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Structural Study of the Compound $[(C_{10}O_8H_2)_2 (C_4N_2H_6)].2H_2O$ Synthesized by Hydrothermal Condition

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Abstract

A new metal–organic compound $[(C_{10}O_8H_2)_2(C_4N_2H_6)].2H_2O$ **(I)** was hydrothermally synthesized from an aqueous solution of Fe $(NO_3)_3$ $9H_2O$, (btec) (btec= 1, 2, 4, 5-benzenetetracarboxylic acid) and piperazine. Compound I crystallizes in the triclinic system with the *P*1 space group. The unit cell parameters are *a* = 8.271 Å, *b* = 8.500 Å, *c* = 9.660 Å, *α* = 87.12°, *β* = 89.53°, *γ* = 70.91°, *Z*= 2, *V*= 640.96(6) Å3 and D_x= 1.602g/cm^3. The refinement converged into *R*= 0.047 and R_W = 0.059. The structure, determined by single crystal X-ray diffraction, consists of two carboxyl group, piperazine and two molecules of water.

Keywords: Hydrothermal synthesis; X-ray diffraction; Crystal structure.

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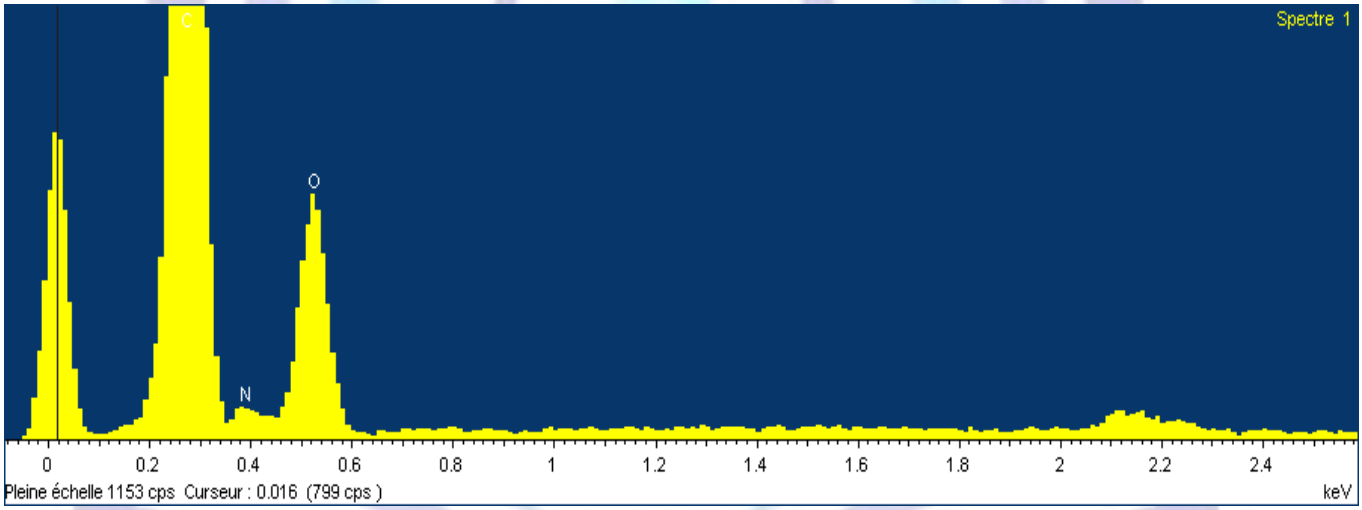
1. Introduction

The design and synthesis of supramolecular coordination polymeric networks, especially those constructed by hydrogen bonding and intermolecular weak interactions have been a field of rapid growth due to their special physical properties and potential application in functional materials. Of particular interest are compounds that are capable of forming very strong hydrogen bonds like some salts of pyromellitic acid. The 1,2,4,5-benzenetetracarboxylic acid, also known as pyromellitic acid, possesses several interesting characteristics: (a) it has four carboxyl groups which may be completely or partially deprotonated, inducing rich coordination modes and allowing interesting structures with higher dimensions; (b) it can act not only as hydrogen-bond acceptor but also as hydrogen-bond donor, depending upon the numbers of deprotonated carboxyl groups **[1-8]**.

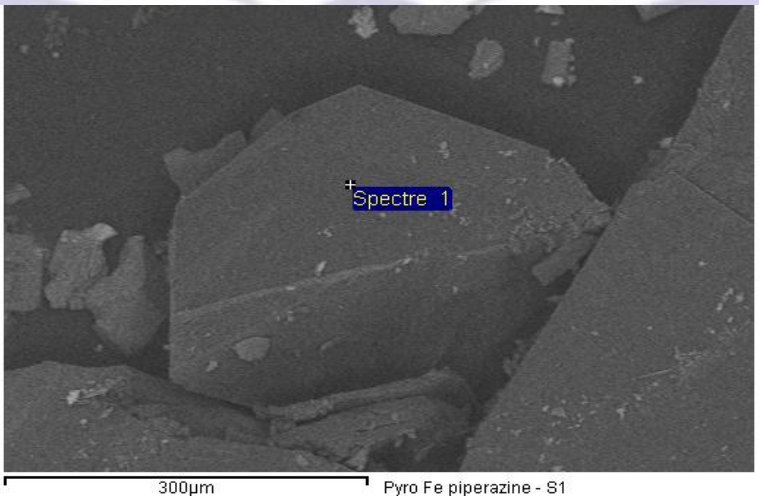
2. Experimental

2.1. Synthesis and initial characterization

The title compound was synthesized under hydrothermal conditions in the presence of piperazine. In a typical synthesis, 0.1655g of 1, 2, 4, 5-benzenetetracarboxylic acid (betc) (Acros Organics) was dispersed in 9 ml of H_2O. To this, 0.1975g of iron nitrate monohydrate (Prolabo) was added under constant stirring. Finally, we add 0.1344g of piperazine (Sigma) and the mixture was homogenized for 15 min at room temperature, was sealed in a 23ml PTFE-lined stainless steel autoclave and heated at 120°C for 60 h. The pH of the initial reaction mixture was ~ 5 and did not change appreciably after the reaction. Then the product obtained is filtered and washed with a small amount of distilled water. The chemical purity of the product was tested by EDAX measurements. **[Figure 1(a)]** presents the EDAX spectrum of $[(C_{10}O_8H_2)_2(C_4N_2H_6)].2H_2O$ which reveals the presence of all non-hydrogen atoms: N, C and O. Elemental analysis give these results: for observed we have C 67.12%, N 3.76%, O 29.13% ; whereas for calculated we find C 72.79%, N 3.49%, O 23.72%. The metal used in this synthesis does not appear in the reaction product and its role remains unexplained. The **[Figure 1(b)]** shows the photograph of scanning electron microscopy (SEM) of the samples $[(C_{10}O_8H_2)_2(C_4N_2H_6)].2H_2O$ at room temperature.



(a)



(b)

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2.2. Single crystal structure determination

The unit-cell dimensions were refined using X-ray diffraction data collected with a Kappa CCD Enraf Noninus diffractometor using Mo Kα radiation .The structure, $[(C_{10}O_8H_2)_2(C_4N_2H_6)].2H_2O$, was analyzed with the crystallographic CRYSTALS program **[9]**. The structure was solved by conventional Patterson and difference-Fourier techniques. The chemical crystal data, the parameters used for X-ray diffraction data collection and strategy used for the crystal structure determination and their results, are listed in **Table 1**. **Table 2** shows the atomic coordinates and equivalent isotropic displacement. The anisotropic displacement parameters are listed in **Table 3**. Selected bond distances and angles are given in **Table 4** and **5**. Structural graphics were created by the DIAMOND program **[10]**. The asymmetric unit is shown in **(Figure 2)**.

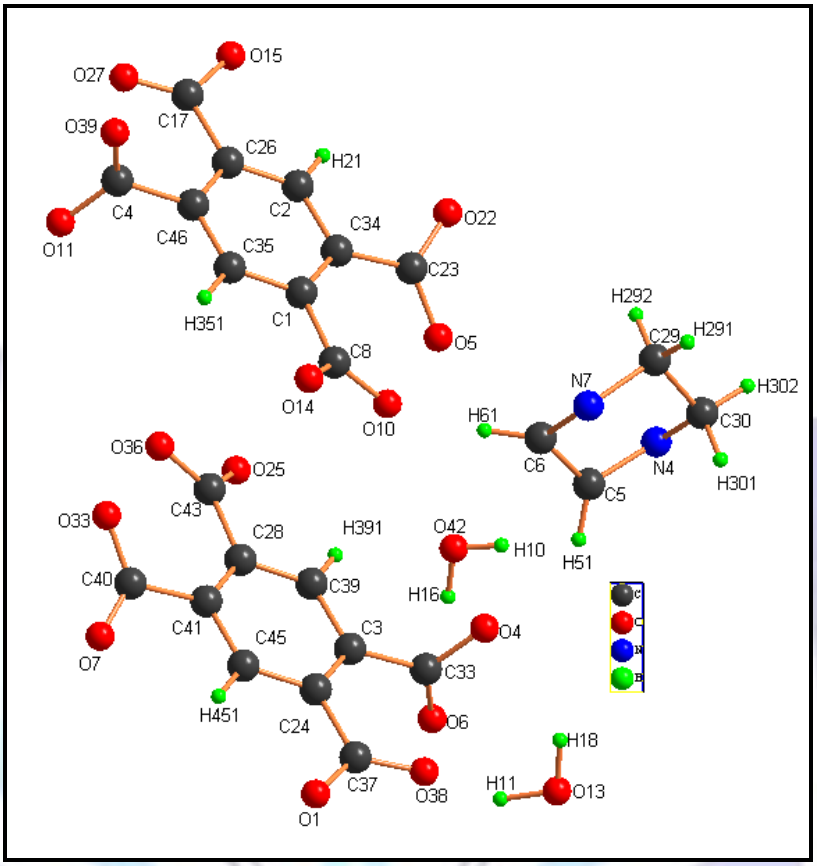

Figure 2. Asymmetric unit of $[(C_{10}O_8H_2)_2(C_4N_2H_6)].2H_2O$

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Table 1. Crystallographic data for $[(C_{10}O_8H_2)_2(C_4N_2H_6)].2H_2O$

Chemical formula $[(C_{10}O_8H_2)_2(C_4N_2H_6)].2H_2O$
Formula weight = 618.38 g mol^{-1}
Crystal system: Triclinic
Space group: *P*1
a = 8.271 Å
b = 8.500 Å
c = 9.660 Å
α = 87.12°
β = 89.53°
γ = 70.91°
V = 640.97 (1) $Å^3$
Z = 1
2θ°max = 30.1°with Mo *Kα*
T = 293 (k)
D_x = 1.602 Mg m^{-3}
-11≤h≤11
-11≤k≤11
-13≤l≤13
μ = 0.14 mm^{-1}
Data collection instrument: Nonius Kappa CCD Diffractometer Radiation, monochromator graphite λ= 0.71073 Å
Measured reflections: 16984
Unique reflections: 3726
R= 0.047 and R_W = 0.059

Table 2. Fractional atomic coordinates and equivalent isotropic displacement for $[(C_{10}O_8H_2)_2(C_4N_2H_6)].2H_2O$

	x	*y*	*z*	U_{iso}*/U_{eq}	Occupancy
C1	0.9906 (6)	0.1589 (6)	−0.0328 (5)	0.0196	1.000
C2	0.7486 (6)	0.4020 (6)	−0.0933 (5)	0.0241	1.000
C3	0.2739 (6)	0.3876 (6)	−0.0977 (5)	0.0201	1.000
O5	0.9305 (6)	0.0808 (5)	−0.3332 (4)	0.0396	1.000
O7	0.3127 (7)	0.3890 (5)	0.4099 (4)	0.0397	1.000
C8	1.1445 (6)	0.0031 (6)	−0.0556 (6)	0.0270	1.000
O10	1.1441 (6)	−0.0799 (5)	−0.1681 (5)	0.0369	1.000
O11	0.7607 (5)	0.3026 (5)	0.3697 (4)	0.0339	1.000
O13	0.1649 (6)	0.1128 (6)	0.4265 (5)	0.0465	1.000
O14	1.2521 (5)	−0.0411 (5)	0.0310 (5)	0.0397	1.000
O15	0.5002 (6)	0.6900 (5)	−0.0268 (4)	0.0397	1.000
C17	0.5868 (6)	0.5964 (6)	0.0822 (5)	0.0215	1.000
O22	0.8039 (6)	0.3551 (5)	−0.3726 (4)	0.0361	1.000

C23	0.8707 (7)	0.2295 (6)	−0.2884 (5)	0.0246	1.000
C24	0.3874 (6)	0.2905 (6)	0.0031 (4)	0.0200	1.000
C26	0.7295 (6)	0.4466 (6)	0.0416 (5)	0.0196	1.000
O27	0.5516 (6)	0.6302 (6)	0.1976 (4)	0.0392	1.000
C33	0.2792 (7)	0.3513 (6)	−0.2487 (5)	0.0243	1.000
C34	0.8751 (6)	0.2590 (6)	−0.1367 (5)	0.0193	1.000
C35	0.9704 (6)	0.2069 (6)	0.1052 (5)	0.0237	1.000
C37	0.5314 (6)	0.1396 (6)	−0.0353 (5)	0.0227	1.000
O4	0.3530 (6)	0.4380 (5)	−0.3282 (4)	0.0366	1.000
O25	−0.1428 (6)	0.7856 (6)	0.0088 (5)	0.0383	1.000
C28	0.1224 (6)	0.5771 (6)	0.0799 (5)	0.0214	1.000
O33	0.1792 (6)	0.6553 (5)	0.3728 (4)	0.0327	1.000
O36	−0.0335 (5)	0.8221 (5)	0.2042 (5)	0.0359	1.000
O38	0.5652 (6)	0.1132 (5)	−0.1609 (4)	0.0315	1.000
C39	0.1418 (6)	0.5318 (6)	−0.0574 (5)	0.0215	1.000
C40	0.2401 (6)	0.5092 (7)	0.3343 (5)	0.0253	1.000
C41	0.2366 (6)	0.4780 (6)	0.1805 (5)	0.0217	1.000

Table 3. Anisotropic displacement parameters (Å^2) for [$(C_{10}O_8H_2)_2(C_4N_2H_6)$].2$H_2O$

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0,0213 (17)	0,0170 (16)	0,0155 (14)	0,0009 (11)	0,0048 (13)	−0,0022 (12)
C2	0,0184 (18)	0,0234 (19)	0,0225 (14)	0,0046 (13)	0,0003 (13)	−0,0037 (13)
C3	0,0220 (18)	0,0189 (18)	0,0155 (13)	−0,0014 (12)	−0,0003 (13)	−0,0013 (12)
O5	0,050 (2)	0,0310 (15)	0,0325 (17)	−0,0040 (16)	−0,0033 (17)	−0,0159 (14)
O7	0,058 (2)	0,0328 (17)	0,0179 (13)	−0,0011 (16)	−0,0001 (15)	−0,0022 (12)
C8	0,0230 (17)	0,0172 (18)	0,034 (2)	0,0030 (12)	0,0036 (14)	−0,0076 (14)
O10	0,0426 (19)	0,0246 (17)	0,0359 (17)	0,0010 (15)	0,0128 (15)	−0,0151 (13)
O11	0,0446 (19)	0,0356 (18)	0,0240 (15)	−0,0175 (15)	0,0056 (14)	0,0030 (13)
O13	0,043 (2)	0,044 (2)	0,043 (2)	0,0007 (18)	−0,0056 (17)	−0,0099 (17)
O14	0,0286 (17)	0,0358 (18)	0,041 (2)	0,0091 (14)	−0,0026 (14)	−0,0097 (17)
O15	0,0357 (19)	0,037 (2)	0,0258 (15)	0,0169 (16)	−0,0043 (16)	−0,0029 (16)
C17	0,0211 (17)	0,0196 (18)	0,0208 (14)	−0,0022 (12)	−0,0009 (13)	−0,0039 (13)
O22	0,0447 (19)	0,0383 (18)	0,0162 (13)	−0,0009 (15)	−0,0024 (14)	−0,0029 (12)
C23	0,029 (2)	0,0257 (16)	0,0193 (14)	−0,0092 (15)	0,0020 (15)	−0,0067 (11)
C24	0,0192 (17)	0,0213 (17)	0,0139 (13)	0,0016 (12)	−0,0007 (12)	−0,0040 (12)
C26	0,0206 (17)	0,0171 (17)	0,0195 (14)	−0,0038 (12)	0,0024 (13)	−0,0009 (13)
O27	0,0403 (19)	0,037 (2)	0,0250 (14)	0,0091 (16)	0,0052 (14)	−0,0120 (14)
C33	0,029 (2)	0,0225 (19)	0,0165 (14)	−0,0009 (14)	−0,0006 (14)	−0,0048 (12)
C34	0,0210 (16)	0,0162 (17)	0,0171 (12)	−0,0008 (12)	0,0028 (12)	−0,0019 (11)

C35	0,025 (2)	0,027 (2)	0,0149 (14)	−0,0029 (14)	0,0038 (14)	−0,0052 (14)
C37	0,0209 (17)	0,0207 (18)	0,0206 (14)	0,0018 (12)	0,0022 (13)	−0,0043 (13)
O4	0,057 (2)	0,045 (2)	0,0144 (12)	−0,0249 (17)	0,0031 (13)	−0,0086 (13)
O25	0,0337 (18)	0,0318 (19)	0,0339 (18)	0,0110 (15)	−0,0051 (14)	−0,0063 (16)
C28	0,0177 (16)	0,0211 (18)	0,0236 (15)	−0,0039 (11)	0,0018 (13)	−0,0015 (13)
O33	0,0407 (18)	0,0323 (15)	0,0223 (15)	−0,0070 (14)	0,0037 (15)	−0,0103 (12)
O36	0,0348 (18)	0,0260 (16)	0,0385 (17)	0,0025 (14)	−0,0072 (14)	−0,0086 (12)
O38	0,0362 (17)	0,0319 (18)	0,0181 (12)	0,0007 (14)	0,0014 (13)	−0,0065 (12)
C39	0,0217 (18)	0,0154 (17)	0,0220 (15)	0,0009 (12)	−0,0046 (14)	0,0020 (13)
C40	0,0241 (19)	0,0312 (17)	0,0154 (13)	−0,0015 (15)	0,0046 (14)	−0,0050 (12)
C41	0,0228 (18)	0,0257 (18)	0,0162 (12)	−0,0070 (13)	0,0006 (13)	−0,0048 (12)
C43	0,0213 (17)	0,0226 (18)	0,0225 (16)	−0,0025 (12)	0,0063 (12)	0,0018 (12)
C45	0,0261 (19)	0,0210 (19)	0,0113 (13)	−0,0054 (13)	−0,0003 (13)	−0,0006 (12)
C46	0,0201 (17)	0,0211 (18)	0,0163 (13)	−0,0045 (12)	0,0023 (12)	−0,0030 (12)
O6	0,049 (2)	0,051 (2)	0,0251 (16)	−0,0269 (18)	0,0031 (16)	−0,0102 (15)
O39	0,0423 (19)	0,040 (2)	0,0299 (16)	−0,0200 (16)	0,0022 (15)	−0,0112 (15)
O42	0,042 (2)	0,042 (2)	0,047 (2)	−0,0016 (18)	0,0004 (18)	−0,0111 (18)
O1	0,037 (2)	0,0345 (19)	0,0212 (15)	0,0131 (15)	0,0023 (16)	−0,0014 (15)
N4	0,0267 (15)	0,0299 (18)	0,0430 (18)	0,0051 (12)	−0,0039 (15)	−0,0123 (14)
N7	0,051 (2)	0,046 (2)	0,0264 (17)	−0,0149 (19)	−0,0051 (18)	−0,0172 (15)
C5	0,044 (2)	0,036 (3)	0,072 (3)	−0,022 (2)	−0,006 (2)	0,0128 (18)
C6	0,0368 (18)	0,060 (3)	0,0309 (18)	−0,0007 (17)	0,0114 (16)	0,0095 (17)
C29	0,051 (2)	0,028 (2)	0,051 (2)	−0,0166 (18)	0,000 (2)	−0,0020 (15)
C30	0,057 (3)	0,051 (2)	0,0242 (17)	−0,022 (2)	0,0044 (18)	0,0000 (16)
C4	0,0173 (17)	0,026 (2)	0,0163 (13)	−0,0034 (13)	0,0005 (12)	−0,0016 (12)

Table 4. Main interatomic distances (Å) for $[(C_{10}O_8H_2)_2(C_4N_2H_6)].2H_2O$

Atoms	distance	atoms	distance
C1—C8	1.531 (6)	C37—O38	1.257 (6)
C1—C34	1.429 (6)	C37—O1	1.263 (6)
C1—C35	1.406 (6)	O25—C43	1.289 (7)
C2—C26	1.369 (6)	C28—C39	1.393 (6)
C2—C34	1.400 (6)	C28—C41	1.399 (7)
C2—H21	1.007 (7)	C28—C43	1.501 (6)
C3—C24	1.392 (6)	O33—C40	1.252 (6)
C3—C33	1.504 (6)	O36—C43	1.256 (6)
C3—C39	1.419 (6)	C39—H391	0.989 (6)
O5—C23	1.293 (6)	C40—C41	1.525 (7)
O7—C40	1.212 (7)	C41—C45	1.391 (6)

C8—O10	1.326 (6)	C45—H451	0.994 (6)
C8—O14	1.181 (7)	C46—C4	1.514 (6)
O11—C4	1.299 (6)	O39—C4	1.183 (6)
O13—H11	1.028 (7)	O42—H10	0.871 (7)
O13—H18	0.988 (6)	O42—H16	0.961 (7)
O15—C17	1.346 (7)	N4—C5	1.469 (9)
C17—C26	1.492 (6)	N4—C30	1.429 (8)
C17—O27	1.176 (7)	N7—C6	1.557 (9)
O22—C23	1.281 (7)	N7—C29	1.502 (9)
C23—C34	1.503 (6)	C5—C6	1.437 (10)
C24—C37	1.498 (6)	C5—H51	1.003 (7)
C24—C45	1.408 (5)	C6—H61	0.995 (7)
C26—C46	1.395 (6)	C29—C30	1.551 (9)
C33—O4	1.317 (7)	C29—H291	1.006 (7)
C33—O6	1.239 (7)	C29—H292	0.987 (9)
C35—C46	1.369 (6)	C30—H301	1.026 (7)
C35—H351	1.011 (7)	C30—H302	0.986 (8)

Table 5. Main bonds angles (deg) for $[(C_{10}O_8H_2)_2(C_4N_2H_6)].2H_2O$

Atoms	Angle	Atoms	Angle
C8—C1—C34	126.6 (4)	C3—C39—H391	118.999
C8—C1—C35	114.2 (4)	C28—C39—H391	119.324
C34—C1—C35	119.1 (4)	O33—C40—O7	124.6 (5)
C26—C2—C34	123.4 (4)	O33—C40—C41	119.1 (5)
C26—C2—H21	118.894	O7—C40—C41	116.2 (4)
C34—C2—H21	117.735	C40—C41—C28	127.0 (4)
C24—C3—C33	124.9 (4)	C40—C41—C45	113.5 (4)
C24—C3—C39	118.8 (4)	C28—C41—C45	119.6 (4)
C33—C3—C39	116.4 (4)	C28—C43—O25	118.9 (4)
C1—C8—O10	117.8 (4)	C28—C43—O36	123.5 (4)
C1—C8—O14	118.4 (5)	O25—C43—O36	117.7 (5)
O10—C8—O14	123.7 (5)	C24—C45—C41	121.6 (4)
H11—O13—H18	106.783	C24—C45—H451	118.553
O15—C17—C26	113.4 (4)	C41—C45—H451	119.831
O15—C17—O27	122.6 (5)	C26—C46—C35	120.1 (4)
C26—C17—O27	124.0 (4)	C26—C46—C4	121.6 (4)
O5—C23—O22	120.6 (5)	C35—C46—C4	118.2 (4)
O5—C23—C34	121.0 (5)	H10—O42—H16	107.385
O22—C23—C34	118.4 (4)	C5—N4—C30	111.2 (5)

C3—C24—C37	120.6 (4)	C6—N7—C29	109.5 (5)
C3—C24—C45	119.3 (4)	N4—C5—C6	113.0 (5)
C37—C24—C45	120.2 (4)	N4—C5—H51	122.738
C17—C26—C2	120.8 (4)	C6—C5—H51	124.274
C17—C26—C46	120.2 (4)	N7—C6—C5	109.8 (5)
C2—C26—C46	119.0 (4)	N7—C6—H61	125.012
C3—C33—O4	114.4 (4)	C5—C6—H61	125.089
C3—C33—O6	122.7 (5)	N7—C29—C30	107.3 (5)
O4—C33—O6	122.8 (4)	N7—C29—H291	108.896
C23—C34—C1	128.0 (4)	C30—C29—H291	110.119
C23—C34—C2	115.1 (4)	N7—C29—H292	110.352
C1—C34—C2	116.9 (4)	C30—C29—H292	110.113
C1—C35—C46	121.5 (4)	H291—C29—H292	109.980
C1—C35—H351	118.816	C29—C30—N4	111.5 (5)
C46—C35—H351	119.712	C29—C30—H301	108.620
C24—C37—O38	119.8 (4)	N4—C30—H301	107.535
C24—C37—O1	114.8 (4)	C29—C30—H302	110.184
O38—C37—O1	125.3 (4)	N4—C30—H302	110.321
C39—C28—C41	119.1 (4)	H301—C30—H302	108.531
C39—C28—C43	113.7 (4)	C46—C4—O11	112.0 (4)
C41—C28—C43	127.2 (4)	C46—C4—O39	121.2 (5)
C3—C39—C28	121.7 (4)	O11—C4—O39	126.5 (5)

Table 6. Distances and hydrogen bond angles in $[(C_{10}O_8H_2)_2(C_4N_2H_6)].2H_2O$.

D*—H···*A	***D*—H**	**H···*A***	***D*—H···*A***
O42—H10···O33^{I}	0.871 (6)	2.053 (4)	179.2 (4)
O13—H11···O5^{II}	1.028 (6)	2.045 (5)	175.4 (3)
O42—H16···O22^{III}	0.961 (5)	2.073 (5)	159.1 (3)
O13—H18···O7	0.988 (5)	2.051 (5)	155.4(3)

3. Results and discussion

In this paper, we recommend a new adduct of piperazine and 1, 2, 4, 5-benzenetetracarboxylic acid (btec), $[(C_{10}O_8H_2)_2(C_4N_2H_6).2H_2O]$ **(I)**, in which there are lots of hydrogen bonds and π–π interactions.

The structure of the title compound comprises two piperazine cations, a 1, 2, 4, 5-benzenetetracarboxylate anion and two water molecules. The structure of the title compound comprises two piperazine cations, two 1, 2, 4, 5-benzenetetracarboxylate anion and two water molecules **(figure 2)**. Two carboxyl groups of 1, 2, 4, 5-benzenetetracarboxylic acid are all deprotonated and a piperazine-ring accepted two proton coming from two 1, 2, 4, 5-benzenetetracarboxylic acid molecule to produce the piperazine cation. It is noteworthy that hydrogen bonding and intermolecular weak interactions play an important role in the structure of the title compound, as shown in **figure 2**. There are several kinds of hydrogen bonding are present in the structure: (a) hydrogen bonding between water molecules are: O(42)-H(10) (0.871Å), O(42)-H(16) (0.961Å), O(13)-H(11) (1.028Å), and O(13)-H(18) (0.988Å); (b) hydrogen bonding of piperazine molecule are in the range 0.986 - 1.026 Å; (c) hydrogen bonding of carboxylate molecules are in the range 0.989 – 1.011 Å.

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In **I**, C-N bonds of the molecule piperazine are C(5)–N(4), C(30)–N(4), C(6)–N(7), C(29)–N(7) are 1.469(9) Å, 1.429(8) Å, 1.557(9) Å and 1.502(9) Å, respectively. These bond distances are shorter enough than the sum of Van der Waals radii between C atom and N atom, C–N bonds (1.47–1.50 Å). The piperazine cyclic bond lengths C(5)–C(6) and C(29)–C(30) are elongated to 1.437(5) Å and 1.551(5) Å, Meanwhile, the C-C distance from first btec are in the range 1.369(6) - 1.429(8) and the second group btec are in the range 1.391(6)- 1.419(6). The connectivity between these units gives rise to a two dimensional hybrid layered structure in the ac plane as shown in (Figurs 3 and 4).

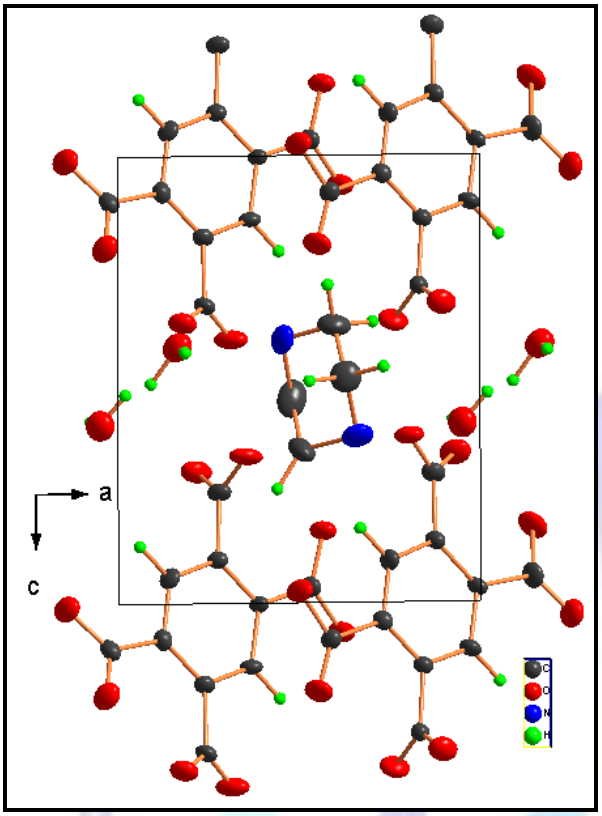

Figure 3. The projection structure of $[(C_{10}O_8H_2)_2(C_4N_2H_6)].2H_2O$, in the *ac* plane.

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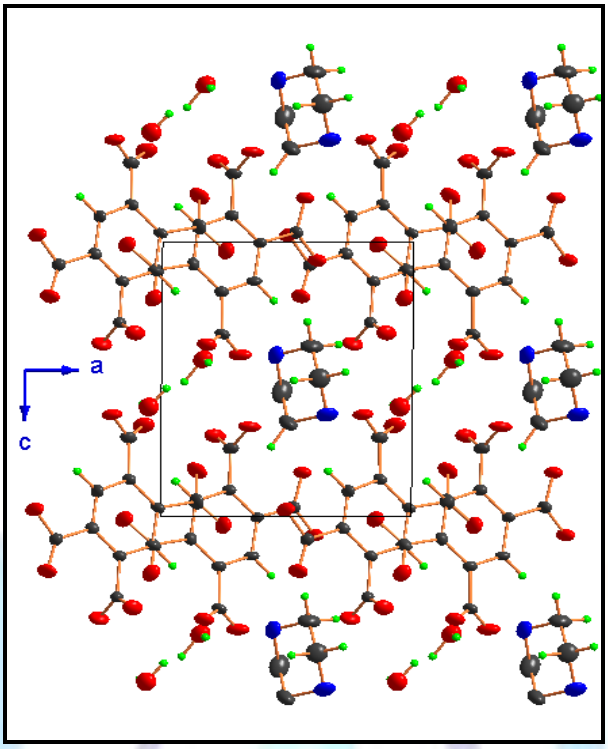

Figure 4. The projection structure of $[(C_{10}O_8H_2)_2(C_4N_2H_6)].2H_2O$, in the *ac* plane showing two layer.

4. Conclusion

In this work, we report a metal–organic complex $[(C_{10}O_8H_2)_2(C_4N_2H_6)].2H_2O$ **(I)**, which is prepared by the hydrothermal synthesis route. In the triclinic system, space group *P*1. Compound **I** exhibits a novel one-dimensional network constructed from bridging btec and piperazine mixed ligand.

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