

Synthesis, crystal structure and properties of tris(benzene-1,2-diamine-N,N')-cadmium naphthalene-1,5-disulfonate trihydrate complex compound

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In this research work, for the first time, tris(benzene-1,2-diamine-N,N')-cadmium naphthalene-1,5-disulfonate trihydrate ($[\text{Cd}(\text{OPD})_3]1,5\text{-NDS}\cdot 3\text{H}_2\text{O}$) complex has been synthesized. The molecular and crystal structures of the synthesized complex have been studied using the RTT method. In the crystal structure of the $[\text{Cd}(\text{OPD})_3]1,5\text{-NDS}\cdot 3\text{H}_2\text{O}$ complex, 3 OPD molecules are chelated to the central Cd(II) ion in the inner sphere. In the outer sphere, there are 1,5-NDS and 3 water molecules. The crystal structure of the complex compound is monoclinic, the spatial group is P21/c, and the spatial shape is distorted octahedral. In the crystal structure of the molecule, there are internal and intermolecular hydrogen bonds of the type O–H...O, N–H...O and C–H...O. For the first time, Hirshfeld surface analysis of this complex compound has been performed using the Crystal Explorer program. In this case, H...H (42%), O...H/H...O (32.6%) and H...N/N...H (23.5%) Van Der Waals forces in the molecule form the main interactions. Also, the physicochemical properties of this complex compound have been studied using IR-spectroscopic and thermal analysis. In the thermal analysis of the complex **2**, endothermic effects are observed, and mass reduction takes place in 4 stages.

Keywords: Complex, *o*-Phenylenediamine, X-ray structure analysis, Hirshfeld surface analysis, Thermal analysis

o-Phenylenediamine is one of the most important ligands in coordination chemistry. Complex compounds obtained based on *o*-phenylenediamine are used to produce fungicides and herbicides in agriculture¹. In medicine, it is used in the treatment of cancer and malaria². The cytotoxic properties of $[\text{PtCl}_4(\text{OPD})]_2\text{H}_2\text{O}$ and $[(\text{RuCl}_2(\text{OPD})(\mu\text{Cl}))_2]\cdot 2\text{H}_2\text{O}$ formed by *o*-phenylenediamine with Pt(IV) and Ru(III) cations were studied. These complexes have been proven to be bioactive against cancer cell strains³. Because $-\text{NH}_2$ groups are located next to each other in the *o*-phenylenediamine molecule, the electron density increases. For this reason, its ability to form complexes is high compared to other isomers^{4,5}. The nitrogen atom is in the $-\text{sp}^3$ hybridized state, and one N–H bond in the $-\text{NH}_2$ group lies in the same plane as the benzene ring. Therefore, the possibility of homolytic cleavage of nitrogen and hydrogen atoms is high, and this serves as the main source of hydrogen in photochemical 3d transition processes^{6,7}.

Various complex compounds formed by *o*-phenylenediamine with some d-metals and the

bonding mechanisms between them were studied. Yann-Teller effect is observed in some complex compounds formed by *o*-phenylenediamine with Cu(II) ion^{8,9}. Also, a conformation phenomenon occurs in the $[\text{Ni}(\text{OPDA})_2(\text{NCS})_2]$ complex formed by *o*-phenylenediamine with Ni(II) cation. There are yellow and purple conformational isomers of this complex, and their properties differ from each other^{10,11}. *o*-Phenylenediamine complexes with 3d metals are mostly octahedral and stable. In the complex formation of OPD with Ni(II) cation $[\text{Ni}(\text{OPD})_2(\text{H}_2\text{O})_2]\cdot \text{NDS}$, two OPD molecules combine with the central atom in a bidentate state. Since OPD forms a chelate complex, the stability of the complex increases further^{10,11}. This can be observed in the complex formed by OPD with Cd(II) cation $[\text{Cd}(\text{C}_6\text{H}_8\text{N}_2)_4](\text{C}_{10}\text{H}_6\text{O}_6\text{S}_2)$ ¹². Due to the strong hydrogen bonding and chelation of OPD with the central atom in the $[\text{Zn}(\text{OPDA})_2(\text{NO}_3)_2]$ complex obtained based on OPD, it was found that the thermal stability and stability of the complex in solvents is much higher. In the complex compound, the

coordination number of the central atom is equal to 6 and it is located in the octahedral position in space^{13,14}. Complex compounds of the Zn(II) cation in most cases form tetrahedral complexes. The electronic structure of this complex compound was thoroughly studied by the DFT method^{15,16}. The spatial form of complex compounds is explained by its electronic structure and the nature of ligands.

In addition, molecular complexes of OPD ligands with other ligands and their crystal structures have been thoroughly studied. In the supramolecular structure of these complex compounds, hydrogen bonds dominate in most cases^{17,18}. In the molecular complex obtained based on L-tartaric acid (TART) and *o*-phenylenediamine ($[(OPD)^+(TART)^-]$), cation (OPD^+) and L-tartaric acid change to anion state as a result of electron transfer of *o*-phenylenediamine to di (TART)¹⁹.

The purpose of the study is to obtain a new complex compound of *o*-phenylenediamine with a mixed ligand of Cd (II) ion and to study the composition, molecular and crystal structure and other physicochemical properties of the obtained complex compound using modern research methods.

Materials and Methods

In this research work, compounds such as cadmium sulfate crystalline hydrate salt, *o*-phenylenediamine ligand and disodium salt of naphthalene 1,5-disulfoacid were used. All chemical reagents were purchased as "chemically pure" from "Merit Chemicals" company.

X-ray structural analysis (RTT)

The composition, molecular and crystal structures of the complex obtained in this research work were determined by the RTT method on an automatic Xcalibur® Oxford Diffraction diffractometer (graphite monochromator, Cu-K α -radiation $\lambda=1.54184$ Å).

IR-analysis

The structure of this obtained complex compound was determined by IR-spectroscopy (Fure spectrometer manufactured in Japan. IR spectroscopic studies were carried out in the powder method on a Shimadzu infrared Fourer spectrometer (range 4000-600 cm^{-1} , dimensions 4 cm^{-1}).

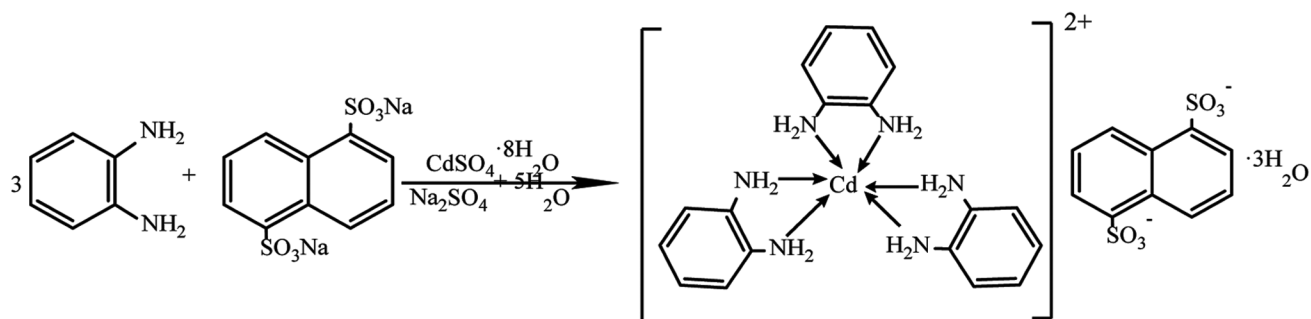
The simultaneous TG-DTA study

The DTG-60 equipment from Shimadzu was used to obtain results from thermogravimetric (TG) and differential thermal analysis (DTA). The tested sample was initially held at 30 °C in an argon atmosphere with a flow rate of 100 mL/min for 10 min, followed by heating at a rate of 10°C/min.

Experimental Section

Complex compound synthesis

In this research work, we used a metal salt and two different ligands. 1 mmol (0.352 g) of cadmium sulfate crystalline hydrate salt ($CdSO_4 \cdot 8H_2O$) was weighed on an analytical balance and an aqueous solution was prepared. A 3 mmol (0.324 g) solution of the *o*-phenylenediamine ligand in ethanol and a 1 mmol (0.332 g) aqueous solution of the disodium salt of naphthalene 1,5-disulfoacid were prepared. First, a solution of *o*-phenylenediamine was added to the cadmium sulfate crystal hydrate salt solution and stirred for 30 minutes at RT using a magnetic stirrer. Then we pour into the reaction mixture a solution of the disodium salt of naphthalene 1,5-disulfoacid, and it was stirred first at RT, and then at 50-55°C for another 2 hours on a magnetic stirrer. The resulting reaction mixture was divided into 5 vials and observed at RT for 12 days ($pH=6.5$). At the end of the process, dark-yellow crystals were obtained at the bottom of the vials with a yield of 80% by slow evaporation (Scheme 1). The molecular and crystal structures of



Scheme 1 — Synthesis reaction of complex compound $[Cd(OPD)_3]_{1,5-NDS} \cdot 3H_2O$

the produced single crystals were determined using the RTT method.

IR spectrum analysis of complex compound

In the IR-spectrum of the OPD molecule depicted in Fig. 1, it is possible to observe the valence vibration of the NH_2 -group in the doublet state in the highly intense region of 3385 and 3361 cm^{-1} . The valence vibration of C-N bonds can be observed in the low-intensity 1273 cm^{-1} region of the spectrum. The valence vibration of the aromatic ring along the plane

of the ring was observed in the absorption part of 1776 cm^{-1} .

In the IR-spectrum of the complex compound $[\text{Cd}(\text{OPD})_3]1,5\text{-NDS}\cdot 3\text{H}_2\text{O}$ described above, it is possible to see the presence of OH groups in the molecule in the broad intensive area of 3385 cm^{-1} and 3325 cm^{-1} . In the absorption region of 3186 cm^{-1} of the spectrum, hydrogen bonds formed by water molecules can be observed (Fig. 2). Deformation vibration of the NH_2 group in the molecule was observed in the absorption region of 1236 cm^{-1} and

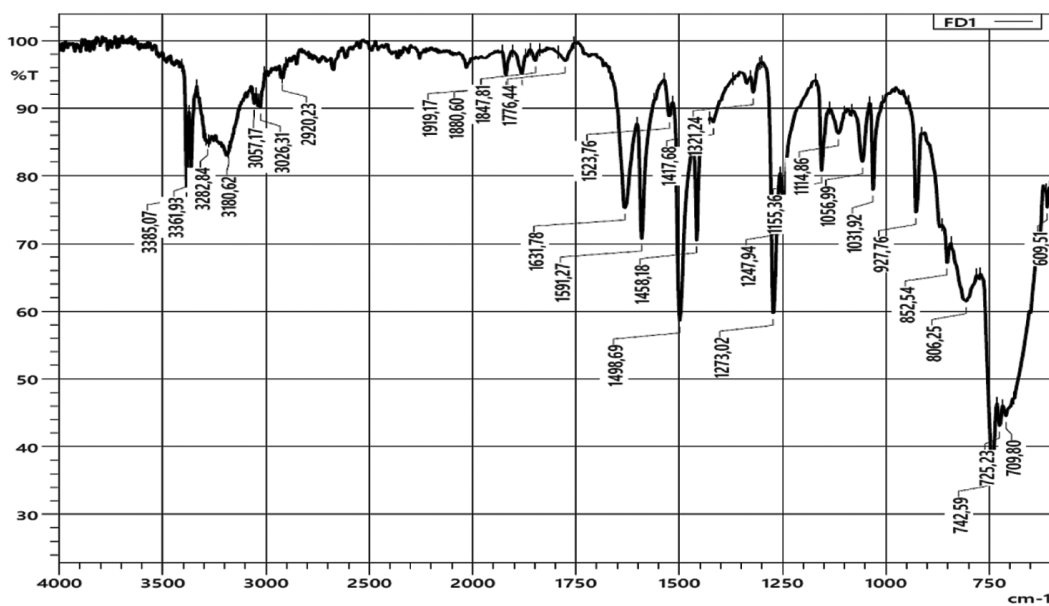


Fig. 1 — IR spectrum of OPD ligand

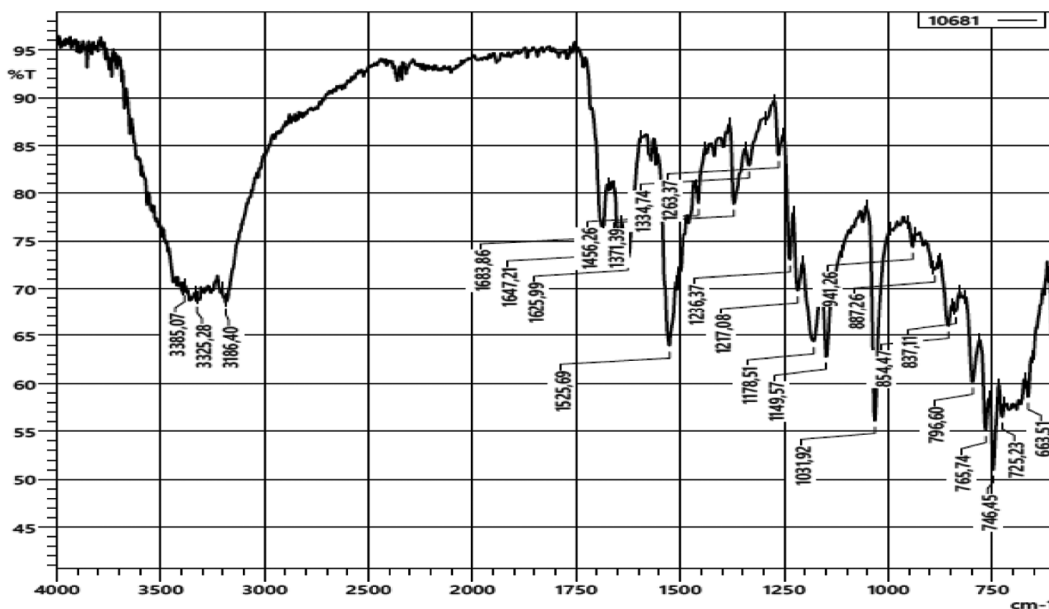


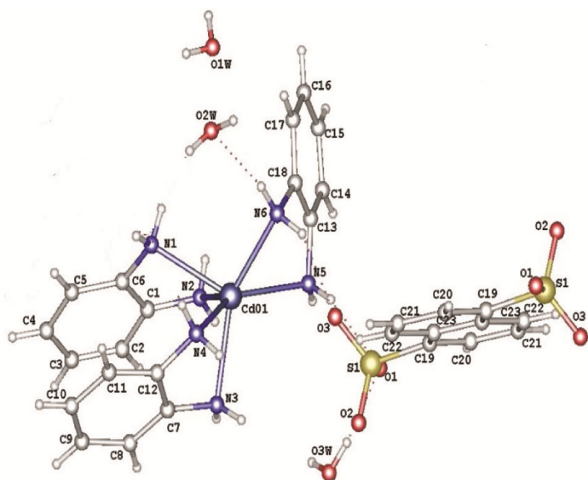
Fig. 2 — IR spectrum of the complex compound $[\text{Cd}(\text{OPD})_3]1,5\text{-NDS}\cdot 3\text{H}_2\text{O}$.

1217 cm^{-1} . The valence vibration of the S=O group in the asymmetric state is observed in the region of 1371 cm^{-1} . The vibration of the benzene ring along the ring plane was observed in the absorption region of 1683 cm^{-1} , and the Cd---N donor-acceptor bonds in the complex compound were observed in the region of 663 cm^{-1} .

Results and Discussion

X-ray crystal structure

[Cd(OPD)₃]₁,5-NDS·3H₂O crystal structure monoclinic and spatial structure P21/c. The crystal occupies a distorted octahedral shape in space. In the crystal structure of [Cd(OPD)₃]₁,5-NDS·3H₂O complex compound, 3 OPD molecules in the inner sphere are connected with cadmium metal in a chelate state using donor-acceptor bonds. In the outer sphere of the complex, 1 NDS molecule is connected with the inner sphere using ionic bonds (Fig. 3a)^{20,21}. The distance between neighbouring nitrogen atoms attached to the central atom (Cd–N) is 2.340 Å–2.447 Å. Also, bidentate nitrogen atoms bound to the central atom form bond angles N1–Cd01–N2 67.74(9)°, N3–Cd01–N4 68.30(9)°, and N5–Cd01–N6 69.35(9)°. 3 water molecules in the outer sphere are connected with the inner and outer spheres using hydrogen bonds. Internal and intermolecular hydrogen bonds of the type O–H...O, N–H...O and C–H...O form the main crystal structure of the molecule in the composition of the complex compound (Table 1 and Table 2). Water molecules in the outer sphere form hydrogen bonds of the type O3W–H3WA...O1, O3W–H3WB...O2, O2W–H2WA...O4 and O2W–H2WB...O1W (Fig. 3b).



Hirschfeld surface analysis

Hirshfeld surface analysis is one of the main methods for expressing the weak interaction of particles in crystalline substances. Hirshfeld surface analysis allows visualization of near and far effects on the van der Waals radii of molecules and the interaction force of particles^{22,23}.

The Hirschfeld surface analysis of this complex compound was studied in the cationic state and the total state. C–H...O, O–H...O and N–H...O intermolecular hydrogen bonds are present in the crystal structure of the complex compound. Also, O2W–H2WA...O4 and N6–H6B...O2W internal hydrogen bonds are arranged in a zigzag pattern in the molecule. The above supramolecular interactions were shown in the form of red spots in the Hirshfeld surface analysis in the CrystalExplorer program. The d_{normal} standard sizes of red and blue s_{spots} are -0.5997 and 1.2276, respectively. Here, the red color indicates bonds closer to the sum of Van Der Waals radius. The blue dots show bonds longer than the sum of the Van Der Waals radius^{24,25}. According to the d_{normal} of this complex compound, the main part of the red spots on the Hirshfeld surface of the molecule are hydrogen bonds (Fig. 4). The Hirschfeld surface area is $S = 774.17 \text{ \AA}^2$ and the volume is $V = 1047.30 \text{ \AA}^3$. The interaction of internal particles in the structure of the crystal was determined using the d_e two-dimensional functions and d_i the interactions on the crystal surface. The main interactions of atoms in the molecule are due to the contribution of H...H (42%), O...H/H...O (32.6%) and H...N/N...H (23.5%) bonds. These effects create spikes with growing tips in a two-dimensional fingerprint (Fig. 5).

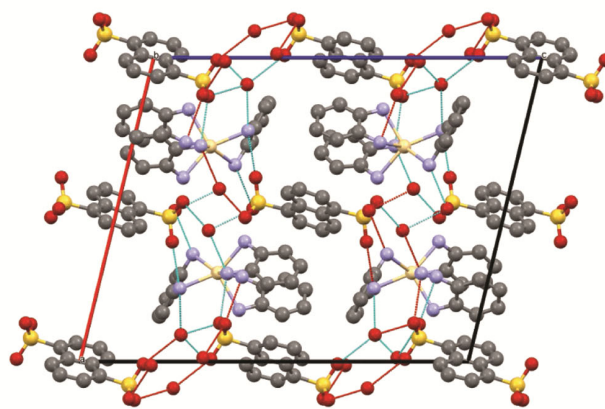


Fig. 3 — (a) Molecular structure of the complex [Cd(OPD)₃]₁,5-NDS·3H₂O. (b) Plotting of the complex compound [Cd(OPD)₃]₁,5-NDS·3H₂O along the c axis (blue lines depict hydrogen bonds).

Table 1 — Geometry of hydrogen bonds of complex compound $[\text{Cd}(\text{OPD})_3]1,5\text{-NDS}\cdot 3\text{H}_2\text{O}$

D-H...A	D-H, Å	H...A, Å	D...A, Å	DHA, angle
O3W-H3WA...O1	0.8500	1.9700	2.812(4)	172.00
N1-H1B...O5	0.89(3)	2.23(3)	3.010(4)	147(3)
O3W-H3WB...O2	0.8500	2.0100	2.849(4)	168.00
N2-H2A...O5	0.89(3)	2.39(3)	3.129(3)	141(3)
N2-H2B...O4	0.89(2)	2.58(2)	3.363(4)	147(3)
O2W-H2WA...O4	0.8500	2.0600	2.852(4)	155.00
N3-H3A...O2	0.89(3)	2.34(3)	3.083(4)	141(3)
O2W-H2WB...O1W	0.8500	2.5600	3.042(6)	117.00
N4-H4A...O4	0.89(2)	2.07(2)	2.914(3)	160(2)
N4-H4B...O3W	0.88(3)	2.17(3)	3.047(4)	173(3)
O1W-H1WA...O5	0.8500	2.2100	3.005(5)	157.00
N5-H5A...O3W	0.89(3)	2.40(3)	3.265(4)	166(3)
N5-H5B...O1	0.89(3)	2.16(3)	3.000(4)	159(2)
O1W-H1WB...O2W	0.8500	2.1400	2.963(5)	163.00
N6-H6A...O3	0.89(3)	2.14(3)	2.940(4)	150(2)
N6-H6B...O2W	0.89(3)	2.04(3)	2.913(4)	165(3)
C20-H20...O1	0.9300	2.4100	2.835(4)	108.00
C22-H22...O3	0.9300	2.4600	3.081(4)	124.00

Symmetry codes: 1-x,1-y,-z; 2-x,1-y,1-z; x,1+y,z; 1-x,1/2+y,1/2-z; 1-x,-1/2+y,1/2-z; x,-1+y,z; x,5/2-y,-1/2+z; 2-x,1/2+y,1/2-z; x,5/2-y,1/2+z; x,3/2-y,1/2+z; 1-x,2-y,-z; 2-x,1/2+y,1/2-z; x,3/2-y,-1/2+z; 1-x,2-y,-z; 2-x,-1/2+y,1/2-z; 2-x,-1/2+y,1/2-z; x,1/2-y,1/2+z; x,1/2-y,-1/2+z.

Table 2 — Crystal data of the complex $[\text{Cd}(\text{OPD})_3]1,5\text{-NDS}\cdot 3\text{H}_2\text{O}$

Parameters	Values	Parameters	Values
		$[\text{Cd}(\text{OPD})_3]1,5\text{-NDS}\cdot 3\text{H}_2\text{O}$	
Formula	$[\text{Cd}(\text{OPD})_3]1,5\text{-NDS}\cdot 3\text{H}_2\text{O}$	Crystal size, mm	$0.20 \times 0.28 \times 0.30$
Molecular mass	777.15	Temperature T, ok	566
Syngonia	monoclinic	Scan interval θ , o	$\theta = 2.6\text{--}71.4^\circ$
Spatial group	P21/c	Interval h,k,l	-21: 21; -8: 10; -26: 27
a, Å	17.6185(2)	Total reflexes	33725
b, Å	8.3960(1)	The number of independent reflexes	6140
c, Å	22.0916(2)	R_{int}	0.059
α°	90	$F^2 \geq 2\sigma(F^2)$	5302
β°	103.787(1)	No. of reflections	6140
γ°	90	No. of parameters	473
V, Å ³	3173.75(6)	R1, wR2(1>2 σ (I))	0.0333, 0.0886, 1.08
Z	4	$\Delta_{\text{pmin/max}} \text{e}\text{Å}^{-3}$	-0.54, 0.53
$D_x/\text{g}/\text{cm}^3$	1.627	CCDC-number	2268254
$\mu(\text{CuK}\alpha)$, mm ⁻¹	7.279		

TG-DTA studies

Thermal analysis of $[\text{Cd}(\text{OPD})_3]1,5\text{-NDS}\cdot 3\text{H}_2\text{O}$ complex compound was carried out up to 800°C temperature. TGA analysis of this complex was carried out in 4 stages (Table 3).

The first stage continued to a temperature of 151°C. An endothermic effect of 91.96°C was observed mainly due to the separation of the water molecule in the outer sphere of the complex. The second stage continued up to a temperature of 237°C.

At this stage, a mass decrease was observed due to the separation of the 1,5-NDS anion in the outer sphere. The third stage continued up to 435°C, and a second endothermic effect was observed at 308°C due to the decomposition of the organic ligand (OPD) in the inner sphere (Fig. 6). At the end of the process, the decomposition of the complex compound continues up to 800°C, and it can be seen that metal oxides and organic residues remain^{26,27}.

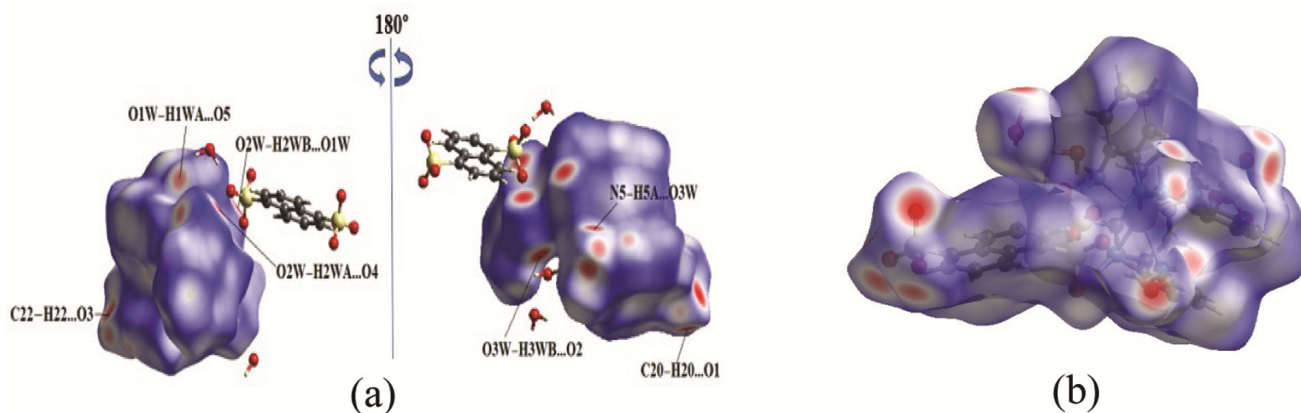


Fig. 4 — (a) Hirschfeld surface of the complex compound $[\text{Cd}(\text{OPD})_3]1,5\text{-NDS}\cdot 3\text{H}_2\text{O}$ by cation. (b) Total Hirschfeld surface area of the complex compound $[\text{Cd}(\text{OPD})_3]1,5\text{-NDS}\cdot 3\text{H}_2\text{O}$

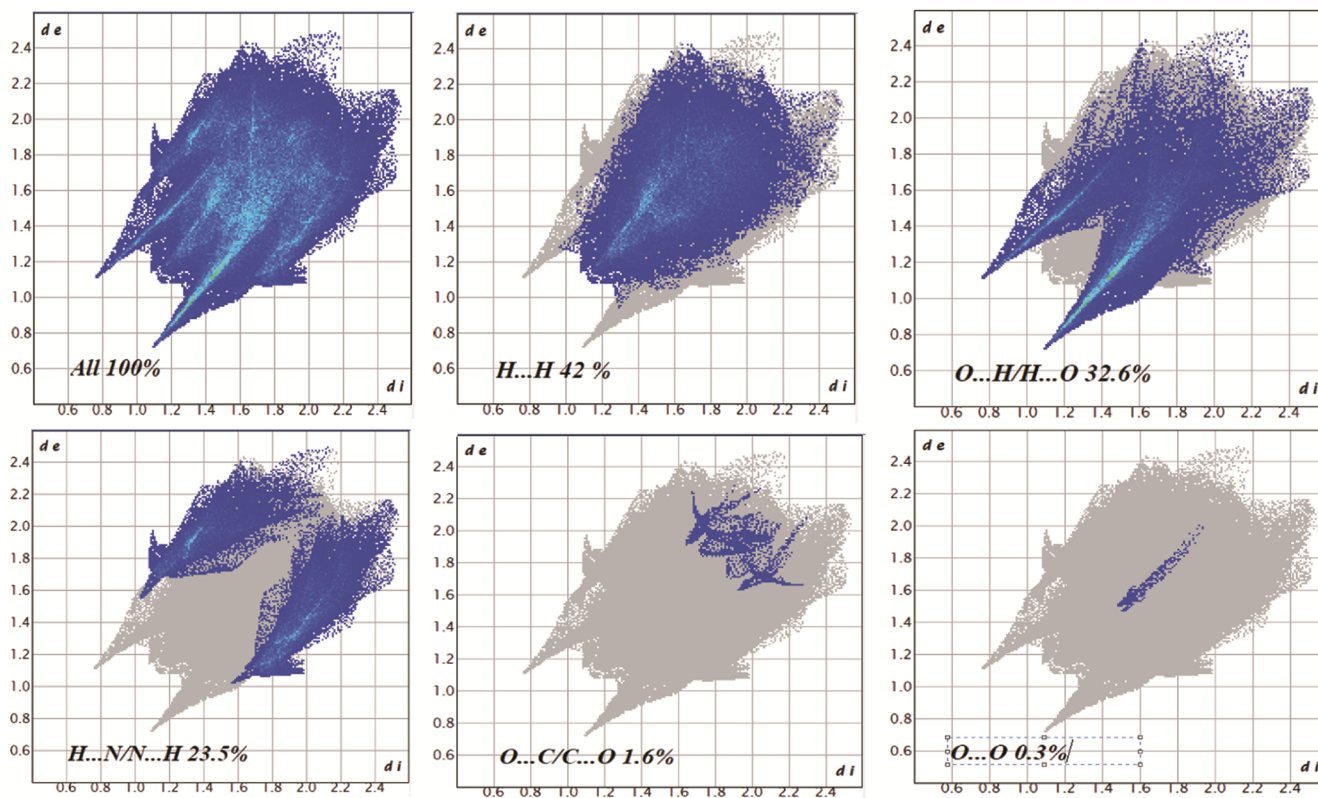


Fig. 5 — 2D fingerprint view of the Hirschfeld surface of the $[\text{Cd}(\text{OPD})_3]1,5\text{-NDS}\cdot 3\text{H}_2\text{O}$ complex.

Table 3 — Thermal analysis of the complex compound $[\text{Cd}(\text{OPD})_3]1,5\text{-NDS}\cdot 3\text{H}_2\text{O}$

No	Temperature, °C	Lost mass, mg.	Mass loss, %	The amount of energy consumed ($\mu\text{V}\cdot\text{s}/\text{mg}$)	Time spent (min)
1	32-151°C	0.235	7.947	16.84	12.78
2	151-237°C	0.341	11.532	14.41	8.76
3	237-435°C	1.37	46.331	2.915	20.16
4	435-800°C	1.025	34.664	8.58	37.65

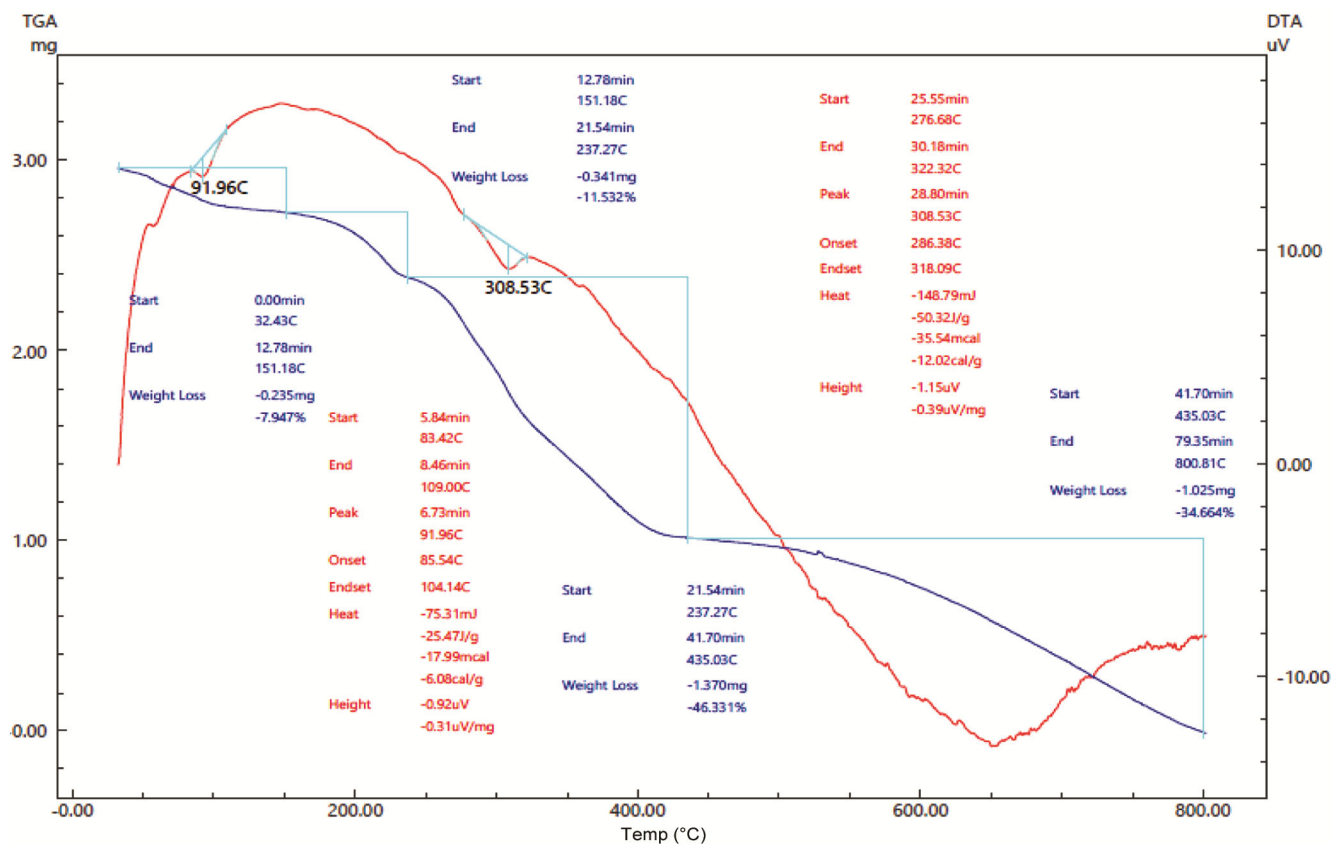


Fig. 6 — Derivatogram of the complex compound $[\text{Cd}(\text{OPD})_3]1,5\text{-NDS}\cdot 3\text{H}_2\text{O}$

Conclusion

In summary, a new mixed-ligand complex compound $[\text{Cd}(\text{OPD})_3]1,5\text{-NDS}\cdot 3\text{H}_2\text{O}$ was synthesized based on OPD with Cd(II) metal. The molecular and crystal structures of the synthesized complex compound were determined using the RTT method. Syngonia of the obtained complex is monoclinic, $b=103.787(1)^\circ$, $a=17.6185(2)\text{\AA}$, $b=8.3960(1)\text{\AA}$ and $c=22.0916(2)\text{\AA}$. Also, the crystal data of this complex was entered into the Cambridge Crystallographic Database (CCDC) and deposit reference number 2268254 was obtained. Thermal analysis of the complex compound showed mass reduction in 4 stages up to 800°C temperature and endothermic effect was observed at 91.96°C and 308.53°C temperatures. Also, in the IR-spectrum analysis of the obtained complex compound, the deformation vibration of the NH_2 -group was observed in 1236 cm^{-1} and 1217 cm^{-1} regions, and the asymmetric vibration of the $\text{S}=\text{O}$ group in 1,5-NDS was observed in the 1371 cm^{-1} region.

Conflict of Interest

The authors declare no conflict of interest.

Authors' Contribution Statement

Ahatov A. A.: data curation and formal analysis. Turaev Kh. Kh.: investigation, methodology, and original draft. Toshkulov A. Kh.: review and editing. Ashurov J. M.: review and editing. Tillaev Kh.R.: review and editing. Ashurov J. M. and Nomozov A. K.: conceptualization, writing (original draft), and supervision. All authors have read and agreed to the published version of the manuscript.

Supplementary Information

Supplementary information is available in the website <http://nopr.niscpr.res.in/handle/123456789/58776>.

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