

L-alanine derived Schiff base Ligated Vanadium(IV) Complex with Phenanthroline as Co-ligand: Synthesis, Crystal Structure and Hirshfeld Surface Analysis

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Abstract

A vanadium(IV) Schiff base complex derived from salicylaldehyde and L-alanine with phenanthroline as co-ligand *viz.* $[V^{IV}O(\text{salala})(\text{phen})].0.5H_2O.CH_3OH$ (where salala = Schiff base derived from salicylaldehyde and L-alanine, phen = 1,10-phenanthroline) was synthesized and its structure determined by single crystal X-ray diffraction. The crystal lattice parameters of the complex was determined by single crystal X-ray diffraction with lattice parameters, $a = 18.4361(5) \text{ \AA}$, $b = 22.4926(6) \text{ \AA}$, $c = 12.4035(6) \text{ \AA}$, $\beta = 126.904(1)^\circ$, $C2$ space group, $Z = 2$. In the crystal, the V(IV) ions are in distorted octahedral geometry by coordinating to two oxygen atoms and one nitrogen atom of Schiff base ligand and two phenanthroline nitrogen atoms. The $\pi \cdots \pi$ stacking interactions as well as C–H \cdots O hydrogen bonds were found to play an important role in the self-assembly of the complex molecules. The non-covalent interactions of the complex were further evaluated by Hirshfield Surface Analysis. Spectroscopic characterization of the complex by Infrared and UV-visible spectroscopic techniques is also reported.

Introduction

The metal complexes involving Schiff base ligands are the most fundamental chelating systems in coordination chemistry.¹ In the past few decades, significant progress has been made in the chemistry of transition metal complexes with Schiff base ligands formed from salicylaldehyde or its analogues and amino acids.² The crystal structures of complexes involving the Schiff base derived from salicylaldehyde and amino acids revealed that the Schiff base ligands mainly act as tridentate ligands, coordinating through the phenolato oxygen, imine nitrogen and carboxylate oxygen.^{3–5}

Supramolecular architectures with coordination bonds, hydrogen bonds and π – π stacking interactions have received much attention for its prospective applications in the fields of host–guest chemistry, biochemistry, electronics, magnetism, optics, gas absorbent, catalysis and molecular recognition.^{6–9} These interactions are becoming increasingly important in supramolecular chemistry and crystal engineering.¹⁰ A large numbers of one-, two- and three-dimensional metal–organic supramolecules formed via self-assembly were prepared by the careful selection of the building blocks and organic ligands with appropriate functional groups for supramolecular interactions.¹¹

In this work we report the crystal structure of an amino acid derived oxovanadium(IV) Schiff base complex with phenanthroline as co-ligand *viz.* $[V^{IV}O(\text{salala})(\text{phen})].0.5H_2O.CH_3OH$ (**1**). The synthesis and crystal structure of $[V^{IV}O(\text{salala})(\text{phen})].0.5H_2O$ was previously reported by Cao *et al* where the authors used DL-alanine to prepare the Schiff base complex.¹² When L-alanine was used instead of DL-alanine in the synthesis described by Cao *et al* the title complex was obtained. The complex crystallizes in $C2$ space group with the asymmetric unit containing two crystallographically unique vanadium(IV) centers along with half water molecule of crystallization and methanol. Complex **1** was further characterized by FT-IR and UV-vis spectroscopic techniques. The complex molecules self-assemble into 2D supramolecular

architecture via $\pi\cdots\pi$ stacking and hydrogen bonding interactions. The supramolecular interactions of the complex were evaluated by Hirshfeld surface analysis.

Synthesis and Characterization

Materials and Methods

All reagents, *viz.* vanadyl sulphate hydrate, salicylaldehyde, L-alanine, 1,10-phenanthroline and methanol used in this work were purchased from Sigma Aldrich and Merk (India) Ltd. and used as received. Deionized water and methanol were used as the reaction medium. Elemental analyses (C, H, and N) were carried out on a Perkin Elmer 2400 Series II CHNS/O Analyzer. A FT-IR spectrum was recorded in a Shimadzu FTIR-8400S spectrophotometer in KBr (4000 – 400 cm^{-1}). A diffuse reflectance UV-vis-NIR spectrum was obtained using a Shimadzu UV-2600 spectrophotometer with BaSO_4 powder as the reference (100% reflectance).

X-ray Structure Determination Procedures

X-ray diffraction data were collected on Bruker SMART APEX II CCD equipped with a graphite monochromator and a MoK α fine-focus sealed tube ($\lambda = 0.71073 \text{ \AA}$) at 296 K. Data integration was made using SAINT.¹³ The structure was solved by a direct method in the monoclinic space group *C*2 with SHELXS and refined by full-matrix least squares techniques with SHELXL-2018/3¹⁴ via WinGX.¹⁵ All the non-hydrogen atoms were refined in the anisotropic approximation against F^2 of all reflections. The hydrogen atoms were refined isotropically, except those attached to oxygen atoms of water molecule and the methyl group of methanol that were located in the difference Fourier maps and refined with isotropic displacement coefficients. The final structure has been deposited with the Cambridge Crystallographic Data Centre (CCDC) under deposition number 2217555. These data can be obtained free of charge under <https://www.ccdc.cam.ac.uk/structures/>.

Results and Discussion

Synthesis

The title complex was obtained by a procedure similar to that reported by Cao *et al* with a slight modification where L-alanine was used instead of DL-alanine in the synthesis.¹² Salicylaldehyde (2 mmol, 0.24 ml) was dissolved in methanol (10 ml) and added slowly to an aqueous solution of L-alanine (2 mmol, 0.179 g). The resulting mixture was stirred for 1 h to obtain a yellow color solution. To this mixture was added vanadyl sulfate hydrate (2 mmol, 0.326 g) followed by methanol solution of 1,10-phenanthroline monohydrate (2 mmol, 0.366 g) and stirred for 4 h to obtain a reddish brown solution. This solution was kept at room temperature for one day, whereupon reddish block shaped crystals suitable for single crystal X-ray diffraction were obtained.

Yield, 81.3% (2.96 g). Anal. calc. for $C_{90}H_{72}N_{12}O_{19}V_4$ (M_r : 1829.36): C, 59.03; H, 3.93; N, 9.18. Found: C, 59.56; H, 4.19; N, 8.72%. FT-IR spectral data (KBr, cm^{-1}): 3398(br), 1612(s), 1529(w), 1438(w sh), 1341(m), 1134(w), 1139(w, sh), 951(s), 846 (s), 804 (m), 727 (s), 608 (w), 538 (w).

Crystal Structure

Table 1
Crystallographic data and structure refinement for 1

Parameters	VO(salala)(phen)].0.5H ₂ O.CH ₃ OH (1)
Formula	C ₈₈ H ₆₆ N ₁₂ O ₁₇ V ₄
Formula weight	1767.28
Temperature (K)	296(2)
Wavelength (Å)	0.71073
Crystal system	Monoclinic
Space group	<i>C</i> 2
<i>a</i> /Å	18.4361(5)
<i>b</i> /Å	22.4926(6)
<i>c</i> /Å	12.4035(6)
α°	90
β°	126.9040(10)
γ°	90
Volume (Å ³)	4112.9(3)
Z	2
Calculated density (Mgm ⁻³)	1.427
F (000)	1812
Crystal size (mm)	0.15 x 0.14 x 0.12
Theta range for data collection (°)	2.053 to 29.613
Limiting indices, <i>h,k,l</i>	-25 ≤ <i>h</i> ≤ 25, -31 ≤ <i>k</i> ≤ 30, -17 ≤ <i>l</i> ≤ 16
Reflections collected	60755
Unique data (<i>R_{int}</i>)	11370 [<i>R</i> (int) = 0.1542]
Refinement method	Full-matrix least-squares on <i>F</i> ²
Data/restraints/ parameters	11370 / 1 / 546

R and *R_w* values are for all unique data; $R = \Sigma |F_o| - |F_c| / \Sigma |F_o|$; $R_w = \{\Sigma [w(F_o^2 - F_c^2)^2] / \Sigma [w(F_o^2)^2]\}^{1/2}$

where $w = 1 / [\sigma^2(F_o^2) + (aP)^2 + bP]$ with $P = [2F_c^2 + \text{Max}(F_o^2, 0)] / 3$.

Parameters	VO(salala)(phen)].0.5H ₂ O.CH ₃ OH (1)
Goodness-of-fit on F^2	0.952
Final R indices [$I > 2\sigma(I)$]	R1 = 0.0524, wR2 = 0.1448
R indices (all data)	R1 = 0.1187, wR2 = 0.1645
Largest diff. peak and hole (e.Å ⁻³)	1.045 and - 0.315
<i>R and R_w values are for all unique data; $R = \Sigma F_o - F_c / \Sigma F_o$; $R_w = \{\Sigma [w(F_o^2 - F_c^2)^2] / \Sigma [w(F_o^2)^2]\}^{1/2}$</i>	
<i>where $w = 1 / [\sigma^2(F_o^2) + (aP)^2 + bP]$ with $P = [2F_c^2 + \text{Max}(F_o^2, 0)] / 3$.</i>	

Details of single crystal XRD and the structure refinement are given in Table 1. The asymmetric unit of complex **1** consists of two crystallographically independent oxovanadium(IV) complexes with one methanol and a half water molecule of crystallization (Fig. 1). The bond lengths and bond angles for the two VO(II) complex units are listed in Table 2. The comparison of main bond lengths and angles shows a close similarity between the two crystallographically independent V(IV) centers. As can be seen from the bond parameters listed in Table 2, both vanadium centers have a distorted octahedral geometry. The V = O bond distances for V1 and V2 units are in the range from 1.593(8) Å to 1.603(8) Å which is in agreement with those reported in the literature.¹⁶⁻¹⁸ The V–N bond distances range from 2.052(9) Å to 2.343(9) Å for the V1 unit and from 2.038(8) Å to 2.375(9) Å for the V2 unit. It is observed that the V–N_{imine} is shorter than the V–N_{phen} in the V1 and V2 centers which implies that the alanine derived Schiff base has stronger chelating ability than phenanthroline ligand.

Table 2
Selected bond lengths (Å) and angles (deg.) in
coordination metal core in **1**

Bond	d, Å	Angle	Degree(°)
1			
V1–O1	1.603(8)	O2–V1–O3	158.2
V1–O2	1.937(8)	N2–V1–N3	160.9
V1–O3	2.011(9)	O1–V1–N1	168.1
V1–N1	2.343(9)	N1–V1–N3	88.2
V1–N2	2.132(9)	O1–V1–N2	96.2
V1–N3	2.052(9)	O1–V1–N3	102.5
		N1–V1–N2	72.7
		O3–V1–N2	94.7
		O1–V1–O3	98.4
V2–O4	1.593(8)	O5–V2–O7	158.9
V2–O5	1.997(9)	N5–V2–N6	159.4
V2–O7	1.957(9)	O4–V2–N4	166.8
V2–N4	2.375(9)	O5–V2–N6	80.2
V2–N5	2.161(9)	O7–V2–N5	91.5
V2–N6	2.038(8)	N6–V2–O7	88.6
		O5–V2–N5	92.7
		O4–V2–N5	94.5
		O4–V2–O5	99.8

As shown in Fig. 2, crystal packing of **1** reveals the formation of $\pi\cdots\pi$ stacking along with C-H \cdots O hydrogen bonding interactions in the direction of crystallographic *bc* plane leading to the formation of a supramolecular 2D assembly. Two types of $\pi\cdots\pi$ interactions were observed in the crystal structure of **1** involving the crystallographically unique VO²⁺ centers. In one type of $\pi\cdots\pi$ interactions that occurs between aromatic ring of salicylaldehyde of V2 center and phenanthroline ring of V1 center [denoted as ($\pi\cdots\pi$)₁] with centroid-centroid separation of 3.813 Å. The second type of $\pi\cdots\pi$ interactions occurs between the aromatic rings of phenanthroline ligands of V1 and V2 centers [denoted as ($\pi\cdots\pi$)₂] with centroid-centroid distance of 3.615 Å. The uncoordinated oxygen atom (O6) of the carboxylate group of Schiff base ligand participates in intermolecular C-H \cdots O hydrogen bonding interaction with the carbon atom (C7) of the phenanthroline ligand with a C-H \cdots O bond distance of 2.794 Å.

In order to investigate the role of lattice water and methanol molecules in the stabilization of the crystal packing of the title complex, we have observed that they are involved in O-H...O, and C-H...O hydrogen bonding interactions Fig. 3. A detail structural analysis reveals that the oxygen atom of lattice water molecule (O8) is involved in strong O-H...O hydrogen bonding interactions with the oxygen atoms O9 and O10 of the methanol and Schiff base ligand respectively. The methanol oxygen atom (O10) is involved in O-H...O and C-H...O hydrogen bonding interactions.

Table 3
Hydrogen bond distances (Å) and angles (deg.) for **1**

D-H...A	d(D-H)	d(D...A)	d(H...A)	< (DHA)	Symmetry Code
C7-H7...O6	0.930 (12)	3.580 (13)	2.794 (8)	142.9	-x + 1/2,+y-1/2,-z + 1
C43-H43B...O1	0.960 (1)	3.853 (13)	2.945 (7)	158.0	-x + 1,+y,-z + 2
C43-H33...O9	0.930(13)	3.494(16)	2.633(10)	154.1	x-1/2,+y + 1/2,+z
C43-H34...O10	0.930(11)	3.504(4)	2.653(4)	152.4	x-1/2,+y + 1/2,+z + 1
O8-H8A...O9	0.868(5)	2.679(11)	2.430(11)	97.1	-x + 1,+y,-z + 1
O8-H8A...O10	0.868(5)	2.650(3)	2.512(3)	89.47	x,y,z

Hirshfeld Surface Analysis

A Hirshfeld surface (HS) analysis was carried out using Crystal Explorer 21¹⁹ to visualize the intermolecular interactions. In the HS plot the normalized contact (d_{norm}) is measured by using d_e (the distance from the point to the nearest nucleus external to the surface), d_i (the distance to the nearest nucleus internal to the surface) and the van der Waals (vdW) radii of the atoms involved in the intermolecular contacts (Fig. 4). The graphical plots of HS with red and blue colors denote contacts with distances shorter (in close contact) or longer (in distinct contact), respectively, than the van der Waals radii, while the white surfaces denote contacts with distances equal to the sum of van der Waals radii.²⁰ The 2D fingerprints were plotted using the translated 1.0–2.8 Å range, including reciprocal contacts.

As described, the crystal packing of **1** is stabilized by $\pi \cdots \pi$ stacking and hydrogen bonding interactions. In order to identify the $\pi \cdots \pi$ stacking interactions the HS were mapped with shaped index and curvedness properties as shown in [Fig. 5(a) and 5(b)]. The presence of adjacent red and blue triangles allows the HS mapped over the shape-index demonstrate the presence of $\pi \cdots \pi$ stacking interactions [Fig. 5(a)]. Figure 6(a) shows the overall two dimensional fingerprint plot. Figure 6b–i show the relative contributions to the Hirshfeld surface of the contacts described into H...H, C...C, H...C/C...H, H...O/O...H, N...H/H...N, C...O/O...C, C...N/N...C and O...O contacts.²¹ With a contribution of 37.7%, the most significant interaction is H...H, which is depicted in Fig. 6(b) and can be visualized as a pair of spikes with the tips at $d_e + d_i = 2.6$ Å. The H...C/C...H contacts involved in intermolecular C-H...O hydrogen bonds with 18.2% contribution [Fig. 6(d)] have the tips at $d_e + d_i = 2.04$ Å. The H...O/O...H contacts involved in O-H...O hydrogen bonds [Fig. 6(e)], which account for 28.3% of the

total, result from the H•••O/O•••H contacts and are shown as a pair of spikes with the tips located at $d_e + d_i = 2.32 \text{ \AA}$. The C•••C contacts involved in π ••• π stacking interactions²¹ with a contribution of 11.1% HS area [Fig. 6(c)] are emerged with an arrow-shaped distribution of points at $d_e = d_i = 1.60 \text{ \AA}$. The N•••H/H•••N contribution of 2.3% to HSs have scattered points of very low densities with tips located at $d_e + d_i = 2.42 \text{ \AA}$. Finally, the contributions of the C•••O/O•••C (1%, Table 2 and Fig. 6g), N•••C/N•••C (0.6% Fig. 6h) and O•••O (0.2%, Fig. 6i) contacts to the HSs have scattered points of very low densities, less than 1.5%, with the tips located at $d_e + d_i = 2.82 \text{ \AA}$, 3.52 \AA and 2.72 \AA , respectively.

Spectral Studies

Infrared Spectroscopy

The functional groups involved in the title complex were observed from the FT-IR spectrum. The FT-IR spectrum of the complex showed a strong stretching band at 1612 cm^{-1} , which was attributed to the $\nu(\text{C} = \text{N})$ imine group. The characteristic strong stretching frequency at 3398 cm^{-1} is due to $\nu(\text{O} - \text{H})$ vibration of lattice water molecule or methanol. The stretching band at 951 cm^{-1} is characteristic of $\nu(\text{V} = \text{O})$, in agreement with related studies.²²⁻²³ Beside this feature, band at 538 cm^{-1} for the complex is due to the $\text{V}-\text{O}_{(\text{phenoxy})}$ stretch.¹⁶ The $[\nu_{\text{as}}(\text{OCO})]$ stretching vibration of carboxylate group observed at 1612 cm^{-1} is merged with imine vibrations while $[\nu_{\text{s}}(\text{OCO})]$ vibrations appears at 1341 cm^{-1} as a medium intensity peak. The difference in asymmetric and symmetric vibration of carboxylate group are greater than 200 cm^{-1} [$\Delta\nu = \nu_{\text{as}}(\text{OCO}) - \nu_{\text{s}}(\text{OCO}) = 271 \text{ cm}^{-1}$] suggests monodentate coordination of carboxylate group of amino acid derived Schiff base which is agreement with the crystal structure result.²⁴ A medium intensity peak at 608 cm^{-1} is attributed to $\delta(\text{COO})$ vibration. The peaks at 1529 and 1438 cm^{-1} are assigned to ring stretching vibrations of pyridine rings of phenanthroline co-ligand. The sharp absorption peak observed at 727 cm^{-1} is attributed to ring breathing vibration of phenanthroline ring.

UV – Visible Spectroscopy

The UV – vis absorption spectrum of the complex is presented in **Fig. 8**. The UV – vis spectrum of the complex was recorded in the wavelength range (200 – 800 nm) using a 0.1 mM sample solution of DMF. In solution, the V(IV) complex exhibited absorption bands at 323 and 383 nm, which are assigned to be $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ transitions, respectively. Moreover, the broad band observed at 482 nm is assigned to the ligand to metal charge transfer (LMCT).²⁵ In the electronic spectrum, d – d transition is not observed which may be attributed to the dominance of the ligand to metal charge transfer (LMCT). The band assignments are presented in **Table 5**.

Solid state UV–vis reflective spectrum for **1** is shown in **Fig. 8(b)**. The bands at 409 and 483 nm for **1** are assigned to $\pi \rightarrow \pi^*$ and the charge–transfer transitions from ligand to vanadium (IV) ions respectively. The weak absorptions can be observed at 738 nm which is attributed to d-d transitions.²⁶

Table 4
The UV-vis absorption peaks of **1** in DMF solution and their molar absorption coefficients

Complex	λ_{\max} (nm)	ϵ (Lmol ⁻¹ cm ⁻¹)
VO(salala)(phen)].0.5H ₂ O.CH ₃ OH (1)	323 (sh)	3.16 x 10 ⁻⁵
	383	3.04 x 10 ⁻⁵
	482	3.03 x 10 ⁻⁶

Conclusions

A novel oxovanadium(IV) Schiff base complex derived from L-alanine and salicylaldehyde with 1,10-phenanthroline as co-ligand have been synthesized and characterized by single crystal X-ray diffraction, UV-vis and FT-IR spectroscopies. The two crystallographically unique vanadium centers of **1** are connected into a 2D supramolecular network structure via $\pi \cdots \pi$ stacking, C-H \cdots O and O-H \cdots O hydrogen bonding interactions. Furthermore, all the supramolecular interactions were identified using Hirshfeld surface analysis.

Declarations

Acknowledgements

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Author contributions

All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

Conflict of interest statement

The authors declare no conflicts of interest regarding this article.

Supplementary Data

Crystallographic data for the complex have been deposited with the Cambridge Crystallographic Data Centre with CCDC No. 2217555. The data can be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge, CB21EZ, UK (fax: +44 1223336 033; e-mail:deposit@ccdc.cam.ac.uk or www: <http://www.ccdc.cam.ac.uk>).

References

1. Liu X, Hamon JR (2019) Recent developments in penta-, hexa- and heptadentate Schiff base ligands and their metal complexes *Coord. Chem. Rev.* 389:94-118
2. Alfonso-Herrera LA, Rosete-Luna S, Hernández-Romero D, Rivera-Villanueva JM, Olivares-Romero JL, Cruz-Navarro JA, Soto-Contreras A, Arenaza-Corona A, Morales-Morales D, Colorado-Peralta R (2022) Transition Metal Complexes with Tridentate Schiff Bases (O N O and O N N) Derived from Salicylaldehyde: An Analysis of Their Potential Anticancer Activity *Chem Med Chem*, 17 e202200367
3. Lahkar S, Borah R, Deori N, Brahma S (2020) (L)-phenylalanine derived Schiff base ligated vanadium(IV) complex as an efficient catalyst for a CO₂ fixation reaction *Polyhedron* 192:114848
4. Das B, Medhi OK (2013) Synthesis, X-ray crystallography, spectroscopic characterization and spectroscopic/electrochemical evidence of formation of phenoxy free radical in active center analogs of galactose oxidase – [Cu(Salgly)H₂O] and [Cu(Salphenylalanine)H₂O] *Spectrochim. Acta A Mol. Biomol. Spectrosc.* 104:352-357
5. Cao Y, Yi C, Liu H, Li H, Li Q, Yuan Z, Wei G (2016) Syntheses, crystal structures and in vitro anticancer activities of oxovanadium(IV) complexes of amino acid Schiff base and 1,10-phenanthroline ligands *Transit Met Chem* 41:531-538
6. Hu J, Liu S (2014) Engineering Responsive Polymer Building Blocks with Host–Guest Molecular Recognition for Functional Applications *Acc. Chem. Res.* 47:2084-2095
7. Jena S, Dutta J, Tulsian KD, Sahu AK, Choudhury SS, Biswal HS (2022) Noncovalent interactions in proteins and nucleic acids: beyond hydrogen bonding and π -stacking *Chem. Soc. Rev.*, 51:4261-4286
8. Zheng L, Hu S, Zhou A (2015) Two three-dimensional supramolecular networks built from metallophenols through hydrogen bonds and coordinate bonds *Transition Met Chem* 40:637-642
9. Yang W, Li B, Wang H-L, Alduhaish O, Alfooty K, Zayed MA, Li P, Arman HD, Chen B-L (2015) A Microporous Porphyrin-Based Hydrogen-Bonded Organic Framework for Gas Separation *Cryst Growth Des* 2000:15
10. Roesky HW, Andruh M (2003) The interplay of coordinative, hydrogen bonding and π - π stacking interactions in sustaining supramolecular solid-state architectures.: A study case of bis(4-pyridyl)- and bis(4-pyridyl-N-oxide) tectons *Coord. Chem. Rev.* 236:91-119
11. Chakrabarty R, Mukherjee PS, Stang PJ (2011) Supramolecular Coordination: Self-Assembly of Finite Two- and Three-Dimensional Ensembles *Chem. Rev.* 111:6810-6918
12. Cao Y-Z, Zhao H-Y, Bai F-Y, Xing Y-H, Wei D-M, Niu S-Y, Shi Z (2011) Aminoacid-derivatized oxidovanadium complexes: Synthesis, structure and bromination reaction activity *Inorg. Chim. Acta* 368:223-230
13. Bruker AXS Inc. (2007) Programme Name (s), Madison, Wisconsin, USA
14. Sheldrick, GM (2008) Crystal structure refinement with SHELXL. *Acta Crystallogr. Sect. A Found. Crystallogr.* 64:112-122
15. Farrugia, LJ (1999) WinGX and ORTEP for Windows: An update *J. Appl. Crystallogr* 32: 837-838.

16. Zhao H-Y, Xing Y-H, Cao Y-Z, Li Z-P, Wei D-M, Zeng X-Q, Ge M-F (2009) Synthesis, structure and properties of three new oxidovanadium complexes containing a tridentate salicylaldehydeglycine J. Mol. Struct. 938:54-64
17. Saha U, Si TK, Nandi PK, Mukherjea KK (2013) An amino acid coordinated vanadium (IV) complex: Synthesis, structure, DFT calculations and VHPO mimicking catalytic bromoperoxidation of organic substrates Inorg. Chem. Commun. 38:43-46
18. Rehder D, Ebel M, Wikete C, Santoni G, Gätjens (2005) Modeling the active site structures of vanadate-dependent peroxidases and vanadate-inhibited phosphatases J. Pure Appl. Chem. 415:1607-1616
19. Spackman PR, Turner MJ, McKinnon JJ, Wolff SK, Grimwood DJ, Jayatilaka D, Spackman, MA. CrystalExplorer: (2021) A program for Hirshfeld surface analysis, visualization and quantitative analysis of molecular crystals. J. Appl. Cryst. 54:1006-1011
20. Venkatesan P, Thamocharan S, Ilangovan A, Liang H, Sundius T (2016) Crystal structure, Hirshfeld surfaces and DFT computation of NLO active (2E)-2-(ethoxycarbonyl)-3-[(1-methoxy-1-oxo-3-phenylpropan-2-yl)amino] prop-2-enoic acid Spectrochim. Acta A Mol. Biomol. Spectrosc. A 153:625-636
21. McKinnon JJ, Jayatilaka D, Spackman MA (2007) Towards quantitative analysis of intermolecular interactions with Hirshfeld surfaces Chem. Commun. 3814-3816
22. Parajón-Costa BS, Piro OE, Pis-Diez R, Castellano EE, González-Baró AC (2006) Crystal structures, spectroscopic characterization and theoretical calculations of the guanidinium and ammonium salts of the insulin-enhancing anion $[\text{VO}_2(\text{dipic})]^-$ Polyhedron 25:2920-2928
23. Dikio CW, Okoli BJ, Mtunzi FM (2017) Synthesis of new anti-bacterial agents: Hydrazone Schiff bases of vanadium acetylacetonate complexes Cogent Chem. 3:1336864
24. Gogoi A, Bharali L (2022) Conversion of Glycine to Oxalate in Presence of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ and Isonicotinamide Russ. J. Inorg. Chem. 67:608-615
25. Szklarzewicz J, Jurowska A, Hodorowicz M, Kazek G, Mordyl B, Menaszek E, Sapa J (2021) Characterization and antidiabetic activity of salicylhydrazone Schiff base vanadium(IV) and (V) complexes Transit. Met. Chem. 46:201-217
26. Deng L, Jin W-T, Weng W-Z, Zhou Z-H (2019) Interactions of vanadium with amino acids— Monodentate coordination of vanadyl proline, lysine and histidine and catalytic degradations of methyl orange Polyhedron 159:375-381

Figures

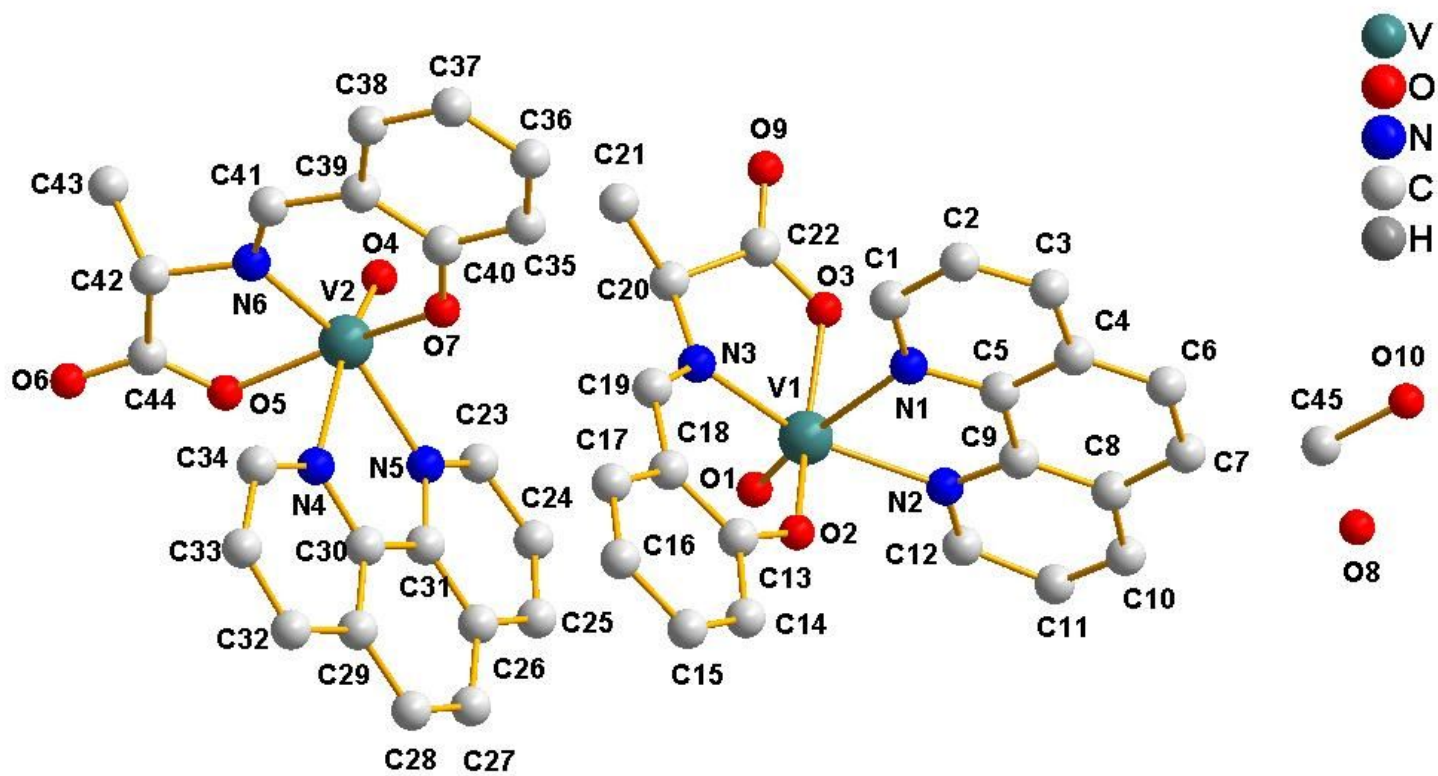


Figure 1

Molecular structure of complex 1 (Hydrogen atoms are omitted for clarity)

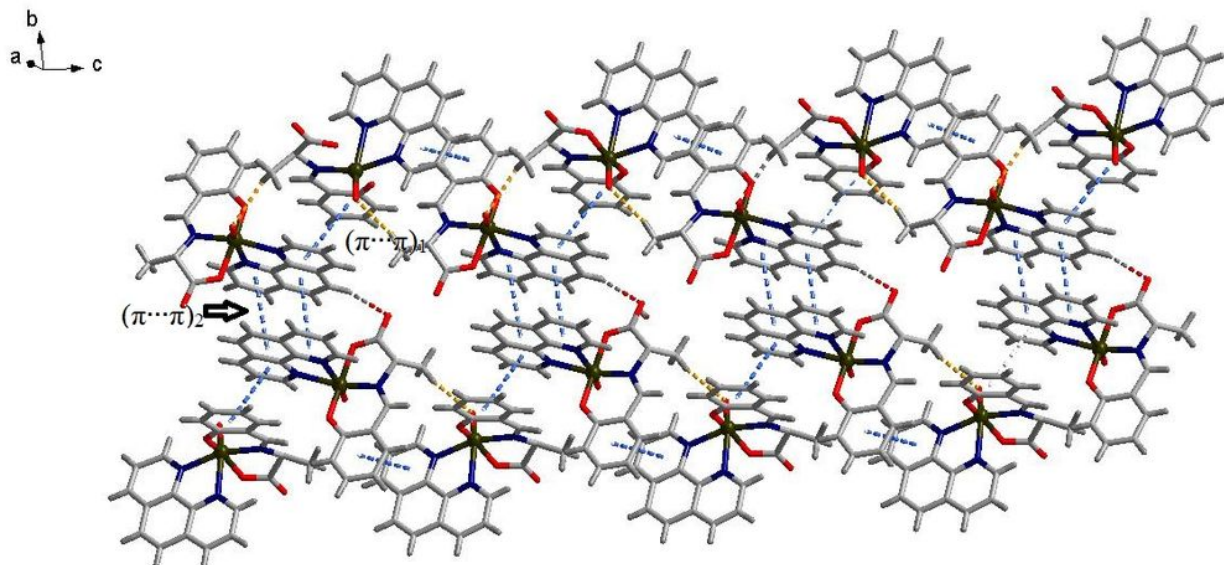


Figure 2

Supramolecular 2D assembly along the *bc* plane

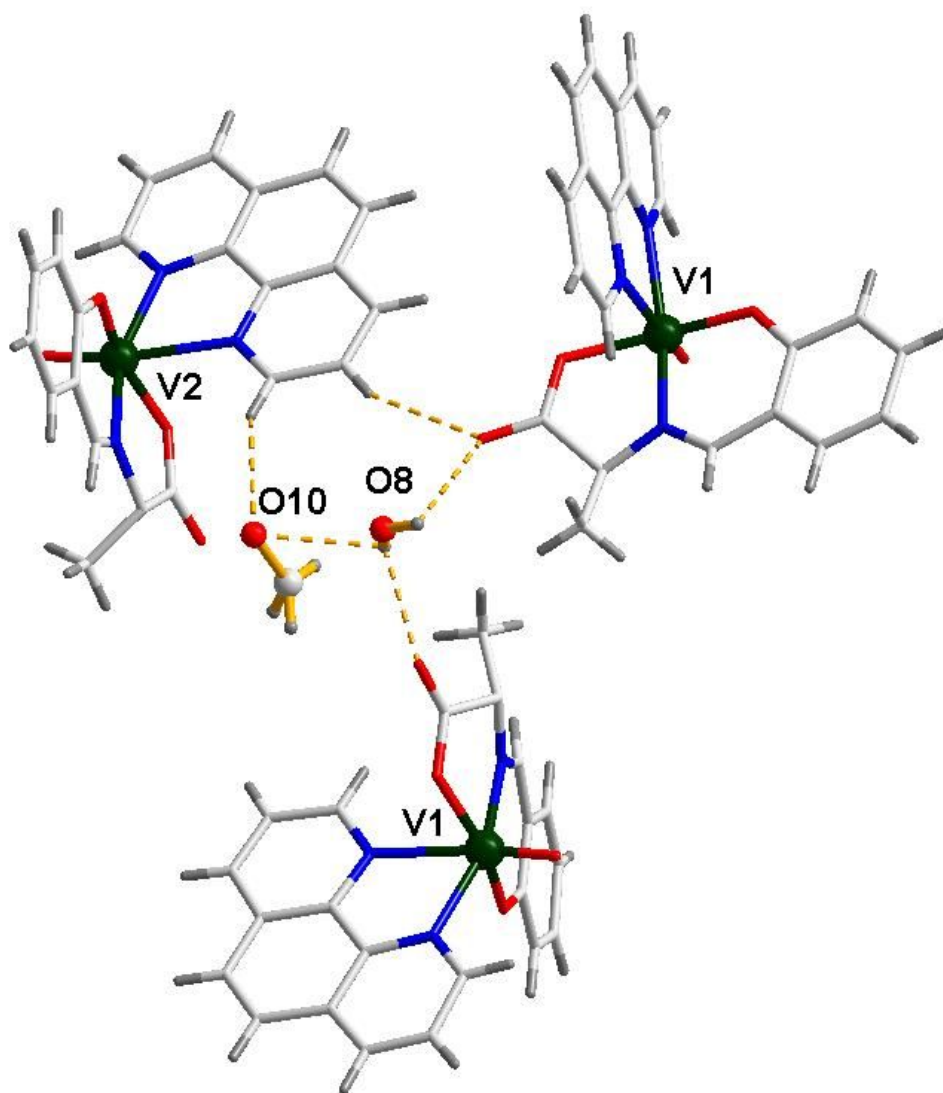


Figure 3

O-H...O and C-H...O hydrogen bonding interactions involving the lattice methanol and water

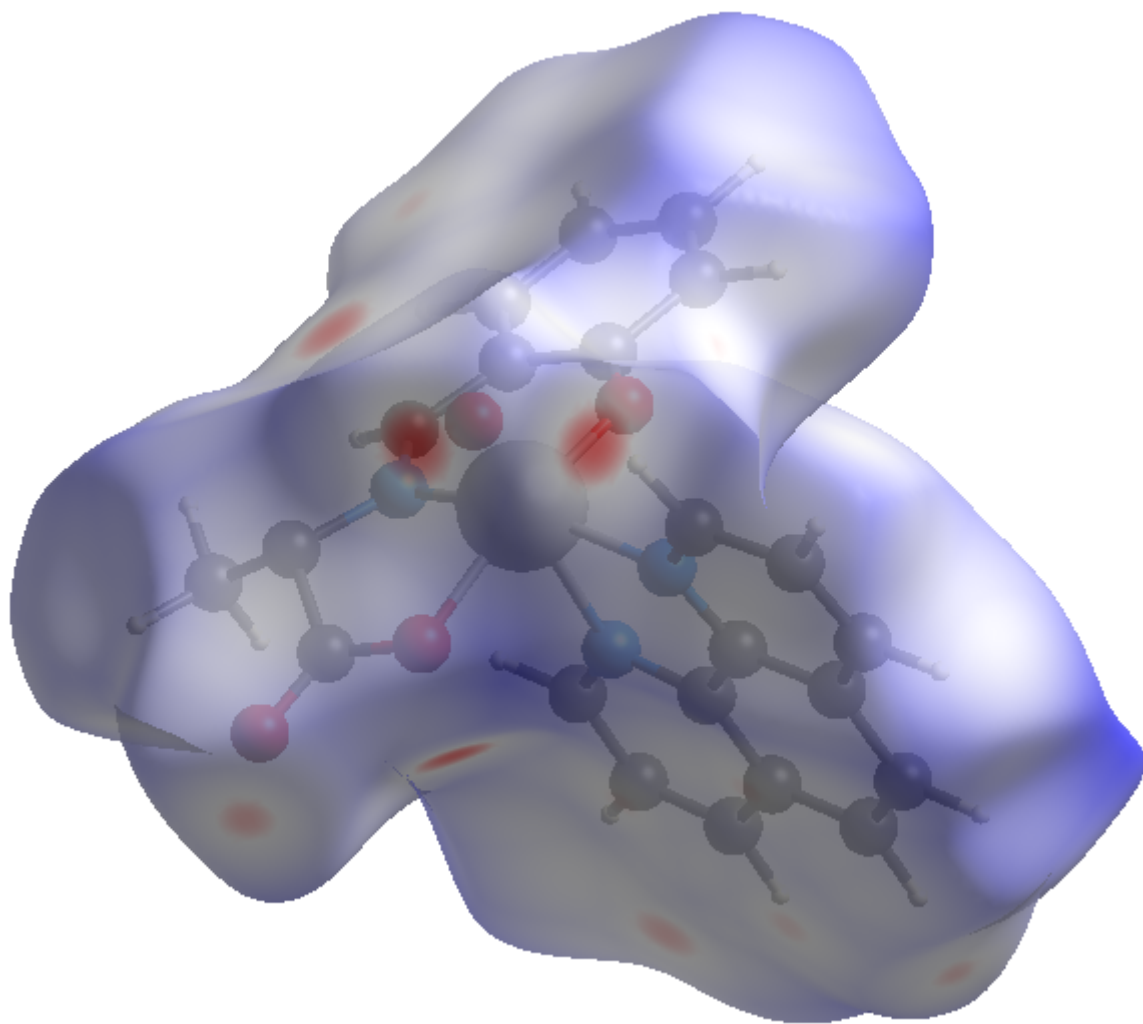


Figure 4

View of the Hirshfeld surfaces of **1** mapped over d_{norm} properties

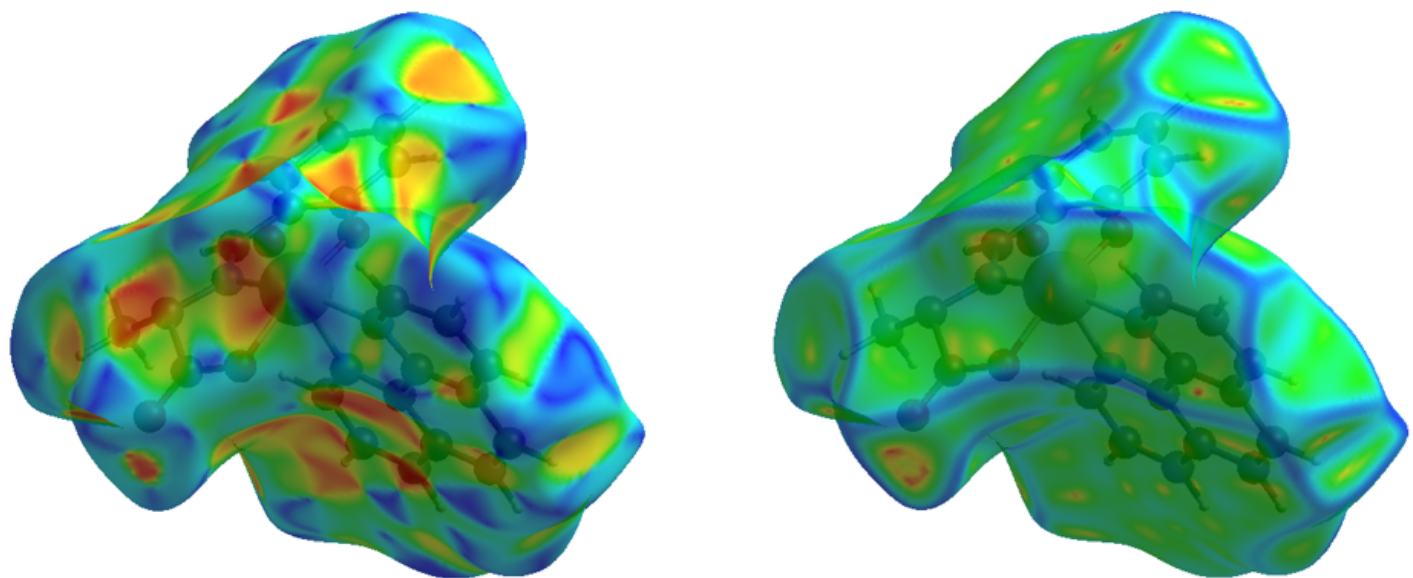


Figure 5

Hirshfeld surfaces of **1** mapped over (a) shape index and (b) curvedness properties

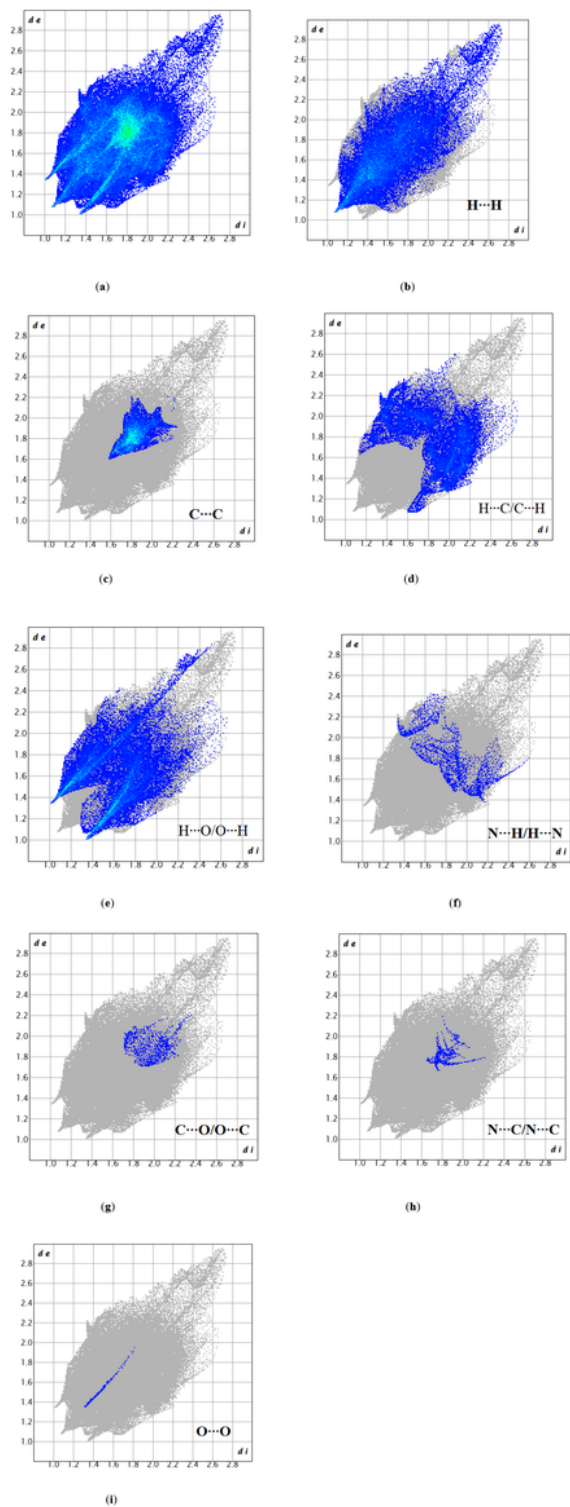


Figure 6

The full two-dimensional fingerprint plots for the title complex, showing **(a)** all interactions, and delineated into **(b)** H...H, **(c)** C...C, **(d)** H...C/C...H, **(e)** H...O/O...H interactions, **(f)** N...H/H...N, **(g)** C...O/O...C, **(h)** N...C/C...N and **(i)** O...O. The d_i and d_e values are the closest internal and external distances (in Å) from given points on the Hirshfeld surface contacts.

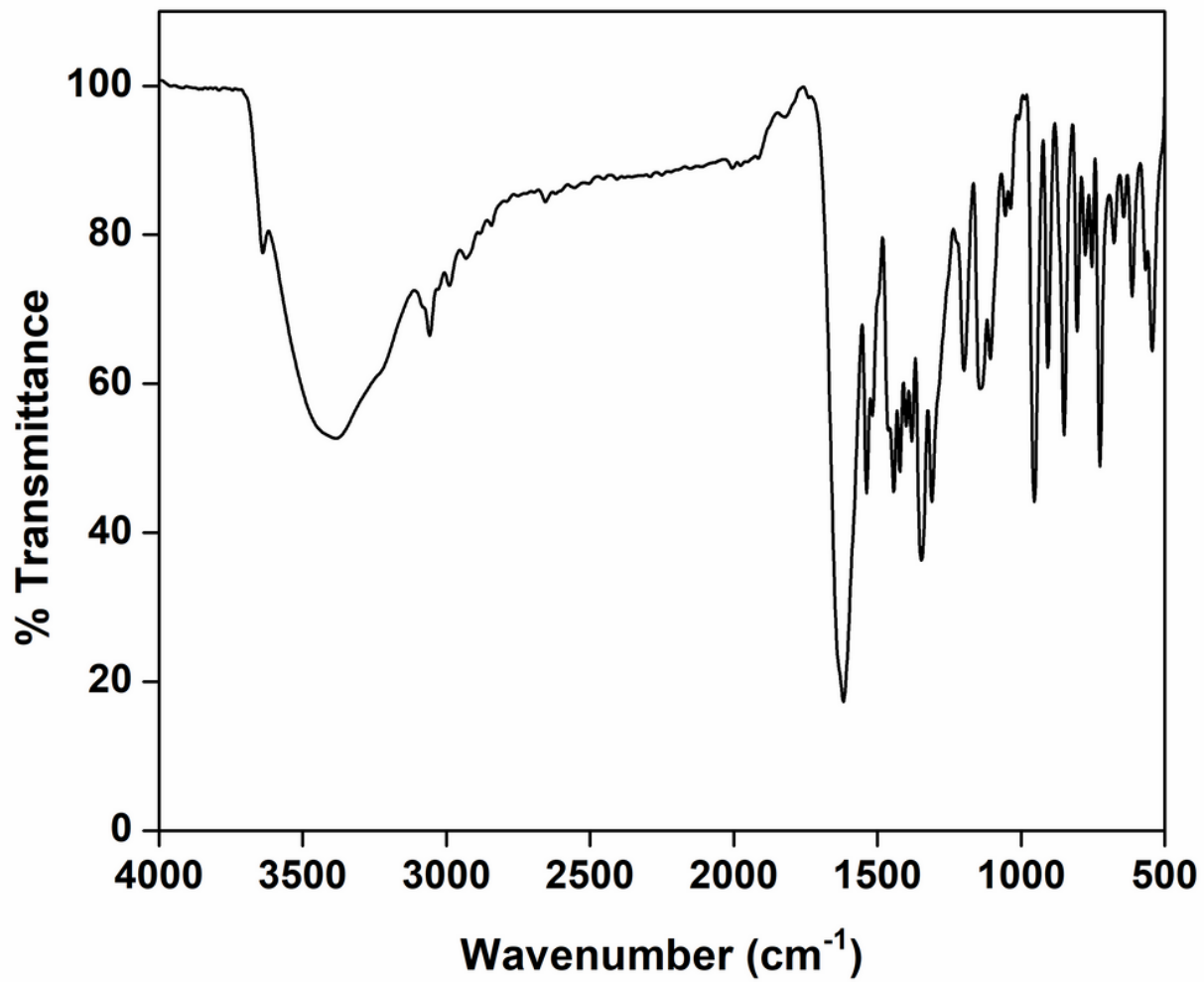
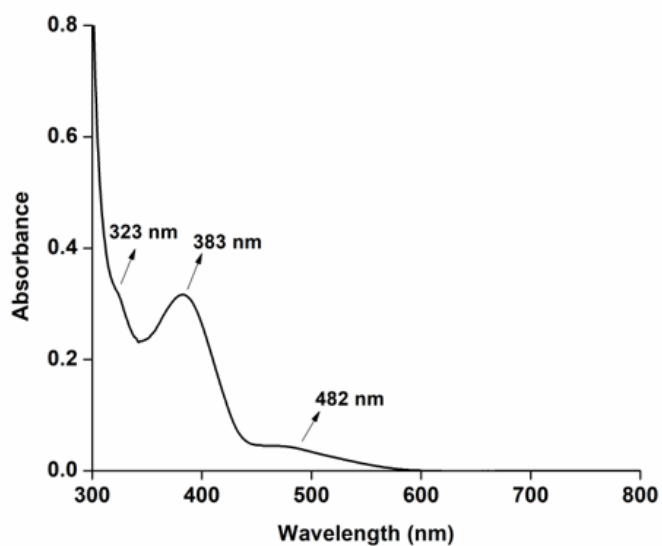


Figure 7

KBr phase FT-IR spectrum of 1

(a)



(b)

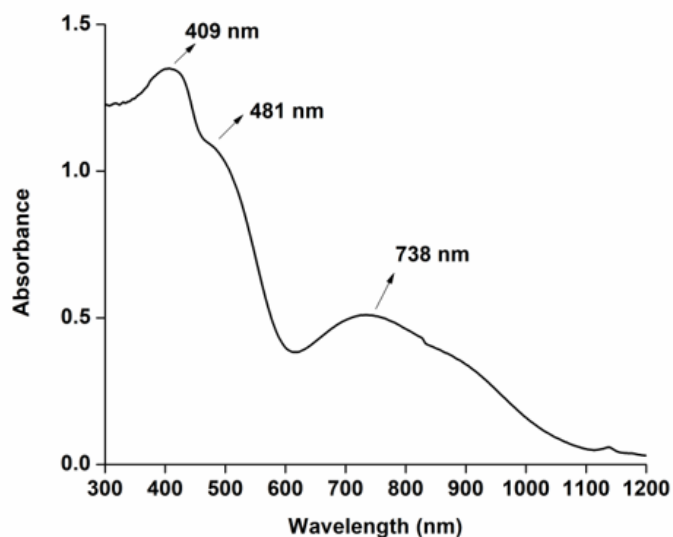


Figure 8

UV-vis spectra of **1** in (a) DMF (10^{-4} M) and (b) solid state

Supplementary Files

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- [IndexAbstract.docx](#)
- [VOsalalaphen.H2O.CH3OH.cif.cif](#)
- [checkcif1.pdf](#)