

Crystal Structures of Two Salts from Piperidine, Naphthalene-1,5-Disulfonic Acid, and 4-Nitrophthalic Acid

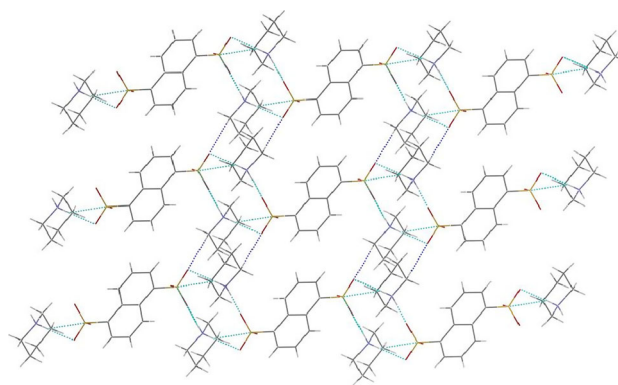
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Abstract Two anhydrous salts of piperidine have been prepared with naphthalene-1,5-disulfonic acid, and 4-nitrophthalic acid by slow evaporation technique, and their structures were fully characterized. These two structures adopted homo or hetero supramolecular synthons. Compound **1** crystallizes in the triclinic, space group P-1, with $a = 7.728(3)$ Å, $b = 8.042(3)$ Å, $c = 9.150(3)$ Å, $\alpha = 80.631(4)^\circ$, $\beta = 79.433(4)^\circ$, $\gamma = 86.531(4)^\circ$, $V = 551.3(3)$ Å³, $Z = 1$. Compound **2** crystallizes in the monoclinic, space group P2(1)/c, with $a = 6.3466(5)$ Å, $b = 24.008(2)$ Å, $c = 9.0390(7)$ Å, $\alpha = 90^\circ$, $\beta = 97.0050(10)^\circ$, $\gamma = 90^\circ$, $V = 1367.01(19)$ Å³, $Z = 4$. Analysis of the crystal packing of **1**, **2** suggests that there are N–H...O hydrogen bonds between the acids and the piperidine in both compounds. Except the classical hydrogen bonds, the secondary propagating interactions including CH₂...O, O...C_π, and CH₂...π interactions also play important roles in structure extension. In conclusion, we have shown that 3D structures can be constructed by collective non-covalent interactions.

Graphical Abstract The crystal structures of the salts from piperidine, naphthalene-1,5-disulfonic acid, and 4-nitrophthalic acid are predominantly stabilized by

classical hydrogen bonds as well as CH₂...O, O...C_π, and CH₂...π interactions.



Keywords Crystal structures · Non-covalent interactions · Piperidine · Organic acids

Introduction

A productive strategy in the crystal engineering is to build supramolecular structures from molecules that are programmed to engage in multiple interactions such as electrostatic forces, hydrogen bonding, cation...π interaction, C–H...π, and π...π interactions [1–6]. Hydrogen bonding system is the main key for the organization of supramolecules between organic acid and organic base containing the hydrogen bonding sites [7, 8]. Intramolecular, and intermolecular hydrogen bonds are responsible for the construction of several complicated self-assembled structures involving N-containing compounds and acidic components [9, 10].

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Through hydrogen bonds cocrystals and organic salts can be generated. In pharmaceuticals, salt formation is often used in order to modify the properties of the compounds [11], such as increasing or decreasing solubility, improving stability and reducing hygroscopicity of a drug product. There are many interesting hydrogen bonded topological structures from infinite 1-D chain to 3-D supramolecular framework [12, 13]. The organic acid bears an important hydrogen bonding functional group for crystal engineering [14–17]. It is interesting to exploit the robust and directional recognition of organic acids with the N-containing basic building blocks [18–25]. In this regard, the most frequently used moieties with hydrogen bonding capability are amine (including aromatic amine and aliphatic amine), and pyridyl derivatives.

Compared with pyridine derivatives, piperidine, besides the NH group, bears the flexible ring with CH₂ units, which can provide more complex nonbonding interactions when it interacts with the carboxylic acids. To the best of our knowledge there are very few reports involving acid–base adducts concerning the piperidine [26–29].

As an extension of our study of the weak interactions (hydrogen bonding, π – π interaction, and halogen bonding) concerning the aromatic N-containing derivatives [30, 31], herein we report the preparation and structures of two organic salts assembled from piperidine (L) and the corresponding organic acids (Scheme 1), respectively. The two organic salts are (piperidine)₂: (naphthalene-1,5-disulfonic acid) [(HL)₂²⁺ · (nds²⁻), nds²⁻ = naphthalene-1,5-disulfonate] (1), and (piperidine): (4-nitrophthalic acid) [(HL) · (Hnpa), Hnpa = hydrogen 4-nitrophthalate] (2).

Experimental Section

Materials and Physical Measurements

The chemicals and solvents used in this work are of analytical grade and available commercially and were used

without further purification. The FT-IR spectra were recorded from KBr pellets in range 4000–400 cm⁻¹ on a Mattson Alpha-Centauri spectrometer. Microanalytical (C, H, N, and S) data were obtained with a Perkin-Elmer Model 2400II elemental analyzer. Melting points of the new compounds were recorded on an XT-4 thermal apparatus without correction.

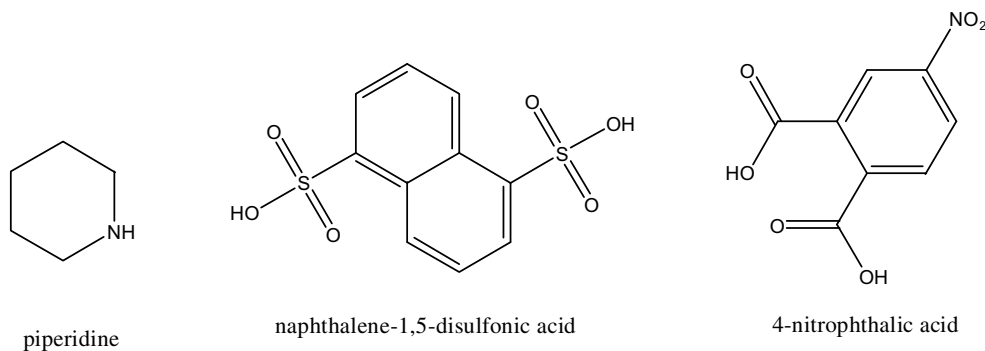
Preparation of the Salts 1–2

(Piperidine)₂: (Naphthalene-1,5-Disulfonic Acid) [(HL)₂²⁺ · (nds²⁻)] (1)

A solution of naphthalene-1,5-disulfonic acid tetrahydrate (36 mg, 0.1 mmol) in methanol (15 ml) was added dropwise to a vigorously stirred solution of piperidine (8.5 mg, 0.1 mmol) in methanol (3 ml) over a period of 5 min. The solution was further stirred for 10 min, then the solution was filtered into a test tube. The solution was left standing at room temperature for several days. Colorless block crystals were isolated after slow evaporation of the methanol solution in air. The crystals were collected and dried in air to give the compound [(HL)₂²⁺ · (nds²⁻)] (1). (yield: 36.0 mg, 78.50 %). mp 184–186 °C. Elemental analysis: Calc. for C₂₀H₃₀N₂O₆S₂ (458.58): C, 52.34; H, 6.54; N, 6.10; S, 13.96. Found: C, 52.28; H, 6.49; N, 6.02; S, 13.88. Infrared spectrum (cm⁻¹): 3389 s(ν_{as} (NH)), 3270 s(ν_s (NH)), 3156, 3090, 2985, 2930, 2852, 1614, 1570, 1523, 1475, 1430, 1386, 1341, 1398, 1255, 1213, 1172, 1128, 1086, 1043, 1001, 958, 914, 870, 827, 784, 743, 697, 655, 630, 612 m.

(Piperidine): (4-Nitrophthalic Acid) [(HL) · (Hnpa)] (2)

A solution of 4-nitrophthalic acid (21.1 mg, 0.1 mmol) in methanol (14 ml) was added dropwise to a vigorously stirred solution of piperidine (8.5 mg, 0.1 mmol) in methanol (3 ml) over a period of 5 min. The solution was further stirred for 10 min, then the solution was filtered into



Scheme 1 Hydrogen bond tectons discussed in this paper

a test tube. The solution was left standing at room temperature for several days. Colorless block crystals were isolated after slow evaporation of the methanol solution in air. The crystals were collected and dried in air to give the compound [(HL) · (Hnpa)] (**2**). (yield: 26.0 mg, 87.75 %). mp 192–193 °C. Elemental analysis: Calc. for C₁₃H₁₆N₂O₆ (296.28): C, 52.65; H, 5.40; N, 9.45. Found: C, 52.60; H, 5.32; N, 9.37. Infrared spectrum (cm⁻¹): 3704 s(v(OH)), 3386 s(v_{as}(NH)), 3268 s(v_s(NH)), 3176, 3130, 3084, 2925, 2853 m, 1674 s(v(C=O)), 1618 s(v_{as}(COO⁻)), 1570 m, 1529 s(v_{as}(NO₂)), 1486 m, 1443 m, 1398 s(v_s(COO⁻)), 1353 m, 1315 s(v_s(NO₂)), 1270 s(v(C–O)), 1225, 1178, 1135, 1089, 1044, 972, 933, 887, 844, 799, 757, 712, 668, 625, 601 m.

X-ray Crystallography and Data Collection

The crystals were mounted on a glass fiber on a Bruker SMART 1000 CCD diffractometer operating at 50 kV and 40 mA using Mo K α radiation (0.71073 Å). Data collection and reduction were performed using the SMART and SAINT software [32]. The structures were solved by direct methods, and the non-hydrogen atoms were subjected to anisotropic refinement by full-matrix least squares on F^2 using SHELXTL package [33].

Hydrogen atom positions for the two structures were located in a difference map and refined independently. Further details of the structural analysis are summarized in Table 1. Selected bond lengths and angles for the compounds **1–2** are listed in Table 2, the relevant hydrogen bond parameters are provided in Table 3.

Results and Discussion

Preparation and General Characterization

The preparation of the compounds **1–2** were carried out with piperidine and the corresponding organic acids in 1:1 ratio in methanolic solution, which was allowed to evaporate at ambient conditions to give the final crystalline products. Both compounds are anhydrous organic salts. The two compounds are not hygroscopic. The molecular structures and their atom labelling schemes for the two structures are illustrated in Figs. 1, 3, respectively. The elemental analysis data for the two crystalline compounds are in good agreement with their compositions. The infrared spectra of the two compounds are consistent with their chemical formulas determined by elemental analysis and further confirmed by X-ray diffraction analysis.

The bands at approximately 3268–3704 cm⁻¹ in the IR spectra of the two compounds arise from the O–H or N–H

stretching frequencies. The medium intensity bands in the regions of 1500–1630 and 600–750 cm⁻¹ are attributed to aromatic ring stretching and bending, respectively. The salt **2** exhibits bands for both COO and COOH groups.

Structural Descriptions

X-ray Structure of (Piperidine)₂: (Naphthalene-1,5-Disulfonic Acid) [(HL)₂²⁺ · (nds)²⁻] (**1**)

Crystallization of piperidine and naphthalene-1,5-disulfonic acid in a 1:1 ratio from methanol gave single crystals suitable for X-ray diffraction. Structure determination (Table 1) revealed that piperidine and naphthalene-1,5-disulfonic acid are present in a 2:1 ratio in the molecular complex **1**. The crystal structure of **1** consists of one protonated L, and half a dianion of naphthalene-1,5-disulfonate in the asymmetric unit (Fig. 1). In the dianion, there exists an inversion centre located at the middle point of the C8–C8A bond. Compound **1** crystallizes in the triclinic space group P-1. The piperidine moiety is protonated at the ring nitrogen. The angle at the protonated ring N atom is 112.06(16)°. The C–N bond lengths in the ring are in the range of 1.489(3)–1.490(2) Å, and the C1–C2, C2–C3, C3–C4, and C4–C5 bond lengths are 1.499(3), 1.515(3), 1.516(3), and 1.507(3) Å, respectively. The piperidine ring adopts the chair conformation. The S–O bond distances in the SO₃⁻ range from 1.4342(16) to 1.4655(15) Å (Δ is 0.0313 Å) which are in the range of the deprotonated SO₃⁻ moiety [34].

The cations and the dianions form 2D sheet extending at the direction that makes an angle of ca. 60° with the *ab* plane (Fig. 2). Herein one H at the cation (NH₂⁺) functions as a double hydrogen bond donor with one O atom at the SO₃⁻ with N–O distance of 2.893(2) Å and the S at the same SO₃⁻ with N–S distance of 3.562(2) Å. The other H at the cation (NH₂⁺) also generates one N–H···O (N(1)–H(1B)···O(2)#3, 2.765(2) Å, 173.4°#3 $-x, -y, -z + 1$), and one N–H···S (N(1)–H(1B)···S(1)#3, 3.7999(19) Å, 161.5°) hydrogen bond with the anion. For the presence of the hydrogen bonds there exists the R₁²(3) ring according to Bernstein [35]. There also exists the CH₂–O (C(5)–H(5A)···O(1)#2, 143.7°, #2 $-x + 1, -y, -z + 1$) association at the sheet between the CH₂ moiety of the piperidine and the SO₃⁻ with C–O distance of 3.260(3) Å. The 2D sheets were further stacked along the direction that was perpendicular with its extending direction by the CH₂– π association between the CH₂ of the piperidine and the naphthalene ring of the anion with C–Cg (Cg is the gravity centre of the naphthalene ring) distance of 3.730(2) Å to form 3D layer network structure. Herein the neighboring sheets were slipped some distances from each other along its extending direction.

Table 1 Summary of X-ray crystallographic data for complexes **1–2**

	1	2
Formula	C ₂₀ H ₃₀ N ₂ O ₆ S ₂	C ₁₃ H ₁₆ N ₂ O ₆
<i>F</i> w	458.58	296.28
<i>T</i> (K)	298(2)	298(2)
Wavelength (Å)	0.71073	0.71073
Crystal system	Triclinic	Monoclinic
space group	P-1	P2(1)/c
<i>a</i> (Å)	7.728(3)	6.3466(5)
<i>b</i> (Å)	8.042(3)	24.008(2)
<i>c</i> (Å)	9.150(3)	9.0390(7)
α (°)	80.631(4)	90
β (°)	79.433(4)	97.0050(10)
γ (°)	86.531(4)	90
<i>V</i> (Å ³)	551.3(3)	1367.01(19)
<i>Z</i>	1	4
<i>D</i> _{calcd} (Mg/m ³)	1.381	1.440
Absorption coefficient (mm ⁻¹)	0.281	0.115
<i>F</i> (000)	244	624
Crystal size (mm ³)	0.45 × 0.35 × 0.28	0.42 × 0.28 × 0.20
θ range (°)	2.57–25.02	2.42–25.02
	–9 ≤ <i>h</i> ≤ 9	–7 ≤ <i>h</i> ≤ 7
Limiting indices	–9 ≤ <i>k</i> ≤ 9	–28 ≤ <i>k</i> ≤ 24
	–8 ≤ <i>l</i> ≤ 10	–7 ≤ <i>l</i> ≤ 10
Reflections collected	2745	6913
Reflections independent (<i>R</i> _{int})	1903 (0.0239)	2417 (0.0425)
Goodness-of-fit on <i>F</i> ²	1.058	0.936
<i>R</i> indices [<i>I</i> > 2σ <i>I</i>]	0.0352, 0.0875	0.0484, 0.1167
<i>R</i> indices (all data)	0.0431, 0.0949	0.0856, 0.1314
Largest diff. peak and hole e (Å ⁻³)	0.186, –0.279	0.372, –0.245

Table 2 Selected bond lengths (Å) and angles (°) for **1–2**

1			
N(1)–C(1)	1.489(3)	N(1)–C(5)	1.490(2)
O(1)–S(1)	1.4559(15)	O(2)–S(1)	1.4655(15)
O(3)–S(1)	1.4342(16)	S(1)–C(7)	1.7793(18)
C(1)–N(1)–C(5)	112.06(16)	O(3)–S(1)–O(1)	114.82(10)
O(3)–S(1)–O(2)	112.83(10)	O(1)–S(1)–O(2)	110.43(9)
2			
N(1)–C(5)	1.478(3)	N(1)–C(1)	1.484(3)
N(2)–C(11)	1.464(3)	O(1)–C(6)	1.222(3)
O(2)–C(6)	1.281(3)	O(3)–C(7)	1.281(3)
O(4)–C(7)	1.219(3)	C(5)–N(1)–C(1)	112.23(19)
O(1)–C(6)–O(2)	122.8(3)	O(4)–C(7)–O(3)	122.5(2)

X-ray Structure of (Piperidine): (4-Nitrophthalic Acid) [(HL) · (Hnpa)] (2)

The compound **2** of the composition [(HL) · (Hnpa)] was also prepared by reacting piperidine and 4-nitro-phthalic

acid with 1:1 ratio in a solution of methanol, in which only one proton of the carboxyl units was transferred to the ring N atom of the piperidine. Thus **2** can be classified as a hydrogenphthalate salt. In the asymmetric unit of **2** there existed one cation of piperidine, and one anion of 4-nitrohydrogenphthalate, as shown in Fig. 3. Salt **2** crystallizes as monoclinic block crystals in the space group P2(1)/c. Structure of **2** is example of a complex with many symmetry-independent components in the asymmetric unit cell (*Z* > 2). In the compound, there is one pair of ion pair with no solvent molecules. The same as the salt **1**, the piperidine ring also adopts the chair conformation.

In the cation, the angle (C(1)–N(1)–C(5)) around the protonated N atom is [112.23(19)°], which is similar to the corresponding angle in **1**. The C–N bond lengths in the piperidine ring are in the range of 1.478(3)–1.484(3) Å, and the C1–C2, C2–C3, C3–C4 and C4–C5 bond lengths are 1.498(3), 1.507(3), 1.511(4), and 1.495(3) Å, respectively. The bond lengths at the cation of **2** are similar to the corresponding values at the cation of salt **1** also.

Table 3 Hydrogen bond distances and angles in studied structures 1–2

D–H...A	d(D–H) (Å)	d(H...A) (Å)	d(D...A) (Å)	<(DHA) (°)
1				
C(5)–H(5A)...O(1)#2	0.97	2.43	3.260(3)	143.7
N(1)–H(1B)...S(1)#3	0.90	2.94	3.7999(19)	161.5
N(1)–H(1B)...O(2)#3	0.90	1.87	2.765(2)	173.4
N(1)–H(1A)...S(1)#4	0.90	2.76	3.562(2)	149.4
N(1)–H(1A)...O(1)#4	0.90	2.00	2.893(2)	173.6
2				
C(5)–H(5B)...O(2)	0.97	2.62	3.148(3)	114.6
C(2)–H(2A)...O(5)#1	0.97	2.60	3.444(3)	145.4
C(1)–H(1D)...O(6)#2	0.97	2.59	3.253(3)	125.8
O(3)–H(3)...O(2)	0.82	1.57	2.383(3)	168.7
N(1)–H(1B)...O(2)#3	0.90	2.53	3.242(3)	136.2
N(1)–H(1B)...O(1)#3	0.90	2.06	2.932(3)	164.4
N(1)–H(1A)...O(4)#4	0.90	2.01	2.854(2)	155.2

Symmetry transformations used to generate equivalent atoms for **1**: #2 $-x + 1, -y, -z + 1$; #3 $-x, -y, -z + 1$; #4 $x, y, z - 1$. Symmetry transformations used to generate equivalent atoms for **2**: #1 $x - 1, -y + 1/2, z + 1/2$; #2 $-x + 1, y + 1/2, -z + 1/2$; #3 $x, -y + 1/2, z - 1/2$; #4 $x - 1, y, z$

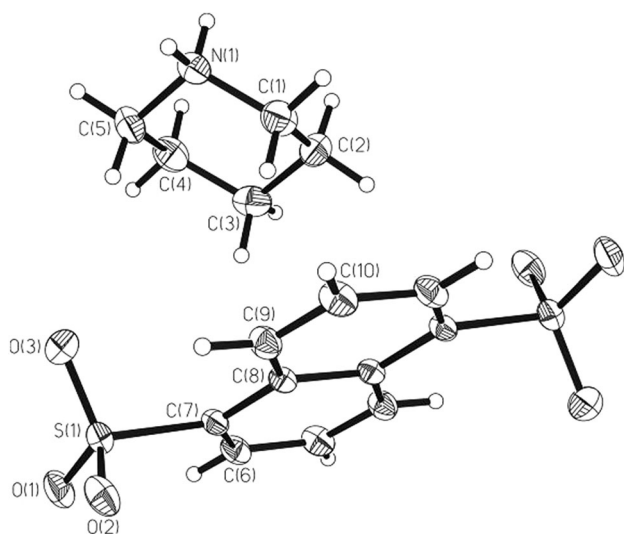


Fig. 1 Molecular structure of **1** showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 30 % probability level

The phenyl ring of the anion with atoms C8–C13 and the piperidine ring with atoms N1–C1–C2–C3–C4–C5 intersects at an angle of $56.4(2)^\circ$ with each other. The carboxylates O1–C6–O2, and O3–C7–O4 are rotated out of the phenyl plane by $19.1(1)^\circ$, and $28.2(1)^\circ$, respectively. The two carboxyl moieties intersect at an angle of $34.9(3)^\circ$ with each other, which is different from the reported adduct of phthalate [36]. The nitro group N6–O5–O6 deviates by $9.9(2)^\circ$ from the phenyl ring of the anion. The nitro group N6–O5–O6 make angles of $18.9(3)^\circ$ and $38.1(1)^\circ$ with the two carboxylates, respectively.

At the cation there is bonded an anion by the bifurcated N–H...O hydrogen bonds between the NH^+ and both O of the carboxylate of the anion with N–O distances of $2.932(3)$ – $3.242(3)$ Å. For the above mentioned bifurcated hydrogen bonds, there generates the $R_1^2(4)$ ring motifs between the anion and the cation. 4-nitrohydrogenphthalate is found the intramolecular O–H...O hydrogen bonds between the COOH and the COO^- with O–O distance of $2.383(3)$ Å, giving an $S_1^1(7)$ ring motif. The cation bonded with the anion are linked together by the CH_2 –O (C(1)–H(1D)...O(6)#2, 125.8° ; C(2)–H(2A)...O(5)#1, 145.4°) contacts between the CH_2 of the cation and the nitro group at the anion with C–O distances of $3.253(3)$ – $3.444(3)$ Å to form a 1D chain (Fig. 4). The 1D chains are connected together by the O– C_π contact between the O atom at the carboxylate and the π –C at the carboxyl group with O–C distance of $3.188(3)$ Å to form 2D sheet extending parallel to the *ac* plane. Herein, the O–C separation is slightly longer than the archived value of $3.019(4)$ Å [37]. The 2D sheets are further combined together by the N–H...O hydrogen bond between the NH^+ and the C=O at the COOH with N–O distance of $2.854(2)$ Å, and CH_2 –O contact (C(5)–H(5B)...O(2), 114.6°) between the CH_2 of the cation and the carboxylate group with C–O distance of $3.148(3)$ Å to form 3D ABAB layer network structure. The geometries of the C(5)–H(5B)...O(2) contact were comparable with those (with D...A distance of $3.154(3)$ Å, and <(DHA) of $115.0(3)^\circ$) found at the reported compound [38]. In this case the adjacent sheet layers are also slipped some distance from each other along the extending direction.

Fig. 2 2D sheet structure of **1** formed by the CH₂–O association extending at the direction that made an angle of ca. 60° with the *ab* plane, the CH₂–O associations were shown in *blue color* (Color figure online)

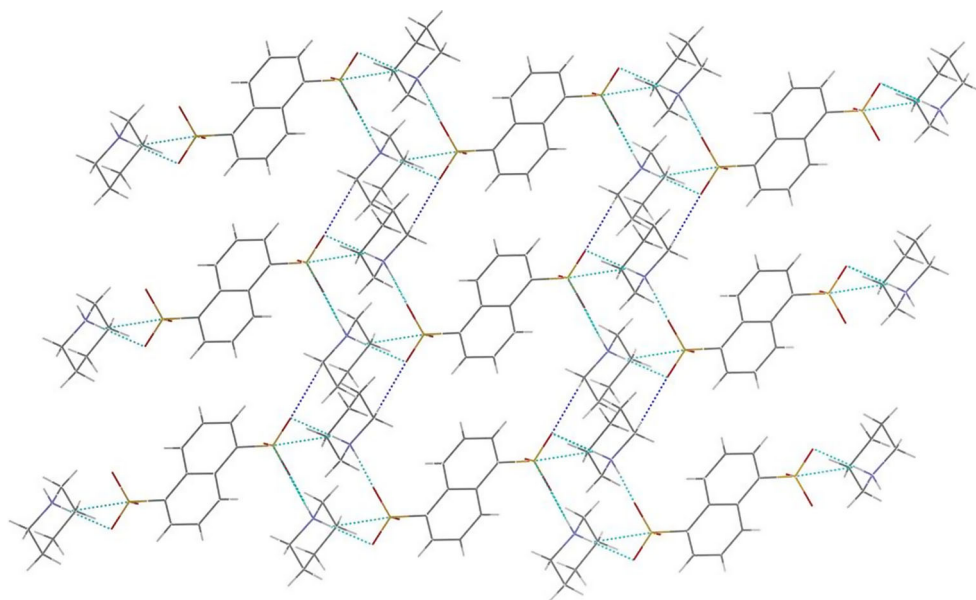


Fig. 3 Molecular structure of **2** showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 30 % probability level

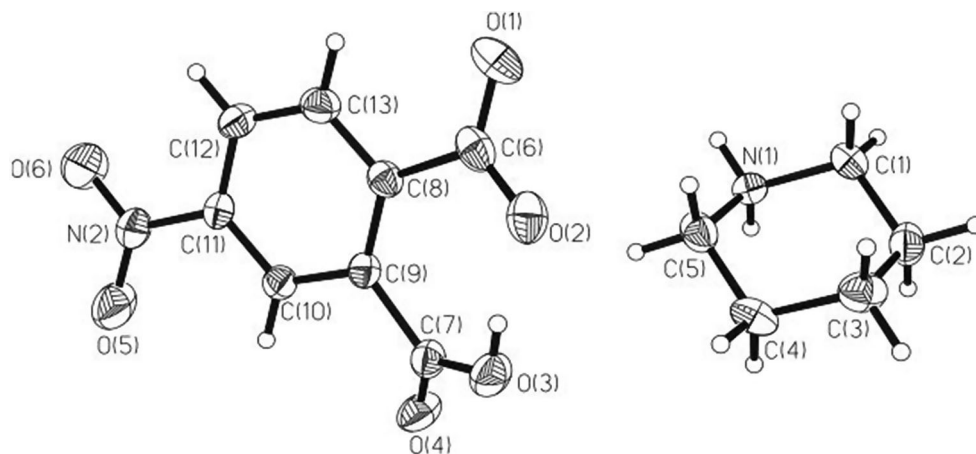
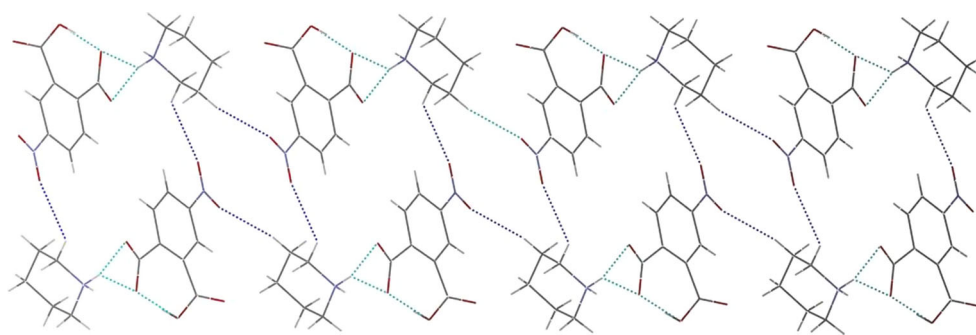


Fig. 4 The 1D chain structure of **2**, the CH₂–O associations were shown in *blue color* (Color figure online)



Supporting Information Available

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic data center, CCDC Nos. 898477 for **1**, and 1059210 for **2**. Copies of this information may be obtained free of charge from the

+44(1223)336-033 or Email: deposit@ccdc.cam.ac.uk or <http://www.ccdc.cam.ac.uk>.

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