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Isoniazid cocrystals with anti-oxidant hydroxy benzoic acids

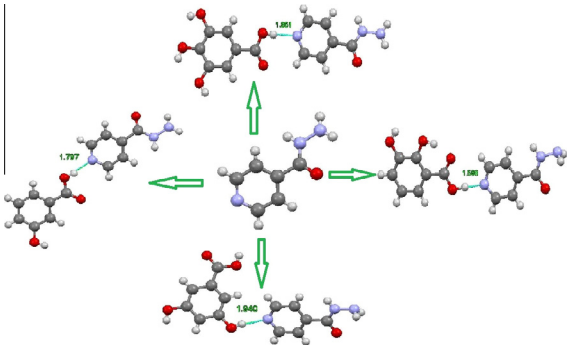
Syed Muddassir Ali Mashhadi [a], Uzma Yunus [a,*], Moazzam Hussain Bhatti [a], Muhammad Nawaz Tahir [b]

[a] Allama Iqbal Open University, Department of Chemistry, Islamabad, Pakistan
[b] University of Sargodha, Department of Physics, Sargodha, Pakistan

HIGHLIGHTS

- Cocrystals of isoniazid with four anti-oxidant hydroxy benzoic acids.
- Single crystal XRD studies of the isoniazid–hydroxy benzoic acid cocrystals.
- Infra-red (IR) studies of the isoniazid–hydroxy benzoic acid cocrystals.
- Differential Scanning Calorimetric (DSC) studies of the Isoniazid–hydroxy benzoic acid cocrystals.

GRAPHICAL ABSTRACT



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ABSTRACT

Isoniazid is the primary constituent of "triple therapy" used to effectively treat tuberculosis. In tuberculosis and other diseases, tissue inflammation and free radical burst from macrophages results in oxidative stress. These free radicals cause pulmonary inflammation if not countered by anti-oxidants. Therefore, in the present study cocrystals of isoniazid with four anti-oxidant hydroxy benzoic acids have been reported. Gallic acid, 2,3-dihydroxybenzoic acid, 3,5-dihydroxybenzoic acid, and 3-hydroxybenzoic acid resulted in the formation of cocrystals when reacted with isoniazid. Cocrystal structure analysis confirmed the existence of pyridine–carboxylic acid synthon in the cocrystals of isoniazid with Gallic acid, 2,3-dihydroxybenzoic acid and 3-hydroxybenzoic acid. While cocrystal of 3,5-dihydroxybenzoic acid formed the pyridine–hydroxy group synthon. Other synthons of different graph sets are formed between hydrazide group of isoniazid and coformers involving N—H···O and O—H···N bonds. All the cocrystals were in 1:1 stoichiometric ratio.

Introduction

Supramolecular Chemistry [1,2] gained attention when Nobel Prize of the year 1987 in chemistry was awarded to the founders of this field. This domain can be portrayed as "chemistry beyond the molecule" [3] and can be applied in the field of crystal engineering. It is described as "the understanding of intermolecular interactions in the context of crystal packing and the utilization of such understanding in the design of new solids with desired physical and chemical properties" [4]. Cocrystallization of two or more pure compounds by crystal engineering to create a new functional material such as pharmaceutical cocrystal is of great academic and industrial interest [5]. Cocrystal can be defined as "a stoichiometric multi component crystal in which all its components are neutral and solid under ambient conditions when in pure form" [6], and can be constructed through several types of interactions, including hydrogen bonding, pi-stacking, and van der Waals forces.

* Corresponding author. Address: Allama Iqbal Open University, Department of Chemistry, Sector H-8 Islamabad, Pakistan. Tel.: +92 519057755.
E-mail address: uzma_yunus@yahoo.com (U. Yunus).

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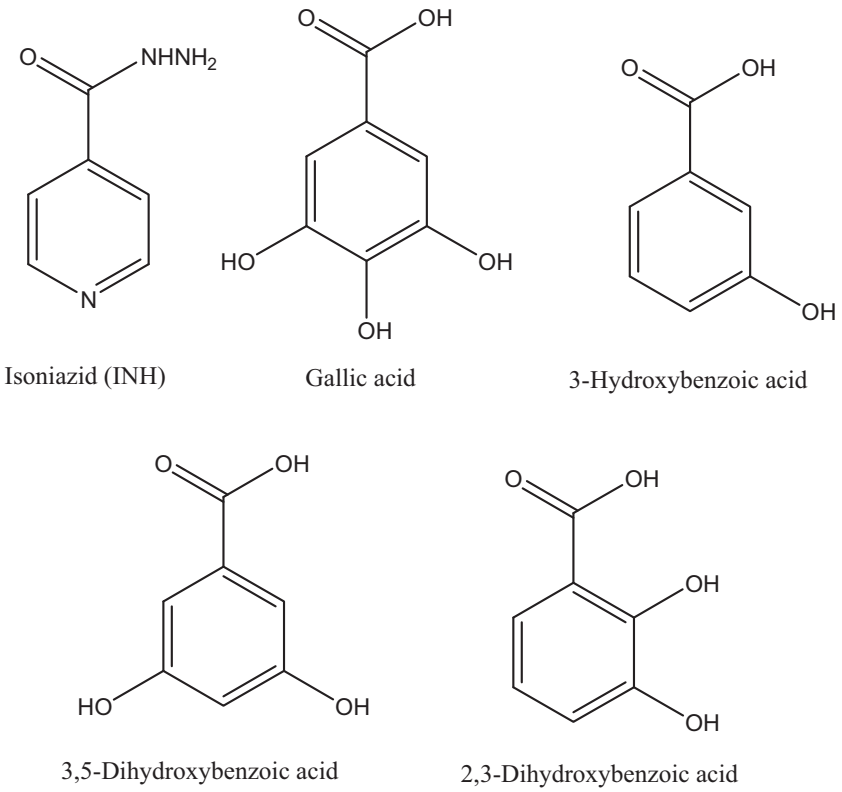

Fig. 1. Structural formulas of cocrystal components.

Pharmaceutical cocrystal is relatively new solid form of Active Pharmaceutical Ingredient (API) that has appealed the scientists as a mean of altering the physicochemical properties of API [7]. Pharmaceutical cocrystallization is an opportunity for the optimization of important physiochemical properties of an API while retaining its molecular structure and having improved bioavailability, increased resistance to hydrate formation, improved compaction properties for tablet formulation of two APIs into one dose. In spite of the fact that relationship between crystal structure and physicochemical properties of cocrystals has been studied [8–11], but still not completely understood. For example, no logical explanation of the variation in melting points of cocrystals can be given because of complicated nature of hydrogen bonding present in a cocrystals [12]. The melting point is also influenced by other intermolecular interactions, molecular conformations, packing, and entropy factors [13,14].

Study of hydrogen bond is heart of all intermolecular interactions [15]; it is energetic and directional [16,17] in nature and has been successfully identified in different environments among specific sets of functional groups having hydrogen bond formation capacity. It is therefore utilized in synthetic schemes to create specific assemblies [18–22]. Carboxylic acid–pyridine hydrogen bond in cocrystals is an established fact. It is due to strong donor and strong acceptor functionality of the carboxylic and pyridine functional groups respectively as described by Etter's rules for the formation of hydrogen bonds [23,24]. A numbers of cocrystals of isoniazid have been made with this hydrogen bond [25–30].

Isoniazid is highly active against *Mycobacterium tuberculosis* and is the primary constituent of "triple therapy" used to effectively treat tuberculosis since 1952. It is very versatile supramolecular reagent to synthesize novel supramolecular structures. Pyridine ring of INH is excellent hydrogen bond acceptor for carboxylic acids and bears possible attaching point for other heterosynthons. The carbohydrazide group of isoniazid has both functionalities of good hydrogen bonding acceptor in form of O and N atoms and donor in the form of three H atoms. Therefore, it is a potentially very important supramolecular reagent to synthesize cocrystals. Cocrystals of isoniazid having carboxylic-pyridine synthons with 4-aminosalicylic acid [31], 2-hydroxybenzoic acid, 4-hydroxybenzoic acid, 2,4-dihydroxybenzoic acid [32], malonic acid, succinic acid, glutaric acid, adipic acid, and pimelic acid [32–34] sebacic acid, suberic acid, cinnamic acid [35], tartaric acid [36], and 2,2-dithiodibenzoic acid have been reported.

Crystalline isoniazid is reported to be stable for long time periods however, its tablet formulations undergo degradation, particularly under high temperature and humid climatic conditions (40 °C, 75% RH) [37,38]. Exposure to light and presence of other drugs (pyrazinamide, ethambutol) being used in combination therapy also enhance isoniazid tablet's degradation [39,40]. It is therefore important to develop stable formulations of isoniazid.

In tuberculosis and other diseases, tissue inflammation and free radical burst from macrophages results in oxidative stress. These free radicals cause pulmonary inflammation if not countered by anti-oxidants [41–43]. On the other hand oxidation reaction is the main reason of degradation of molecules (APIs). The shelf life of pharmaceutical formulations depends on their ability to resist to oxidation of APIs. Anti-oxidants are used to stop the oxidation processes. Therefore, in the present study cocrystals of isoniazid with gallic acid, 2,3-dihydroxy benzoic acid, 3,5-dihydroxy benzoic acid and 3-hydroxy benzoic acid, which are known anti-oxidants, have been developed by slow evaporation method, and characterized by Fourier transform infrared spectroscopy (FTIR), single crystal X-ray diffraction (XRD) and Differential Scanning Calorimetry (DSC). Fig. 1 shows the structural formulas of the cocrystal components.

Table 1
IR spectral data of isoniazid and cocrystals.

Functional groups	Isoniazid (υ cm^{-1})	C-1 (υ cm^{-1})	C-2 (υ cm^{-1})	C-3 (υ cm^{-1})	C-4 (υ cm^{-1})
Asymmetric-NH_2 stretching	3430	3441	3420	3285	3303
Aromatic C—H stretching	3172	3140	3198	3145	3195
C=O stretching	1670	1674	1672	1700	1677
C=N stretching	1600	1614	1585	1608	1608
Aromatic ring vibration	1500, 1465	1523, 1469	1511, 1464	1534, 1493	1522, 1454
Pyridine ring	1410	1408	1412	1412	1410

Table 2
Single crystal XRD data of cocrystals (C-1–C-4).

Cocrystal	C-1	C-2	C-3	C-4
Empirical formula	$C_{26}H_{26}N_6O_{12}$	$C_{13}H_{13}N_3O_5$	$C_{13}H_{13}N_3O_5$	$C_{13}H_{13}N_3O_4$
Formula weight	614.53	291.26	291.26	275.26
Temperature (K)	296(2)	296(2)	296(2)	296(2)
Wavelength (Å)	0.71073	0.71073	0.71073	0.71073
Crystal system	Triclinic	Monoclinic	Monoclinic	Orthorhombic
Space group	P-1	P 21/c	P 21/n	P c a 21
Cell formula unit-Z	2	4	4	8
Unit cell dimensions				
a (Å)	8.7178(7)	13.638(10)	3.7659 (5)	25.165(2)
b (Å)	11.9273(8)	7.0669(5)	24.345(3)	4.8636(3)
c (Å)	13.396(10)	13.6823(11)	14.018(16)	21.2572(15)
α (°)	93.696(3)	90	90	90
β (°)	98.707(4)	95.126(4)	91.390(7)	90
γ (°)	108.217(4)	90	90	90
Volume (Å^3)	1298.54(17)	1313.4(17)	1284.8(3)	2601.7
Absorption coefficient (mm^{-1})	0.127	0.107	0.118	0.107
R factor (%)	5.81	3.95	4.97	4.87

Table 3
Hydrogen bond distances in isoniazid-cocrystals (C-1–C-4).

Atoms	D—H (A°)	H···A (A°)	D···A (A°)	<D—H···A (°)
Cocrystal C-1				
O1—H1···N6	0.280	1.826	2.645	176.37
N4—H4A···O3	1.937	2.525	2.853	103.6
N5—H5A···O10	0.860	2.525	2.937	132.4
O4—H4···N4	0.820	1.937	2.736	164.3
Cocrystal C-2				
O3—H3A···N3	1.040	1.600	2.636	176.0
N1—H1···O4	0.860	2.299	2.940	131.52
O5—H5A···N2	0.890	1.880	2.715	156.0
Cocrystal C-3				
O3—H3A···N1	0.820	1.907	2.714	168.2
O4—H4···N3	0.819	1.941	2.750	169.1
O1—H1···O3	0.821	1.855	2.651	172.5
Cocrystal C-4				
O1—H1···N3	0.820	1.846	2.649	166.4
N2—H2···O4	0.860	2.130	2.931	154.7

Experimental section

Substrates and reagents

All the chemicals were used as received from the supplier without any further purification. Solvents were dry distilled according to standard procedures before use. Isoniazid was purchased from Alfa Aeser, gallic acid, 2,3-dihydroxy benzoic acid, 3,5-dihydroxy benzoic acid and 3-hydroxy benzoic acid were purchased from Acros.

Melting point range was studied by using a Gallenkamp (UK) 50 Hz 220/240 volt melting point apparatus. The results were in close agreement with the results of Differential Scanning Calorimetry (DSC). DSC experiments were performed with Mettler Toledo instrument. The samples (15–20 mg) were heated in open aluminum pans at a rate of 10 °C/min in nitrogen (flow of 20.0 mL/min). DSC experiments were carried out to study the thermal behavior of the cocrystals. Melting point can also be determined from the melting curve. If the low temperature side of melting peak is almost a straight line, the melting point corresponds to the onset and if melting curve are concave in shape the melting point are characterized by the peak maxima. The Infrared spectra were recorded on Varian 640-IR spectrophotometer in ATR mode. Single crystal X-ray data was collected by using Bruker Kappa APEX II CCD diffractometer equipped with a graphite monochromator at 296 K. Fine focus of molybdenum Kα tube was used. Data was collected using APEX2 software, SAINT for indexing the reflections and determining the unit cell parameters. The structure was solved by direct methods and refined by full-matrix least square calculations using SHELXL-97 software. Structures of the cocrystals were drawn and other calculations were carried out by using Mercury 3.1 software.

Slow evaporation cocrystallization

Isoniazid–gallic acid cocrystal (C-1)

Isoniazid (0.137 g, 1 mmol) and gallic acid (0.170 g, 1 mmol) were dissolved separately in 20 ml of methanol each then mixed together at room temperature (22 °C). The solution was kept for slow evaporation for 15 days. Brown Prism crystals were isolated by filtration and dried in the air. Melting point was determined to be 221 °C.

Isoniazid–2,3-dihydroxybenzoic acid cocrystal (C-2)

Isoniazid (0.137 g, 1 mmol) and 2,6-dihydroxybenzoic acid (0.154 g,1 mmol) were dissolved separately in 20 ml solvent (etha-

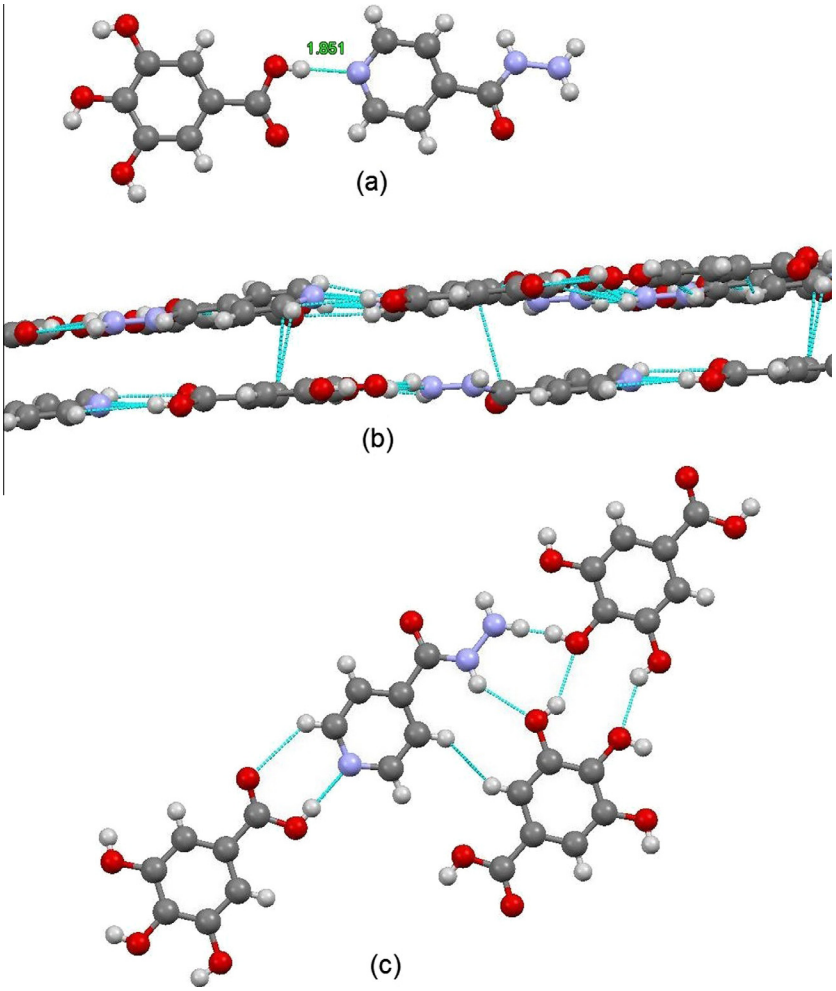

Fig. 2. The cocrystal structure of Isoniazid–gallic acid (C-1). (a) Hydrogen bonding between nitrogen of pyridine ring and hydrogen of carboxylic acid group, (b) the packing and layer structure of motif (C-1) and (c) graph sets in the cocrystal structure of (C-1).

nol:acetonitrile, 2:1) and then mixed together and heated up to 70 °C for 10 min. The solution was left for slow evaporation. Brown needle crystals were isolated after 11 days. Melting point of this cocrystal was 180 °C.

Isoniazid–3,5-dihydroxybenzoic acid cocrystal (C-3)

Isoniazid (0.137 g, 1 mmol) and 3,5-dihydroxybenzoic acid (0.154 g, 1 mmol) were separately dissolved in 20 ml each of the mixture of ethanol and acetonitrile (2:1) then mixed together. Solution was heated up to 70 °C for 10 min and then kept for slow evaporation for 09 days. White needle like crystals were isolated. Calculated melting point was 225 °C.

Isoniazid–3-hydroxybenzoic acid cocrystal (C-4)

Isoniazid (0.137 g, 1 mmol) and 3-hydroxybenzoic acid (0.138 g, 1 mmol) were separately dissolved in 20 ml of the mixture of ethanol and acetonitrile (2:1). The two solutions were mixed together and heated up to 70 °C for 10 min and then kept for slow evaporation for 09 days. Brown needle crystals were separated by filtration. Melting point of cocrystal was 132 °C.

Results and discussion

Cocrystallization of isoniazid with gallic acid, 2,3-dihydroxybenzoic acid, 3,5-dihydroxybenzoic acid and 3-hydroxybenzoic acid produced cocrystals. These were characterized by infra-red spectroscopy, single-crystal X-ray diffraction and thermal analysis. IR spectral data is given in Table 1. Crystallographic information is shown in Table 2 and information regarding hydrogen bonds of cocrystals is presented in Table 3. Crystal structures were deposited at the Cambridge Crystallographic Data Centre. The data have been assigned the following deposition numbers, CCDC 1005087-100509.

Isoniazid–gallic acid cocrystal (C-1)

The IR spectrum of cocrystal (C-1) shows stretching of N—H bond in the high wave number region with a sharp band of weak intensity at 3372 cm^{-1}. Another sharp band is present at 3140 cm^{-1}, which is attributed to the C—H (aromatic) stretching. C=O stretching vibration is present at 1674 cm^{-1} and C=N stretching is observed at 1614 cm^{-1}. Aromatic ring vibrations are attributed at 1523 cm^{-1} and 1469 cm^{-1}. C=N of pyridine ring shows stretching vibration at 1408 cm^{-1}. Cocrystal shows a distinct pattern than isoniazid and coformer.

The cocrystal (C-1) crystallizes in the triclinic space group P-1. Stoichiometry shows a discrete 1:1 adduct as gallic acid is hydrogen bonded to N of pyridine ring of isoniazid through O—H···N (Fig. 2a) having bond length 1.860 A°. The extended packing of these discreet adducts forms layers (Fig. 2b). The angle between the carboxyl group plane and the pyridine ring plane is 1.64° and results in a R_2^2 (7) ring motif. Another graph set of R_2^2 (10) results

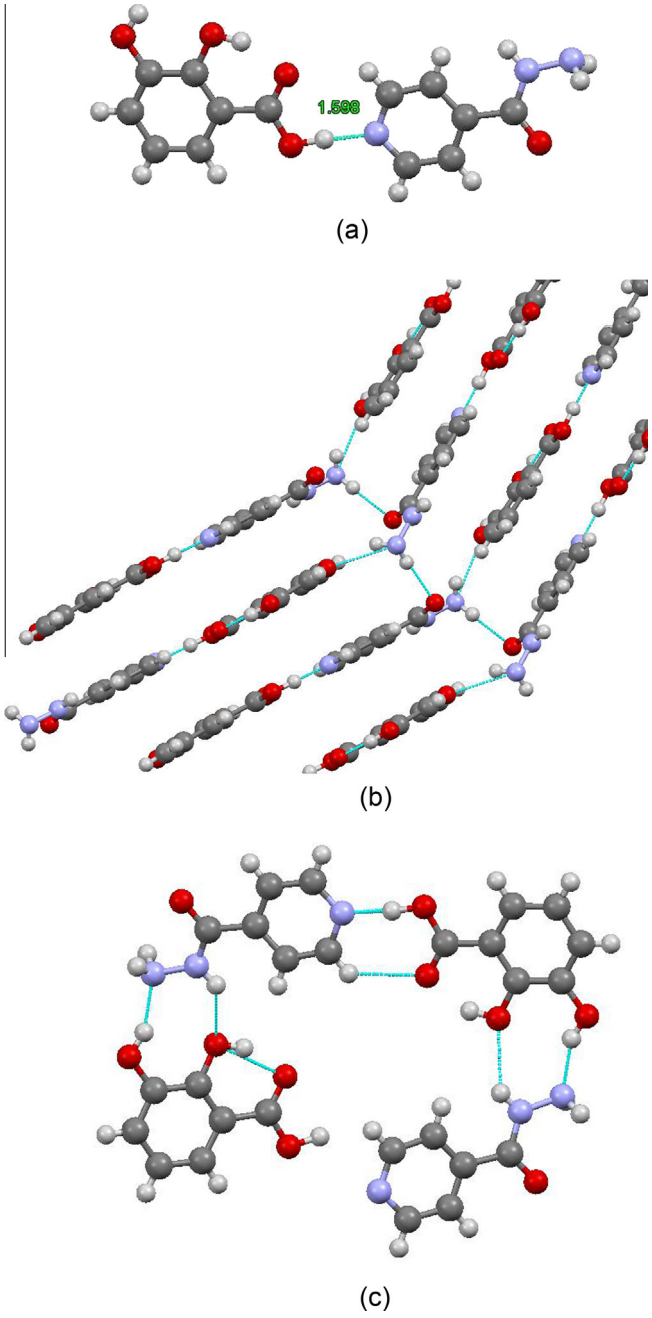

Fig. 3. Cocrystal of isoniazid and 2,3-dihydroxybenzoic acid (C-2). (a) Hydrogen bonding between nitrogen of pyridine ring and hydrogen of carboxylic acid group, (b) the packing and layer structure of motif (C-2) and (c) graph sets in the cocrystal structure of (C-2).

between two homosynthons of carboxylic acid. A heterosynthon is formed by the hydrogen bond of the terminal NH_2 and its adjacent NH with hydroxy groups of two gallic acid molecules having a graph set R_3^3 (7) (Fig. 2c).

The DSC thermogram of cocrystal (C-1) shows endothermic peak at 232 °C, thermal profile of molecular cocrystal is distinct showing different melting transition from either of the individual components. Onset value 221 °C is considered to be melting point which is in close agreement to the value taken by melting point apparatus. This indicates the formation of novel molecular complex. This single endothermic transition indicates the absence of any unbound or absorbed solvent or water and also demonstrates the stability of the phase until the melting point.

Isoniazid–2,3-dihydroxybenzoic acid cocrystal (C-2)

The IR spectrum of cocrystal (C-2) shows a sharp band at 3420 cm^{-1} for N—H bond stretching while sharp band at 3198 cm^{-1} is attributed to the C—H (aromatic) stretching vibrations. C=O stretching vibration is present at 1672 cm^{-1} and C=N stretching at 1585 cm^{-1}. Aromatic ring vibrations are observable at 1511 cm^{-1} and 1464 cm^{-1}. C=N bond of pyridine ring in cocrystal is identified at 1412 cm^{-1}.

The cocrystal (C-2) has monoclinic space group *P*21/c with molecular formula $C_{13}H_{13}N_3O_5$ and molar mass 291.26 amu. The cocrystal stoichiometry is a discrete 1:1 adduct where distinct hydrogen bonding is formed between nitrogen of pyridine ring of isoniazid and hydrogen of carboxylic group of acid (Fig. 3a) having bond length 1.602 A°. The extended packing of these discreet adducts forms layers (Fig. 3b). These findings correlate well with the predicted hydrogen bond interactions as found in the structures of previously reported cocrystals [31–36]. Intramolecular hydrogen bonding is also present. 2,3-Dihydroxy benzoic acid is hydrogen bonded to N of pyridine ring of isoniazid through O—H···N. The angle between the carboxyl group plane and the pyridine ring plane is 2.52° and results in a R_2^2 (7) ring motif. A hetero synthon is formed by the hydrogen bond of the terminal NH_2 and its adjacent NH with hydroxy groups of 2,3-dihydroxy benzoic acid having a graph set R_2^2 (8) (Fig. 3c).

DSC results of isoniazid and 2,3-dihydroxybenzoic acid cocrystal (C-2) shows endothermic peak at 189 °C. Onset value 180 °C is considered to be melting point which is in close agreement with the measured melting range in the melting point determination. The thermal profile of molecular cocrystal is distinct, with a different melting transition from either of the individual components. This indicates the formation of novel molecular complex. This single endothermic transition also indicates the absence of any unbound or absorbed solvent or water and also demonstrates the stability of the phase until the melting point.

Cocrystal of isoniazid and 3,5-dihydroxybenzoic acid (C-3)

The N—H bond in the high frequency region of the IR spectrum of cocrystal (C-3) shows a sharp band of medium intensity at 3285 cm^{-1}. Sharp band at 3145 cm^{-1} is due to the C—H (aromatic) stretching vibrations. C=O stretching vibration is present at 1700 cm^{-1} and C=N stretching at 1608 cm^{-1}. Aromatic ring vibrations are attributed at 1534 cm^{-1}, 1493 cm^{-1}. Pyridine C=N stretching is identified at 1412 cm^{-1}.

Cocrystal (C-3) crystallizes in the monoclinic crystal system having space group *P 21/n*. Molecular formula of cocrystal is determined to be $C_{13}H_{13}N_3O_5$ and molar mass 291.26 amu. Distinct 1:1 adduct is resulted by hydrogen bonding between nitrogen of pyridine ring of isoniazid and hydroxy group of 3,5-dihydroxybenzoic-acid (Fig. 4a) instead of carboxylic group with bond length 1.941 A°. Cocrystal shows distinct behavior as other cocrystals of isoniazid are formed by the hydrogen bonding between N of pyridine ring and carboxylic acid through O—H···N which is not the case for this particular cocrystal. The extended packing of these discreet adducts forms layers (Fig. 4b). The angle between the hydroxy group plane and the pyridine ring plane is 7.67°. A heterosynthon is formed by the hydrogen bond of the terminal NH_2 and its adjacent NH of isoniazid with carboxyl group of one molecule of 3,5-dihydroxybenzoic acid and hydroxy group of other molecule having a graph set R_3^3 (9) (Fig. 4c).

The DSC thermogram of isoniazid and 3,5-dihydroxybenzoic acid cocrystal (C-3) shows endothermic peak at 231 °C. Onset value 225 °C is considered to be melting point which is in close agreement to the value taken by melting point apparatus. The thermal profile of cocrystal is distinct, with a different melting transition

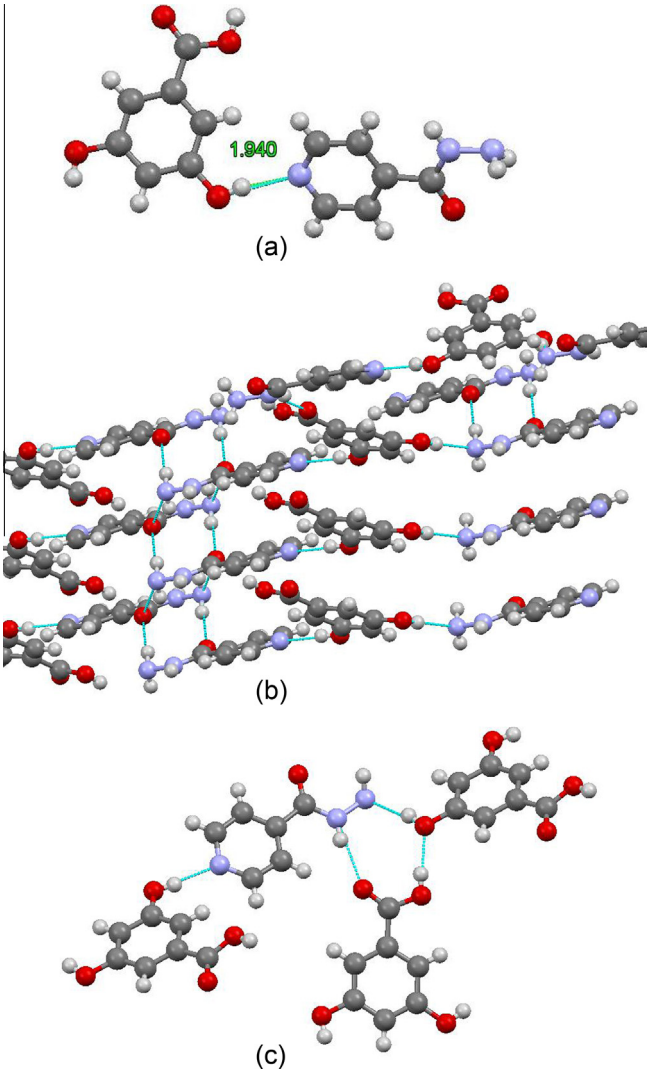

Fig. 4. Cocrystal of isoniazid and 3,5-dihydroxy benzoic acid (C-3). (a) Hydrogen bonding between nitrogen of pyridine ring and hydroxy group, (b) the packing and layer structure of motif (C-3) and (c) graph sets in the cocrystal structure of (C-3).

from either of the individual components. This indicates the formation of novel molecular complex. This single endothermic transition indicates the absence of any unbound or absorbed solvent or water and also demonstrates the stability of the phase until the melting point.

Cocrystal of isoniazid and 3-hydroxybenzoic acid (C-4)

IR spectrum of cocrystal (C-4) shows the stretching of N—H bond in the high frequency region at wave number 3303 cm^{-1}. Another sharp band is present at 3195 cm^{-1}, which is attributed to the C—H (aromatic) stretching vibrations. C=O stretching vibration is present at 1677 cm^{-1} and C=N stretching is observed at 1608 cm^{-1}. Aromatic ring vibrations are present at 1522 cm^{-1}, 1454 cm^{-1} while C=N stretching of pyridine is identified at 1410 cm^{-1}.

Cocrystal (C-4) crystallizes in the orthorhombic crystal system having space group *P c a 21*. Molecular formula of the cocrystal is $C_{13}H_{13}N_3O_4$ with molar mass 275.26 amu. The stoichiometry of cocrystal is a discrete 1:1 adduct where distinct hydrogen bonding is formed between nitrogen of pyridine ring of isoniazid and hydrogen of carboxylic group of acid having bond length 1.846 A°

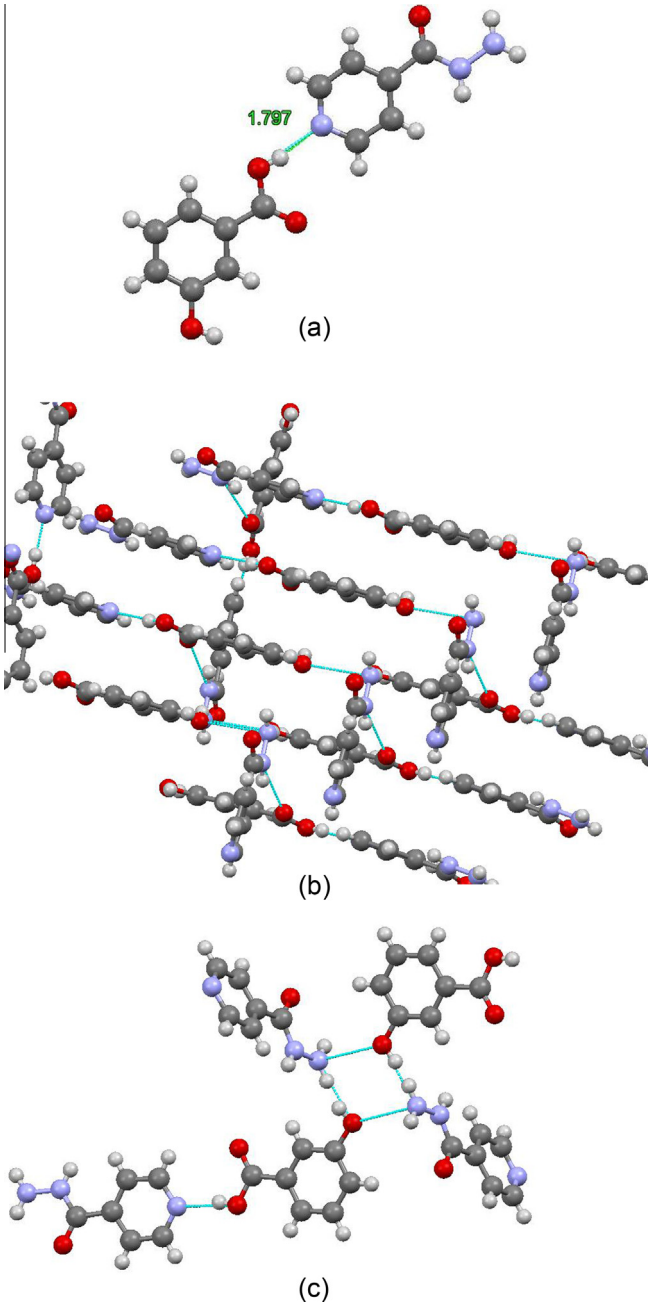

Fig. 5. Cocrystal of isoniazid and 3-hydroxybenzoic acid (C-4). (a) Hydrogen bonding between nitrogen of pyridine ring and hydrogen of carboxylic acid group, (b) the packing and layer structure of motif (C-4) and (c) graph sets in the cocrystal structure of (C-4).

(Fig. 5a). The extended packing of these adducts forms layers (Fig. 5b). 3-Hydroxybenzoic acid is hydrogen bonded to N of pyridine ring of isoniazid through O—H···N. The angle between the carboxyl group plane and the pyridine ring plane is 1.64° and results in a R_2^2 (6) graph set (Fig. 5c).

The distinct thermal profile of cocrystal shows endothermic peak at 140 °C. Onset value 132 °C is considered to be melting point which is in close agreement to the value obtained during melting point determination and show different pattern from either of the individual components. This confirms the formation of novel molecular complex and indicates the absence of any unbound or absorbed solvent or water and also demonstrates the stability of the phase until the melting point.

Conclusion

In the present study four cocrystals of isoniazid with hydroxybenzoic acids have been developed and characterized by infra-red spectroscopy, Differential Scanning Calorimetry and single crystal XRD. Cocrystal structure analysis reveals that formation of pyridine–carboxylic acid synthon and pyridine–hydroxy group synthon are the main reason of cocrystal development. The study reveals that cocrystals of isoniazid with anti-oxidant hydroxybenzoic acids can be developed easily and may serve better in reducing oxidative stress in tuberculosis patients during treatment as well as increasing stability of isoniazid tablets by decreasing oxidation processes due to the presence of anti-oxidants in the formulation.

Appendix A. Supplementary material

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.molstruc.2014.07.070.

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