1386, 1.13
Max. and Av. Shift/Error	0.00, 0.00
Min. and Max. Resd. Dens. [e/Å ³]	-0.39, 0.69
CCDC	2515357

Table 2. Crystal data and structure refinement for $[\text{Co}(\text{HCO}_2)_3]_n \cdot n(\text{C}_3)_2\text{N}$

The d_{norm} values are mapped onto the Hirshfeld surface using a red-white-blue color scale. Intense red regions correspond to close contacts shorter than the sum of the van der Waals radii, indicating strong interactions such as hydrogen bonding or metal-ligand-assisted contacts between adjacent polymeric units. White regions represent contacts approximately equal to the van der Waals separations, while blue regions indicate longer, weaker intermolecular interactions.

To further quantify the intermolecular contacts, three-dimensional (3D) fingerprint plots were generated, providing a comprehensive breakdown of the contributions from different types of interactions (e.g., $H\cdots H$, $O\cdots H/H\cdots O$, $C\cdots H/H\cdots C$). These plots enable a comparative evaluation of the dominant interactions governing the packing and stabilization of the polymeric coordination network. All Hirshfeld surface calculations and fingerprint analyses were performed using CrystalExplorer 21.5, which allowed detailed visualization and quantitative analysis of short-range intermolecular interactions within the polymeric coordination compound [16].

Hirshfeld analysis

In recent years, in the field of coordination chemistry, scientific interest in polymer complexes formed on the basis of transition metals has significantly increased. In particular, such compounds are a relevant object of research in terms of crystal engineering, magnetic and electronic properties, as well as potential biological and catalytic activity. In this work, the crystalline structure and intermolecular interactions of the coordination polymer katena- (dimethylammonium tris (μ_2 -formiato) -cobalt (II)) synthesized in the presence of cobalt (II) ions were studied in detail and thoroughly analyzed based on Hirshfeld surface analysis. According to crystallographic data, this complex has a three-dimensional (3D) infinite-chain polymer structure, in which Co (II) ions act as an μ_2 -bridge through the formate ($HCOO^-$) ligands. The ligand of each format binds two cobalt centers, leading to a spatially ordered arrangement of metal ions. As a result, a catene-type structural motif is formed in the crystal lattice, consisting of repeating metal-ligand-metal fragments.[17]

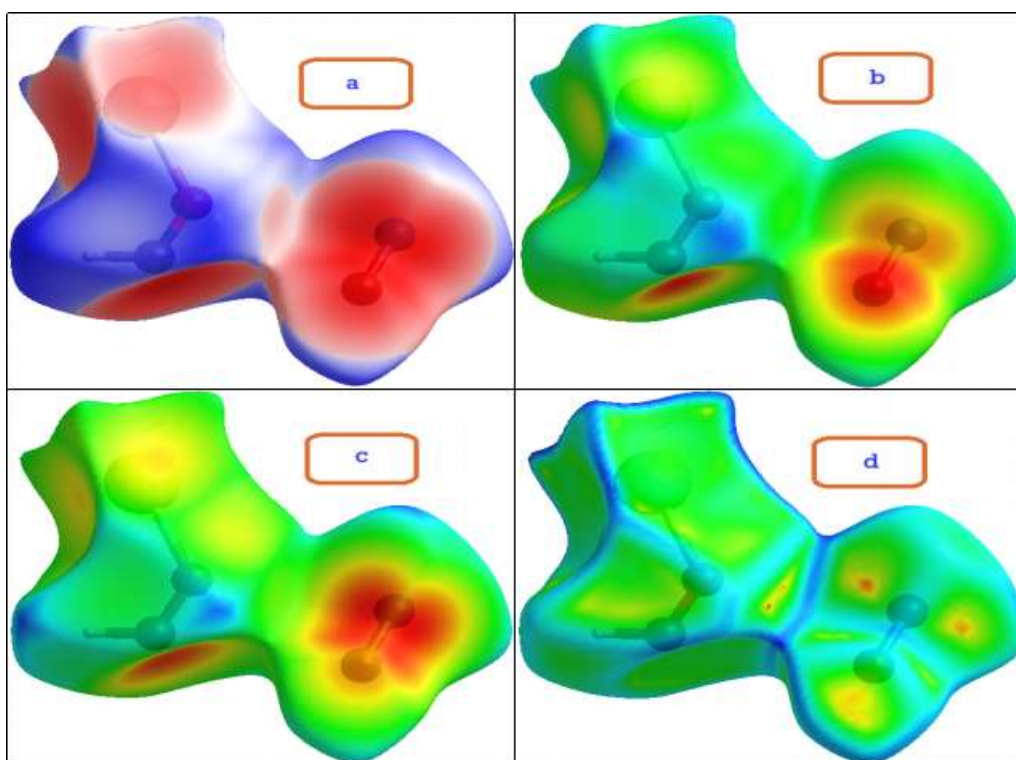


Figure 3. Hirshfeld surface maps: (a) d_{norm} , (b) d_i , (c) d_e , (d) curvedness

A coordination medium consisting of six oxygen atoms is formed around each Co (II) center, which indicates that the metal center is in a configuration close to distorted octahedral geometry.

The bidentate and bridge-forming properties of formate ligands cause a certain degree of asymmetry in the Co-O bond lengths and O-Co-O angles. This geometry influences the electronic environment of the metal ion, contributing to the overall stability of the complex. Dimethylammonium cations participate in the complex as counter ions, ensuring charge balance. At the same time, their role in the crystal lattice is not limited to electrostatic compensation.[18] For a deeper assessment of intermolecular interactions within the crystal structure, a Hirshfeld surface analysis was conducted. This method allows for a visual and quantitative description of interatomic contacts based on the electron density around the molecule or ion located within the crystal. The red intense regions observed on d_{norm} maps indicate the presence of short-distance and energetically important contacts. These regions mainly occupy a dominant position in the $\text{O}\cdots\text{H}/\text{H}\cdots\text{O}$ complex.

The distances with atoms on the inner and outer sides of the Hirshfeld surface were analyzed, respectively, using d_i and d_e maps. The results showed that oxygen atoms have the largest share outside the surface, actively interacting with hydrogen atoms. According to the table data, the contribution of O atoms outside the surface is the highest value, which once again confirms the decisive role of ligands of this format at the supramolecular level.

Table 3. Contribution of atoms of the same element inside and outside the surface to the formation of the Hirshfeld surface and their differences.

	Co	O	C	H	N
Inside	25.2.	13.2.	24.8	15.4.	21.4.
Outside	0.0	51.7	20.7.	17.7.	9.9.

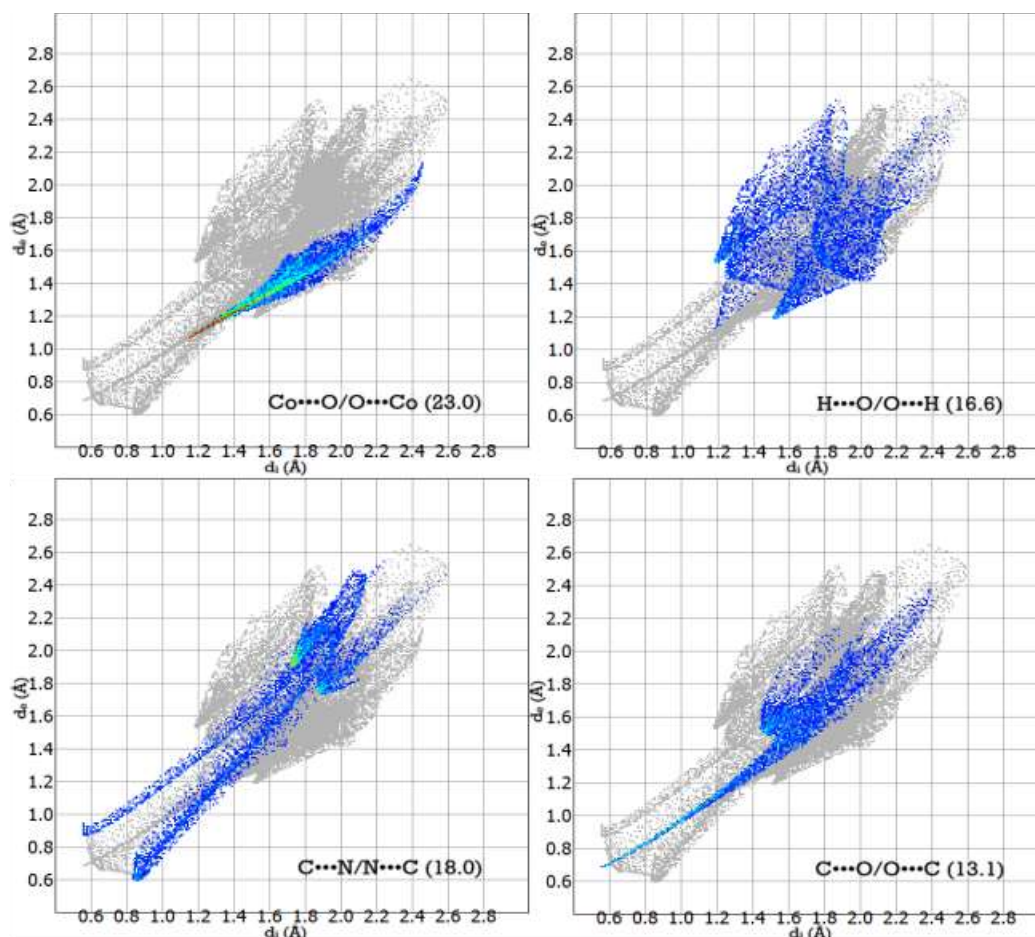


Figure 4. 2D representation of the Hirshfeld surface and the contributions of interatomic interactions to surface formation.

2D fingerprint diagrams accurately reflect the percentage distribution of intermolecular contacts. In these diagrams, $\text{H}\cdots\text{O}/\text{O}\cdots\text{H}$ interactions occupy the largest share, which indicates that the stability of the crystal structure is ensured mainly due to hydrogen bonds. In addition, there are weak Van der Waals interactions of the $\text{C}\cdots\text{O}$ types, which contribute to the density of the crystal lattice and the overall energy stability. The contribution of cobalt atoms to the Hirshfeld surface is mainly concentrated in the inner part of the surface, which indicates that the metal centers form strong coordination bonds with ligands. Nitrogen atoms are located in dimethylammonium cations, and their participation is mainly manifested through hydrogen bonds. In conclusion, the catena-(dimethylammonium tris (μ_2 -formiato) -cobalt (II)) complex is a well-ordered, structurally stable coordination polymer, in which metal-ligand coordination bonds and hydrogen bonds form a complex supramolecular system. Analysis of the Hirshfeld surface clearly demonstrated the leading role of oxygen atoms and hydrogen bonds in this complex. The obtained results can serve as an important scientific basis for the targeted design of new coordination polymers based on cobalt (II), predicting their physicochemical and functional properties.

Conclusion

In summary, the crystal structure of the cobalt(II) formate compound has been unambiguously elucidated by single-crystal X-ray diffraction analysis, revealing a purely polymeric coordination

architecture. Each formate ligand adopts a symmetric μ_2 -bridging coordination mode, linking adjacent Co^{2+} centers through oxygen atoms. As a result, each cobalt ion is uniformly connected to six neighboring cobalt centers via six equivalent μ_2 -formate bridges, leading to the formation of an extended and highly regular coordination polymer. The equivalence of all Co-O bond distances is enforced by the combined effect of the symmetric bridging mode of the formate ligands and the crystallographic symmetry of the lattice, confirming a well-ordered coordination environment. The μ_2 -formate bridges propagate along multiple crystallographic directions, generating infinite catena-type motifs that are further interconnected to produce a robust three-dimensional coordination framework. The absence of terminal formate ligands or additional coordinating donor groups further confirms the exclusively polymeric nature of the structure. Hirshfeld surface analysis provided deeper insight into the intermolecular interactions governing the supramolecular assembly of the crystal. The d_{norm} surfaces exhibit pronounced red regions corresponding to short and energetically significant intermolecular contacts, which are predominantly associated with $\text{O}\cdots\text{H}/\text{H}\cdots\text{O}$ interactions. Analysis of the d_i and d_e maps, together with the fingerprint plot data, demonstrates that oxygen atoms make the largest contribution to contacts outside the Hirshfeld surface, highlighting their dominant role in stabilizing the crystal packing.

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