APPENDIX-II

List of Publications

1. Bis[2-(4,5-Diphenyl-1 *H* -Imidazol-2-Yl)-4-Nitrophenolato]Copper(II) Dihydrate: Crystal Structure and Hirshfeld Surface Analysis., **Sailesh Chettri**, Dhiraj Brahman, Biswajit Sinha, Mukesh.M.Jotani and Edward R.T.Tiekink *Acta Crystallographica Section E Crystallographic Communications* **2019**, ***75*** **(11), 1664–1671.**
2. Exploration of Inhibitory Action of Azo Imidazole Derivatives against COVID-19 Main Protease (Mpro): A Computational Study., Abhijit Chhetri, **Sailesh Chettri**, Pranesh Rai, Biswajit Sinha and Dhiraj Brahman *Journal of Molecular Structure* **2021, *1224*, 129178.**
3. Synthesis, Characterization and Computational Study on Potential Inhibitory Action of Novel Azo Imidazole Derivatives against COVID-19 Main Protease (Mpro: 6LU7)., Abhijit Chhetri, **Sailesh Chettri**, Pranesh Rai, Dipu Kumar Mishra, Biswajit Sinha and Dhiraj Brahman *Journal of Molecular Structure* **2021, *1225*, 129230.**
4. Environmentally Benign Approach towards C–S Cross-Coupling Reaction by Organo-Copper(II) Complex., Rabindranath Singha, **Sailesh Chettri**, Dhiraj Brahman, Biswajit Sinha and Pranab Ghosh *Molecular Diversity.,* **2022**, ***26*** **(1), 505–511.**
5. [Diaquo{bis(p-hydroxybenzoato-κ1O1)}(1-methylimidazole-κ1N1)}copper(II)]: Synthesis, crystal structure, catalytic activity and DFT study., Amarjit Kamath, Dhiraj Brahman, **Sailesh Chettri**, Patrick McArdle and Biswajit Sinha *Journal of Molecular Structure* **2022, 1247, 131323.**

APPENDIX-III

List of Communicated Articles

1. Copper Borate (CuB_4O_7) catalyzed multi-component green synthesis of 2,4,5 Tri-aryl imidazole derivatives and evidence of In-situ conversion of Copper Borate to Copper Acetate in presence of NH_4OAc. **(Communicated)**
2. DFT, Molecular Docking and Pharmacokinetic study of some selected 2, 4, 5-Tri-arylimidazole derivatives. **(Communicated)**
3. Synthesis, DFT, Molecular Docking and Pharmacokinetic study of some selected 3, 4-dihydropyrimidin-2(1H)-one (DHPM) derivatives. **(Communicated)**
4. Synthesis, DFT, Molecular Docking and Pharmacokinetic study of some selected 1- hydroxy-2-arylimidazole-3-oxide derivatives. **(Communicated)**

APPENDIX-IV

List of Seminars, Webinars, Symposiums and Conferences Attended

1. National Seminar on "Frontiers in Chemistry-2019" organized by the Department of Chemistry, University of North Bengal, Darjeeling and CRSI North Bengal Local Chapter on 22nd May 2019.
2. International Seminar on the "International Year of the Periodic Table of Chemical Elements-2019" organized by the Department of Chemistry, University of North Bengal, Darjeeling on 22-23rd November 2019. **(Presented a Poster and got the award for One of the Best Poster Presentations).**
3. National Seminar on "Frontiers in Chemistry-2020" organized by the Department of Chemistry, University of North Bengal on 5th March 2020. **(Presented a Poster)**
4. National Seminar on "Material Chemistry-Today & Tomorrow" organized by the Indian Chemical Society, Kolkata & Department of Chemistry, Jadavpur University, Kolkata on 28th February – 1st March 2021. **(Presented a Poster).**
5. Interdisciplinary International Web Seminar on "Modern Trends in Humanities, Science & Technology and Social Sciences for Sustainable Development" organized by A.P.C. Roy Govt. College, Siliguri in collaboration with UGC-Human Resource Development Centre, University of North Bengal, Darjeeling on 23rd – 24th September 2021**. (Presented an oral presentation).**

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INDEX

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Bis[2-(4,5-diphenyl-1*H*-imidazol-2-yl)-4-nitrophenolato]copper(II) dihydrate: crystal structure and Hirshfeld surface analysis

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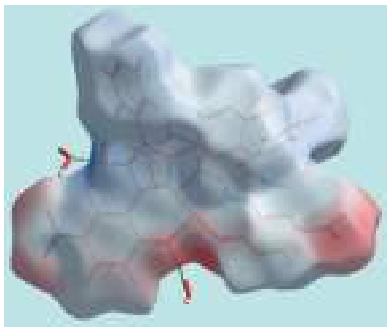
The crystal and molecular structures of the title Cu^{II} complex, isolated as a dihydrate, $[Cu(C_{21}H_{14}N_3O_3)_2]\cdot 2H_2O$, reveals a highly distorted coordination geometry intermediate between square-planar and tetrahedral defined by an N_2O_2 donor set derived from two mono-anionic bidentate ligands. Furthermore, each six-membered chelate ring adopts an envelope conformation with the Cu atom being the flap. In the crystal, imidazolyl-amine-N—H···O(water), water-O—H···O(coordinated, nitro and water), phenyl-C—H···O(nitro) and π(imidazolyl)–π(nitrobenzene) [inter-centroid distances = 3.7452 (14) and 3.6647 (13) Å] contacts link the components into a supramolecular layer lying parallel to (101). The connections between layers forming a three-dimensional architecture are of the types nitrobenzene-C—H···O(nitro) and phenyl-C—H···π(phenyl). The distorted coordination geometry for the Cu^{II} atom is highlighted in an analysis of the Hirshfeld surface calculated for the metal centre alone. The significance of the intermolecular contacts is also revealed in a study of the calculated Hirshfeld surfaces; the dominant contacts in the crystal are H···H (41.0%), O···H/H···O (27.1%) and C···H/H···C (19.6%).

1. Chemical context

The title copper(II) complex, (I), was isolated during an ongoing research programme on the catalytic activity of copper borate (CuB_4O_7) for C—N heterocyclic bond formation reactions. Complex (I) was formed during the attempted synthesis of a triarylimidazole derivative using benzil and the respective aromatic aldehyde with copper borate, using ammonium acetate as a nitrogen source. The single-crystal analysis of the synthesized product revealed that in the copper(II) complex, the triarylimidazole moiety acts as a bidentate ligand for the copper atom. During the successful synthesis of the triarylimidazole, the desired product formed in good yield at a temperature in the range 100–110 °C. However, when the reaction was conducted at 130 °C and above, the title copper(II) complex formed instead of the targeted triarylimidazole. The crystal and molecular structures of (I) are described herein, along with a detailed analysis of the molecular packing *via* an analysis of the calculated Hirshfeld surfaces.



2. Structural commentary

The crystallographic asymmetric unit of (I) comprises a complex molecule and two water molecules of crystallization.

Table 1
Selected geometric parameters (Å, °).

Cu—O1	1.9291 (17)	Cu—N1	1.9586 (19)
Cu—O2	1.9304 (17)	Cu—N2	1.957 (2)
O1—Cu—O2	89.36 (7)	O2—Cu—N1	144.41 (8)
O1—Cu—N2	147.34 (8)	O2—Cu—N2	93.56 (7)
O1—Cu—N1	92.83 (8)	N1—Cu—N2	103.14 (8)

The copper(II) centre in (I), Fig. 1, is bis-*N*,*O*-chelated by two 2-(4,5-diphenyl-1*H*-imidazol-2-yl)-4-nitrophenolate monoanions. The resulting N_2O_2 donor set defines a highly distorted coordination geometry, as seen in the angles included in Table 1 and in the view of Fig. 2. The angles range from a narrow 89.36 (7)°, for O1—Cu—O2, to a wide 147.34 (8)°, for O1—Cu—N2. The distortion is highlighted in the dihedral angle between the best planes through the two chelate rings of 49.82 (7)°. The value of τ_4 is a geometric measure of the distortion of a four-coordinate geometry (Yang *et al.*, 2007). For (I), the value computes as 0.48 which is almost exactly intermediate between the values of τ_4 = 0 for an ideal tetrahedron and τ_4 = 1.0 for an ideal square-planar geometry. In fact, the six-membered chelate rings are not planar, each adopting an envelope conformation with the Cu atom being the flap atom. In this description, the r.m.s. deviation for the least-squares plane through the O1/N1/C1/C2 atoms is 0.036 Å with the Cu atom lying 0.410 (3) Å out of the plane. The comparable parameters for the O2-chelate ring are 0.033 and 0.354 (3) Å, respectively. The dihedral angle formed between the two planar regions of the chelate rings is 49.38 (8)°. The dihedral angles between the best plane through the O1-chelate ring and each of the fused six- and five-membered rings are 9.18 (12) and 5.54 (14)°, respectively; the equivalent angles for the O2-chelate rings are 8.44 (8) and 2.71 (9)°, respectively. The N1-imidazol-2-yl ring forms dihedral angles of 41.20 (11) and 37.46 (10)° with the C10- and C16-phenyl substituents, respectively, and the dihedral angle between the phenyl rings is 59.92 (8)°, *i.e.* all indicating splayed relationships. A similar situation pertains to the N2-imidazol-2-yl ring, where the comparable dihedral angles formed with the C31- and C37-phenyl rings are 38.29 (10), 48.5 (9) and 50.84 (7)°, respectively. Finally, the nitro groups are not strictly coplanar with the benzene rings to which they are connected, as seen in the dihedral angles of 14.2 (4)° for C1–C6/N4/O3/O4 and 5.9 (3)° for C22–C27/N6/O5/O6.

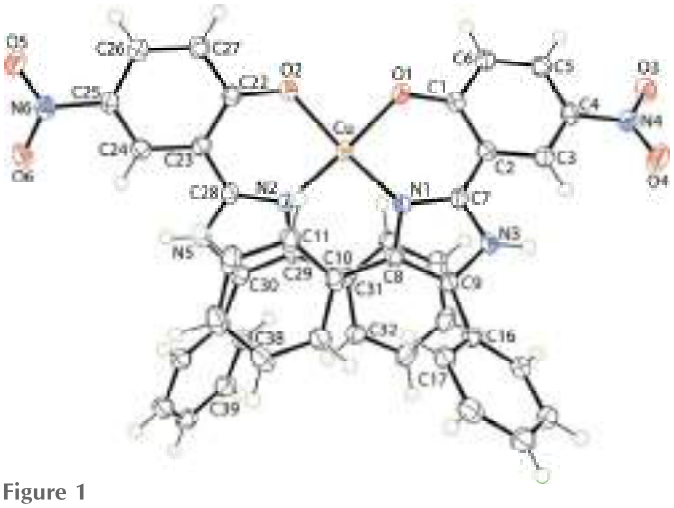


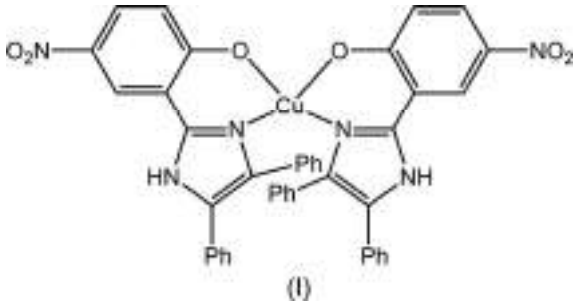
Figure 1
The molecular structure of the complex molecule in (I), showing the atom-labelling scheme and with displacement ellipsoids drawn at the 70% probability level.

Table 2
Hydrogen-bond geometry (Å, °).

*Cg*1 is the ring centroid of the C16–C21 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3*N*···O1*W*^{i}	0.89 (2)	1.91 (2)	2.790 (3)	173 (3)
N5—H5*N*···O2*W*	0.88 (2)	1.95 (2)	2.822 (3)	172 (3)
O1*W*—H1*W*···O2	0.85 (2)	1.92 (2)	2.745 (2)	164 (2)
O1*W*—H2*W*···O2*W*^{ii}	0.85 (2)	2.21 (2)	2.868 (2)	134 (2)
O2*W*—H3*W*···O1^{ii}	0.84 (2)	2.01 (2)	2.841 (2)	172 (2)
O2*W*—H4*W*···O3^{iii}	0.84 (2)	2.27 (2)	2.938 (2)	136 (2)
C3—H3···O1*W*^{i}	0.95	2.57	3.435 (3)	151
C33—H33···O5^{iv}	0.95	2.48	3.345 (3)	151
C5—H5···O6^{v}	0.95	2.50	3.361 (3)	151
C34—H34···*Cg*1^{vi}	0.95	2.49	3.426 (3)	168

Symmetry codes: (i) $-x+2, -y+1, -z+1$; (ii) $-x+1, -y+1, -z+1$; (iii) $x-1, y, z-1$; (iv) $-x+1, y+\frac{1}{2}, -z+\frac{1}{2}$; (v) $x+1, y, z+1$; (vi) $x, -y+\frac{1}{2}, z-\frac{1}{2}$.



(I)

3. Supramolecular features

As each component of the asymmetric unit has hydrogen-bonding functionality, conventional hydrogen bonds are found in the crystal of (I); the geometric parameters characterizing the identified intermolecular interactions operating in the crystal of (I) are collated in Table 2. Each of the imidazolyl-amine-N—H atoms forms a donor interaction to a water molecule to generate a three-molecule aggregate. The O1*W* water molecule forms donor interactions to the coordinated O2 atom and to a symmetry-related O2*W* water molecule. The O2*W* water molecule connects to the coordinated O1 atom as well as to a nitro-O3 atom. Hence, the O2*W* water molecule is involved in four hydrogen-bonding interactions. The fourth contact involving the O1*W* water molecule, a C—H···O acceptor contact, is provided by the nitrobenzene ring. There is also a phenyl-C—H···O(nitro) contact of note, Table 2. The aforementioned interactions combine to stabilize a supramolecular layer lying parallel to (101), as shown in Fig. 3(*a*). There are also π–π stacking and C—H···O interactions in the crystal, Fig. 3(*b*). Within layers, there are π–π interactions occurring between the imidazolyl and nitrobenzene rings

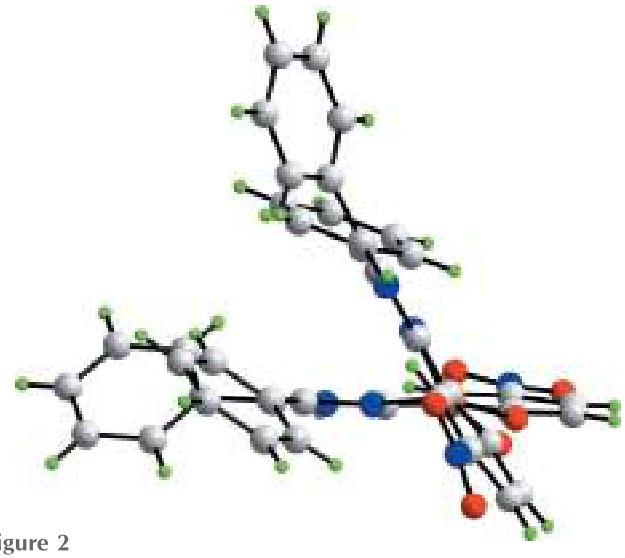


Figure 2
A view of the molecular structure of the complex molecule in (I), highlighting the distorted coordination geometry about the copper(II) atom.

[inter-centroid distances: *Cg*(N1/N3/C7–C9)···*Cg*(C1–C6) = 3.7452 (14) Å and angle of inclination = 9.70 (13)° for symmetry operation (−*x* + 2, −*y* + 1, −*z* + 1); *Cg*(N2/N5/C28–C30)···*Cg*(C22–C27) = 3.6647 (13) Å and angle of inclination = 8.15 (12)° for (−*x* + 1, −*y* + 1, −*z* + 1)]. The connections between layers along [010] are of the type nitrobenzene-C—H···O(nitro) and phenyl-C—H···π(phenyl), as detailed in Table 2.

4. Hirshfeld surface analysis

The Hirshfeld surface calculations for (I) were performed with *CrystalExplorer17* (Turner *et al.*, 2017) and published protocols (Tan *et al.*, 2019), and serve to indicate the significant role of the two water molecules in the supramolecular association in the crystal. The involvement of both the water molecules in hydrogen bonds, Table 2, are evident as bright-red spots near the respective atoms on the Hirshfeld surfaces mapped over d_{norm} for the O1*W*-, Fig. 4(*a*), and O2*W*-water, Fig. 4(*b*), molecules. In addition, the presence of faint-red spots near the O1*W*, O2*W* and H1*W* atoms in Figs. 4(*a*) and 4(*b*) are indicative of the other contacts of these atoms with those of the Cu^{II} complex molecule (Table 2). The donors and acceptors of the hydrogen bonds involving atoms of the complex molecule are also apparent as bright-red spots near the participating atoms in the views of the Hirshfeld surfaces calculated for the complex molecule shown in Figs. 4(*c*)–(*e*).

The presence of a short interatomic C···C contact between atoms C22 and C28 (Table 3) arises from π–π stacking between symmetry-related imidazole and nitrobenzene rings, and is observable as the faint-red spots near these atoms on the d_{norm}-mapped Hirshfeld surface in Fig. 4(*c*). The pair of faint-red spots appearing near the phenyl-C36 and H36 atoms, and also near the nitro-O5 atom on the surface indicating short interatomic contacts that characterize the weak C—H···O interaction, Table 3. The influence of the C—H···π contact on the molecular packing is recognized from the three faint-red spots in the phenyl-(C16–C21) ring and another near atom H34 in Fig. 4(*e*). The donors and acceptors of this interaction are also evident as the blue bump and a bright-orange spot enclosed within the black circle on the Hirshfeld surface mapped with the shape-index property in Fig. 5(*a*). The bright-orange region enclosed within a black circle in Fig. 5(*b*) is also an indication of the O2*W*—H4*W*···*Cg*(C16–C21) contact. The Hirshfeld surfaces mapped over the calculated electrostatic potential for the water and complex molecules in Fig. 6 also illustrate the donors and acceptors of

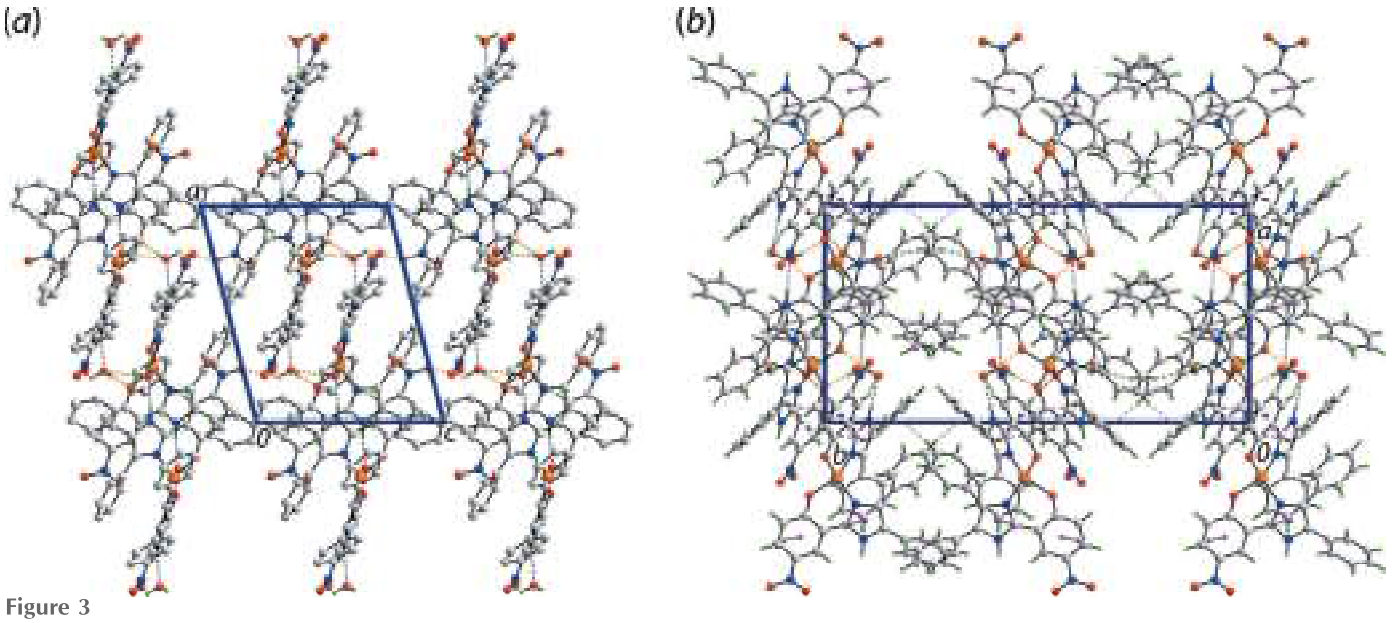


Figure 3
The molecular packing in the crystal of (I): (*a*) a supramolecular layer parallel to (101) sustained by O—H···O, N—H···O and C—H···O interactions shown as orange, blue and green dashed lines, respectively, and (*b*) a view of the unit-cell contents in projection down the *c* axis, with π–π and C—H···π interactions shown as purple and pink dashed lines, respectively.

intermolecular interactions through blue and red regions corresponding to positive and negative electrostatic potentials, respectively. The π–π stacking between symmetry-related imidazolyl and nitrobenzene rings are viewed as the flat regions enclosing them on the Hirshfeld surfaces mapped over curvedness in Fig. 7. On the Hirshfeld surfaces mapped over d_{norm} illustrated in Figs. 4(*c*)–(*e*), faint-red spots also appear near other atoms indicating their involvement in other short interatomic contacts, as summarized in Table 3.

The Hirshfeld surfaces also provide an insight into the distortion in the coordination geometry formed by the N_2O_4 donor set about the copper(II) centre in the complex molecule. This is performed by considering the Hirshfeld surface about the metal centre alone (Pinto *et al.*, 2019). The distortion in the coordination geometry is observed on the Hirshfeld surface mapped with the shape-index property as the bright-orange patches of irregular shape covering a major region for the Cu—O bonds in Fig. 8(*a*) and the small orange regions on the surface relatively far from the Cu—N bonds in Fig. 8(*b*). The different curvature of the Hirshfeld surfaces coordinated by the N_2O_4 donor set in Figs. 8(*c*) and 8(*d*) also support this observation. The Cu—O and Cu—N bonds are rationalized in the two-dimensional fingerprint plot taking into account only the Hirshfeld surface for the copper atom shown in Fig. 9. The distribution of points in the fingerprint plot through the pair of aligned red points at different inclinations from $d_e + d_i \sim 2.0$ Å for the Cu—N bonds (upper region) and the Cu—O bonds (lower region) are indicative of the distorted geometry (Pinto *et al.*, 2019).

The overall two-dimensional fingerprint plot for (I), *i.e.* the entire asymmetric unit, Fig. 10(*a*), and those delineated into H···H, O···H/H···O, C···H/H···C, C···C and C···O/O···C contacts are illustrated in Figs. 10(*b*)–(*f*), respectively. The percentage contribution from different interatomic contacts to the Hirshfeld surfaces of the complex molecule and for overall (I) are summarized in Table 4. The presence of water molecules in the crystal of (I) increases the percentage contribution from O···H/H···O contacts (Table 4) to the Hirshfeld surface of the asymmetric unit compared with the complex molecule alone. This results in slight decreases in the percentage contributions from other interatomic contacts for (I) (Table 4). A single conical tip at $d_e + d_i \sim 1.9$ Å in the fingerprint plot delineated into H···H contacts shown in Fig. 10(*b*) is the result of the involvement of the H12 atom in a short interatomic H···H contact, Table 3. The points due to short interatomic contacts between amine hydrogen-H3N and water hydrogen atoms, H1*W* and H2*W*, Table 3, are merged within the plot. Although the molecular packing of (I) is influenced by several intermolecular O—H···O and C—H···O interactions, the presence of a pair of long spikes at d_e +

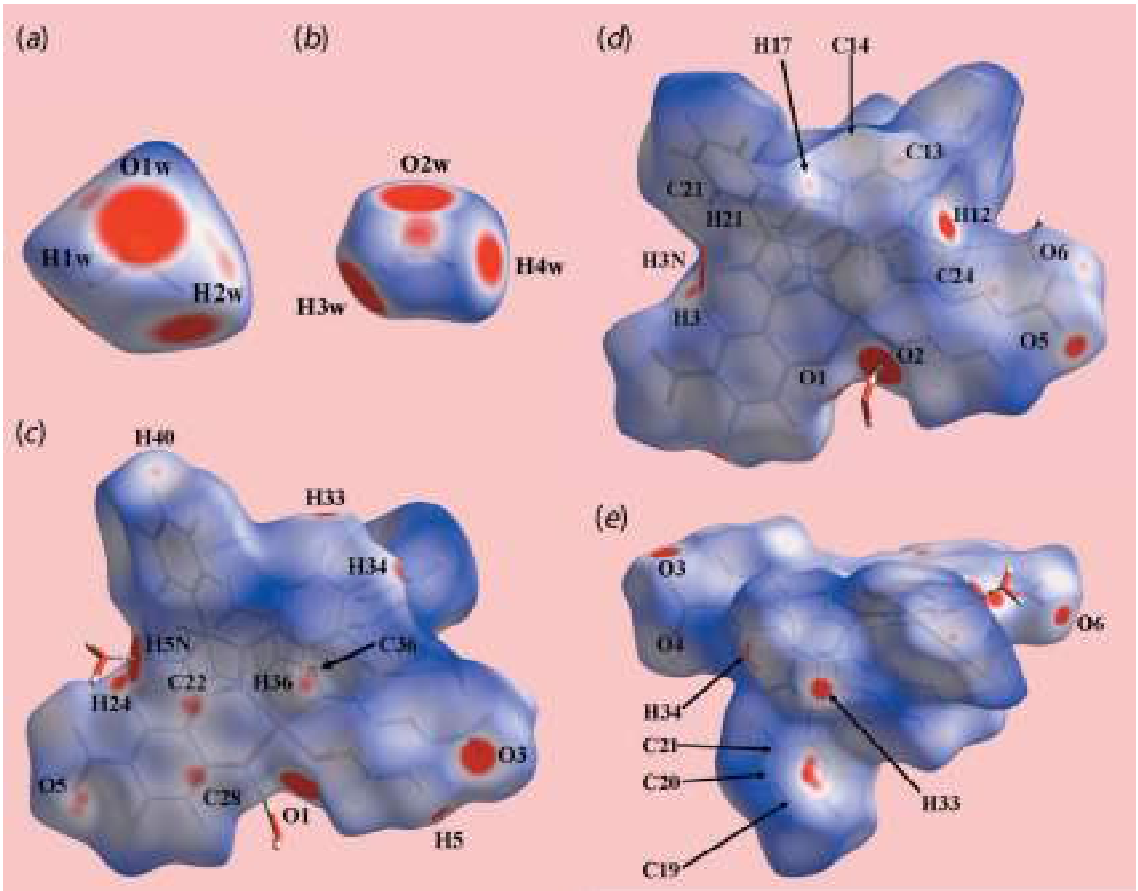


Figure 4
Different views of the Hirshfeld surfaces for the constituents of (I) mapped over d_{norm} for the (*a*) water-O1*W* molecule [in the range −0.2369 to +1.2173 arbitrary units (au)], (*b*) water-O2*W* molecule (−0.2114 to + 0.7500 au) and (*c*)–(*e*) complex molecule (−0.1170 to + 1.6287 au).

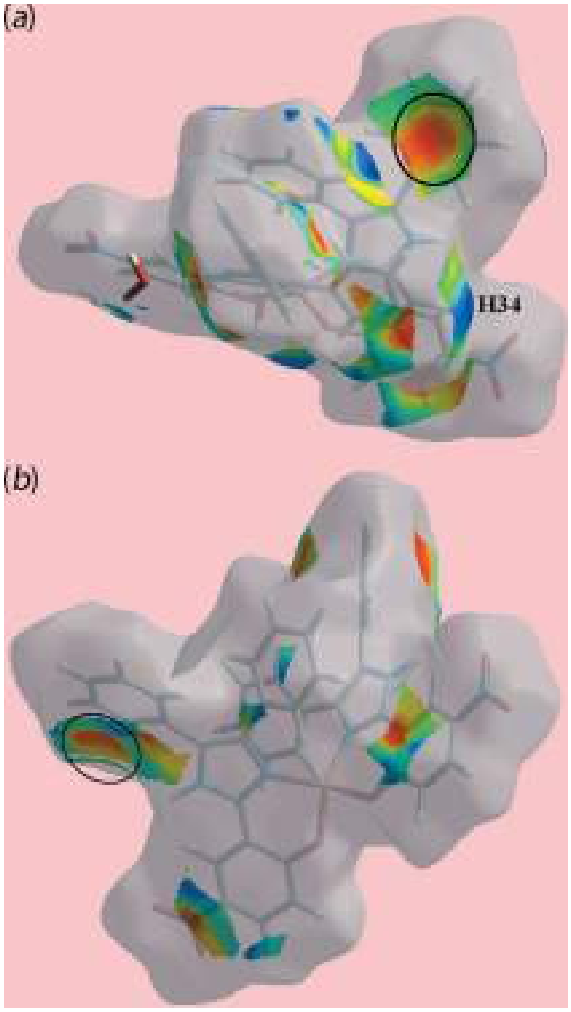


Figure 5
Two views of the Hirshfeld surface mapped with the shape-index property for the complex molecule in (I) from −1.0 to +1.0 arbitrary units highlighting (*a*) the donor and acceptor atoms of the C—H···π interaction through a blue bump near the H34 atom and bright-orange curvature, enclosed within the black circle, and (*b*) the O2*W*—H4*W*···π interaction by the bright-orange region enclosed within the black circle.

$d_i \sim 1.8$ Å in the plot delineated into O···H/H···O contacts, Fig. 10(*c*), arise from the N—H···O hydrogen bond, while the merged points correspond to other interactions at greater interatomic distances. The significant contribution from interatomic C···H/H···C contacts (Table 4) to the Hirshfeld surface of (I) reflect the combined influence of intermolecular C—H···π interactions (Table 2) and the short interatomic C···H/H···C contacts, summarized in Table 3, and viewed as the distribution of points in the form of characteristic wings in Fig. 10(*d*). The presence of short interatomic C···C contacts are evident as the points near a rocket shape tip at $d_e + d_i \sim$ 3.2 Å in the respective delineated fingerprint plot, Fig. 10(*e*), while the points corresponding π–π stacking between the imidazole and nitrobenzene rings are distributed about $d_e = d_i = 1.7$ Å in the plot. The small, *i.e.* 2.7%, contribution from C···N/N···C contacts to the surface is also due to these π–π stacking interactions (delineated plot not shown). The

Table 3
Summary of short interatomic contacts (Å) in (I)[a].

Contact	Distance	Symmetry operation
H12···H12	1.92	$-x+1, -y+1, -z+1$
H1*W*···H3N	2.22	$-x+2, -y+1, -z+1$
H2*W*···H3N	2.26	$-x+2, -y+1, -z+1$
O4···H40	2.54	$x+1, -y+\frac{3}{2}, z+\frac{1}{2}$
C1···H3*W*	2.74	$-x+1, -y+1, -z+1$
C6···O6	3.206 (3)	$-x+1, -y+1, -z+1$
C12···H12	2.55	$-x+1, -y+1, -z$
C13···C25	3.347 (3)	$-x+1, -y+1, -z$
C14···O5	3.197 (3)	$-x+1, -y+1, -z$
H17···O6	2.55	$-x+1, -y+1, -z$
C19···H34	2.68	$x, -y+\frac{3}{2}, z-\frac{1}{2}$
C20···H34	2.60	$x, -y+\frac{3}{2}, z-\frac{1}{2}$
C21···H34	2.67	$x, -y+\frac{3}{2}, z-\frac{1}{2}$
C21···H2*W*	2.64	$-x+2, -y+1, -z+1$
C21···O1*W*	3.161 (3)	$-x+2, -y+1, -z+1$
C22···C28	3.267 (3)	$-x+1, -y+1, -z+1$
C36···O5	3.146 (3)	$-x+1, -y+1, -z+1$
H36···O5	2.49	$-x+1, -y+1, -z+1$
C41···H20	2.76	$-x+1, y, z$

Notes: (*a*) the interatomic distances are calculated in *CrystalExplorer17* (Turner *et al.*, 2017), whereby the *X*—H bond lengths are adjusted to their neutron values.

contribution of 3.2% from C···O/O···C contacts is due to the presence of short interatomic contacts involving nitro-O atoms, Table 2, and are apparent as the pair of parabolic tips at $d_e + d_i \sim 3.2$ Å in the delineated plot of Fig. 10(*f*). The

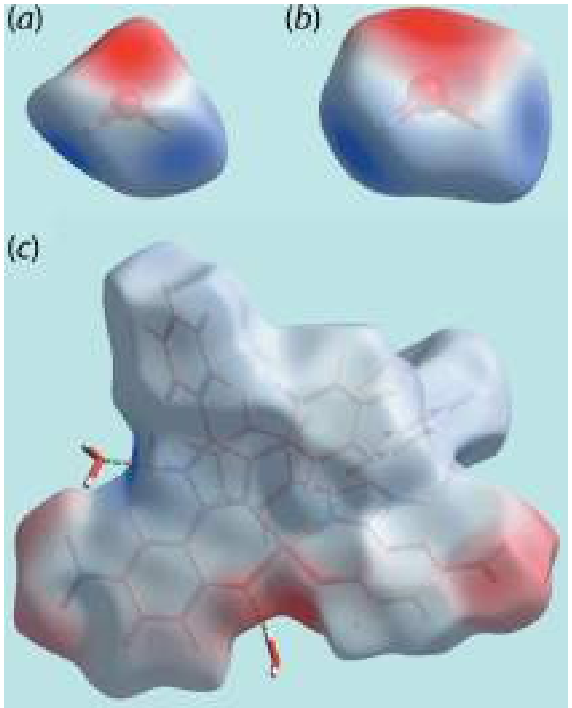


Figure 6
Different views of the Hirshfeld surfaces for the constituents of (I) mapped over the electrostatic potential (the red and blue regions represent negative and positive electrostatic potentials, respectively) for the (*a*) water-O1*W* molecule [in the range −0.1001 to +0.1943 atomic units (a.u.)], (*b*) water-O2*W* molecule (−0.1013 to +0.1751 a.u.) and (*c*) complex molecule (−0.1209 to +0.2076 a.u.). The hydrogen bonds involving water molecules in (*c*) are indicated by green dashed lines.

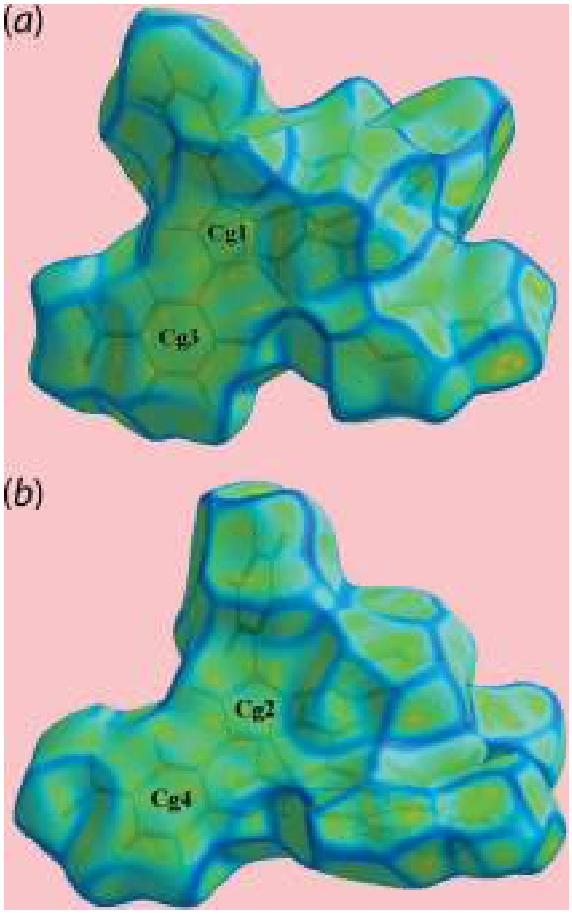


Figure 7
Two views of the Hirshfeld surface mapped over curvedness for the complex molecule in (I), highlighting flat regions enclosing symmetry-related imidazole and nitrobenzene rings involved in π–π stacking, labelled *Cg*1 and *Cg*3 for one pair of rings in (*a*), and *Cg*2 and *Cg*4 for the other pair in (*b*).

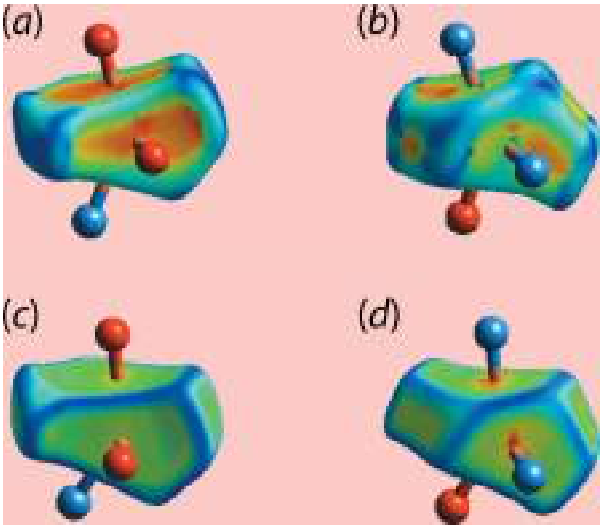


Figure 8
Different views of the Hirshfeld surfaces calculated for the copper(II) centre in (I) highlighting the coordination by the N_2O_4 donor set mapped over (*a*)/(*b*) shape-index in the range −1.0 to +1.0 arbitrary units and (*c*)/(*d*) curvedness in the range −4.0 to +0.4 arbitrary units.

Table 4
Percentage contributions of interatomic contacts to the Hirshfeld surface for the complex molecule in (I) and overall (I).

Contact	Percentage contribution	
	complex molecule	(I)
H···H	41.3	41.0
O···H/H···O	25.6	27.1
C···H/H···C	19.8	19.6
C···C	3.5	3.3
C···O/O···C	3.4	3.2
C···N/N···C	2.8	2.7
N···H/H···N	2.2	2.1
O···O	0.6	0.5
N···O/O···N	0.2	0.2
Cu···O/O···Cu	0.0	0.3
Cu···C/C···Cu	0.3	0.0

contribution from other interatomic contacts to the surface summarized in Table 4 have negligible influence on the molecular packing.

5. Database survey

There are five crystal structures of copper complexes with related 2-(4,5-diphenyl-1*H*-imidazol-2-yl)phenolate ligands in the literature [Cambridge Structural Database (CSD): Groom *et al.*, 2016]. The first of these is the 4-bromo derivative of (I), isolated as a dimethylformamide solvate [(II); CSD refcode YUKSOO] (Parween *et al.*, 2015). The remaining four structures are 2,4-(*t*-Bu)$_2$-phenolate derivatives, three of which are copper(II) complexes and the other, a copper(III) complex. Three of these four species have no additional substitution (Benisvy *et al.*, 2003). One was isolated as a methanol trisolvate [(III); JADZUK], another as a dimethylformamide tetrasolvate [(IV); NEPLAV01] and the third an oxidized species, *i.e.* a copper(III) complex, was isolated as a tetrafluoroborate salt/dichloromethane disolvate [(V); NEPLEZ01]; complex (IV) has crystallographic twofold symmetry. The final structure, a copper(II) complex (Benisvy *et al.*, 2006), has additional 4-methoxyphenyl substituents on the imidazol-

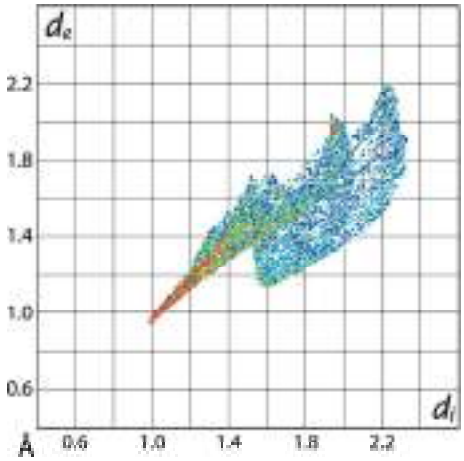


Figure 9
The two-dimensional fingerprint plot taking into account only the Hirshfeld surface calculated about the copper(II) atom.

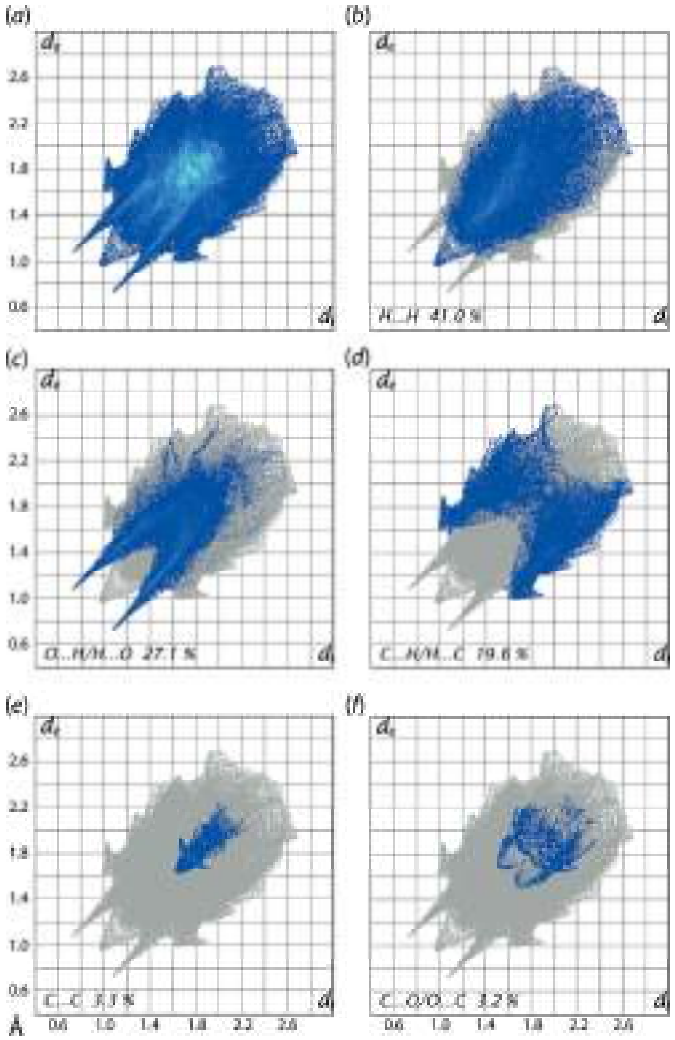


Figure 10
(*a*) A comparison of the full two-dimensional fingerprint plot for (I) and those delineated into (*b*) H···H, (*c*) O···H/H···O, (*d*) C···H/H···C, (*e*) C···C and (*f*) C···O/O···C contacts.

2-yl rings and was isolated as a methanol disolvate [(VI); JEBRUE]. The common feature of all the structures is the '*cis*'-N_2O_2 set but the coordination geometries are highly distorted, as seen in the sequence of τ_4 values for (I)–(VI) of 0.48, 0.53, 0.44, 0.37, 0.47 and 0.35, respectively.

6. Synthesis and crystallization

In a typical procedure, benzil (0.3 g, 1 mmol), ammonium acetate (0.19 g, 2.5 mmol), 2-hydroxy-5-nitrobenzalaldehyde (0.167 g, 1 mmol) and copper(II) borate (0.218 mg, 1 mmol) were ground in an agate mortar with a pestle. To this mixture, about 1.5 g of dried silica gel (column chromatography, 60–120 mesh) was added and the reaction mixture was ground again for 30 min. The whole reaction mixture was then transferred to a 100 ml round-bottomed flask and heated at 130 °C with constant stirring for 4 h. The reaction mixture was then extracted with dry acetone and dried over $MgSO_4$. After a few days, a dark-brown solid was obtained. The product was recrystallized from dry dimethylformamide and, after 5 d, light-blue crystals of (I) were obtained (yield 60%; m.p. > 300 °C).

Table 5
Experimental details.

Crystal data	
Chemical formula	$[Cu(C_{21}H_{14}N_3O_3)_2]\cdot 2H_2O$
M_r	812.27
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	100
a, b, c (Å)	13.2752 (2), 25.1602 (4), 11.1166 (2)
β (°)	104.256 (1)
V (Å^3)	3598.68 (10)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	1.42
Crystal size (mm)	0.14 × 0.11 × 0.07
Data collection	
Diffractometer	XtaLAB Synergy, Dualflex, AtlasS2
Absorption correction	Gaussian (*CrysAlis PRO*; Rigaku OD, 2018)
T_{min}, T_{max}	0.757, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	46023, 7490, 6420
R_{int}	0.058
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.631
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.047, 0.128, 1.05
No. of reflections	7490
No. of parameters	532
No. of restraints	8
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.61, −0.74

Computer programs: *CrysAlis PRO* (Rigaku OD, 2018), *SHELXS* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*), *ORTEP-3 for Windows* (Farrugia, 2012), *DIAMOND* (Brandenburg, 2006) and *publCIF* (Westrip, 2010).

7. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 5. Carbon-bound H-atoms were placed in calculated positions (C—H = 0.95 Å) and were included in the refinement in the riding-model approximation, with U_{iso}(H) values set at 1.2U_{eq}(C). The O- and N-bound H atoms were located in a difference Fourier map but were refined with distance restraints of O—H = 0.84 ± 0.01 Å and N—H = 0.88 ± 0.01 Å, respectively, and with U_{iso}(H) set at 1.5U_{eq}(O) or 1.2U_{eq}(N).

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