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Issue Article

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SYNTHESIS AND PHYSICO-CHEMICAL CHARACTERIZATION OF MIXED LIGAND COMPLEX OF CADMIUM WITH 1,10-PHENANTHROLINE AND SALICYLIC ACID

Abstract: *The molecular salt [Cd($C_7H_5O_3$)($C_{12}H_8N_2$)2(H_2O)]($C_7H_5O_3$) was synthesized from cadmium chloride, 1,10-phenanthroline (phen) and 2-hydroxybenzoic (salicylic) acid in a 1:2:2 molar ratio and characterized by single-crystal X-ray diffraction, powder X-ray diffraction (PXRD), Raman spectroscopy and scanning electron microscopy with energy-dispersive X-ray analysis (SEM-EDS). The compound crystallized in the monoclinic space group $P2_1/n$. The Cd(II) atom is six-coordinate in a distorted CdN_4O_2 octahedron formed by four N atoms of two chelating phen ligands, one carboxylate O atom of a monodentate salicylate ligand and one aqua O atom. O-H···O and C-H···O hydrogen bonds connect the ions into a three-dimensional network. PXRD confirms a highly crystalline pure material. Raman spectroscopy reveals the characteristic vibrations of the aromatic phen and salicylate fragments, SEM shows blocky, layered crystals and EDS confirms the presence of Cd, C, N and O.*

Key words: *crystal structure, 1,10-phenanthroline, salicylate, 2-hydroxybenzoate, hydrogen bonding, powder X-ray diffraction, Raman spectroscopy, SEM-EDS.*

Language: *English*

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Introduction

Aromatic carboxylic acids and their derivatives are widely used as building blocks for the assembly of metal-organic architectures because of their versatile coordination modes, their ability to act as multidentate linkers, and their capacity to behave as both hydrogen-bond donors and acceptors [1,2]. 2-Hydroxybenzoic (salicylic) acid is of particular interest as a complexing agent in coordination and analytical chemistry, and it is also biologically relevant, with many of its functions being mediated through metal-ion complexation [3].

Chelating bipyridyl-type ligands such as 2,2′-bipyridine and 1,10-phenanthroline (phen) are frequently employed as auxiliary ligands in such systems, since the rigid, planar phen unit both saturates the coordination sphere of the metal and promotes supramolecular organization through π-π stacking [4,5]. As part of our continuing studies on the coordination chemistry of hydroxybenzoic acid derivatives with d-block metals [6,3], we report here the synthesis, crystal structure, powder X-ray diffraction, Raman spectroscopic and SEM-EDS characterization of the title cadmium salicylate-phenanthroline complex (next compound (I)).

1. Literature review

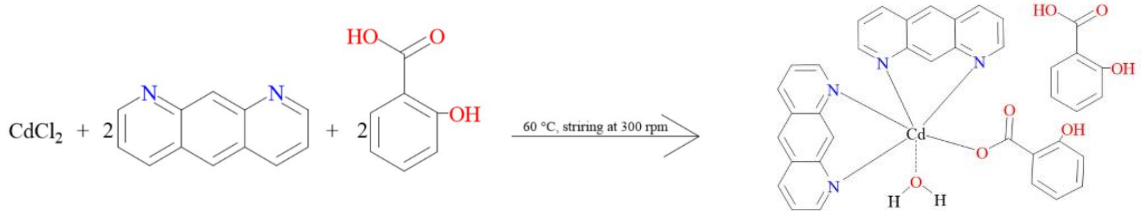
A survey of the Cambridge Structural Database (CSD version 5.45 [7]; Mercury [8]) shows that the [M(carboxylate)(phen)$_2$(H_2O)]$^+$ cation is a recurring structural motif for first- and second-row divalent metals. The most closely related structure is aqua(3-hydroxybenzoato-κO)bis(1,10-phenanthroline)cobalt(II) 3-hydroxybenzoate pentahydrate [9], which is essentially isostructural at

the cation level, the Co(II) atom adopting the same distorted cis-MN4O2 octahedral coordination and the structure being held together by an extensive O-H···O hydrogen-bonded network with π-π stacking between parallel phen rings.

Analogous monocationic complexes with the same coordination motif have been reported for the cobalt(II) salts of hippuric acid [10] and 3-methoxycinnamic acid [11]. For cadmium(II), structurally characterized phen complexes are also well represented [4,5,12], although examples combining two chelating phen ligands with a monodentate salicylate and an aqua ligand remain comparatively rare. These metric and supramolecular features are comparable with those reported for related $[M(carboxylate)(phen)_2(H_2O)]^+$ cations [9-11].

2. Research methodology

Synthesis and crystallization. The title compound was prepared by the reaction of cadmium chloride, 1,10-phenanthroline and 2-hydroxybenzoic (salicylic) acid in a 1:2:2 molar ratio. The reactants were combined in aqueous medium and the mixture was heated at 60 °C with continuous stirring at 300 rpm. The resulting clear solution was left to evaporate slowly at room temperature, light-yellow needle-shaped crystals of (I) suitable for single-crystal X-ray diffraction were obtained after several days.



Scheme 1. Synthesis of the title compound (I).

Single-crystal X-ray diffraction. Intensity data were collected at 293 K on a Rigaku XtaLAB Synergy diffractometer equipped with a HyPix3000 detector using Cu Kα radiation (λ=1.54184 Å). Data were processed and a multi-scan absorption correction was applied with CrysAlis PRO [13]. The structure was solved by dual-space methods with SHELXT [14] and refined by full-matrix least-squares on F^2 with SHELXL [15] within the OLEX2 interface [16]. All non-H atoms were refined anisotropically. A Hirshfeld surface analysis was carried out with CrystalExplorer [17].

Powder X-ray diffraction (PXRD). The powder diffraction pattern was recorded on a Rigaku diffractometer with Cu Kα radiation ($K\alpha_1$ = 1.540593 Å, K_{α_2}=1.544414 Å) operated at 40 kV and 15 mA, in a θ/2θ scan over the 2θ range 3-90° with a step of 0.01° (Table 1).

Raman spectroscopy. Raman spectra were recorded on a HORIBA spectrometer (LabSpec 6 software) using 785 nm laser excitation, a 685 grating, an acquisition time of 1s and 1 accumulation, with the intensity-correction system (ICS) on, over the 200-3600 cm^{-1} range.

Scanning electron microscopy and EDS. Morphology was examined with a JEOL JSM-IT210 scanning electron microscope in high-vacuum mode. Elemental analysis was performed by energy-dispersive X-ray spectroscopy (EDS) at an accelerating voltage of 15.0 kV (working distance 15.2 mm).

Table 1. Crystal Data and Details of the Structure Determination for (I)

Crystal Data	
Formula	$C_{31}H_{23}CdN_4O_4,C_7H_5O_3$
Formula Weight	765.04
Crystal System	monoclinic
Space group	P21/n(No. 14)
a, b, c [Å]	10.2873(1) 13.7512(1) 23.1956(2)
alpha, beta, gamma [°]	90 94.508(1) 90
V [$Å^3$]	3271.16(5)
Z	4
D(calc) [g/cm^3]	1.554
Mu(CuKα) [mm]	5.839
F(0)	1552

Crystal Size [mm]	0.11 x 0.12 x 0.14
Data Collection	
Temperature (K)	293
Radiation [Å]	CuKα 1.54184
Theta Min-Max [Deg]	3.7, 68.2
Data set	-12: 12 ; -16: 16 ; -27: 24
Tot., Uniq. Data, R(int)	32360, 5947, 0.122
Observed Data [I > 0.0 sigma(I)]	5511
Refinement	
Nref, Npar	5947, 455
R, wR2, S	0.0450, 0.1249, 1.02
Max. and Av. Shift/Error	0.00, 0.00
Min. and Max. Resd. Dens. [e/Å^3]	-1.03, 1.80

3. Results and discussion

Structural commentary. The asymmetric unit of (I) consists of one $[Cd(phen)_2(Hbz)(H_2O)]^+$ complex cation and one uncoordinated salicylate anion (Hbz^-). The compound is therefore a 1:1 molecular salt (Fig. 1). The Cd(II) atom is six-coordinate and lies in a distorted octahedral CdN4O2 environment built from four N atoms (N1, N2, N3 and N4) of two chelating phen ligands, one carboxylate O atom (O4) of a monodentate salicylate ligand and one water O atom (O1).

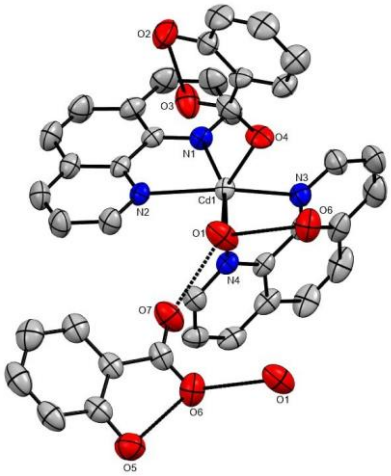


Figure 1. The molecular structure of (I) shown with displacement ellipsoids at the 50% probability level. Dashed lines indicate O-H···O hydrogen bonds. H atoms not involved in hydrogen bonding are omitted for clarity.

The Cd-O bond lengths Cd1-O4 = 2.245 Å and Cd1-O1 = 2.274 Å are shorter than the Cd-N bond lengths, which span 2.356-2.420 Å (Table 2). The two phen ligands chelate the metal atom with acute bite angles N1-Cd1-N2 = 70.60° and N3-Cd1-N4 = 69.65°, which are responsible for the principal deviations from ideal octahedral geometry. The largest trans-type angles are O1-Cd1-N1 = 162.12°, O4-Cd1-N4 = 150.88° and N2-Cd1-N3 = 143.11°, confirming the marked distortion of the coordination polyhedron. The salicylate ligand coordinates through only one carboxylate O atom, in both the coordinated and the uncoordinated salicylate units the phenol -OH group forms an intramolecular O-H···O hydrogen bond closing a six-membered S(6) ring characteristic of ortho-hydroxybenzoate species.

Table 2. Selected bond lengths (Å) and angles (°)

Bond	Value	Bond/Angle	Value
Cd1-O4	2.245 (2)	Cd1-N3	2.369 (3)
Cd1-O1	2.274 (2)	Cd1-N1	2.414 (2)

Cd1-N2	2.356 (2)	Cd1-N4	2.420 (2)
O4-Cd1-N4	150.88 (9)	O4-Cd1-N2	122.44 (9)
O1-Cd1-N1	162.12 (10)	O1-Cd1-N3	113.00 (12)
N2-Cd1-N3	143.11 (8)	O4-Cd1-O1	81.04 (9)
N1-Cd1-N2	70.60 (8)	N3-Cd1-N4	69.65 (8)

Supramolecular features. In the crystal, the complex cation and the salicylate anion are linked into a charge-assisted ion pair by two hydrogen bonds donated by the aqua ligand: O1-H1A···O7 and O1-H1B···O6 (Table 3), both acceptor atoms belonging to the carboxylate group of the uncoordinated salicylate anion. These O-H···O interactions, together with weaker C-H···O contacts involving the phen H atoms C8-H8···O3, C10-H10···O7 and C35-H35···O4, connect the ions into a three-dimensional supramolecular network.

Table 3. Hydrogen Bonds for (I)

D-H···A	d(D-H)	d(H···A)	d(D···A)	<(DHA)
O1-H1A-O7	0.86	2.19	2.646(3)	113
O1-H1B-O6	0.86	1.92	2.734(4)	156
O2-H2-O3	0.82	1.81	2.535(4)	146
O5-H5A-O6	0.82	1.83	2.552(4)	146
C8-H8-O3	0.93	2.51	3.248(4)	136
C10-H10-O7	0.93	2.57	3.407(5)	151
C35-H35-O4	0.93	2.60	3.379(5)	142

Hirshfeld surface analysis. A Hirshfeld surface analysis of the complex cation shows that the most important contributions to the surface arise from H···H contacts (36.9%) and H···C/C···H contacts (34.9%), the latter reflecting C-H···π interactions associated with the phen ligands. H···O/O···H contacts account for 18.4% and correspond to the O-H···O and C-H···O hydrogen bonds. Smaller contributions come from C···C (6.4%), N···H/H···N (2.5%), N···C/C···N (0.6%) and O···C/C···O (0.4%); the C···C term is consistent with π-π interactions between the aromatic phen systems.

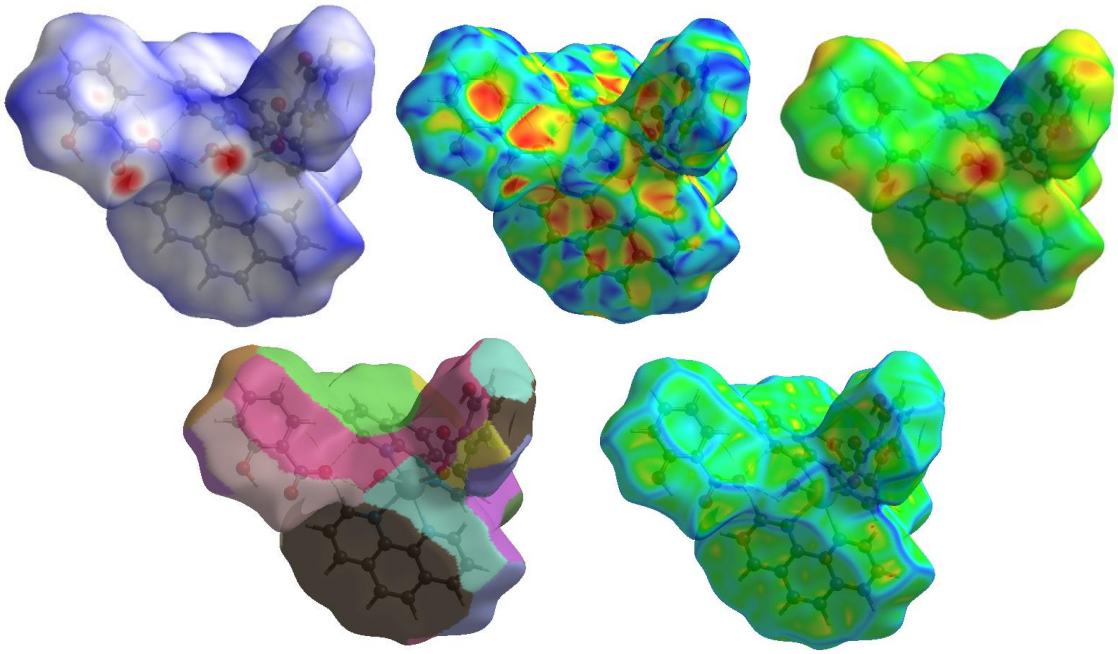


Figure 5. Hirshfeld surface maps of (I): (a) d□ₒ□□ (b) shape index, (c) dᵢ, (d) fragment patch, and (e) curvedness.

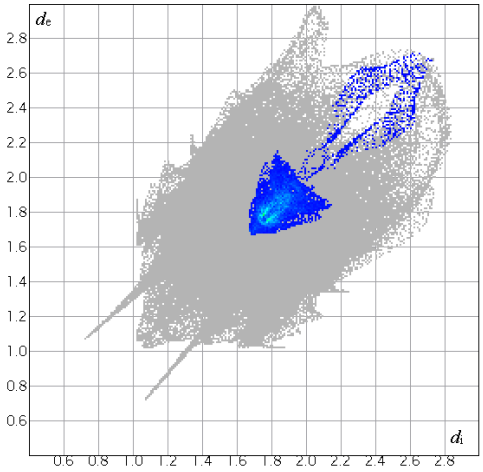


Figure 6. Two-dimensional fingerprint plot of the Hirshfeld surface of (I). The full fingerprint (all contacts) is shown.

Powder X-ray diffraction. The PXRD pattern of the bulk sample (Fig. 2) displays numerous sharp, well-resolved reflections, indicating that the product is a highly crystalline solid. The diffracted intensity is concentrated in the low-angle region (2θ < 30°), with the dominant reflection at 2θ=11.44° (d =7.73 Å) and further strong reflections at 8.38° (10.54 Å), 8.71° (10.14 Å), 9.59° (9.22 Å) and 22.97° (3.87 Å). The prominence of these low-angle reflections is consistent with the relatively large monoclinic unit cell obtained from the single-crystal analysis a=10.2873, b=13.7512, c=23.1956 Å, β=94.508°.

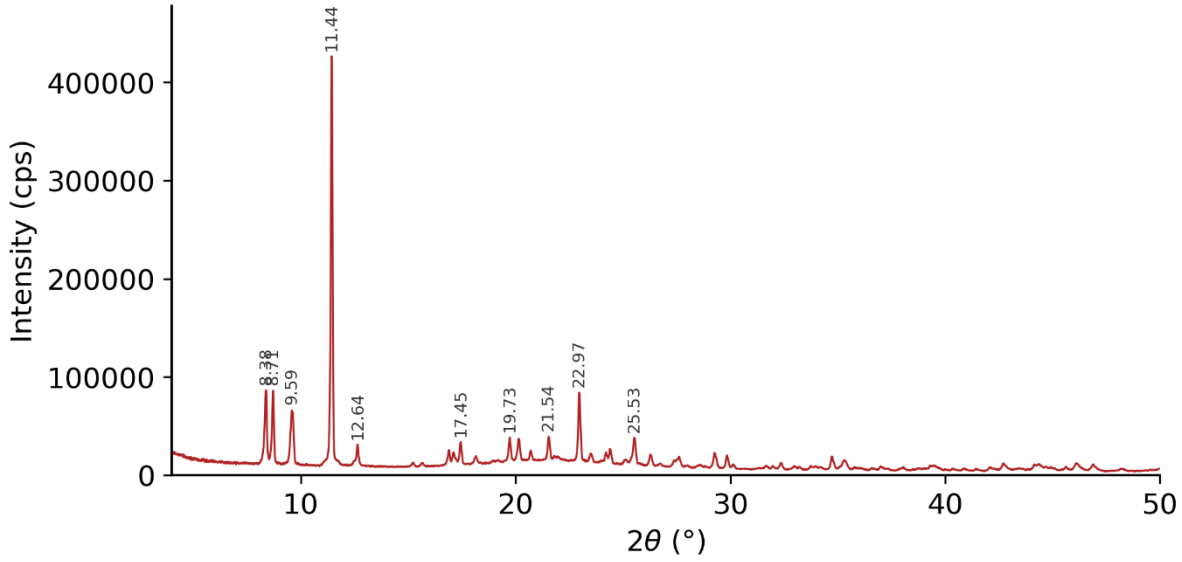


Figure 2. Powder X-ray diffraction pattern of (I)

Raman spectroscopy. The Raman spectrum of (I) (Fig. 3) shows the vibrational bands expected for a compound containing aromatic 1,10-phenanthroline and salicylate components. The observed band positions and their tentative assignments are summarized in Table 4. The strong, sharp band at 1003 cm^{-1} is characteristic of an aromatic ring-breathing mode, while the bands in the 1500-1600 cm^{-1} region (1520 and 1563 cm^{-1}) correspond to aromatic C=C /C=N ring-stretching vibrations of the phen and salicylate rings. The band at 1775 cm^{-1} lies in the region of carboxyl related stretching, and the band at 3023 cm^{-1} is assigned to aromatic C-H stretching. The bands below 800 cm^{-1} (240, 473, 543 and 792 cm^{-1}) arise from ring-deformation and skeletal modes. The gradual rise of the baseline above 3000 cm^{-1}, together with the broad feature near 3360-3520 cm^{-1}, is attributed to O-H stretching of the water. Under 500 cm^{-1} spectres demonstrate M-O and M-N connections.

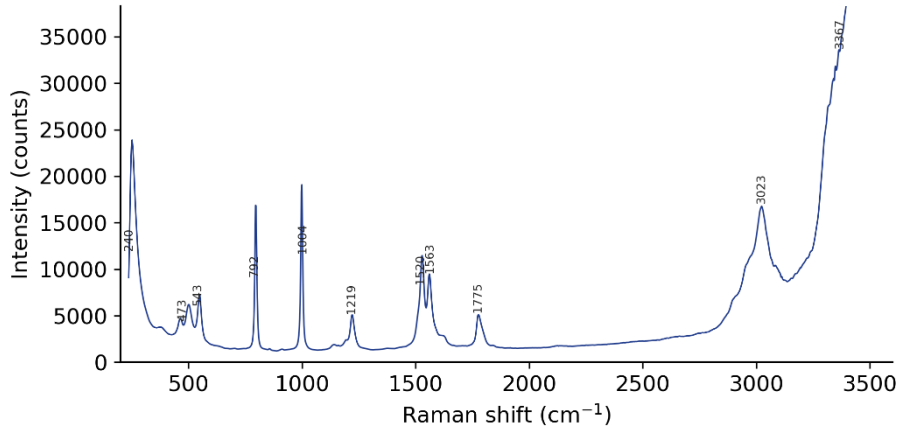


Figure 3. Raman spectrum of (I)

Table 4. Observed Raman bands for (I)

Raman shift (cm^{-1})	Tentative assignment
240, 473	M-O/N bonds
1003	aromatic ring-breathing mode
1219	aromatic C-H in-plane bending
1520, 1563	aromatic C=C/C=N ring stretching (phen, salicylate)
1775	carboxyl/carbonyl-related stretching
3023	aromatic C-H stretching
3360-3520 (broad)	O-H stretching (aqua/hydroxy) on a fluorescence background

SEM and EDS analysis. Scanning electron microscopy (Fig. 4) shows that the sample consists of compact, blocky crystals built from stacked plate-like crystallites with smooth fracture faces, layered surfaces and sharp, well-defined edges, tens of micrometres in size. Morphology typical of a well-crystallized bulk solid. The elemental composition determined by EDS (Table 5) confirms the presence of cadmium together with carbon, oxygen and nitrogen, which the constituent elements expected for the [Cd(phen)$_2$(salicylate)(H$_2$O)](salicylate) complex. The measured atomic ratios (C 76.98, O 15.14, Cd 5.74 and N 2.14 at.%) reproduce the qualitative composition of the compound; the relatively low apparent nitrogen content reflects the well-known limitations of standardless ZAF quantification for light elements present at low concentration, so the EDS data are taken as confirming the elemental identity rather than as a precise quantitative measure.

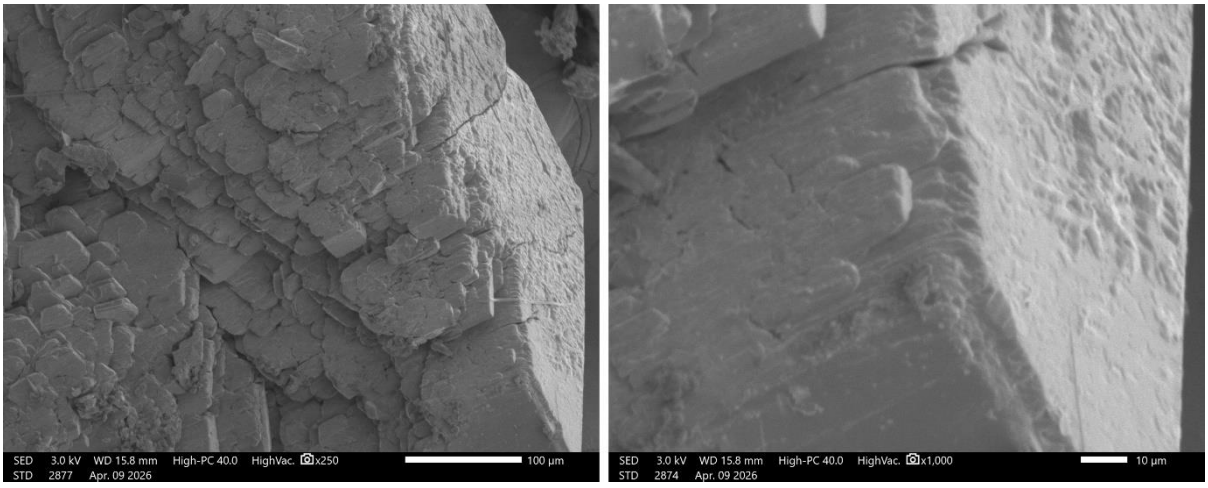


Figure 4. SEM micrographs of (I)

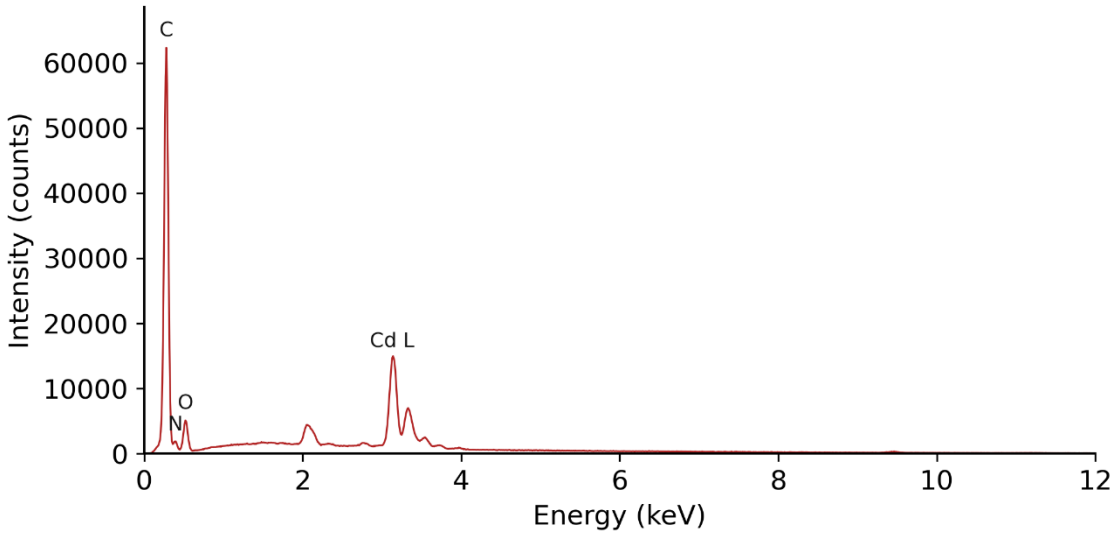


Figure 7. EDS spectrum of (I), showing the C, N, O and Cd lines

Table 5. EDS elemental composition of (I)

Element	Mass %	Atom %
C	50.18 ± 0.07	76.98 ± 0.11
N	1.63 ± 0.07	2.14 ± 0.09
O	13.15 ± 0.12	15.14 ± 0.14
Cd	35.04 ± 0.15	5.74 ± 0.02
Total	100.00	100.00

4. Conclusion

A cadmium(II) salicylate-phenanthroline complex $[Cd(C_7H_5O_3)(C_{12}H_8N_2)\ 2(H_2O)](C_7H_5O_3)$, was synthesized and characterized by complementary structural, diffraction, spectroscopic and microscopic methods. Single-crystal X-ray diffraction established a monoclinic ($P2_1/n$) structure in which the Cd(II) atom adopts a distorted CdN4O2 octahedral coordination, and O-H···O and C-H···O hydrogen bonds assemble the cations and anions into a three-dimensional network, as supported by the Hirshfeld surface analysis. Powder X-ray diffraction confirmed that the bulk product is highly crystalline, with intense low-angle reflections consistent with the large monoclinic unit cell. Raman spectroscopy revealed the characteristic vibrational signatures of the aromatic phenanthroline and salicylate fragments, while SEM showed a well-crystallized, layered morphology and EDS confirmed the presence of Cd, C, N and O.

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