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Investigation of supramolecular synthons in the crystals of N-(aryl)-succinamic acids and N-(aryl)-maleamic acids: A case study of 4-oxo-4-(pyridin-2-ylamino)butanoic acid

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ABSTRACT

4-Oxo-4-(pyridin-2-ylamino)butanoic acid crystallizes with two symmetry independent molecules (A and B) in the asymmetric unit. The crystal structure features hetero $R_2^2(8)$ synthon comprising of N–H···O = C(acid) and O–H···$N_{pyridyl}$ hydrogen bonds between the molecules A and B resulting in one-dimensional (1D) helical columns running down the a axis which are further stabilized by C–H···O = C(acid) interactions. The C–H···O = C(amide) interactions characterized by $R_2^2(14)$ rings between the molecules of the neighbouring channels result in a 1D architecture. Hirshfeld surface analysis comprising of d_{norm} surface and 2D Fingerprint plot (FP) analyses revealed that the maximum contribution to the Hirshfeld surfaces of A and B are from H···H contacts followed by O···H/H···O contacts. In the crystal structure of the reported N-(aryl)-succinamic/maleamic acids, the most repetitive supramolecular synthons are either amide…acid C(7) chains or a combination

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of acid...acid $R_2^2(8)$ homodimers and amide...amide C(4) chains, which are very different from the one observed in the present structure.

Specifications Table

Subject area	*X-ray crystallography*
Compounds	*4-oxo-4-(pyridin-2-ylamino)butanoic acid or N-(pyridin-2-yl)-succinamic acid*
Data category	*Crystallographic data*
Data acquisition format	*CIF for crystallography*
Data type	*Analyzed*
Procedure	*A rod-like single crystals of 4-oxo-4-(pyridin-2-ylamino)butanoic acid dimensions (in mm) 0.22 × 0.19 × 0.17 obtained by slow solvent evaporation technique using ethanol was mounted on a Bruker Proteum2 CCD diffractometer equipped with an X-ray generator operating at 45 kV and 10 mA, using Cu-Kα radiation of wavelength 1.54178 Å. Data were collected for 24 frames per set with different settings of φ (0° and 90°), keeping the scan width of 0.5°, exposure time of 5 s, the sample-to-detector distance of 45.10 mm, and 2θ value at 64.4°. The diffraction data was collected at 296 K.*
Data accessibility	*Crystallographic data for the structures reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC-1,811,797. URL:* https://summary.ccdc.cam.ac.uk/structure-summary-form

1. Rationale

It is reported that various substituted succinamic acids exhibit dual antitumor and anti-inflammatory activities [1-4]. Further, these class of compounds serve as starting materials for the synthesis of N, N' -diarylsuccinamides; a class of compound known to exhibit varied biologically activities such as antimycobacterial [5], antialgal [5] and antitumor [6]. Identification of robust supramolecular synthons in the solid state is a decisive step in crystal engineering [7]. A rational approach to design novel solid-state structures is to gather a prior knowledge of the supramolecular synthons of a particular hydrogen bond motif present in a series of compounds and modifications brought about in them by small chemical modifications [7]. In this view, the study of the crystal structure of 4-oxo-4-(pyridin-2-ylamino)butanoic acid i.e., N-(pyridin-2yl)-succinamic acid is very appropriate as it offers the possibility of formation of different hydrogen bonds having different patterns owing to the presence of multiple hydrogen bond acceptors and donors in the molecule.

2. Procedure

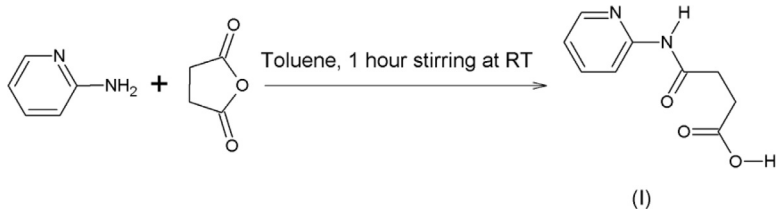
2.1. Synthesis

To a hot solution of succinic anhydride (0.0025 moles, 0.25 g) in toluene (10 ml) was added dropwise a hot solution of 2-aminopyridine (0.0025 moles, 0.235 g) also in toluene (15 ml) with constant stirring (Scheme 1). Solid of (I) separated out of the solution immediately. Stirring was continued for about one hour. The solid was separated by filtration and was later treated with dilute hydrochloric acid (10 ml). The solid of (I) was filtered under suction and washed thoroughly with water to remove the unreacted succinic anhydride and succinic acid. The crude product obtained (0.33 g, yield = 68.0%) was recrystallized to constant melting point from ethanol (M.P. = 453 K).

Single crystals of (I) suitable for single crystal X-ray studies were obtained from slow solvent evaporation technique at room temperature using ethanol as the solvent.

2.2. X-ray crystallographic study

The X-ray intensity data were collected at temperature of 296 K on a Bruker Proteum2 CCD diffractometer equipped with an X-ray generator operating at 45 kV and 10 mA, using Cu-Kα radiation of



(I)

Scheme 1. Synthesis of 4-oxo-4-(pyridin-2-ylamino)butanoic acid.

Table 1
Crystal data and structure refinement parameters for the title compound.

CCDC deposition number	1,811,797
Empirical formula	$C_9H_{10}N_2O_3$
Formula weight	194.19
Temperature/K	296(2)
Radiation	CuKα ($\lambda = 1.54178$)
Crystal system	triclinic
Space group	*P*-1
a/Å	5.0762(6)
b/Å	13.1162(17)
c/Å	13.8245(17)
α/°	88.724(8)
β/°	89.581(8)
γ/°	88.313(8)
Volume/Å^3	919.8(2)
Z/Z'	4/2
ρ_{calc}/g cm^{-3}	1.402
μ/mm^{-1}	0.902
Crystal size/mm^3	0.25 × 0.22 × 0.19
2θ range/°	6.394 to 128.81
Reflections collected	8786
Data[I ≥ 2σ(I)]/ restraints/parameters; R_{int}	3011/4/269
R_1; wR_2 [I ≥ 2σ(I)]	$R_1 = 0.0517$, $wR_2 = 0.1457$
R_1; wR_2[all data]	$R_1 = 0.0726$, $wR_2 = 0.1646$
Largest diff. peak/hole/e Å^{-3}	0.21/−0.24

wavelength 1.54178 Å. Data were collected for 24 frames per set with different settings of φ (0° and 90°), keeping the scan width of 0.5°, exposure time of 5 s, the sample-to-detector distance of 45.10 mm, and 2θ value at 64.4°. Image processing and data reduction were done using SAINT-Plus and XPREP [8]. The structure was solved by direct methods using SHELXS-97 [9]. All the non-hydrogen atoms were revealed in the first-difference Fourier map itself and were refined anisotropically. All the hydrogen atoms were positioned geometrically. In All carbon bound H atoms were positioned geometrically, with C–H = 0.93 Å for aromatic H, and C–H = 0.97 Å for methylene H, and refined using a riding model with $U_{iso}(H) = 1.2U_{eq}(C)$ for aromatic as well as methylene H atoms. The nitrogen and oxygen bound H atoms were located in a difference map and were refined isotropically with the bond length restraint N–H = 0.86(2) Å and O–H = 0.85(2) Å. All the geometrical calculations were carried out using the program *PLATON* [10] within the WinGX suite [11]. The molecular and packing diagrams were generated using the software *MERCURY* [12]. The crystallographic data and refinement parameters are summarized in Table 1.

2.3. Hirshfeld surface calculations

Hirshfeld surface analyses were carried out and finger print plots were plotted using the software CrystalExplorer 3.0 [13]. The d_{norm} plots were mapped with colour scale in between −0.18 au (blue) and 1.4 au (red). The 2D fingerprint plots [14] were displayed by using the expanded 0.6–2.8 Å view with the d_e and d_i distance scales displayed on the graph axes. When the cif file of (I) was uploaded

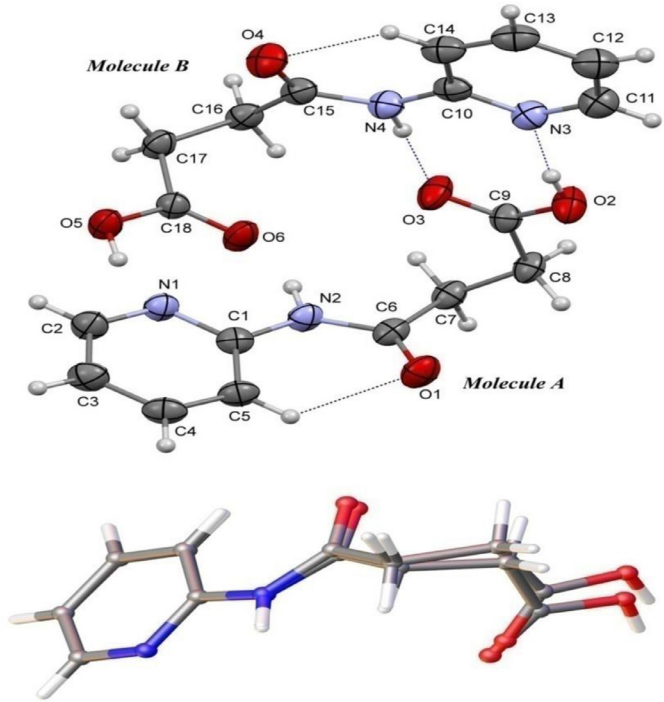


Fig. 1. a) An ORTEP view of the title compound drawn at 50% ellipsoidal probability. Dotted lines depict intramolecular interactions. (b) An overlay of the A and B molecules.

into the CrystalExplorer software, all bond lengths to hydrogen were automatically modified to typical standard neutron values i.e., C–H = 1.083 Å and N–H = 1.009 Å.

3. Data, value and validation

3.1. Results

3.1.1. Molecular conformation

The *ORTEP* view of the compound is shown in Fig. 1. Crystallographic data of the compound is presented in Table 1, bond lengths and angles are listed in Table 2 whereas, hydrogen bond geometries are listed in Table 3. The compound crystallizes in the triclinic system and *P*-1 space group, with two molecules in the asymmetric unit (A and B). The two symmetry independent molecules slightly differ in the conformation of the side chains as described in the overlay of the two molecules in Fig. 1. The side chain of the molecule is bent at the C7 and C16 atoms respectively in Mol. A and B, the dihedral angles between the C1-N2-C6(O1)-C7-C8 and the C8-C9-O2(O3) segment in A being 79.79(2)º, and, between C10-N4-C15(O4)-C16-C17 and the C17-C18-O5(O6) segment in B being 81.57(2)º. Further, the pyridyl rings and the -C1-N2-C6(O1)-C7-/-C10-N4-C15(O4)-C16- amide segments in A and B are almost planar with dihedral angles between the two being 9.95(1)º in A and 5.19° in B. In both A and B, the O–H bond of the –COOH group points in the direction opposite to the -CH_2- segment of the side chain, and, the conformation of both the C = O bonds are anti to the H atoms of their adjacent -CH_2- groups. Also, the C = O and O–H bonds of the acid group are in syn position to each other. The molecular conformations in both A and B are stabilized by intramolecular interactions; C5-H5···O1 in A and C14-H14···O4 in B, both of which close into S(6) loops [15]. Contrary to this, 4-oxo-4-(pyridin-3-ylamino)butanoic acid [16], a positional isomer of the title compound crystallizes in the monoclinic crystal system [$P2_1/n$ space group] with a single molecule in the asymmetric unit (Fig. 1). The overlay

Table 2
Bond lengths, angles and torsions in the title compound.

C1—N1	1.343 (3)	C10—C14	1.396 (4)
C1—C5	1.389 (3)	C11—N3	1.335 (3)
C1—N2	1.393 (3)	C11—C12	1.377 (4)
C2—N1	1.339 (3)	C12—C13	1.380 (4)
C2—C3	1.377 (4)	C13—C14	1.365 (4)
C3—C4	1.377 (4)	C15—O4	1.222 (3)
C4—C5	1.371 (4)	C15—N4	1.367 (3)
C6—O1	1.222 (3)	C15—C16	1.510 (4)
C6—N2	1.360 (3)	C16—C17	1.517 (3)
C6—C7	1.504 (3)	C17—C18	1.490 (3)
C7—C8	1.518 (3)	C18—O6	1.208 (3)
C8—C9	1.499 (3)	C18—O5	1.323 (3)
C9—O3	1.205 (3)	C10—N3	1.341 (3)
C9—O2	1.315 (3)	C10—N4	1.389 (3)
N1—C1—C5	121.9 (2)	C13—C12—C11	117.2 (3)
N1—C1—N2	113.2 (2)	C14—C13—C12	120.6 (3)
C5—C1—N2	124.8 (2)	C13—C14—C10	118.7 (3)
N1—C2—C3	123.0 (3)	O4—C15—N4	123.4 (3)
C4—C3—C2	118.3 (3)	O4—C15—C16	121.9 (2)
C5—C4—C3	119.8 (2)	N4—C15—C16	114.7 (2)
C4—C5—C1	118.8 (2)	C15—C16—C17	110.7 (2)
O1—C6—N2	124.1 (2)	C18—C17—C16	111.1 (2)
O1—C6—C7	121.8 (2)	O6—C18—O5	122.5 (2)
N2—C6—C7	114.1 (2)	O6—C18—C17	122.8 (2)
C6—C7—C8	111.53 (19)	O5—C18—C17	114.7 (2)
C9—C8—C7	112.08 (19)	C2—N1—C1	118.2 (2)
O3—C9—O2	122.9 (2)	C6—N2—C1	129.0 (2)
O3—C9—C8	122.8 (2)	C11—N3—C10	118.5 (2)
O2—C9—C8	114.2 (2)	C15—N4—C10	129.0 (2)
N3—C10—N4	113.5 (2)	N4—C10—C14	125.2 (2)
N3—C10—C14	121.3 (3)	N3—C11—C12	123.7 (3)
N1—C2—C3—C4	−0.1 (4)	C15—C16—C17—C18	−67.3 (3)
C2—C3—C4—C5	−0.5 (4)	C16—C17—C18—O6	−15.8 (4)
C3—C4—C5—C1	1.1 (4)	C16—C17—C18—O5	165.3 (2)
N1—C1—C5—C4	−1.1 (4)	C3—C2—N1—C1	0.1 (4)
N2—C1—C5—C4	179.3 (2)	C5—C1—N1—C2	0.5 (4)
O1—C6—C7—C8	−45.6 (3)	N2—C1—N1—C2	−179.8 (2)
N2—C6—C7—C8	135.2 (2)	O1—C6—N2—C1	3.3 (4)
C6—C7—C8—C9	−63.0 (3)	C7—C6—N2—C1	−177.5 (2)
C7—C8—C9—O3	−15.2 (4)	N1—C1—N2—C6	168.0 (2)
C7—C8—C9—O2	164.5 (2)	C5—C1—N2—C6	−12.4 (4)
N3—C11—C12—C13	0.6 (4)	C12—C11—N3—C10	0.5 (4)
C11—C12—C13—C14	−0.9 (4)	N4—C10—N3—C11	179.0 (2)
C12—C13—C14—C10	0.0 (4)	C14—C10—N3—C11	−1.5 (4)
N3—C10—C14—C13	1.2 (4)	O4—C15—N4—C10	1.3 (4)
N4—C10—C14—C13	−179.3 (2)	C16—C15—N4—C10	−179.4 (2)
O4—C15—C16—C17	−40.9 (3)	N3—C10—N4—C15	173.7 (2)
N4—C15—C16—C17	139.8 (2)	C14—C10—N4—C15	−5.9 (4)

of the two isomers shown in Fig. 2 indicates the major differences observed in the conformations of the side chains especially at the alkyl carbon atoms adjacent to the amide carbonyl carbons in the two molecules. Also, the O–H bond in the positional isomer is anti to the carboxylic C = O bond which is very contrary to that observed in the title compound (Fig. 1).

3.1.2. Crystal structure

The crystal structure of the title compound features alternating hetero $R_2^2(8)$ synthons comprising of N–H···O = C(–OH) and (O = C)–O–H···$N_{pyridyl}$ hydrogen bonds [O2–HO2···N3 + N4–HN4···O3 and N2–HN2…O6 + O5–HO5···N1] between the molecules A and B resulting in 1D helical column running down the a axis (Fig. 3a and b, Table 3). The channels can be also viewed as $C_2^2(18)$ and

Table 3
Geometric parameters for hydrogen-bonds and other intermolecular contacts (Å, º) operating in the crystal structure of the title compound.

D–H…A	D-H	H…A	D…A	D-H…A
O2–HO2···N3	0.88	1.81	2.6845	171
O5–HO5···N1[i]	0.87	1.83	2.7025	175
N2–HN2···O6[ii]	0.86	1.98	2.8299	171
N4–HN4···O3	0.90	1.97	2.8657	175
C4–H4···O1[iii]	0.93	2.52	3.3716	152
C7–H7A···O6[ii]	0.97	2.57	3.4193	146
C5–H5···O1[#]	0.93	2.35	2.9130	119
C14–H14···O4[#]	0.93	2.32	2.8963	120

[i] 1 + x,y,z
[ii] 1 + x, y, z
[iii] 1 − x, − y, 1 − z
[#] Intra

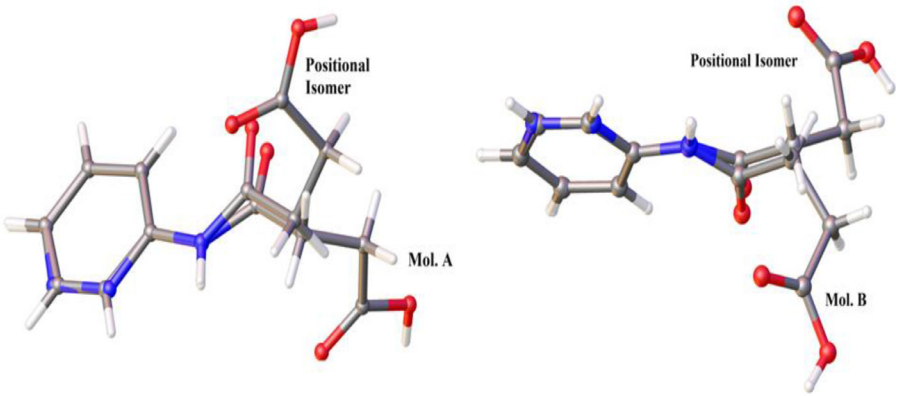


Fig. 2. An overlay of Mol. A and B of (I) with the reported positional isomer.

$C_2^2(14)$ [15] hydrogen bonded chains running down the a axis. The 1D column is stabilized by bifurcating C–H···O = C(–OH) interactions viz C7-H7A···O6 between the A and B molecules, bifurcated at the acceptor oxygen atom (Fig. 3b, Table 3). Finally, C4-H4···O1 interactions characterized by $R_2^2(14)$ rings between the molecules of the neighbouring channels result a 1D architecture.

3.1.3. Hirshfeld surface analysis

The Hirshfeld surface analysis comprising d_{norm} surfaces and 2D fingerprint plots (FP) were generated for both A and B molecules in order to verify the various intermolecular interactions and to provide quantitative data for the relative contributions to the surfaces. In the d_{norm} surface of A (Fig. 4a and b), dark red spots are observed close to pyridine N1 atom, N2 bound H atom, O3 atom and O2 bound H atoms arising from strong O5–HO5···N1, N4–HN4···O3, O2–HO2···N3 and N2–HN2···O6 hydrogen bonds, while, a faint red spot close to H7A atom is due to the weaker C7–H7A···O_6 interactions. Similarly, the red spots in the proximity of O1 and H4 atoms (Fig. 4b) in the d_{norm} surface of A is evident for the ring motif generated by C4-H4···O1 interactions. Similarly, the dark red spots near N3 atom, N4 bound H atom, O5 bound H atom and O6 atom in the d_{norm} surface of B molecule are due to the strong N–H···O and O–H···N hydrogen bonds while, a faint red spot near O6 atom is due to the bifurcated C7-H7A···O6 interactions.

The quantitative analysis of the hydrogen bonds/intermolecular interactions is made by analyzing the FP of A and B molecules (Fig. 5). The FP of both the molecules features two pair of long, sharp spikes characteristic of strong hydrogen bonds. Sharp spikes at $d_i + d_e \approx 1.8$ and 2.0 Å respectively in

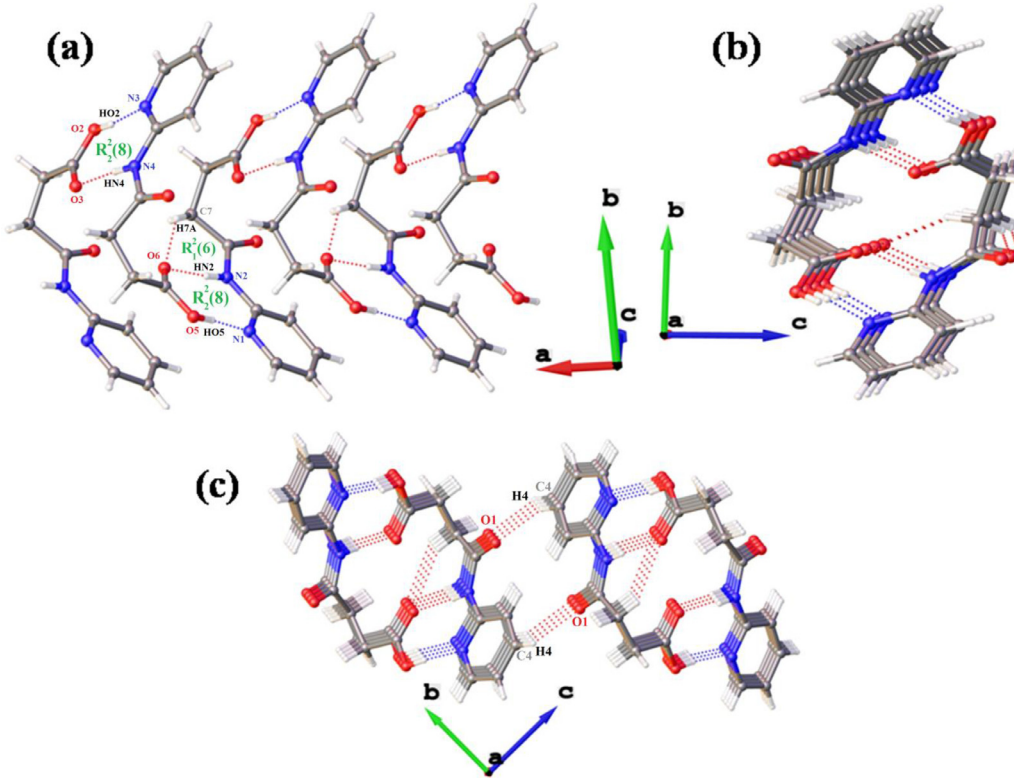


Fig. 3. Views of the crystal packing formed via N–H···O, O–H···N and C–H···O interactions.

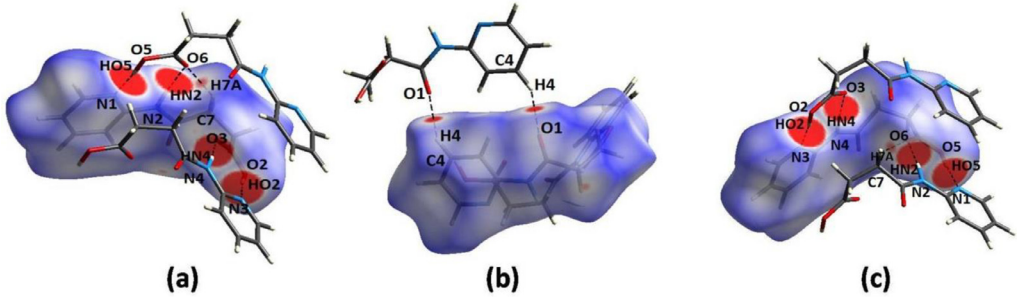


Fig. 4. d_{norm} surfaces of Molecule A (4a and 4b) and Molecule B (4c) of the title compound.

the N···H/H···N and H···O/O···H FP of A and B are very close to the H···A distances for the different O–H···N and N–H···O hydrogen bonds observed in the crystal structure (Table 3).

The FP for different atom…atom contacts and the percentage contribution of each contacts to the Hirshfeld surfaces in both A and B reveals that in each molecules the contribution to the Hirshfeld surfaces from different contacts is almost similar (Fig. 5). In both, the highest contribution is from H···H contacts: 38.9% in A and 41.7% in B. This is followed by O···H/H···O contacts arising from N–H···O hydrogen bonds and C–H···O interactions contributing 30.4% in A and 27.1% in B. The H···C/C···H contacts contribute 13.2% and 13.1% respectively in A and B while, H···N/N···H contacts arising due to O–H···N hydrogen bonds contribute 9.9% and 9.2% to the d_{norm} surfaces of A and B respectively.

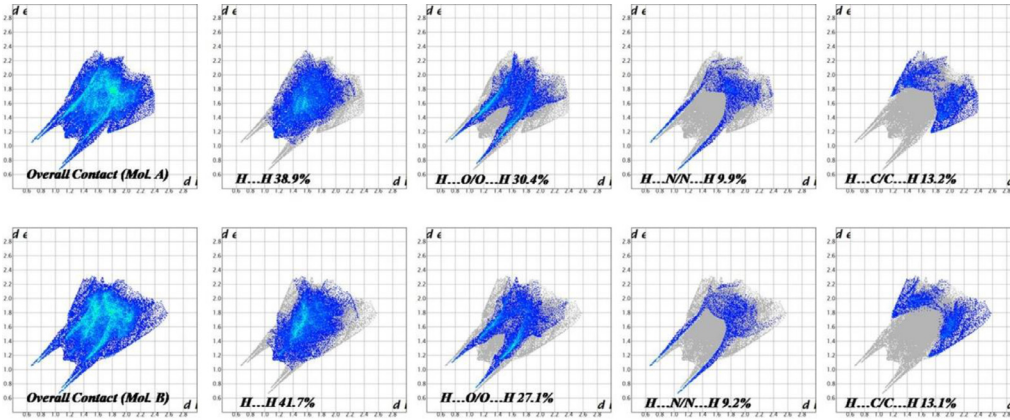
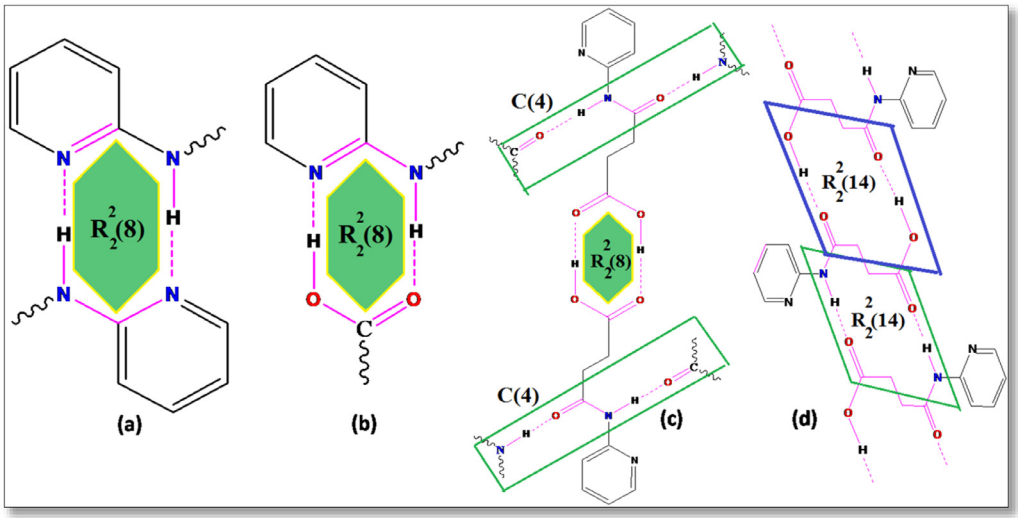


Fig. 5. FP of overall and individual atom…atom contacts in Molecule A (top) and B (below).

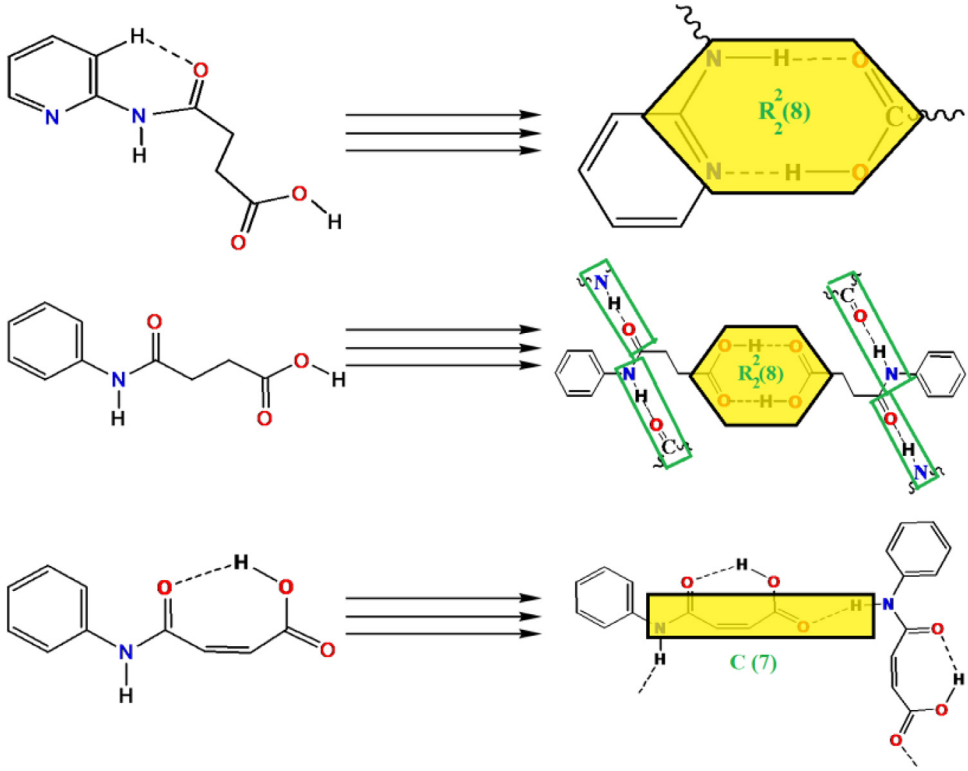


Scheme 2. Supramolecular synthons constructed via different homo- and heteromeric hydrogen bonds possible in 4-oxo-4-(pyridin-2-ylamino)butanoic acid.

3.2. Discussion

Recently, we had reported the detailed analysis of the crystal structure of the positional isomer of (I) viz., 4-oxo-4-(pyridin-3-ylamino)butanoic acid [16]. Also, seventeen closely related N-(phenyl/mono/di-substitutedphenyl)-succinamic acids [17–33] and nineteen N-(phenyl/mono/di-substitutedphenyl)-maleamic acids [34–52] have been reported. A comparison of the crystal structures of these closely related compounds and, a study of how minor chemical modifications [such as substituent effects] would affect the nature and pattern of different supramolecular synthons would be very useful to design new solid state structures with desired properties.

Some of the possible supramolecular aggregations are collectively described in Scheme 2. In motif-a, the supramolecular aggregation is via amide…2-AMP (2-AMP = 2-Aminopyridine) homomeric interactions comprising of N–H···N hydrogen bonds forming $R_2^2(8)$ network [7]. In motif-b, a hetero $R_2^2(8)$ dimeric motif is formed via acid…2-AMP and amide…acid interaction involving >C=O–H···N



Scheme 3. Supramolecular synthon observed in the crystal structure of the title compound and the most repetitive synthons observed in N-(aryl)-succinamic and maleamic acids.

and N–H···O = C(–OH) hydrogen bonds [7]. Motif-c is a 1D ribbon constructed via acid…acid interaction forming O–H···O hydrogen bonded $R_2^2(8)$ motif which is further linked via amide…amide interactions involving N–H···O = C hydrogen bonded C4 chains. Motif-d consists of two homomeric dimers constructed via two acid…amide interactions comprising N–H···O = C(–OH) and O–H…O = C(-NH) hydrogen bonds forming fused $R_2^2(14)R_2^2(14)$ ribbon. In the title compound, motif-b is observed, while, in the positional isomer [16] none of the above pattern is observed. The crystal structure features O–H···$N_{pyridyl}$ and amide N–H···O hydrogen bonded chains.

The supramolecular synthon and the molecular aggregation observed in the reported N-(aryl)-succinamic acids and N-(aryl)-maleamic acids is very different than that observed in the title compound. In the reported N-(aryl)-succinamic acids [17-33], the most reproducible pattern is the $R_2^2(8)$ homosynthon consisting of carboxylic acid dimers (O = C)-O–H···O = C(-OH) which are further extended into 1D ribbons via C(4) chains of amide N–H···O = C hydrogen bonds i.e., motif-c. In the crystals of 13 out of the 17 reported N-(aryl)-succinamic acids, namely, N-(phenyl)-succinamic acid [17] and N-(2–chloro/3–chloro/4–chloro/2-methyl/3-methyl/4-methyl/2,3-dimethyl/2,6-dimethyl/3,4-dimethyl/2,5-dichloro/2,6-dichloro/2,4,6-trimethylphenyl)-succinamic acids [18-29], motif-c is observed. In N-(3,5-dimethyl/3,5-dichlorophenyl)-succinamic acids [30,31], motif-d is observed while, in the monohydrates of N-(2,5-dimethyl/3,4-dichlorophenyl)-succinamic acids [32,33], pair of amide N–H···O hydrogen bonded C(4) chains are interconnected by fused $R_4^4(12)R_4^2(8)$ tetramers consisting of O_{acid}-H.. O_{water}-H···O_{acid} hydrogen bonds forming 1D ribbons. Thus, it can be generalized that motif-c is the robust and most repetitive supramolecular synthon appearing in the crystal structures of N-(aryl)-succinamic acids.

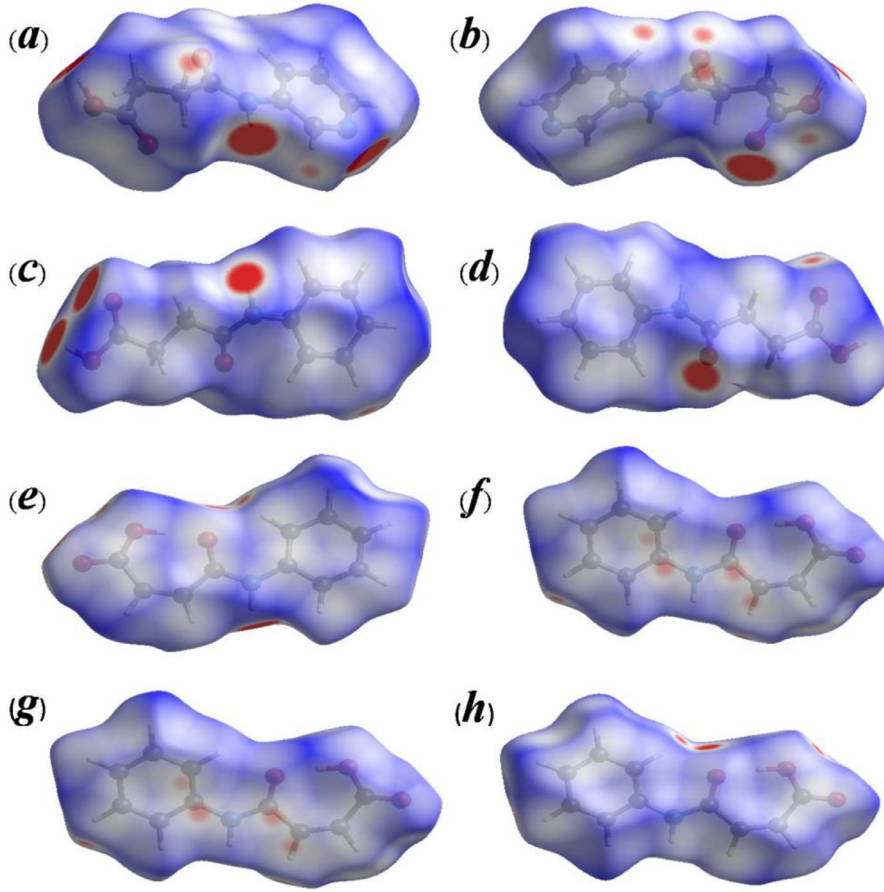


Fig. 6. Two views of d_{norm} surfaces of reference compounds: 4-oxo-4-(pyridin-3-ylamino)butanoic acid (a and b); N-phenyl succinamic acid (c and d); N-phenyl maleamic acid (Mol. 1 and 2, e-h).

Nineteen N-(aryl)-maleamic acids [34–52] are reported in literature. The crystal structure of sixteen compounds, namely, N-phenyl-maleamic acid [34] and N-(3–chloro/2-methyl/4-methyl/3-nitro/4-nitro/4-acetyl/4–methoxy/2,6-dimethyl/3,4-dimethyl/2,5-dichloro/3,4-dichloro/3,5-dichloro/4–chloro–2methyl/2,4,6-trimethyl/2,4,5-trichloro/2,4,6-trichlorophenyl)-maleamic acids [35–50] features C(7) chains of N–H···O = C(-OH) hydrogen bonds forming a 1D architecture (Scheme-3). However, the crystal structure of N-(2-aminophenyl)-maleamic acid [51] features N_{amide}-H···N_{amino} hydrogen bonded $R_2^2(8)$ dimers which are extended into three dimension via $N_{aniline}$–···O = C(-OH) hydrogen bonds and C–H···O interactions. In the crystal structure of the reported monohydrate of N-(2–chloro–4-nitrophenyl)-maleamic acid [52], amide N–H···O = C and O_{water}–H···O = C(-OH) hydrogen bonds link the molecules into a three-dimensional network. The molecular conformation of all the reported maleamic acids is stabilized by an intramolecular O–H···O = Camide hydrogen bond which closes into an S(7) loop [15].

The Hirshfeld surface analysis was performed on the reported positional isomer [16] and the parent compounds of the two series i.e., N-phenyl succinamic acid [17] and N-phenyl maleamic acid [34]. The d_{norm} surfaces of the related compounds are shown in Fig. 6 and FP in Fig. 7. The d_{norm} surfaces and FP of these compounds are very different from each other and from that of the title compound. This indicates the difference in the nature of the intermolecular interactions and differ-

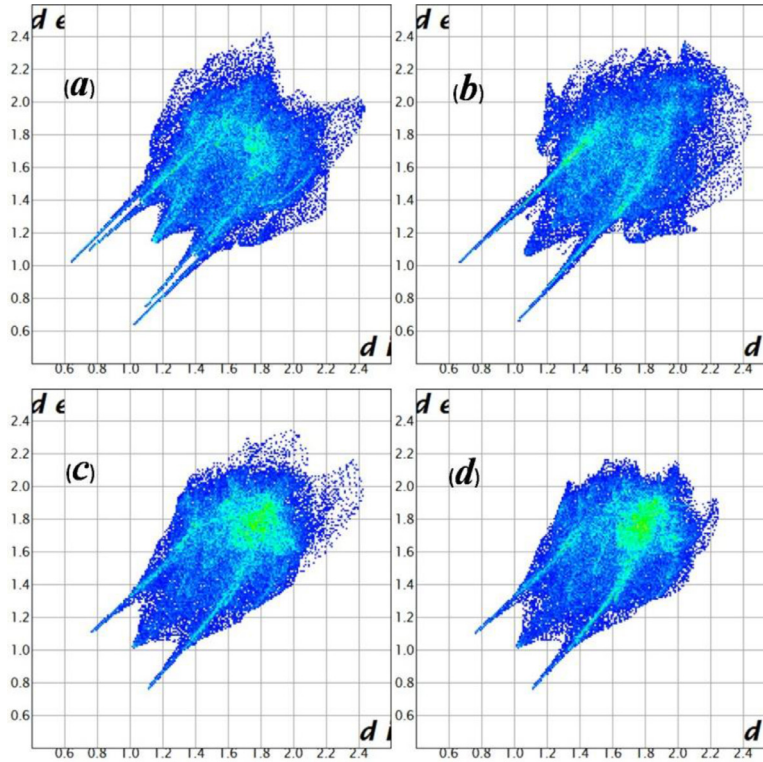


Fig. 7. FP of overall contacts of reference compounds: 4-oxo-4-(pyridin-3-ylamino)butanoic acid (a); N-phenyl succinamic acid (b); N-phenyl maleamic acid (Mol. 1 and 2, c and d).

ence in the supramolecular synthons existing in these structures. The FP analysis of the positional isomer [16] shows that the relative contribution of different atom…atom contacts to the surface is almost same as that in the title compound. The H···C/C···H contacts contributes 19.1% to the Hirshfeld surface of N-phenyl succinamic acid [17], which is slightly high than that observed in the title compound. This is due to the additional C–H···π interactions exhibited in N-phenyl succinamic acid (Fig. 7) [17]. Similarly, C···C contacts in the two molecules of N-phenyl maleamic acid [34] contribute 9.8 and 10.2% respectively to the Hirshfeld surface, which is very high compared to that observed in other compounds (0.5 – 1.2%). These high values observed can be attributed to the $\pi\cdots\pi$ interactions between the molecules in the crystal structure of N-phenyl maleamic acid [34].

From the above observations the following conclusions can be drawn:

1) The crystal structures of N-(aryl)-succinamic and maleamic acids are not largely influenced by the nature and position of the substituents present on the aromatic ring. Putative hydrogen bonding patterns are observed in each series which differ from one another [Scheme-3].
2) The molecular conformations of the sidechains of all the maleamic acids are stabilized by an intramolecular O–H···O hydrogen bond while, there are no intramolecular hydrogen bonding in succinamic acids.
3) The crystal structure of the title compound and its positional isomer [16] [where an aryl ring is replaced by a pyridyl ring] are very different from that of the reported succinamic and maleamic acids. The N_{amide}–H···O_{acid} and O–H_{acid}···$N_{pyridyl}$ hydrogen bonding in the title compound and N–H···N hydrogen bonding in the positional isomer is preferred over the putative hydrogen patterns observed in succinamic and maleamic acid series. Also, the molecular structure of the title compound is stabilized by an intramolecular hydrogen bond which is not observed in any of the

reported succinamic acids. Further, the nature of the intramolecular hydrogen bonds is very different from those present in the maleamic acids [Scheme 3].

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi: 10.1016/j.cdc.2018.02.002.

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