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STUDY OF THE SUPRAMOLECULAR INTERACTIONS OF CARBOXYLIC ACIDS USED AS VERSATILE LIGANDS IN COORDINATION CHEMISTRY

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Carboxylate ligands are very versatile because they can adopt different coordination modes towards metal cations, such as monodentate, bidentate-chelate, bidentate bridging and monoatomic bridging. Because of this versatility, these ligands have been widely used in coordination chemistry. In this study the three dimensional structure of the crystal arrangement of 2,5-thiophenedicarboxylic (**H_2TPD**), trans-3-(3-pyridyl)acrylic (**HPYA**), and 4,4′-sulfonyldibenzoic (**H_2SFD**) acids are determined in order to investigate their supramolecular properties for coordination polymers and metal-organic framework (MOF) constructions.

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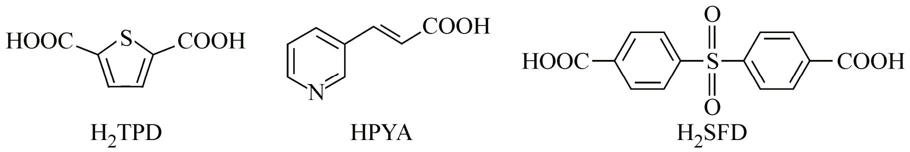
INTRODUCTION

In the last decade, carboxylate ligands have been widely explored in coordination chemistry, through the formation of coordination polymers (CPs) and supramolecular arrangement with different and interesting coordination modes in respect to a metallic center [1]. CPs are defined as metal-organic polymers in which the repeating coordination entities, build up with metal ion centers (nodes) and organic molecules (spacers or linkers), are linked through covalent bonds [2]. CPs have potential applications in gas storage [3—5], separation processes [6], luminescence and magnetism [7—9], electricity [10], sensor technology [11, 12] and catalysis [12—15]. The supramolecular chemistry is based on hydrogen bonds, van der Waals interactions, and π—π interactions. In the last years, the formation of supramolecular arrangements have been explored due to their properties and applications in catalysis [16], magnetic [17, 18] electrical-conductive [19], and host-guest chemistry [20]. Indeed, the combination of coordination chemistry and non-covalent interactions provides a powerful method for creating complex and interesting structures from simple building blocks [21].

Multidentate carboxylate ligands that, apart from the O donor, also have N and/or S donors are candidates to form 1D, 2D, and 3D CPs through of the coordination to a metallic center [22]. Due to these characteristics, 2,5-thiophenedicarboxylic acid (**H_2TPD**), trans-3-(3-pyridyl)-acrylic acid (**HPYA**), and 4,4′-sulfonyldibenzoic acid (**H_2SFD**) shown in Scheme 1, have already been used in coordination chemistry [1, 23—27].

H_2TPD, is a multicarboxylate ligand with an S donor, generally considered to be aromatic, that obeys the $4n+2p$ electron rule. Owing to the larger radius of sulfur compared to carbon, nitrogen, and oxygen atoms, a sulfur electron pair is easily delocalized over the heterocycle, and the ligand exhibits a good charge-transfer ability [28]. The chemistry of H_2TPD-like derivatives is consequently domi-



Scheme 1. Structures of H_2TPD, HPYA, and H_2SFD carboxylic acids

nated by this effect. In the industry, H_2TPD and some derivatives are used in the preparation of fluorescent brightening agents [29, 30].

HPYA belongs to a representative class of hybrid spacers commonly used in CPs and supramolecular arrangements [21]. The presence of both pyridine and carboxylate functions gives to this ligand the maximum ability to cope with the demands of different metals and adapt to different forms of metal connections necessary for the formation of CPs. The pyridine donor can be further adjusted by the nature and position of the substituent on the heterocyclic ring, whilst the ligand character of the carboxylate group is also influenced by the electronic state of the pyridine ring. Furthermore, the rotation freedom of 120° of the acrylate group relative to the pyridyl moiety imparts to this ligand a large variety of coordination modes [26]. In conjunction, the chemical and stereo chemical properties of HPYA offer a simple and yet powerful mechanism to meet various geometric demands of metals and the topological demands of the network [31].

Among the various classes of multidentate ligands, aromatic dicarboxylates such as H_2SFD have received the attention because of their ability to provide both structural rigidity and necessary charge balance [32]. H_2SFD acid is a V-shaped building block that presents six potential donor atoms, allowing the formation of complexes with various forms of coordination, different dimensions and topologies [25].

In this work, we report the crystal structure and the study of the supramolecular arrangement of H_2TFD, HPYA, and H_2SFD carboxylic acids.

EXPERIMENTAL

Materials and physical measurements. All the reagents and solvents were used as purchased without further purification. Infrared spectra were recorded under ambient conditions using KBr pellets in the wavenumber range 4000—400 cm^{-1} in a Bomen MB102 spectrophotometer with an average of 64 scans and 4 cm^{-1} spectral resolution. Thermogravimetric analysis was performed in the nitrogen atmosphere (flow rate = 100 ml/min). Elemental analyses for C, H, N, and S were carried out using a Perkin-Elmer 2400CHN analyzer. The ^1H and ^{13}C nuclear magnetic resonance spectra were recorded on Bruker AC-300 operating at frequencies of 300 Hz,13 Hz, and 75.43 Hz respectively.

Crystallization of H_2TPD. In an attempt to prepare a coordination polymer of H_2TPD and Co(II) ions, 2,5-thiophenedicarboxylic acid (30 mg, 0.2 mmol) were dissolved in 10 ml of DMF and an aqueous solution containing $CoCl_2 \cdot 6H_2O$ (40 mg, 0.2 mmol) was added through a diffusion method. After few weeks colorless single crystals of pure, 2,5-thiophenedicarboxylic acid suitable for the X-ray diffraction analysis were obtained. Yield: 32 %. Anal. Calcd. for $C_6H_4O_4S$: C, 41.86; H, 2.34; S, 18.63 wt %. Found: C, 41.16 and H, 2.35. IR (KBr, cm^{-1}): 3560(s), 3090(m), 2857(m), 2650(m), 2526(m), 1690(s), 1663(s), 1577(w), 1528(s), 1480(m), 1415(s), 1273(s), 1231(m), 1112(m), 1039(m), 933(m), 855(m), 756(s). 1H NMR data (δ, ppm; DMSO-d_6): 7.70 (2H, aromatic ring H).

Crystallization of HPYA. In an attempt to prepare a coordination polymer with mixed ligands and Cu(II) ions, *trans*-3-(3-pyridyl)-acrylic acid (75 mg, 0.5 mmol) and 3,4-pyridinedicarboxylic acid (84 mg, 0.5 mmol) were dissolved in 7 ml of DMSO. An ethanolic solution containing $CuCl_2 \cdot 6H_2O$ (43 mg, 0.25 mmol) was added to the acid solution through a diffusion method. After few weeks colorless single crystals of pure, *trans*-3-(3-pyridyl)-acrylic acid suitable for X-ray diffraction analysis were obtained. Yield: 16 %. Anal. Calcd. for $C_8H_7NO_2$: C, 64.42; H, 4.73; N, 9.39 wt. %. Found: C, 64.17; H, 4.47; N, 9.34. IR (KBr, cm^{-1}): 3490(br), 3049(w), 2921(w), 2804(m), 2447(m), 1705(s), 1641(s), 1578(m), 1418(m), 1313(vs), 1288(s), 1220(vs), 1173(s), 1049(s), 983(m), 721(w), 646(s).

^{1}H NMR data (δ, ppm; DMSO-d_6): 6.68 (1H, H—C=C—H), 7.58 (1H, H—C=C—H), 8.14(1H, aromatic C—H), 8.54 (1H, C—H), 8.56 (1H, aromatic C—H) and 8.81 (1H, aromatic C—H).

Crystallization of H$_2$SFD. In an attempt to prepare a coordination polymer of H$_2$SFD and Zn(II) ions, 4,4′-sulfonyldibenzoic acid (30 mg, 0.01 mmol) were dissolved in 5 ml of DMF and an ethanolic solution containing ZnCl$_2$ (13 mg, 0.01 mmol) was added through a diffusion method. After few weeks colorless single crystals of pure, 4,4′-sulfonyldibenzoic acid suitable for the X-ray diffraction analysis were obtained. Yield: 24 %. Anal. Calcd. for $C_{14}H_{10}O_6S$: C, 54.9; H, 3.29; S, 10.47 wt. %. Found: C, 54.46 and H, 3.1. IR (KBr, cm^{-1}): 3451(br), 3096(s), 2987(s), 2873(s), 2688(s), 2542(s), 1695(vs), 1578(m), 1493(m), 1424(s), 1331(m), 1310(s), 1286(s), 1161(s), 1125(m), 1100(s), 1069(m), 1015(m), 932(m), 865(s), 771(m), 722(s). ^{1}H NMR data (δ, ppm; DMSO-d_6): 8.06—8.14 (8H, aromatic CH).

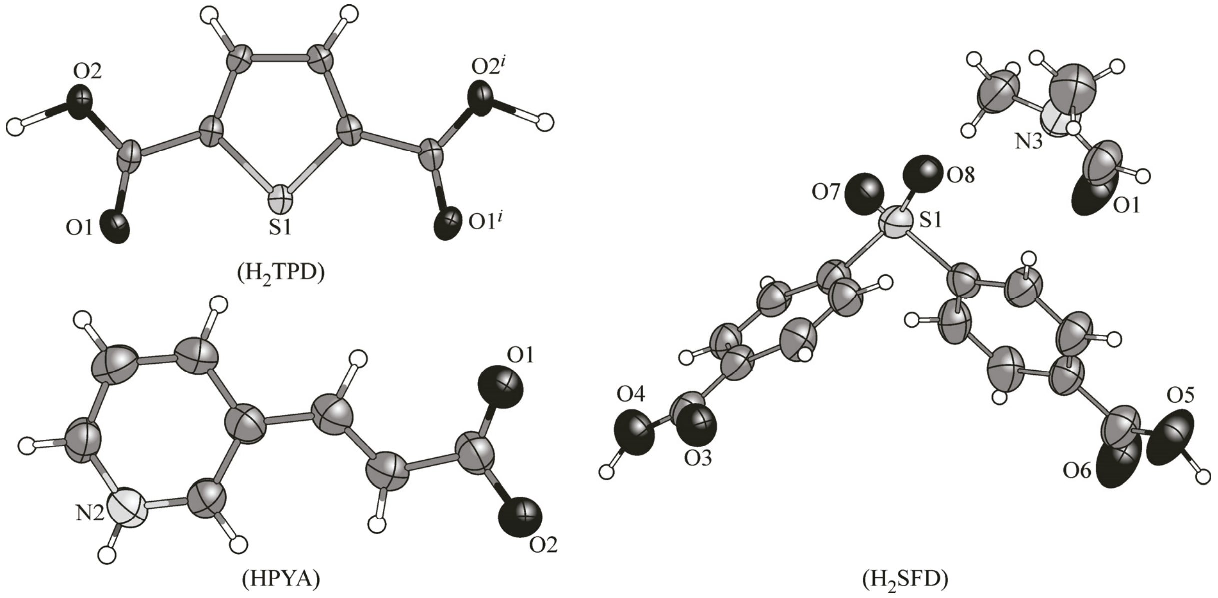
Single crystal X-ray diffraction. For all investigated samples, the X-ray diffraction data integration and scaling of reflection intensities were performed using the *Crysalis* suite [33]. Final unit cell parameters were based on the fitting of all measured reflection positions. Analytical [34] and semi-empirical [35] absorption corrections were performed using the *Crysalis* suite [33]. The XPREP program [36] was used in the space group identification and final data reduction. The structures of all compounds were solved by direct methods using the SIR92 program [37]. For each compound, the positions of all but hydrogen atoms could be unambiguously assigned on consecutive difference Fourier maps. Refinements were performed using SHELXL2013 [38] based on F^2 through a full-matrix least squares routine. All but hydrogen atoms were refined with anisotropic atomic displacement parameters. The hydrogen atoms in the compounds were added in the structure in idealized positions and further refined according to the riding model [39] $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{O})$ for aromatic molecules and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for methyl groups.

The sample characteristics, data collection, and refinement parameters for representative temperature measurements are indicated in Table 1. ORTEP-3 [40] for Windows and Mercury [41] were used to construct the illustrations of the molecules.

Table 1

Crystal data of H$_2$TPD, HPYA, *and* H$_2$SFD *acids*

Compounds	H$_2$TPD	HPYA	H$_2$SFD
Formula	$C_6H_4O_4S$	$C_8H_7NO_2$	$C_{17}H_{17}NO_7S$
Formula weight, g·mol^{-1}	172.16	149.15	379.37
Temperature, K	298(2)	290(2)	270(2)
Crystal system; space group	Monoclinic; *C*2/*c*	Monoclinic; *Pc*	Triclinic; *P*-1
a, Å	12.2136(17)	3.9852(4)	7.4666(4)
b, Å	5.8945(7)	7.3896(6)	8.0513(5)
c, Å	9.7129(15)	12.1600(10)	16.6868(9)
α, deg.	90	90	77.633(5)
β, deg.	107.608(17)	92.817(9)	88.519(5)
γ, deg.	90	90	64.508(6)
V, Å^3; *Z*	666.50(17); 4	357.67(5); 2	881.96(10); 2
Crystal size, mm	0.25×0.19×0.07	0.88×0.39×0.05	0.30×0.13×0.03
D_{calc}, g·cm^{-3}	1.716	1.385	1.429
μ(MoK_α), cm^{-1}	0.440	0.101	1.998
Transmission factors (min / max)	0.728 / 0.918	0.656 / 0.957	0.502 / 0.896
Reflections measured / unique / observed	2088 / 688 / 493	4215 / 1424 / 1082	17011 / 3090 / 2318
No. of parameters refined	60	110	248
$R[F_0 > 2\sigma(F_0)]$ / $wR[F_0^2 > 2\sigma(F_0)^2]$	0.0528 / 0.1233	0.0420 / 0.1014	0.0453 / 0.1150
S; RMS peak	1.082; 0.078	1.074; 0.032	1.043; 0.046



(H_2TPD) (HPYA) (H_2SFD)

Fig. 1. Molecular structure of (H_2TPD), (HPYA), and (H_2SFD). Displacement ellipsoids are drawn at the 50 % probability level

RESULTS AND DISCUSSION

Crystallizations. Single crystals investigated in the present work were obtained through diffusion reactions between ethanolic solutions containing metal ions (Co^{2+}, Cu^{2+}, and Zn^{2+}) and DMSO or DMF solutions containing the carboxylic acids at room temperature.

The crystal structure of H_2TPD acid was not found in the literature. HPYA acid was crystallized in the zwitterionic form in the *Pc* space group. However, the neutral form of HPYA acid in the $P2_1/c$ ($Z = 4$) space group with different lattice parameters has been already known (CCDC CIKXOK) [42]. The H_2SFD acid structure is reported in the literature (CCDC YIFNAD) [43], but in this work, DMF molecules are present in the net, together with H_2SFD acid, resulting in a new supramolecular arrangement.

Crystal structures. In an attempt to obtain coordination polymers, the H_2TPD, HPYA, and H_2SFD ligands were crystallized and their structures determined by the single crystal X-ray diffraction analysis are shown in Fig. 1.

The supramolecular arrangement of H_2TPD acid is formed by average hydrogen bonds of the O—H···O type (O1—H1···O3 and O4—H4···O2 with heteroatomic distances of 2.627(9) Å and 2.645(9) Å respectively) and weak bonds of the C2—H2···S3 type with a C···S distance of 3.467(6) Å. The acid molecules interact with each other by hydrogen bonds of the O—H···O type, interspersed in opposite sides, forming dimers in the *ac* diagonal plane. The dimers form a ring by hydrogen bonds, whose pattern of the network can be described by the $R_2^2(8)$ graph-set symbol [44], as shown in Fig. 2. These molecules interact with each other forming a three-dimensional supramolecular network.

In HPYA compound the formation of a 2D supramolecular arrangement occurs through weak hydrogen bonds of the C—H···O type (d(C···O) = 3.515 Å) and N—H···O (d(N···O) = 2.6362 Å), Fig. 3. It was also observed that the zwitterionic form of this compound is predominant. The HPYA molecule adopts a planar conformation, with a maximum derivation from the mean molecular plane of 0.011 Å and the intramolecular C—H···O hydrogen bond (C3···O2 = 2.849 Å and C3—H3···O2 = = 105.30°), which leads to the formation of a ring described by $S(5)$. Unlike its acid form, the zwitterionic form shows an intermolecular hydrogen bond, generating two closed paths whose graph-set symbols can be described as $R_3^2(16)$ and $R_2^2(12)$, Fig. 3, The acid form described in the literature [42] presented three closed paths, symbolized by $R_2^2(7)$, $R_2^2(14)$ and $R_6^4(28)$. Both molecular forms gene-

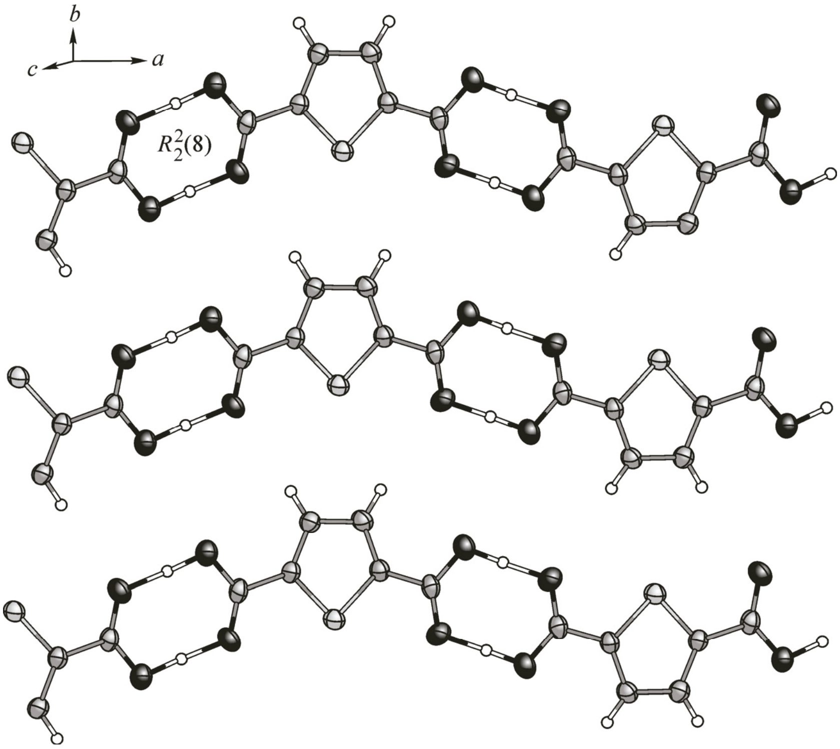


Fig. 2. Crystal packing of (H_2TPD), showing the hydrogen-bonded chain and the rings formed

rate *zigzag* supramolecular ribbons along the *b* direction; these interactions induce the self-assembly of parallel ribbons in an alternating head-to-tail fashion, resulting in a two-dimensional array in the *bc* plane. Only in the neutral form these 2D sheets interact with other by π—π stacking, generating a 3D supramolecular array along the *a* axis.

The supramolecular arrangement of H_2SFD acid presents DMF molecules embedded in the network. The acid structure shows the dihedral angle between two phthalic moieties of 80.44°. The acid molecule is a typical V-shaped dicarboxylate ligand, whose central S1 atom presents a tetrahedral arrangement, with the bond angles O8—S1···O7 = 119.78(12)°, O8—S1···C1 = 108.16(11)°, O7—S1···C1 = 108.34(12)°, O8—S1···C6 = 108.08(13)°, O7—S1···C6 = 107.95(11)°, and C1—S1···C6 = = 103.32(11)°. The presence of DMF molecules in the crystal lattice prevented the formation of a supramolecular arrangement similar to that reported in the literature [43], however, one of the carboxylate groups is connected by the hydrogen bonding to the neighboring molecule through a O5—H5w···O1 dimer while the other carboxylic group interacts with DMF molecules forming a 3D supramolecular network in the *zigzag* chain form (Fig. 4).

Thermal behavior. Hydrogen bonds of O—H···O and C—H···S types in the (HPYA) com

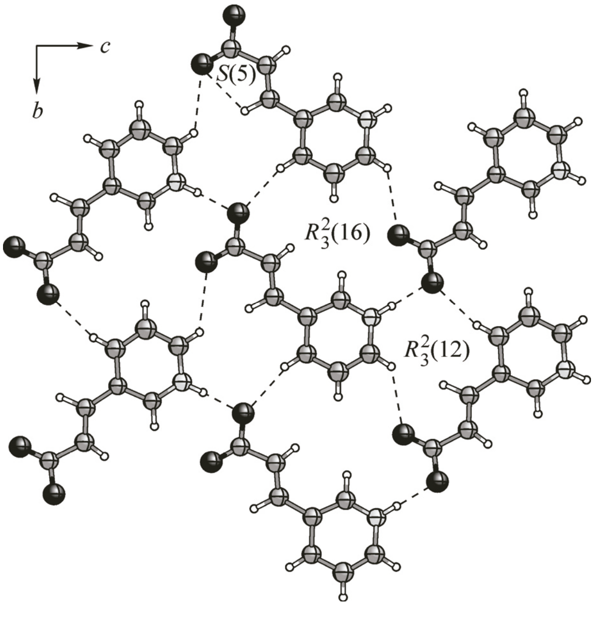


Fig. 3. A view in the *bc* plane of the hydrogen-bonded network observed in the crystal structure of (HPYA), showing the rings formed

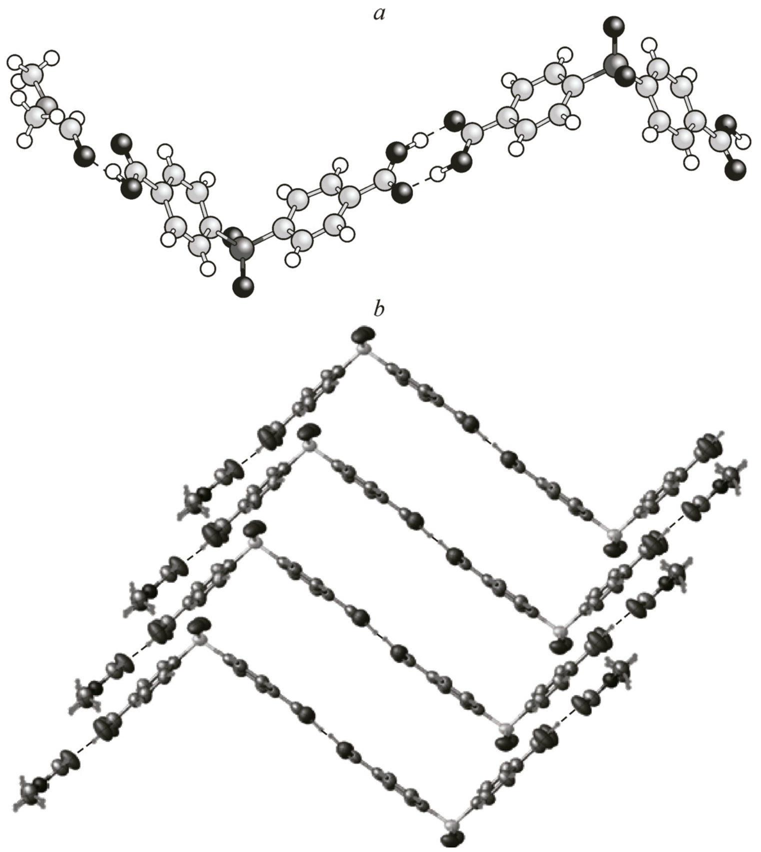
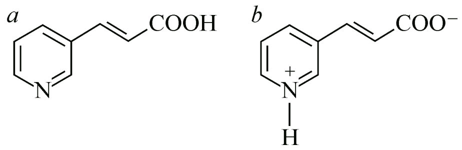


Fig. 4. 1D-chain formed from hydrogen bonds between carboxylate groups and DMF molecules (*a*), 3D arrangement of (H_2SFD) acid (*b*)

pound are responsible for the acid decomposition at a higher temperature range between 167 °C and 275 °C. The TG curve of the H_2SFD compound has two weight losses; the first of 17.31 %, observed between 96 °C and 118 °C corresponding to the network DMF release (calc. 19.26 %) and the second to the acid decomposition.

Infrared study. The structure of HPYA acid revealed the presence of the tautomeric form (b) (Scheme 2) where the nitrogen atom of the pyridine ring is protonated and the carboxylate oxygen atom appears deprotonated.



Scheme 2. Tautomeric forms of HPYA acid

In the infrared spectra of all acids, the bands at 1705 cm^{-1} referring to the carboxyl group νC=O vibrations are observed. In addition, for the spectrum of HPYA acid, bands at 2453 cm^{-1} and 1641 cm^{-1}, referring to νNH and δNH, respectively, corroborate the conclusion that the pyridine nitrogen atom is protonated. The spectrum of H_2SFD acid shows a band at 1603 cm^{-1} assigned to νCN vibrations in amides, indicating the presence of DMF in the network.

Crystallographic data for the structures reported in this paper have been deposited with the Cambridge Crystallographic Data Center. Copies of the data (CCDC 1400530-1400532) can be obtained free of charge upon application to CCDC, 12, Union Road, Cambridge CB2 1EZ, UK (e-mail: deposit@ccdc.cam.ac.uk).

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