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Molecular structures of five adducts assembled from p-dimethylaminobenzaldehyde and organic acids

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ABSTRACT

Five adducts **1**–**5** derived from p-dimethylaminobenzaldehyde have been prepared and characterized by X-ray diffraction analysis, IR, mp, and elemental analysis. Of the five adducts two are organic salts (**1**, and **2**) and the other three (**3**–**5**) are cocrystals. In salts **1**, and **2**, the L molecules are protonated.

The supramolecular architectures of the adducts **1**–**5** involve extensive intermolecular N–H···O, O–H···O, O–H···S, and C–H···O hydrogen bonds as well as other non-covalent interactions. The role of weak and strong non-covalent interactions in the crystal packing is ascertained. The complexes displayed 2D/3D framework structure for the synergistic effect of the various non-covalent interactions.

The results presented herein tell that the strength and directionality of the N–H···O, O–H···O, and O–H···S hydrogen bonds between organic acids and p-dimethylaminobenzaldehyde are sufficient to bring about the formation of binary cocrystals or organic salts.

1. Introduction

Crystal engineering using multiple interactions, such as electrostatic forces, hydrogen bonding, cation···π interaction, C–H···π, and π···π interactions, is a constantly growing field of research [1]. Multiple hydrogen-bond interactions are widely used in the design of self-assembled structures from organic acid and organic base [2]. The intramolecular, and intermolecular hydrogen bonds are responsible for the construction of several complicated self-assembled structures involving N-containing compounds and acidic components [3,4].

Organic acids represent one of the most prevalent functional groups in crystal engineering because they possess a hydrogen bond donor and acceptor with a geometry that facilitates self-association through supramolecular homosynthons *via* centrosymmetric dimer or catemer [5–7]. Furthermore, it is now recognized that organic acids are nice candidates for multicomponent crystals since they form persistent supramolecular heterosynthons with a number of different complementary functional groups such as amine, and aromatic nitrogen etc. For instance, much has been said about the use of organic acid and pyridyl groups in the design of supramolecular systems [8–13]. Besides the acidic group, the functional groups such as CH_3, NO_2, and phenol OH groups are all good groups in forming organic solids through non-covalent interactions [14]. As an amine derivative, besides the N group, p-dimethylaminobenzaldehyde contains the CH_3, –CHO, and phenyl groups, which can provide more complex nonbonding interactions when it interacted with the acidic compounds. To the best of our knowledge, there were few reports of the organic adducts involving p-dimethylaminobenzaldehyde and organic acids [15].

In order to understand the interaction modes p-dimethylaminobenzaldehyde exhibits in binding with the organic acids, we began to study the p-dimethylaminobenzaldehyde-acids system, also aiming to find the role the weak non-covalent interactions played in forming the final supramolecular frameworks. Thus, in the following, we report the preparation and crystal structures of five adducts assembled *via* nonbonding interactions between organic acids and p-dimethylaminobenzaldehyde (L) (Scheme 1). In this study, the five adducts are p-dimethylaminobenzaldehyde: (picric acid) $[(HL^+)\cdots(pic^-)]$ **(1)**, p-dimethylaminobenzaldehyde: (p-toluenesulfonic acid): (H_2O) $[(HL^+)\cdot(tsa^-)\cdots H_2O]$ (**2**), p-

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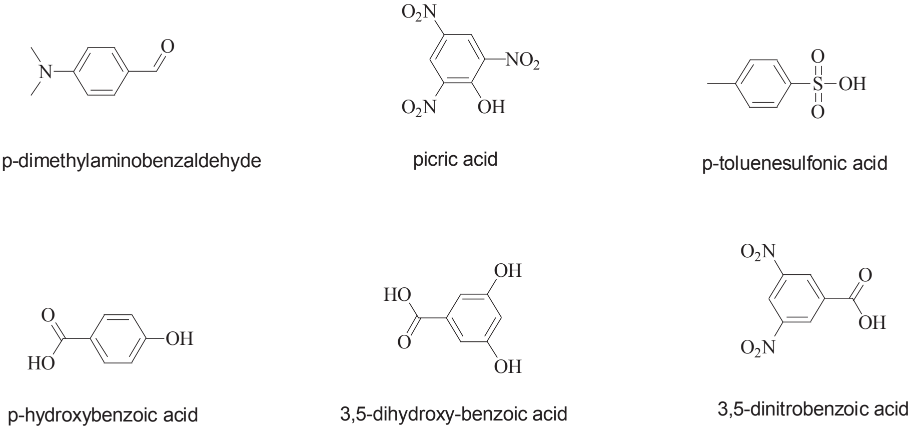


Scheme 1. The building blocks discussed in this paper.

dimethylaminobenzaldehyde: (p-hydroxybenzoic acid) [(L)···(phba)] **(3)**, p-dimethylaminobenzaldehyde: (3,5-dihydroxybenzoic acid)$_2$: $(H_2O)_2$ [(L)···(dhba)$_2$···$(H_2O)_2$] (**4**), and p-dimethylaminobenzaldehyde: (3,5-dinitrobenzoic acid) [(L)···(Hdba), Hdba = 3,5-dinitrobenzoic acid] (**5**), respectively (Scheme 2).

2. Experimental section

2.1. Materials and methods

The chemicals and solvents used in this work were of analytical grade and available commercially and were used without further purification. The C, H, N, and S microanalysis were carried out with a Carlo Erba 1106 elemental analyzer. The FT-IR spectra were recorded from KBr pellets in range 4000–400 cm^{-1} on a Mattson Alpha-Centauri spectrometer. Melting points of new compounds were recorded on an XT-4 thermal apparatus without correction.

2.2. Preparation of supramolecular compounds

2.2.1. p-dimethylaminobenzaldehyde: (picric acid) [$(HL^+)\cdot(pic^-)$] *(1)*

p-dimethylaminobenzaldehyde (14.9 mg, 0.1 mmol) was dissolved in 2 mL methanol. To this solution was added picric acid (22.9 mg, 0.1 mmol) in 20 mL methanol. Yellow block crystals were afforded after several days by slow evaporation of the solvent (yield: 28.0 mg, 74.02%, based on L). mp 91–92 °C. Elemental analysis: Calc. for $C_{15}H_{14}N_4O_8$(378.30): C, 47.58; H, 3.70; N, 14.80. Found: C, 47.54; H, 3.68; N, 14.76. Infrared spectrum (KBr disc, cm^{-1}): 3377s(ν_{as}(NH)), 3269s(ν_s(NH)), 3122m, 3071m, 2981m, 2968m, 2800m, 1753s, 1627m, 1584m, 1533s($\nu_{as}(NO_2)$), 1488m, 1445m, 1402m, 1361m, 1318s($\nu_{as}(NO_2)$), 1272m, 1227m, 1177m, 1133m, 1091m, 1048m, 1003m, 955w, 912m, 867w, 824m, 775w, 730m, 684w, 643m, 626w, 602m.

2.2.2. p-dimethylaminobenzaldehyde: (p-toluenesulfonic acid): (H_2O) [$(HL^+)\cdot(tsa^-)\cdot H_2O$] *(2)*

p-dimethylaminobenzaldehyde (14.9 mg, 0.1 mmol) was dissolved in 2 mL methanol. To this solution was added p-toluenesulfonic acid (19.0 mg, 0.1 mmol) in 6 mL ethanol. Colorless block crystals were afforded after several days by slow evaporation of the solvent (yield: 27.0 mg, 79.55%, based on L). mp 82–83 °C. Elemental analysis: Calc. for $C_{16}H_{21}NO_5S$ (339.40): C, 56.57; H, 6.19; N, 4.12; S, 9.43. Found: C, 56.54; H, 6.15; N, 4.08; S, 9.41. Infrared spectrum (KBr disc, cm^{-1}): 3712w(br, ν(OH)), 3391s(ν_{as}(NH)), 3282s(ν_s(NH)), 3055m, 2972m, 2924m, 2876m, 1692s, 1604m, 1578m, 1531m, 1486m, 1441m, 1400m, 1360m, 1318m, 1276m, 1234m, 1189m, 1068m, 1023m, 979w, 934m, 891w, 848m, 800m, 757m, 714m, 666w, 631m, 618w.

2.2.3. p-dimethylaminobenzaldehyde: (p-hydroxybenzoic acid) [(L)·(phba)] *(3)*

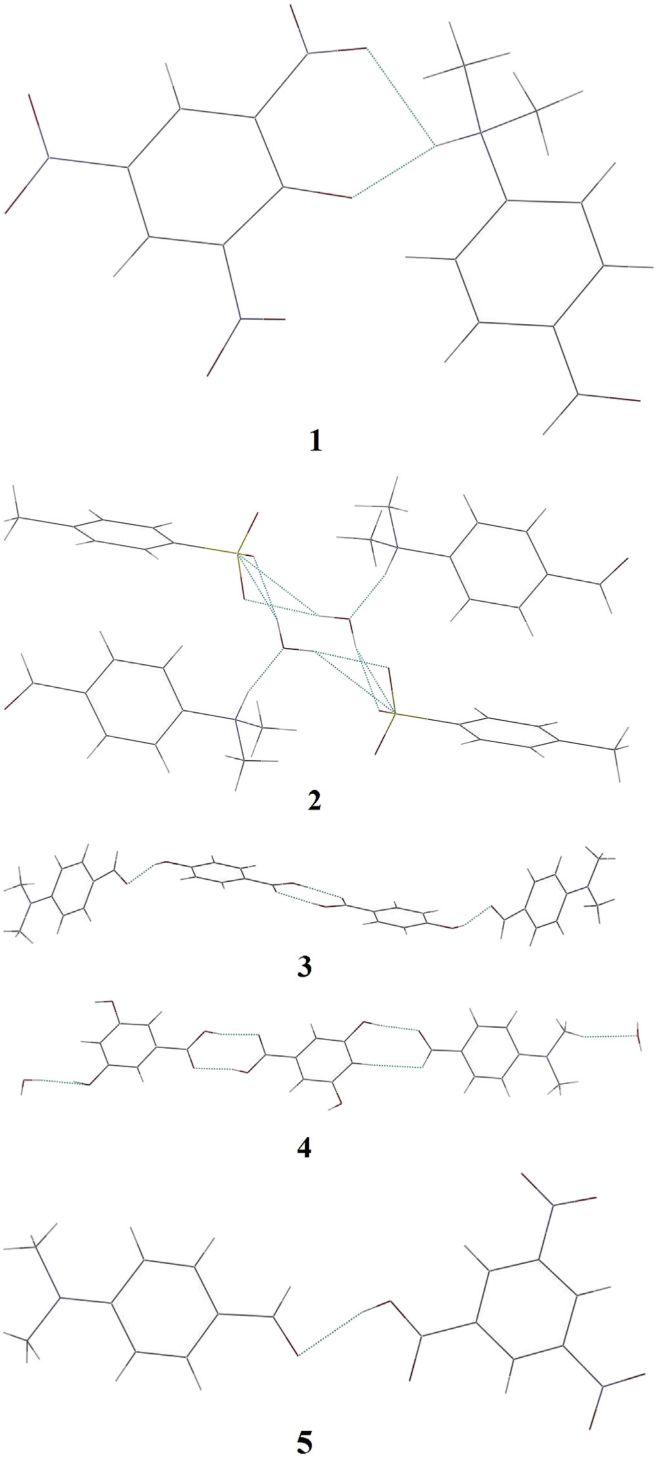
p-dimethylaminobenzaldehyde (14.9 mg, 0.1 mmol) was dissolved in 2 mL methanol. To this solution was added p-hydroxybenzoic acid (14.9 mg, 0.1 mmol) in 8 mL ethanol. Colorless block crystals were afforded after several days by slow evaporation of the solvent (yield: 22 mg, 76.57%, based on L). mp 154–156 °C. Elemental analysis: Calc. for $C_{16}H_{17}NO_4$ (287.31): C, 66.83; H, 5.92; N, 4.87. Found: C, 66.78; H, 5.89; N, 4.84. Infrared spectrum (KBr disc, cm^{-1}): 3671w(br, ν(OH)), 3129m, 3046m, 2991m, 2916m, 2879m, 1687s, 1638s(ν(C=O)), 1595m, 1550m, 1508m, 1465m, 1424m, 1378m, 1333m, 1290s(ν(C–O)), 1247m, 1203m, 1159m, 1117m, 1076m, 1033m, 990m, 948m, 905m, 862m, 820m, 779m, 732m, 691m, 646m, 622m, 600m.

2.2.4. p-dimethylaminobenzaldehyde: (3,5-dihydroxybenzoic acid)$_2$: $(H_2O)_2$ [(L)·(dhba)$_2$·$(H_2O)_2$] *(4)*

p-dimethylaminobenzaldehyde (14.9 mg, 0.1 mmol) was dissolved in 2 mL methanol. To this solution was added 3,5-dihydroxybenzoic acid (15.4 mg, 0.1 mmol) in 10 mL ethanol. Colorless prisms were afforded after several days of slow evaporation of the solvent, yield: 34 mg, 68.90%. mp 140–142 °C. Elemental analysis: Calc. for $C_{23}H_{27}NO_{11}$ (493.46): C, 55.93; H, 5.47; N, 2.84. Found: C, 55.88; H, 5.45; N, 2.79. Infrared spectrum (KBr disc, cm^{-1}): 3630s(ν(OH)), 2963s, 2855m, 1706s, 1652s(ν(C=O)), 1599m, 1547m, 1501m, 1459m, 1418m, 1376m, 1338m, 1296s(ν(C–O)), 1253m, 1210m, 1168m, 1124m, 1082m, 1039m, 994m, 952m, 899m, 855m, 813m, 768m, 725m, 684m, 652m, 617m.

2.2.5. p-dimethylaminobenzaldehyde: (3,5-dinitrobenzoic acid) [(L)·(Hdba), Hdba = 3,5-dinitrobenzoic acid] (5)

p-dimethylaminobenzaldehyde (14.9 mg, 0.1 mmol) was dissolved in 2 mL methanol. To this solution was added 3,5-dinitrobenzoic acid (21.2 mg, 0.1 mmol) in 10 mL ethanol. Colorless block crystals were afforded after several days by slow evaporation of the solvent (yield: 26 mg, 71.96%, based on L). mp 124–125 °C. Elemental analysis: Calc. for $C_{16}H_{15}N_3O_7$ (361.31): C, 53.14; H, 4.15; N, 11.62. Found: C, 53.11; H, 4.09; N, 11.58. Infrared spectrum (KBr disc, cm^{-1}): 3678ν(OH), 3112m, 3064m, 2960m, 2881m, 1722s, 1661(ν_{as}(C=O)), 1616m, 1602m, 1526s($\nu_{as}(NO_2)$), 1458m, 1415m, 1373m, 1332s($\nu_s(NO_2)$), 1289(ν_s(C–O)), 1245m,



Scheme 2. The five compounds described in this paper, **1**–**5**.

1203m, 1160m, 1117m, 1076m, 1034m, 993m, 945m, 900m, 856m, 814m, 763m, 722m, 678m, 634m, 601m.

2.3. X-ray crystallography

Suitable crystals were performed on a Bruker SMART 1000 CCD diffractometer using Mo Kα radiation ($\lambda = 0.71073$ Å). Data collections and reductions were performed using the SMART and SAINT software [16,17]. 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 [18].

Hydrogen atom positions for all structures were located in difference Fourier maps but were allowed to ride on the parent atoms at geometrically determined positions with O—H = 0.82–0.85 Å, N—H = 0.91 Å, C—H = 0.93 Å (aldehyde group; formyl H—C=O and aromatic) or 0.96 Å (methyl) and with $U_{\text{iso}} = 1.2U_{\text{eq(C,N)}}$ and $U_{\text{iso}} = 1.5U_{\text{eq(O)}}$.

Further details of the structural analysis are summarized in Table 1. Selected bond lengths and angles for the compounds **1**–**5** are listed in Table 2; the relevant hydrogen bond parameters are provided in Table 3.

3. Results and discussion

3.1. Syntheses and general characterization

p-dimethylaminobenzaldehyde has good solubility in common organic solvents, such as CH_3OH, C_2H_5OH, CH_3CN, $CHCl_3$, and CH_2Cl_2. For the preparation of **1**–**5**, the acids were mixed directly with the p-dimethylaminobenzaldehyde in methanol and/or ethanol solvents in 1:1 ratio, which was allowed to evaporate at ambient conditions to give the final crystalline products. The molecular structures and their atom labelling schemes for the five structures are shown in Figs. 1, 3, 5, 7 and 9, respectively.

The elemental analyses for the five compounds are in good agreement with their compositions. The infrared spectra of **1**–**5** are consistent with their chemical formulas determined by elemental analysis and further confirmed by X-ray diffraction analysis. The very strong and broad features at 3678–3269 cm^{-1} arise from O—H or N—H stretching frequencies. Aromatic ring stretching and bending are in the regions of 1500–1630 cm^{-1} and 600–750 cm^{-1}, respectively. Compounds **3**, **4**, and **5** display strong IR peaks for the COOH groups. The bands at 1526 and 1332 cm^{-1} were attributed to the $\nu_{as}(NO_2)$ and $\nu_s(NO_2)$, respectively [18].

3.1.1. X-ray structure of p-dimethylaminobenzaldehyde: (picric acid) [(HL$^+$)·(pic$^-$)] (1)

Compound **1** was prepared by reacting of a methanol solution of p-dimethylaminobenzaldehyde and picric acid in 1: 1 ratio, which crystallizes as monoclinic pale yellow crystals in the space group P2(1)/c. In the crystal structure of the picrate salt [(HL$^+$)···(pic$^-$)] **(1)**, the asymmetric unit contains one cation, and one anion. The structure of **1** with the atom numbering scheme is shown in Fig. 1. This is a salt in which the —OH groups of the picric acid molecules are ionized by proton transfer to the nitrogen atoms [N(1)] of the p-dimethylaminobenzaldehyde moieties, which is also confirmed by the bond distances of O(1)-C(10) [1.257(2) Å] for the phenolates comparing with the expected values of 1.24 ± 0.01 Å [19]. The bond length of the O(8)-C(1) (1.208(3) Å) displays characteristic bond for the aldehyde group. The angles involving the protonated N are 110.77(18)–113.20(18)°, which are smaller than the corresponding values found at the crystal of L [20].

The rms deviation of the phenyl ring of the cation is 0.0022 Å. The rms deviation of the picrate ring defined by C10—C15 is 0.0046 Å. Both rings form a dihedral angle of 6.2(2)° with each other. The *ortho*-nitro groups of the anion (N2—O2—O3 and N4—O6—O7) are rotated slightly out of the benzene plane of the anions and form dihedral angles of 33.9(2)°, and 19.2(1)°, respectively with the benzene plane, these values being similar to the corresponding values found in the reported picrate salt [21]. The two *ortho*-nitro groups of the anion intersected at an angle of 23.9(3)° with each other. The *para*-nitro group [N3—O4—O5] also deviated slightly from the benzene plane of the anion [6.5(2)°],

Table 1
Summary of X-ray crystallographic data for compounds **1**–**5**.

	1	**2**	**3**	**4**	**5**
Formula	$C_{15}H_{14}N_4O_8$	$C_{16}H_{21}NO_5S$	$C_{16}H_{17}NO_4$	$C_{23}H_{27}NO_{11}$	$C_{16}H_{15}N_3O_7$
Fw	378.30	339.40	287.31	493.46	361.31
T, K	298(2)	298(2)	298(2)	298(2)	298(2)
Wavelength, Å	0.71073	0.71073	0.71073	0.71073	0.71073
Crystal system	Monoclinic	Triclinic	Triclinic	Triclinic	Triclinic
space group	P2(1)/c	P-1	P-1	P-1	P-1
a, Å	9.821(4)	6.288(5)	7.6678(5)	7.3322(6)	7.5790(6)
b, Å	10.847(5)	7.757(6)	9.2975(8)	11.1451(9)	8.7530(8)
c, Å	15.849(7)	18.372(15)	11.4249(9)	15.6486(13)	13.9060(12)
α, deg.	90	99.018(10)	77.4110(10)	105.352(2)	101.0800(10)
β, deg.	90.1530(10)	92.048(9)	84.529(2)	97.1670(10)	91.7900(10)
γ, deg.	90	98.081(10)	65.9930(10)	103.924(2)	112.180(2)
V, $Å^3$	1688.4(13)	874.7(12)	726.13(10)	1172.39(17)	832.84(12)
Z	4	2	2	2	2
D_{calcd}, Mg/m^3	1.488	1.289	1.314	1.398	1.441
Absorption coefficient, mm^{-1}	0.123	0.208	0.095	0.112	0.115
F(000)	784	360	304	520	376
Crystal size, mm^3	0.41 × 0.40 × 0.38	0.44 × 0.21 × 0.08	0.42 × 0.37 × 0.33	0.40 × 0.21 × 0.12	0.43 × 0.28 x 0.13
θ range, deg	2.57–25.02	2.25–25.02	2.75–25.02	2.73–25.02	2.58–25.02
Limiting indices	$-11 \le h \le 11$	$-7 \le h \le 6$	$-9 \le h \le 9$	$-8 \le h \le 8$	$-8 \le h \le 9$
	$-10 \le k \le 12$	$-7 \le k \le 9$	$-11 \le k \le 11$	$-8 \le k \le 13$	$-10 \le k \le 6$
	$-18 \le l \le 14$	$-21 \le l \le 21$	$-13 \le l \le 9$	$-18 \le l \le 16$	$-16 \le l \le 16$
Reflections collected	8269	4479	3646	5899	4271
Reflections independent (R_{int})	2976 (0.0310)	3042 (0.0361)	2499 (0.0143)	4050 (0.0642)	2892 (0.0536)
Goodness-of-fit on F^2	0.981	0.932	0.996	0.876	0.886
R indices [$I > 2\sigma I$]	0.0398, 0.0892	0.0593, 0.1412	0.0449, 0.1162	0.0902, 0.2362	0.1040, 0.2695
R indices (all data)	0.0821, 0.1078	0.0867, 0.1564	0.0799, 0.1401	0.2296, 0.2966	0.2056, 0.3359
Largest diff. peak and hole, e.$Å^{-3}$	0.184, −0.147	0.444, −0.451	0.186, −0.184	0.601, −0.420	0.329, −0.262

these being larger than the corresponding values reported by Muthamizhchelvan et al. [21]. The *para*-nitro group made dihedral angles of 32(1)° and 22.6(4)° with the two *ortho*-nitro groups, respectively.

One cation and one anion formed a bicomponent heteroadduct. In the bicomponent heteroadduct there found the bifurcated N–H···O hydrogen bonds from the NH cation, the O of the phenolate and one o-nitro group with N–O distances of 2.762(3) Å, and 3.013(3) Å, respectively. There also existed the bifurcated CH–O associations between the phenyl CH of the cation, the phenolate, and the other o-nitro group with C–O distances of 3.025(2) Å, and 3.231(3) Å, respectively, and the bifurcated CH_3–O associations from the same O atom of the o-nitro group that was involved in the N–H···O hydrogen bond and both CH_3 groups of the cation with C–O distances of 3.139(3)–3.194(3) Å. The intramolecular O–O contacts were formed between the phenolate and both o-nitro groups with O–O distances of 2.670(4)–2.751(3) Å. For the presence of these nonbonding interactions there were $R^2_1(6)$, $R^1_2(5)$, $R^1_2(6)$, and $R^2_2(9)$ ring motifs in the bicomponent adduct. Two bicomponent adducts were held together by the pair of CH–O association between the phenyl CH of the anion and one o-nitro group with C–O distance of 3.472(3) Å to generate a tetracomponent aggregate. Under the pair of CH–O associations there found a $R^2_2(10)$ ring motif. The tetracomponent aggregates were linked together by the CH–O association between the phenyl CH of the cation and the carbonyl unit of the cation with C–O distance of 3.394(3) Å to form 1D chain. The 1D chains were joined together by the interchain CH_3–O association between the CH_3 of the cation and the p-nitro group with C–O distance of 3.367(4) Å to form 2D sheet extending parallel to the *ab* plane (Fig. 2). The 2D sheets were further stacked along the *c* axis direction by the intersheet CH_3–O associations between the CH_3 group of the cation and the p-nitro group with C–O distances of 3.533(3)–3.631(3) Å, O-π association between the p-nitro group and the phenyl ring of the anion with O–Cg distance of 3.110(2) Å, C-π association between the π-C atom of the carbonyl group of the cation and the phenyl ring of the anion with C-Cg distance of 3.305(2) Å, O–N contact between the p-nitro groups with O–N distance of 2.908(3) Å, and N-C contact between the p-nitro group and the π-C atom of the carbonyl group of the cation with N-C distance of 3.246(4) Å to form 3D layer network structure. In this case the O–Cg distance is comparable to the archived data (3.12 Å) [22]. Herein the neighboring sheets were slipped some distances from each other along the *a* and *b* axis directions, respectively.

3.1.2. *X-ray structure of p-dimethylaminobenzaldehyde: (p-toluenesulfonic acid): (H_2O) [(HL^+)·(tsa^-)·H_2O]* ***(2)***

Similar to **1**, the compound **2** of the composition [(HL^+)···(tsa^-)···H_2O] was prepared by reacting equal mol of L and p-toluenesulfonic acid, in which the proton of the p-toluenesulfonic acid was also transferred to the N atom of the L. Thus **2** can be classified as an organic salt. This structure is a solvate. In the asymmetric unit of **3** there existed one cation of HL, one anion of p-toluenesulfonate, and one water molecule, as shown in Fig. 3. Salt **2** crystallized as triclinic block crystals in the space group P-1.

The S–O bond distances of the SO_3^- group of the p-toluenesulfonate are ranging from 1.446(2) to 1.457(2) Å. The O–S bond lengths are comparable to those in other 4-toluenesulfonate salts [23], indicating the ionized SO_3H group. The C–N angles around the protonated N atoms are 110.9(2)–113.1(2)°, which are similar to the corresponding angles at the salt **1**. The C–O bond length of the CHO group at the cation was 1.185(5) Å. The rms deviation of the phenyl ring of the cation is 0.0041 Å. The rms deviation of the phenyl ring of the anion is 0.0044 Å. The interplane angle between the phenyl ring of the anion and the phenyl ring of the cation is 46.7(2)°.

The water molecule was bonded to the cation by the N–H···O hydrogen bond from the NH cation with N–O distance of 2.760(3) Å. The cation attached with the water molecule associated with the anion by the O–H···O hydrogen bond between the water molecule and the O atom at the SO_3^- with O–O distance of 2.944(4) Å, O–H···S hydrogen bond between the same H of the water molecule and the S atom at the SO_3^- with O-S distance of 3.593(3) Å, and

Table 2
Selected bond lengths [Å] and angles [°] for **1**–**5**.

1			
N(1)–C(5)	1.487(3)	N(1)–C(9)	1.491(3)
N(1)–C(8)	1.499(3)	N(2)–O(2)	1.216(2)
N(2)–O(3)	1.222(2)	N(2)–C(11)	1.468(3)
N(3)–O(4)	1.231(2)	N(3)–O(5)	1.231(2)
N(3)–C(13)	1.453(3)	N(4)–O(7)	1.222(2)
N(4)–O(6)	1.226(2)	N(4)–C(15)	1.461(3)
O(1)–C(10)	1.257(2)	O(8)–C(1)	1.208(3)
C(5)–N(1)–C(9)	110.77(18)	C(5)–N(1)–C(8)	113.20(18)
C(9)–N(1)–C(8)	110.82(19)		
2			
N(1)–C(5)	1.473(3)	N(1)–C(8)	1.493(3)
N(1)-C(9)	1.503(4)	O(1)–C(1)	1.185(5)
O(2)–S(1)	1.457(2)	O(3)–S(1)	1.455(2)
O(4)–S(1)	1.446(2)	S(1)–C(10)	1.771(3)
C(5)–N(1)–C(8)	113.1(2)	C(5)–N(1)–C(9)	112.2(2)
C(8)–N(1)–C(9)	110.9(2)	O(4)–S(1)–O(3)	113.05(14)
O(4)–S(1)–O(2)	112.71(14)	O(3)–S(1)–O(2)	111.73(14)
3			
N(1)–C(5)	1.358(2)	N(1)–C(8)	1.442(3)
N(1)–C(9)	1.446(3)	O(1)–C(1)	1.222(8)
O(1′)–C(1′)	1.230(9)	O(2)–C(10)	1.278(2)
O(3)–C(10)	1.261(2)	O(4)–C(14)	1.351(2)
C(5)–N(1)–C(8)	121.5(2)	C(5)–N(1)–C(9)	121.5(2)
C(8)–N(1)–C(9)	116.7(2)	O(3)–C(10)–O(2)	122.10(18)
4			
N(1)–C(5)	1.332(7)	N(1)–C(9)	1.442(8)
N(1)–C(8)	1.454(8)	O(1)–C(1)	1.125(11)
O(2)–C(10)	1.268(7)	O(3)–C(10)	1.249(7)
O(4)–C(15)	1.372(6)	O(5)–C(13)	1.352(7)
O(6)–C(17)	1.255(7)	O(7)–C(17)	1.269(7)
O(8)–C(20)	1.357(7)	O(9)–C(22)	1.376(6)
C(5)–N(1)–C(9)	121.6(6)	C(5)–N(1)–C(8)	120.8(6)
C(9)–N(1)–C(8)	117.6(6)	O(3)–C(10)–O(2)	123.5(5)
O(6)–C(17)–O(7)	122.9(5)		
5			
N(2)–O(4)	1.203(7)	N(2)–O(5)	1.215(7)
N(2)–C(13)	1.480(7)	N(1)–C(5)	1.352(8)
N(1)–C(8)	1.432(9)	N(1)–C(9)	1.468(8)
N(3)–O(6)	1.198(8)	N(3)–O(7)	1.218(8)
N(3)–C(15)	1.489(9)	O(1)–C(1)	1.208(12)
O(2)–C(10)	1.225(11)	O(3)–C(10)	1.225(11)
O(4)–N(2)–O(5)	125.6(6)	C(5)–N(1)–C(8)	122.6(6)
C(5)–N(1)–C(9)	121.3(6)	C(8)–N(1)–C(9)	116.1(6)
O(6)–N(3)–O(7)	126.7(8)	O(3)–C(10)–O(2)	122.4(9)

Table 3
Hydrogen bond distances and angles in studied structures **1**–**5**.

D-H···A	d(D–H) [Å]	d(H···A) [Å]	d(D···A) [Å]	<(DHA)[°]
1				
N(1)–H(1)···O(7)	0.91	2.47	3.013(3)	118.6
N(1)–H(1)···O(1)	0.91	1.87	2.762(3)	166.0
2				
O(5)–H(5D)···S(1)#1	0.85	2.89	3.651(3)	150.6
O(5)–H(5D)···O(2)#1	0.85	1.94	2.780(3)	172.8
O(5)–H(5C)···S(1)	0.85	2.86	3.593(3)	145.7
O(5)–H(5C)···O(3)	0.85	2.10	2.944(4)	173.0
N(1)–H(1)···O(5)	0.91	1.87	2.760(3)	164.7
3				
O(2)–H(2)···O(3)#1	0.82	1.83	2.632(2)	167.6
4				
O(11)–H(11D)···O(5)#1	0.85	2.02	2.867(9)	171.3
O(11)–H(11C)···O(5)#2	0.85	2.34	3.184(10)	173.7
O(10)–H(10D)···O(11)#1	0.85	1.83	2.673(9)	169.5
O(10)–H(10C)···O(4)#3	0.85	2.05	2.886(8)	169.7
O(9)–H(9)···O(10)#4	0.82	1.80	2.618(7)	172.9
O(8)–H(8)···O(1)#5	0.82	1.94	2.755(7)	174.6
O(6)–H(6)···O(3)#6	0.82	1.81	2.610(5)	166.8
O(5)–H(5)···O(11)#7	0.82	2.38	3.184(10)	167.2
O(4)–H(4)···O(9)#8	0.82	1.89	2.703(5)	173.5
O(2)–H(2)···O(7)#6	0.82	1.82	2.627(5)	168.4
5				
O(3)–H(3)···O(1)#1	0.82	2.06	2.865(9)	167.7

Symmetry transformations used to generate equivalent atoms for **2**: #1 −x+1, −y+1, −z+1. Symmetry transformations used to generate equivalent atoms for **3**: #1 −x, −y+1, −z. Symmetry transformations used to generate equivalent atoms for **4**: #1 −x+1, −y+1, −z+1; #2 x, y, z+1; #3 x, y−1, z; #4 x, y+1, z; #5 −x, −y+1, −z; #6 −x+1, −y+2, −z+1; #7 x, y, z−1; #8 −x+1, −y+2, −z. Symmetry transformations used to generate equivalent atoms for **5**: #1 −x, −y, −z.

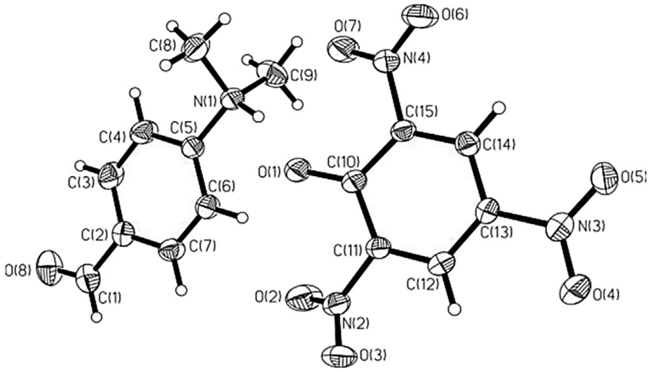


Fig. 1. Molecular structure of **1** showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

CH_3–O associations between both CH_3 groups of the cation and the other two O atoms of the SO_3^- with C–O separations of 3.343(2)–3.655(2) Å to form a tricomponent adduct. Two tricomponent adducts were joined together by the O–H···O hydrogen bond between the water molecule and the O atom at the SO_3^- with O–O distance of 2.780(3) Å, and O–H···S hydrogen bond between the same H of the water molecule and the S at the SO_3^- with O-S distance of 3.651(3) Å to form a six-component aggregate. At the six-component aggregate the two tricomponent adducts were inversionally related. The six-component aggregates were linked together by the CH–O association between the phenyl CH of the cation and the SO_3^- with C–O separation of 3.397(4) Å, and CH_3–O associations between both CH_3 groups of the cation and the O atoms of the SO_3^- with C–O separations of 3.465(2)–3.506(2) Å to form 1D chain. The 1D chains were connected together by the CH_3–O association between the CH_3 of the cation and the CHO group of the cation with C–O distance of 3.486(3) Å to form 2D sheet extending at the direction that made an angle of *ca.* 60° with the *bc* plane (Fig. 4). The 2D sheets were further stacked along the direction that was perpendicular with its extending direction by the CH_3–O association between the CH_3 group of the cation and the SO_3^- group of the anion with C–O distance of 3.404(2) Å to form 3D network structure.

3.1.3. X-ray structure of p-dimethylaminobenzaldehyde: (p-hydroxybenzoic acid) [(L)·(phba)] ***(3)***

Compound **3** was also prepared by reacting equal mol of p-dimethylaminobenzaldehyde and p-hydroxybenzoic acid. The crystals of a new cocrystal, [(L)···(phba)], are built up from one p-dimethylaminobenzaldehyde and one p-hydroxybenzoic acid molecule, which is shown in Fig. 5. The compound **3** crystallizes in the triclinic space group P-1. The aldehyde group of the p-dimethylaminobenzaldehyde was disordered over two positions with equal occupancy. Different from the above adducts, here the N atom of the p-dimethylaminobenzaldehyde is not protonated by the p-hydroxybenzoic acid. Thus **3** is a cocrystal. The angles concerning the N1 atom are 116.7(2)–121.5(2)°, which are larger than the corresponding angles at the salts **1** and **2**. The C–O bond involving the CHO unit is also of typical length for the aldehyde unit. The rms deviation of the ring with C2–C7 is 0.0012 Å, the rms deviation of the ring with C11–C16 is 0.0055 Å, both rings intersected at an

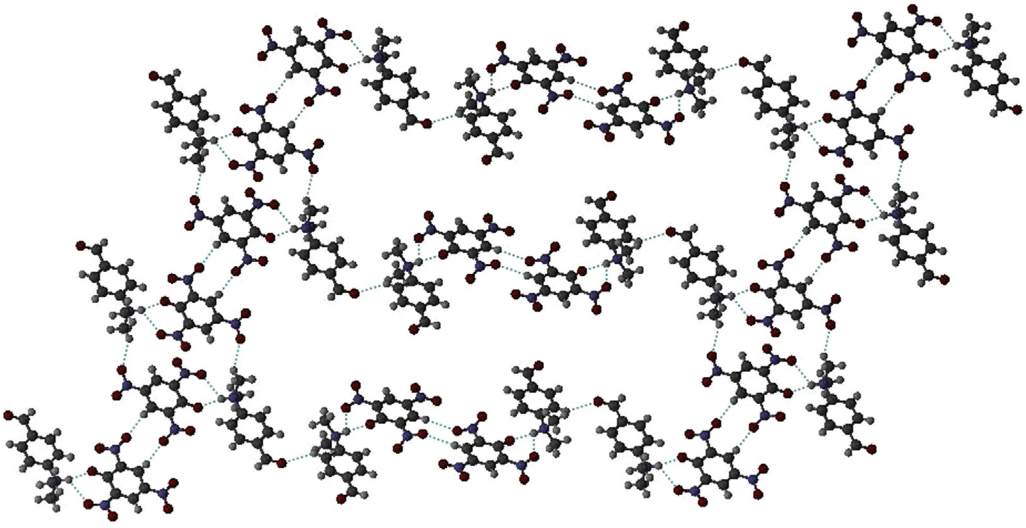


Fig. 2. 2D sheet structure of **1** extending parallel to the *ab* plane.

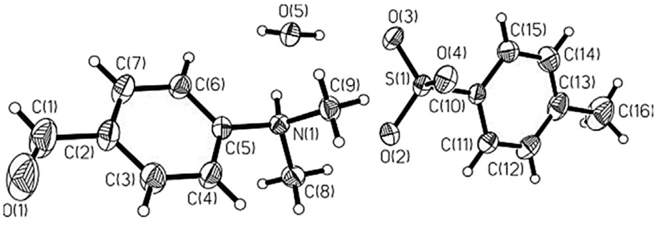


Fig. 3. Molecular structure of **2**, showing the atom-numbering scheme. Displacement ellipsoids were drawn at the 30% probability level.

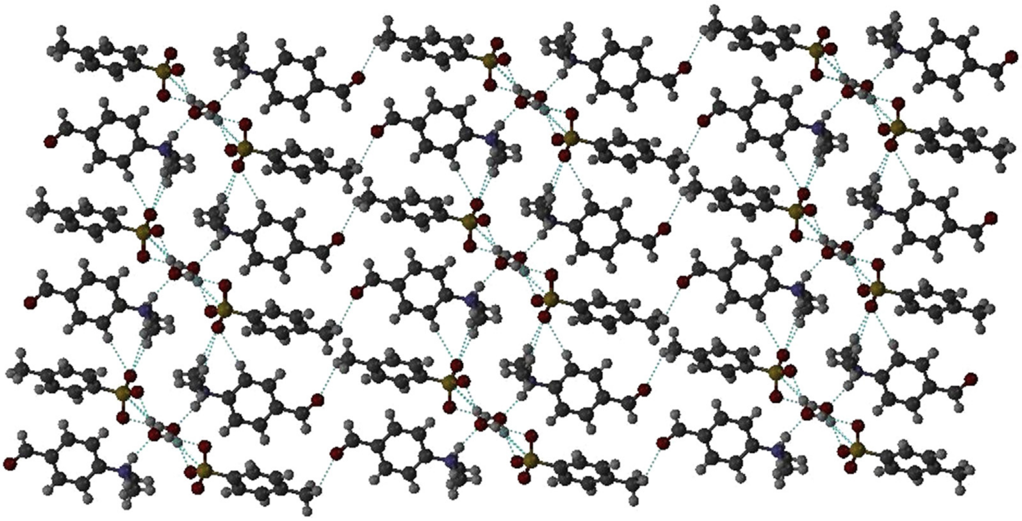


Fig. 4. The 2D sheet structure of **2** formed *via* the CH_3–O association extending at the direction that made an angle of *ca.* 60° with the *bc* plane.

angle of 85.6(2)° with each other.

At the p-dimethylaminobenzaldehyde there was bonded a p-hydroxybenzoic acid by the O–H···O hydrogen bond between the p-hydroxyl group of the p-hydroxybenzoic acid and the aldehyde group of the p-dimethylaminobenzaldehyde with O–O distance of 2.702(2) Å, and CH–O association between the 3-CH of the p-hydroxybenzoic acid and the aldehyde group of the p-dimethylaminobenzaldehyde with C–O distance of 3.317(3) Å to generate a heteroadduct. Two heteroadducts were joined together by the O–H···O hydrogen bonds between the COOH groups of the p-hydroxybenzoic acids with O–O distance of 2.632(2) Å to form a tetracomponent adduct. In the tetracomponent adduct there existed a symmetry centre located at the middle point of the two COOH groups. Herein the tetracomponent adduct displayed the $R_2^1(6)$, and $R_2^2(8)$ rings. The tetracomponent adducts were combined together by the CH–O association between the 2-CH of the p-hydroxybenzoic acid and the OH of the COOH with C–O distance of 3.497(4) Å to form 1D chain running along the *a* axis direction (Fig. 6). The $R_2^2(8)$ rings were formed for the CH–O associations. The chains extending on the same plane formed a 2D sheet, yet there were no bonding interactions between these chains. The 2D sheets were further stacked along the direction that was perpendicular with its extending direction by the CH–O association between the 2-CH of the p-dimethylaminobenzaldehyde and the carbonyl unit

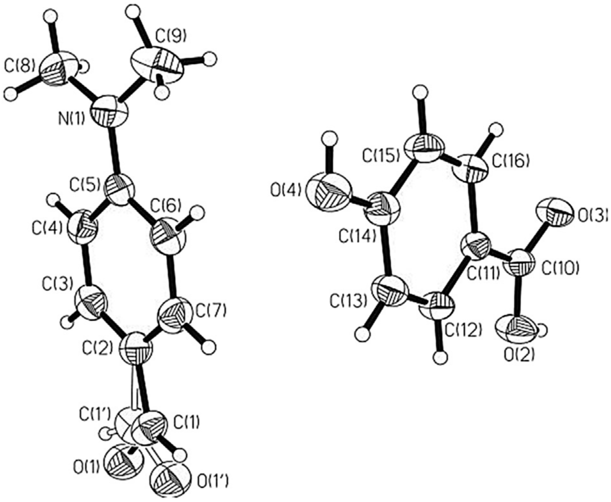


Fig. 5. Molecular structure of **3** showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

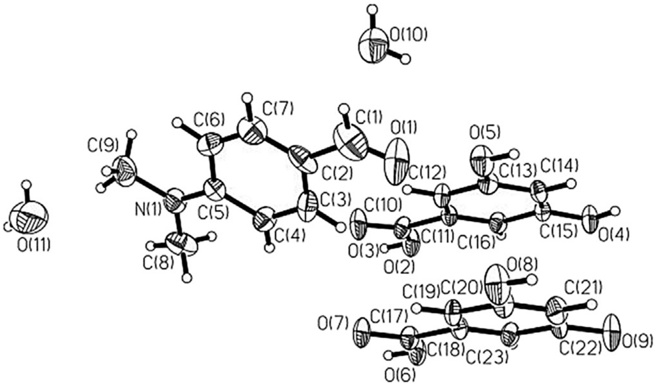


Fig. 7. Molecular structure of **4** showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

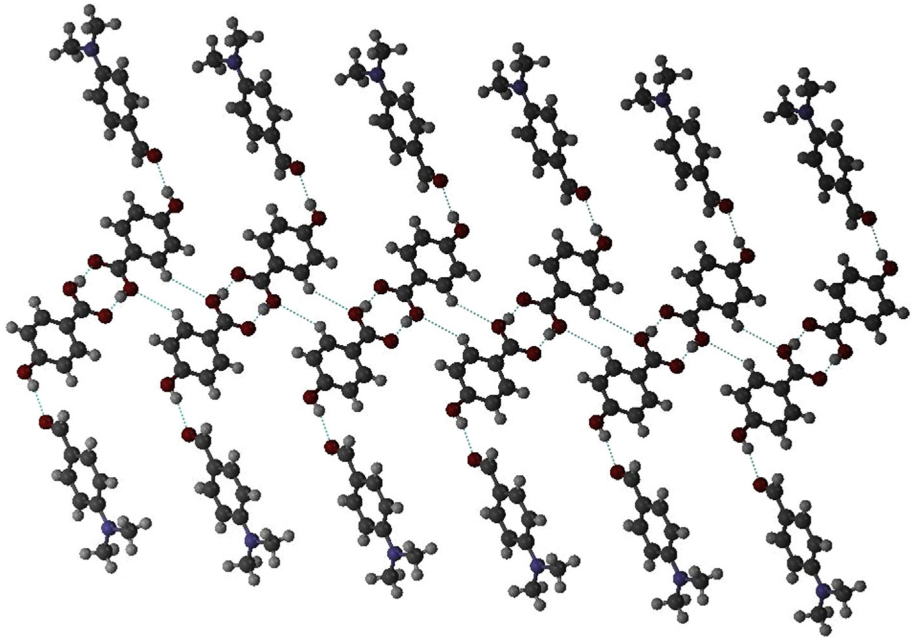


Fig. 6. The 1D chain of **3** generated by the CH–O association running along the *a* axis direction.

of the p-hydroxybenzoic acid with C–O distance of 3.594(4) Å to form 3D layer network structure. Herein the neighboring sheets were slipped some distance from each other along the extending direction.

3.1.4. X-ray structure of p-dimethylaminobenzaldehyde: (3,5-dihydroxybenzoic acid)$_2$: (H$_2$O)$_2$ [(L)·(dhba)$_2$·(H$_2$O)$_2$] **(4)**

Compound **4** was also prepared by reacting equal mol of p-dimethylaminobenzaldehyde and 3,5-dihydroxybenzoic acid. The crystals of a new p-dimethylaminobenzaldehyde cocrystal, [(L)···(dhba)$_2$···(H$_2$O)$_2$], are built up from one L, two 3,5-dihydroxybenzoic acid molecules, and two lattice water molecules, which is shown in Fig. 7. The compound **4** crystallizes in the triclinic space group P-1. For **4**, only crystals of bad quality could be grown with the largest residual density being relatively high.

The angles around the N1 are similar to the corresponding values at the compound **4**. The O(1)–C(1) bond was 1.125(11) Å. The rms deviation of the phenyl ring with C2–C7 is 0.0038 Å, the rms deviation of the phenyl ring with C11–C16 is 0.0059 Å, both rings made an angle of 32.7(2)° with each other. The rms deviation of the phenyl ring with C18–C23 is 0.0014 Å, this ring made dihedral angles of 34.3(1)° and 2.1(1)° with the above two rings, respectively.

The two OH groups at one kind of 3,5-dihydroxybenzoic acid are arranged in a *syn–syn* orientation with respect to the H atom in the *para* position. While in the other kind of 3,5-dihydroxybenzoic acid molecule the two OH groups are arranged in a *syn-anti* orientation with respect to the H atom in the *para* position, which are different from the reported organic adduct containing the 3,5-dihydroxybenzoate [24].

Two 3,5-dihydroxybenzoic acid molecules were held together by a pair of O–H···O hydrogen bonds between the COOH groups with O–O distances of 2.610(5)–2.627(5) Å to form a carboxyl dimer. In the dimer there existed the hydrogen bonded motif with

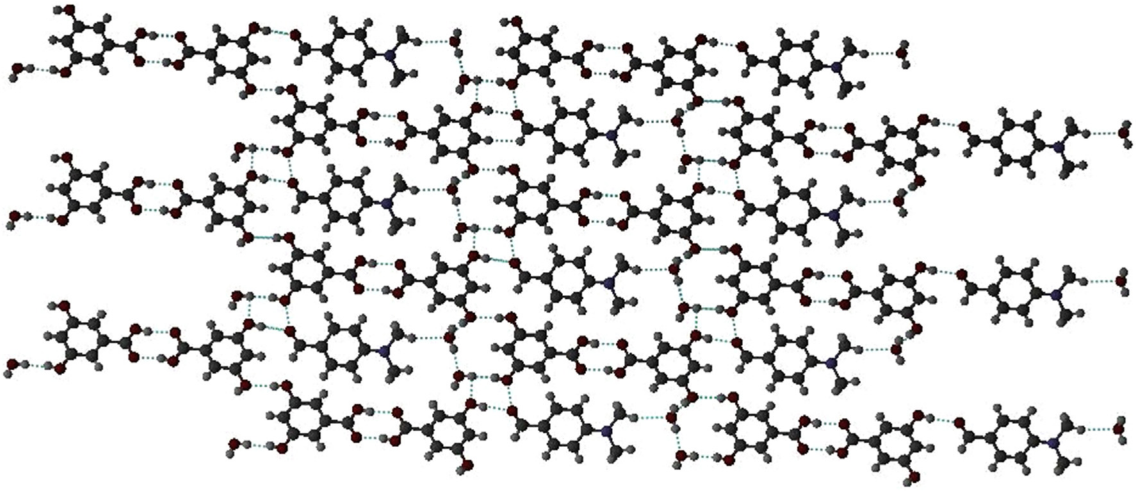


Fig. 8. 2D sheet structure of **4** extending along the direction that made an angle of *ca.* 60° with the *bc* plane.

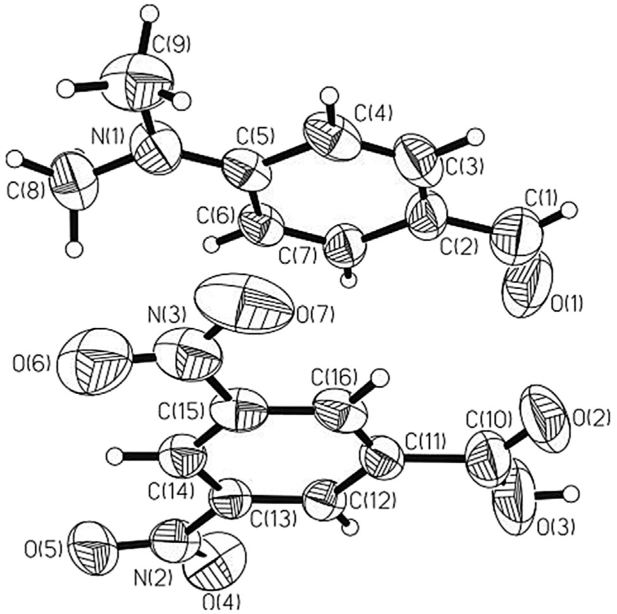


Fig. 9. Molecular structure of **5** showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

graph set description of $R_2^2(8)$. At the carboxyl dimer there was bonded a water *via* the O—H···O hydrogen bond between the hydroxyl group of the 3,5-dihydroxybenzoic acid and the water molecule with O—O distance of 3.184(10) Å to form a tricomponent adduct. At the p-dimethylaminobenzaldehyde there was also bonded a water molecule by the CH_3—O association between the CH_3 of the p-dimethylaminobenzaldehyde and the water molecule with C—O distance of 3.450(6) Å, producing a bicomponent adduct. The tricomponent adduct and the bicomponent adduct were combined together by the O—H···O hydrogen bond between the hydroxyl group of the 3,5-dihydroxybenzoic acid and the C=O of the p-dimethylaminobenzaldehyde molecule with O—O distance of 2.755(7) Å to form a five-component aggregate. The five-component aggregates were linked together by the O—H···O hydrogen bond between the water molecule attached to the CH_3 and the water molecule attached to the hydroxyl group of the 3,5-dihydroxybenzoic acid molecule with O—O distance of 2.673(9) Å to form 1D chain. The 1D chains were connected together by the O—H···O hydrogen bond between the hydroxyl groups of the 3,5-dihydroxybenzoic acid molecules with O—O distance of 2.703(5) Å, O—H···O hydrogen bonds between the hydroxyl group(donor) of the 3,5-dihydroxybenzoic acid molecule and the water molecule(-acceptor) with O—O distances of 2.618(7)–2.727(4) Å, and O—O contact between the O atom at the CHO and the O atom of the hydroxyl group with O—O distance of 2.896(2) Å to form 2D sheet extending along the direction that made an angle of *ca.* 60° with the *bc* plane (Fig. 8). The 2D sheets were further stacked along the direction that was perpendicular to its extending direction by the O—H···O hydrogen bond between the water molecule (donor) and one hydroxyl group (acceptor) of the 3,5-dihydroxybenzoic acid molecule with O—O distance of 2.886(8) Å, and CH_3-π association between one CH_3 of the p-dimethylaminobenzaldehyde and the phenyl ring of the p-dimethylaminobenzaldehyde with C-Cg distance of 3.719(2) Å to form a double sheet. In this case the chains of the two sheets were slipped some distance from each other along the extending direction. The double sheets were further stacked along the direction that was perpendicular with its extending direction by the O—H···O hydrogen bond between the water molecule (donor) and one hydroxyl group of the 3,5-dihydroxybenzoic acid molecule with O—O distance of 2.867(9) Å, and CH_3-π association between one CH_3 unit of the p-dimethylaminobenzaldehyde and the phenyl ring of the p-dimethylaminobenzaldehyde with C-Cg distance of 3.785(5) Å to form 3D network structure.

3.1.5. X-ray structure of p-dimethylaminobenzaldehyde: (3,5-dinitrobenzoic acid) [(L)·(Hdba), Hdba = 3,5-dinitrobenzoic acid] (5)

The compound **5** of the composition [(L) · (Hdba)] was prepared by reacting equal mol of p-dimethylaminobenzaldehyde and 3,5-dinitrobenzoic acid in 1:1 ratio, which crystallizes as triclinic colorless crystals in the space group P-1. The structure of **5** with the atom numbering scheme is shown in Fig. 9. This is a cocrystal where the COOH groups of 3,5-dinitrobenzoic acids are unionized by proton transfer to the nitrogen atoms of the p-dimethylaminobenzaldehyde moiety. New crystals were obtained for **5** and used for a new data acquisition and refinement, unfortunately, the obtained R is not better than that obtained for the previous study.

One p-dimethylaminobenzaldehyde and one 3,5-dinitrobenzoic acid molecule formed a bicomponent adduct by the O—H···O hydrogen bond between the OH of the COOH and the C=O of the p-

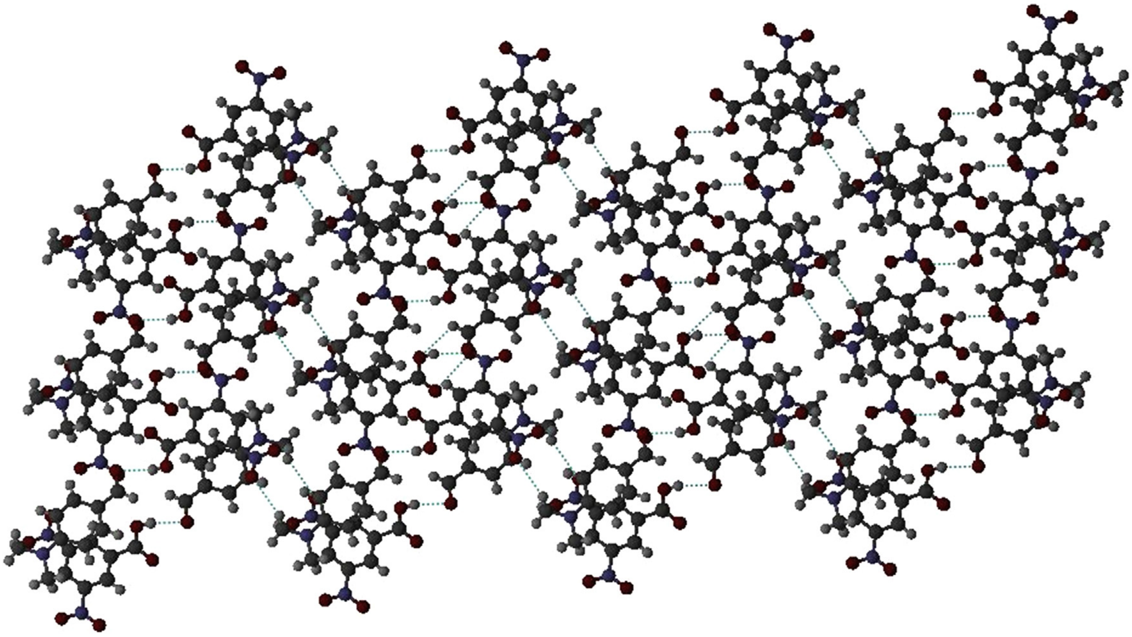


Fig. 10. 2D sheet structure of **5** extending at the direction that made an angle of *ca.* 60° with the *bc* plane.

dimethylaminobenzaldehyde with O—O distance of 2.865(9) Å, one CH—O association between the CH at the CHO and the OH of the COOH with C—O distance of 3.128(2) Å, and O—O contact between the O of the carbonyl at the COOH and the O atom of the C═O at the p-dimethylaminobenzaldehyde with O—O distance of 2.998(3) Å to generate a bicomponent adduct. The bicomponent adducts were connected together by the CH_3—O association between the CH_3 of the p-dimethylaminobenzaldehyde and the nitro group with C—O distance of 3.393(2) Å to form 1D zigzag chain. Two 1D chains were joined together by the CH_3—O association between the CH_3 of the p-dimethylaminobenzaldehyde and the nitro group with C—O distance of 3.524(2) Å to form double chain. The double chains were combined together by the O—O contact between the O atom of the C═O at the p-dimethylaminobenzaldehyde and the O of the nitro group with O—O distance of 3.036(2) Å to produce a 2D sheet extending at the direction that made an angle of *ca.* 60° with the *bc* plane (Fig. 10).

4. Conclusion

The five crystalline adducts **1**–**5** from p-dimethylaminobenzaldehyde have been prepared and characterized by X-ray diffraction analysis, IR, mp, and elemental analysis. The different hydrogen bond interaction modes in these compounds lead to a variety of different structures such as 2D sheet, 3D layer network structure, and 3D network structure.

Of the five adducts the N atoms at **1**, and **2** are fully protonated and then act as H-donor linking the cation and the anion together. While the N at **3**, **4**, and **5** do not take part in the non-covalent interactions giving rise to the supramolecular organization of these cocrystals. In **3** and **5**, the water molecules are also participated in hydrogen bonding. For the acids in **1**, and **2** are stronger than the acids used at the compounds **3**, **4**, and **5**, and the acidic H at **1**, and **2** are ionized in the solution and transferred to the N at L forming the carboxylate-ammonium pairs, while this fact does not fit for **3**, **4**, and **5**.

This study has demonstrated that the classical hydrogen bonds (N—H···O, O—H···O and O—H···S) are the major intermolecular forces in a family of structures with the OH···p-dimethylaminobenzaldehyde synthons. Except the classical hydrogen bonds, the secondary propagating interactions also play an important role in structure extension. Compounds **1**–**5** possess weak C—H···O associations. Two types of auxiliary C—H···O associations were observed based upon their geometric preferences, intra- and interchain interactions. Based upon an analysis of the metrics displayed by each set of interactions, it seems that intra- and interchain C—H···O interactions are of equal structural importance.

There are also CH_3···O associations in the compounds **1**, **2**, **4**, and **5**. The CH_3-π interactions were found in **4**. There existed the O—O contacts at compounds **1**, **4**, and **5**. Compound **1** bears the O-π, O—N, N—C, and C-π associations.

In conclusion, we have shown that higher dimensional structures can be constructed by the collective non-covalent interactions of strong directional hydrogen bonds and other non-classical hydrogen bonds.

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Appendix A. Supplementary data

Supplementary data related to this article can be found at http://dx.doi.org/10.1016/j.molstruc.2016.03.027.

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