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SYNTHESIS, STRUCTURE AND HIRSHFELD SURFACE ANALYSIS OF THE COMPLEX COMPOUND BASED ON Cd(II) SALT AND O-PHENYLENEDIAMINE

In this paper studying the synthesis, crystal structure and Hirshfeld surface analysis of a new complex compound containing $[Cd(OPD)_3SO_4] \cdot H_2O$ obtained based on o-phenylenediamine (OPD) are given. The molecular and crystal structure of this complex compound was determined using the RTT method. In the inner sphere of the complex, two molecules of OPD in the chelate state and one molecule of OPD in the monodentate state are attached to the central atom. The complex compound forms a distorted octahedral shape in space. $[Cd(OPD)_3SO_4] \cdot H_2O$ complex compound syngonia is monoclinic, space group P21/c and crystal parameters are $a = 22.5631(5) \text{ \AA}$, $b = 8.1292(2) \text{ \AA}$, $c = 11.6094(4) \text{ \AA}$. In the crystal structure of the complex, 5 Cd–N (2.350(5) Å – 2.448(5) Å) and 1 Cd–O (2.355(5) Å) bonds form an inner sphere. The crystal structure of the molecule is formed by intra- and intermolecular hydrogen bonds of the type N---H...O and O---C...O. In the Hirshfeld surface analysis of $[Cd(OPD)_3SO_4] \cdot H_2O$ complex compound, the main interactions are H...H (54.4%), O...H/H...O (26.7%) and H...C/C...H (16.7%) organizes.

Keywords: o-phenylenediamine, complex compound, hydrogen bond, Hirshfeld surface analysis, crystal parameter.

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Cd(II) тұзы мен о-фенилендиамин негізіндегі күрделі қосылыстардың бетін Гиршфельд бойынша синтезі, құрылымы және талдауы

Бұл ұсынылған жұмыста о-фенилендиаминнен (OPD) алынған $[Cd(OPD)_3SO_4] \cdot H_2O$ бар жаңа күрделі қосылыс синтезі, кристалдық құрылымы және Хиршфельд беттік талдауының нәтижелері берілген. Бұл күрделі қосылыстың молекулалық және кристалдық құрылымы RTT әдісі арқылы анықталды. Кешеннің ішкі сферасында орталық атомға хелат күйіндегі екі OPD молекуласы және моноденттік күйдегі бір OPD молекуласы бекітілген. Кешенді қосылыс кеңістікте бұрмаланған октаэдрлік пішінді құрайды. $[Cd(OPD)_3SO_4] \cdot H_2O$ комплексті қосылыс жүйесі моноклиникалық, кеңістіктік тобы P21/c, ал кристалдық параметрлері $a = 22,5631(5) \text{ \AA}$, $b = 8,1292(2) \text{ \AA}$ және $c = 11,6094(4) \text{ \AA}$ болып табылады. Кешеннің кристалдық құрылымында 5 Cd–N байланысы 5 Cd – N (2,350(5) Å – 2,448(5) Å) және 1 Cd – O (2,355(5) Å) байланысы ішкі сфераны құрайды. Молекуланың кристалдық құрылымы N---H...O және O---C...O типті ішкі және молекулааралық сутектік байланыстар арқылы түзіледі. $[Cd(OPD)_3SO_4] \cdot H_2O$ күрделі қосылыстың беттік Хиршфельд талдауында негізгі өзара әрекеттесулер H...H (54,4%), O...H/H...O (26,7%) және H...C/C...H (16,7%) болып табылады.

Түйін сөздер: о-фенилендиамин, комплексті қосылыс, сутектік байланыс, Хиршфельд бетінің анализі, кристалдық параметр.

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Синтез, структура и анализ поверхности по Хиршфельду комплексного соединения на основе соли Cd(II) и о-фенилендиамина

В данной работе приведены результаты синтеза, кристаллической структуры и анализа поверхности по Хиршфельду нового комплексного соединения, содержащего $[\text{Cd}(\text{OPD})_3\text{SO}_4] \cdot \text{H}_2\text{O}$, полученного на основе о-фенилендиамина (OPD). Молекулярная и кристаллическая структура этого комплексного соединения была определена с использованием метода РТТ. Во внутренней сфере комплекса к центральному атому присоединены две молекулы OPD в хелатном состоянии и одна молекула OPD в монодентатном состоянии. Комплексное соединение образует в пространстве искаженную октаэдрическую форму. Сингония комплексного соединения $[\text{Cd}(\text{OPD})_3\text{SO}_4] \cdot \text{H}_2\text{O}$ является моноклинной, пространственная группа P21/c и параметры кристалла составляют $a = 22,5631(5) \text{ \AA}$, $b = 8,1292(2) \text{ \AA}$ и $c = 11,6094(4) \text{ \AA}$. В кристаллической структуре связей 5 Cd–N (2,350(5) \AA – 2,448(5) \AA) и связь 1 Cd–O (2,355(5) \AA) образуют внутреннюю сферу. Кристаллическая структура молекулы образована внутри- и межмолекулярными водородными связями типа N---H...O и O---C...O. При поверхностном Хиршфельд анализе комплексного соединения $[\text{Cd}(\text{OPD})_3\text{SO}_4] \cdot \text{H}_2\text{O}$ основными взаимодействиями являются H...H (54,4%), O...H/N...O (26,7%) и H...C/C...H (16,7%).

Ключевые слова: о-фенилендиамин, комплексное соединение, водородная связь, поверхностный анализ Хиршфельда, параметр кристалла.

Introduction

The OPD molecule is important in the chemistry of organic and complex compounds due to its easy oxidation and reduction properties [1]. Complex compounds obtained on the basis of O-phenylenediamine are used in medicine to obtain drugs against cancer cells and against malaria [2,3]. In addition, OPD is used as sorbents in industrial wastewater treatment [4]. It is known that OPD and its complex compounds have been used as fungicides and herbicides in agriculture for many years [5,6]. It has been proven that the amino group in OPD participates in an active oxidation-reduction reaction due to easy protonation or deprotonation [7]. Also, due to the adjacent arrangement of amino groups in OPD, it increases the possibility of forming chelate complexes with its metal atoms [8]. Jan-Teller effect is observed in the $[\text{Cu}(\text{SO}_4)(\text{C}_6\text{H}_8\text{N}_2)_2] \cdot \text{H}_2\text{O}$ complex formed by OPD with Cu(II) ion. When studying single crystals of this complex compound by RTT method, it was found that the length of N—H bonds is 0.92 \AA , Cu1—N2 2.014 \AA , Cu1—N4 2.020 \AA , Cu1—N1 2.022 \AA , Cu1—N3 2.023 \AA , and Cu1—O1 2.2433 \AA [9]. A conformation phenomenon is observed in some complex compounds of OPD. For example, the complex compound $[\text{Ni}(\text{opda})_2(\text{NCS})_2]$ forms yellow and purple complexes. Both monocrystals have spatial group P21/c and the

presence of N—H...S hydrogen bonds in the crystal structure. Yellow single crystals are heated in the temperature range of 110–120°C and quickly turn into purple crystals [10]. Thermal and solution stability of $[\text{Ni}(\text{C}_6\text{H}_8\text{N}_2)_2(\text{H}_2\text{O})_2](\text{C}_{10}\text{H}_6\text{O}_6\text{S}_2)$ complex formed by OPD of Ni(II) ion was proved. In the structure of this complex, 2 OPD and 2 water molecules are located in the inner sphere. The disodium salt of naphthalene 1,5-disulfoacid is located in the outer sphere [11]. Chelate complexes are more stable than monodentate complexes [12].

$[\text{Cd}(\text{C}_6\text{H}_8\text{N}_2)_4](\text{C}_{10}\text{H}_6\text{O}_6\text{S}_2)$ complex obtained on the basis of Cd(II) ion and OPD also forms a chelate complex. The central atom is surrounded by 4 OPD molecules. 2 OPD molecules are connected to the central atom in bidentate and 2 OPD molecules in monodentate state [13].

The aim of study the crystal structure of substances by X-ray diffraction is to determine the position of its atoms in space [14,15]. If the exact position of the atoms in the substance is determined, it will be possible to determine the distance between atoms, bond angles, bond energies and other parameters. X-rays are generated through an X-ray tube [16]. The free electrons coming from the heated cathode in the X-ray tube have a high potential between the cathode and the anode and fall to the Cu or Mo electrode anode. In this process, X-rays are generated with high voltage. The anode is always

cooled by a cooling device. Generated X-rays are processed automatically [17,18].

We used an automatic Xcalibur® Oxford Diffraction diffractometer (graphite monochromator, Cu-K α -radiation $\lambda = 1.54184 \text{ \AA}$) to determine the structure and composition of the single crystal obtained in this research work.

Experimental part

This complex compound, $[\text{Cd}(\text{OPD})_3\text{SO}_4] \cdot \text{H}_2\text{O}$, was synthesized by reacting o-phenylenediamine with cadmium sulfate crystalline hydrate in a 1:3 molar ratio (M:L) (Fig.1).

First, 0.176 g (0.5 mmol) of cadmium sulfate crystalline hydrate salt ($\text{CdSO}_4 \cdot 8\text{H}_2\text{O}$) was taken and a 0.1 M aqueous solution was prepared. 0.054 g (0.5 mmol) of OPD ligand was taken and its solution in 0.1 M ethanol (96%) was prepared. The reaction mixture was first stirred at room temperature and then at 60°C on a magnetic stirrer for 80 minutes. At the end of the process, the reaction mixture was brought

to room temperature and left for 10 days in a thermostat with constant temperature ($28 \pm 1^\circ\text{C}$). Dark-yellow single crystals obtained by slow evaporation method were obtained with 80% yield and the crystal structure was studied using the RTT method (Fig.2). The elemental analysis of the obtained $[\text{Cd}(\text{OPD})_3\text{SO}_4] \cdot \text{H}_2\text{O}$ complex is as follows: determined (%) Cd 20.36, C 39.27, H 4.72, N 15.27, O 14.56, S 5.82; found (%) Cd 20.34, C 39.19, H 4.71, N 15.21, O 14.45, S 5.79.

Asymmetric and symmetric valence vibrations of NH_2 groups in o-phenylenediamine are in the doublet state at 3385 cm^{-1} , 3361 cm^{-1} , 3282 cm^{-1} and 3180 cm^{-1} , and the deformation vibrations of this group are at 1631 cm^{-1} and 1591 cm^{-1} observed. The frequencies of valence vibration of the C-N bond are observed in the region of 1273 cm^{-1} . In the $3063\text{-}3057 \text{ cm}^{-1}$ region of the spectrum, there are bands caused by the valence vibrations of the C-H bonds of the aromatic ring. We can observe the valence vibration of the aromatic ring along the plane of the ring in the range of $1776\text{-}1919 \text{ cm}^{-1}$ (Fig.3).

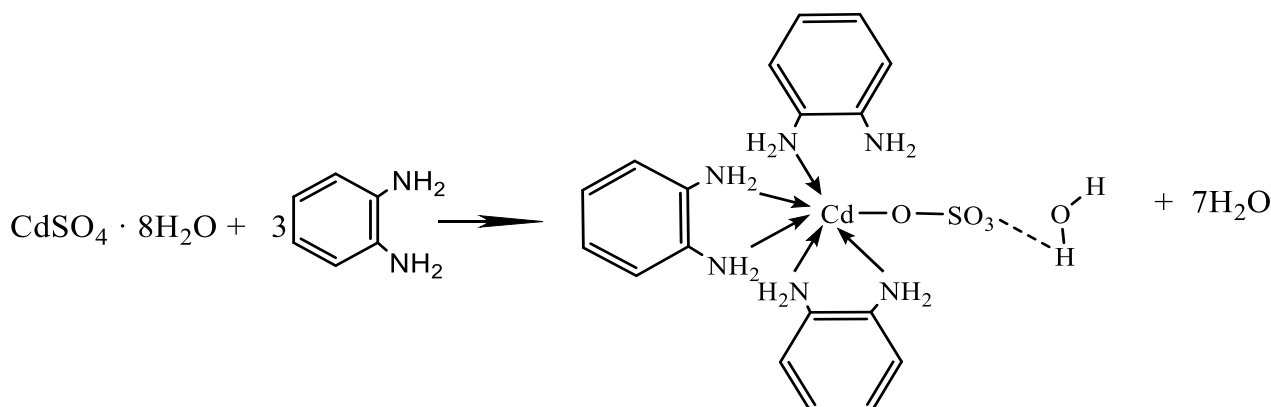


Figure 1 – Schem.1. Synthesis reaction of complex compound $[\text{Cd}(\text{OPD})_3\text{SO}_4] \cdot \text{H}_2\text{O}$

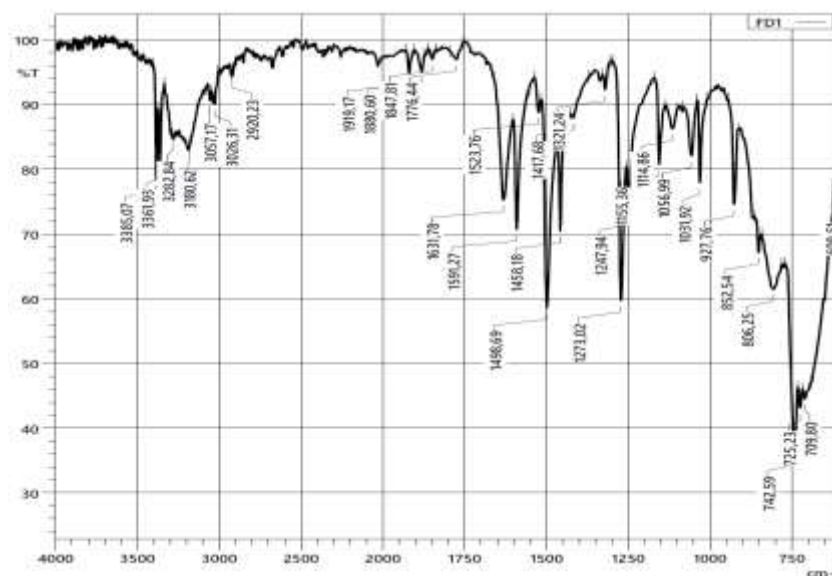


Figure 2 – IR spectrum of OPD

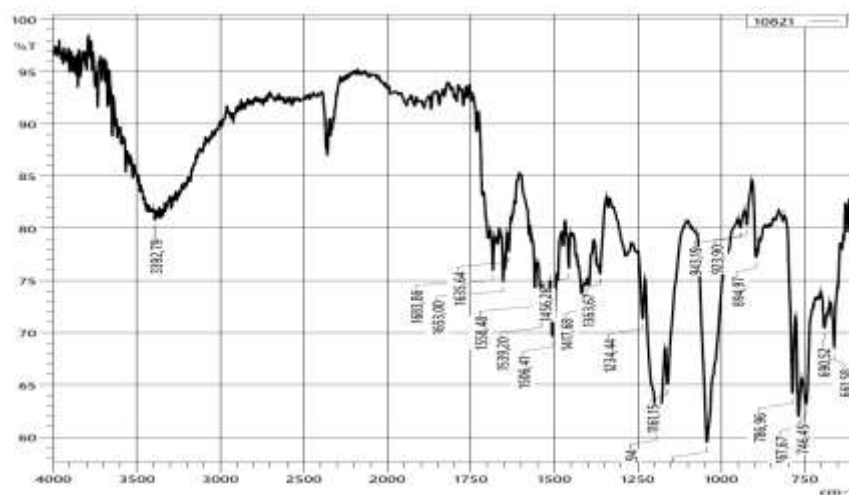


Figure 3 – IR spectrum of $[\text{Cd}(\text{OPD})_3\text{SO}_4]\cdot\text{H}_2\text{O}$ complex compound

In the analysis of the IR spectrum depicted in the picture above, we can conclude that there are OH groups located in the outer sphere of the molecule in the high-frequency 3392 cm^{-1} short region of the spectrum. In the part of the spectrum in the doublet area of 1456 cm^{-1} and 1506 cm^{-1} , we see the deformation vibration of the NH_2 group. Aromatic rings vibrate due to overtone frequencies in the absorption regions of 1653 cm^{-1} and 1683 cm^{-1} along the ring plane. We can see the valence vibration of S-O groups between the absorption areas of 1363 cm^{-1} and 1417 cm^{-1} .

Crystal structure

In the crystal structure of the $[\text{Cd}(\text{OPD})_3\text{SO}_4]\cdot\text{H}_2\text{O}$ complex compound (Fig.4), there are three OPD molecules a sulfate anion in the

inner sphere, and one water molecule in the outer sphere. Two of the OPD molecules are in the chelate state and one is in the monodentate state attached to the central atom. The coordination number of the central atom is 6. Nitrogen atoms are connected to the central atom with the help of donor-acceptor bonds, and the oxygen atom forms a covalent bond. In the supramolecular structure of the molecule, the distance between atoms attached to the central atom is Cd–O1 $2.355(5)\text{ \AA}$, Cd1–N1 $2.373(5)\text{ \AA}$, Cd1–N2 $2.403(6)\text{ \AA}$, Cd1–N3 $2.448(5)\text{ \AA}$, Cd1–N4 is $2.375(5)\text{ \AA}$, Cd1–N5 is $2.350(5)\text{ \AA}$. Also, O1–Cd1–N1 $98.96(15)^\circ$, O1–Cd1–N2 $166.88(16)^\circ$, O1–Cd1–N3 $103.79(17)^\circ$, O1–Cd1–N4 $77.97(17)^\circ$ form in the molecule. In the crystal structure of the molecule, there are internal and intermolecular hydrogen bonds of the type N---H...O and O---C...O (Tab. 2).

Table 1 – Crystal parameters of the $[\text{Cd}(\text{OPD})_3\text{SO}_4]\cdot\text{H}_2\text{O}$ complex

Parameters	Values	Parameters	Values
$[\text{Cd}(\text{OPD})_3\text{SO}_4]\cdot\text{H}_2\text{O}$			
Formula	$[\text{Cd}(\text{OPD})_3\text{SO}_4]\cdot\text{H}_2\text{O}$	Crystal Size, mm	0.00 x 0.00 x 0.00
Molecular Weight	551.46	Temperature T, °K	293
Syngonia	monoclinic	Theta Min-Max [Deg] 0, °	4.0, 76.1
Space group	P21/c	Dataset h,k,l	-27: 24 ; -10: 8 ; -13: 14
a, Å	22.5631(5)	Total reflexes	8493
b, Å	8.1292(2)	The number of independent	4265
c, Å	11.6094(4)	R_{int}	0.063
α°	90	$F^2 \geq 2\sigma(F^2)$	3349
β°	99.340(3)	No. of reflections	4265
γ°	90	No. of parameters	284
V, Å ³	2101.17(10)	R1, wR2(1>2σ(I))	0.0538, 0.1395, 1.03
Z	9	$\Delta\rho_{\text{min/max}}\text{ e}\text{\AA}^{-3}$	-1.67, 1.84
$D_x/\text{g}/\text{cm}^3$	1.742		
$\mu(\text{CuK}\alpha)$, mm ⁻¹	9.645		

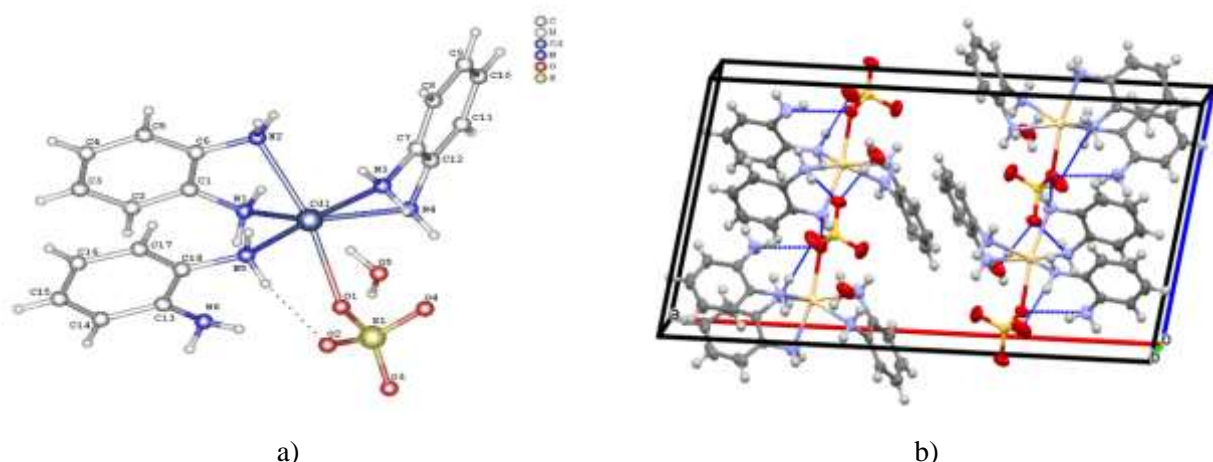


Figure 4 – (a) Molecular structure of the complex $[\text{Cd}(\text{OPD})_3\text{SO}_4]\cdot\text{H}_2\text{O}$ and (b) Plotting of the complex compound $[\text{Cd}(\text{OPD})_3\text{SO}_4]\cdot\text{H}_2\text{O}$ along the c axis (blue lines depict hydrogen bonds)

Table 2 – Geometry of hydrogen bonds of complex compound $[\text{Cd}(\text{OPD})_3\text{SO}_4]\cdot\text{H}_2\text{O}$

$D-H\cdots A$	$D-H, \text{Å}$	$H\cdots A, \text{Å}$	$D\cdots A, \text{Å}$	$DHA, \text{burchak}$
N1---H1A...O3	0.8900	2.1200	2.986(8)	164.00
N2---H2A...O2	0.8900	2.3100	3.085(8)	145.00
N2---H2B...O1	0.8900	2.2200	3.024(6)	150.00
N4---H4B...O5	0.8900	2.0600	2.947(9)	174.00
N5---H5A...O2	0.8900	2.0800	2.915(8)	156.00
N5---H5B...O2	0.8900	2.5700	3.345(8)	146.00
N5---H5B...O3	0.8900	2.3100	3.137(7)	154.00
O5---H5C...O3	0.8500	1.8800	2.713(9)	167.00
O5---H5D...O2	0.8500	2.2900	3.118(9)	165.00
O5---H5D...O4	0.8500	2.4900	3.139(10)	134.00
N6---H6A...O1	0.8700	2.3200	3.019(6)	137.00

Symmetry codes: $x, 3/2-y, -1/2+z \# x, 1/2-y, -1/2+z \# 1-x, 1-y, 2-z \# x, 3/2-y, 1/2+z \# x, 1/2-y, 1/2+z \# x, 1+y, z \# 1-x, -1/2+y, 5/2-z \# -x, 1/2+y, 3/2-z \# 1-x, 1/2+y, 5/2-z \# -x, -1/2+y, 3/2-z \# x, -1+y, z$

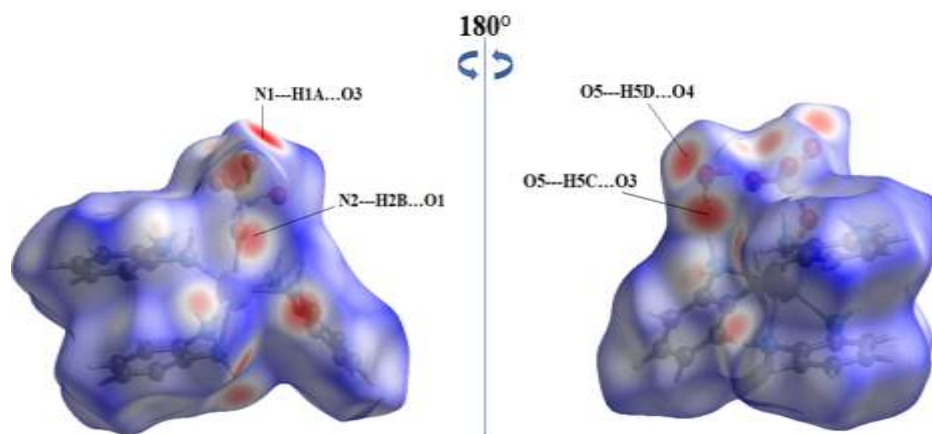


Figure 5 – 3D Hirshfeld surface of the complex compound $[\text{Cd}(\text{OPD})_3\text{SO}_4]\cdot\text{H}_2\text{O}$ by dnorm

Hirschfeld surface analysis allows visualization of weak interactions within a molecule. It also represents the share of the particles in the molecule in the total structure of the molecule through electron densities [19,20]. Hirschfeld surface analysis of this complex compound was performed using Crystal Explorer 21.5 software. The red spots on the

Hirschfeld surface represent interactions close to the surface of the molecule. Also, these spots represent hydrogen bonds in the crystal structure of the molecule. In the complex compound $[\text{Cd}(\text{OPD})_3\text{SO}_4]\cdot\text{H}_2\text{O}$, there are N1---H1A...O3 hydrogen bonds between the third oxygen atom located in the inner sphere and the NH2- group of the

molecule. Also, between the water molecule located in the outer sphere of the complex and the inner sphere of the molecule, there are intermolecular hydrogen bonds of the type O5---H5D...O4. In the inner sphere of the complex compound, there are internal hydrogen bonds of the N2---H2B...O1 type between the sulfate anion and the NH₂ group. On the Hirshfeld surface of the complex compound, the standard sizes of red and blue spots according to d_{norm} are -0.6676 and 1.2204, respectively. On the Hirshfeld surface, the van der Waals radii of the red colour molecule indicate close interactions. The blue spots show interactions far from van der Waals radii [16]. In this complex compound, hydrogen bonds

form the main part of the red spots located on the surface of Hirshfeld. The Hirshfeld surface area of the complex is $S = 414.80 \text{ \AA}^2$ and the volume $V = 517.63 \text{ \AA}^3$ (Fig.5). Also, the interaction of internal particles in the structure of the molecule was determined using the two-dimensional functions d_i and the interactions on the crystal surface. The main interactions in the molecule are H...H (54.4%), O...H/H...O (26.7%) and H...C/C...H (16.7%). These interactions create spikes with tip and side growth in the two-dimensional fingerprint image (Fig. 6). Usually, the percentage of hydrogen bonds is higher on the sides of the fingerprint image [21, 22].

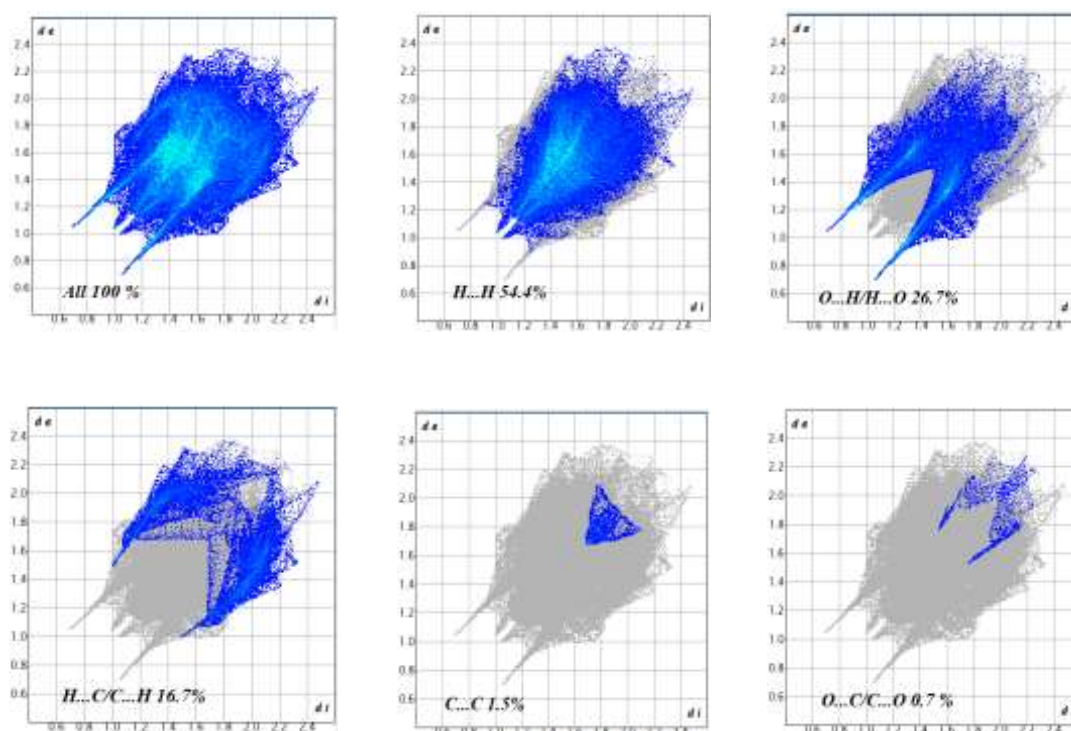


Figure 6 – 2D fingerprint diagram of the $[\text{Cd}(\text{OPD})_3\text{SO}_4] \cdot \text{H}_2\text{O}$ complex

Conclusion

In short, for the first time, a complex compound with the composition $[\text{Cd}(\text{OPD})_3\text{SO}_4] \cdot \text{H}_2\text{O}$ was synthesized based on OPD and its single crystals were obtained. The molecular and crystal structure of the obtained single crystals was studied using the RTT method. The bond angles, the distance between bonds, spatial shape and other crystal parameters of this complex compound were determined. Hirshfeld surface analysis of the obtained complex compound was performed in the Crystal Explorer 21.5 program. In the crystal structure of the complex, hydrogen bonds are represented by red

spots in the Hirshfeld surface analysis. The interaction of the particles in the molecule and the van der Waals radii were determined. Also, in the IR-spectrum analysis of the formed complex compound, the deformation vibration of the OH group in the 3392 cm^{-1} region and the doublet state deformation vibration of the NH₂ group in the lower frequency 1456 cm^{-1} and 1506 cm^{-1} regions of the spectrum is observed.

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