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B··· π -aromatic and C–H···B interactions in co-crystals of aromatic amine *N*-oxides with *p*-phenylenediboronic acid

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ABSTRACT

Co-crystals of *p*-phenylenediboronic acid with four aromatic *N*-oxides namely pyridine *N*-oxide, quinoline *N*, *N*-dioxide, isoquinoline *N*, *N*'-dioxide and 4,4'-dipyridine *N*, *N*'-dioxide are prepared and are structurally characterized. The stacking pattern in each case is found to be different. An interesting stacking effect that, sandwiches the boronic acid groups by pyridine *N*-oxide rings and associated through η^2 and η^3 type B- π interactions is presented. In the structures of co-crystals of aromatic amine *N*-oxides with *p*-phenylenediboronic acid B··· π aromatic and C-H···B interactions are observed.

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1. Introduction

The weak interactions are of general interest in chemistry and biology [1–4]. Information gathered from structural studies of a particular class of compounds helps in establishing new interactions. The compounds containing boronic acid functional group are important as precursor for organic transformations [5-11] and they are also used in medicine [12-13]. Phenylboronic acids are toxic to microorganisms but they are relatively harmless to higher animals [14–17]. The hydroxy group of boronic acid can be replaced by various diols and based on this reactivity towards diols, sensors for sugars are devised [18–19]. Despite susceptibility of boronic acid towards organic reactions, a good numbers of hostguest chemistry of boronic acids are available [20-25]. The selfassemblies of boronic acid have structural features that are similar to analogous carboxylic acid and structural comparisons are being made [26–30]. The framework structures of boronic acids with 4,4'-bipyridine type linkers are studied [31]. On the other hand the *N*-oxides are known to form inclusion compounds [32–34] and some N-oxide co-crystals are of medicinal importance [35-36]. Boron as a covalent center in a molecule has high affinity for oxygen, thus, framework structures constructed with N-oxides are expected to have new weak interactions. A nucleophile would interact with boronic acid at the electron deficient boron center and also with OH groups to form H-bond, thereby leading to chemical reactions or host-guest complexes. With these objectives we have studied the structural aspects of four co-crystals of aromatic amine *N*-oxide with *p*-phenylenediboronic acid.

2. Materials and methods

2.1. General

The *p*-phenylenediboronic acid, pyridine *N*-oxide, quinoline *N*-oxide, isoquinoline *N*, N'-dioxide, and 4,4'-bipyridine *N*, N'-dioxide were obtained from Sigma–Aldrich Chemical Co., USA and directly used without further purifications.

UV-visible spectra were recorded on a Perkin-Elmer Lambda 750 UV/vis spectrometer.

¹H NMR spectra were collected in a Varian 400 MHz FT-NMR instrument. The X-ray single crystal diffraction data were collected at 296 K with Mo_{Ka} radiation ($\lambda = 0.71073$ Å) using a Bruker Nonius SMART CCD diffractometer equipped with graphite monochromator. The SMART software was used for data collection and also for indexing the reflections and determining the unit cell parameters; the collected data were integrated using SAINT software. The structures were solved by direct methods and refined by full-matrix least-squares calculations using SHELXTL software. All the non-H atoms were refined in the anisotropic approximation against F^2 of all reflections. The H-atoms, except those attached to oxygen atoms were placed at their calculated positions and refined in the isotropic approximation; those attached to oxygen were located in the difference Fourier maps, and refined with isotropic displacement coefficients. The crystal data for the co-crystals are given in Table 1.



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Table 1		
Crystallographic	parameters of	the co-crystals.

Compound No.	А	В	C	D
Formulae	$C_{16}H_{18}B_2N_2O_6$	$C_{24}H_{22}B_2N_2O_6$	$C_{42}H_{36}B_2N_4O_8$	$C_{16}H_{16}B_2N_2O_6$
Mol. wt.	355.94	456.06	746.37	353.93
Crystal system	Monoclinic	Triclinic	Triclinic	Triclinic
Space group	P21/c	PĪ	PĪ	ΡĪ
a/Å	10.685 (13)	6.807 (16)	8.104 (5)	6.669 (8)
b/Å	7.111 (9)	7.777 (2)	10.057 (6)	7.598 (10)
<i>c</i> /Å	12.140 (14)	11.578 (4)	12.627 (7)	8.285 (11)
α/°	90.00	106.447 (12)	84.15 (4)	100.16 (11)
β/°	115.05 (3)	99.51 (12)	78.47 (4)	106.99 (9)
γl°	90.00	101.72 (9)	67.19 (4)	92.94 (9)
V/Å ³	835.6 (18)	559.2 (3)	929.17 (9)	392.85 (9)
Ζ	2	1	1	1
Density/Mgm ⁻³	1.415	1.354	1.334	1.496
Abs. Coeff. /mm ⁻¹	0.106	0.096	0.092	0.112
Abs. correction	None	None	None	None
F(000)	372	238	390	184
Total no. of reflections	6351	4359	5296	2963
Reflections, $I > 2\sigma(I)$	1632	2695	2431	1424
Max. 2 $ heta/^\circ$	26.50	28.38	23.10	25.48
Ranges (h, k, l)	−13 <= <i>h</i> <= 13, −6 <= <i>k</i> <= 8,	<i>−</i> 9 <= <i>h</i> <= 3, <i>−</i> 8 <= <i>k</i> <= 10,	<i>−</i> 8 <= <i>h</i> <= 8, <i>−</i> 11 <= <i>k</i> <= 11,	<i>−</i> 8 <= <i>h</i> <= 7, <i>−</i> 9 <= <i>k</i> <= 9,
	−15 <= <i>l</i> <= 14	−15 <= <i>l</i> <= 15	−13 <= <i>l</i> <= 13	−10 <= <i>l</i> <= 10
Complete to 2θ (%)	94.9	96.1	93.3	97.6
Refinement method	Full-matrix least-squares on F ²	Full-matrix least-squares on F ²	Full-matrix least-squares on F ²	Full-matrix least-squares on F^2
Data/restraints/ parameters	1632/0/118	2695/0/156	2431/0/256	1424/0/120
Goof (F^2)	1.529	1.074	1.069	1.067
R indices $[I > 2\sigma(I)]$	0.1093	0.0458	0.0710	0.0454
R indices (all data)	0.1304	0.0626	0.0821	0.0622

2.2. Spectroscopic data for the co-crystals

By mixing *p*-phenylenediboronic acid (0.165 g, 1 mmol) and pyridine N-oxide (0.190 g, 2 mmol) in a mixed solvent methanol/toluene (4:1) gave the corresponding 1:1 co-crystals (A). The crystals were obtained after seven days. Yield (crystals) 46%. IR (KBr, cm⁻¹): 3209 (b), 1615 (m), 1510 (m), 1472 (m), 1423 (m), 1401 (m), 1369 (s), 1340 (s), 1235 (s), 1192 (s) 1165 (s), 1192 (m), 1139 (m), 1094 (m), 1058 (m), 836 (s), 759 (m). ¹H NMR (DMSO-d₆, 400 MHz): 8.2 (2H, d, J = 8.0 Hz), 8.0 (4H, s), 7.7(4H,s), 7.4 (2H, t, J = 8.0 Hz), 7.3 (1H, t, J = 8.0 Hz). Layering an aqueous solution of pphenylenediboronic acid (0.165 g, 1 mmol) over a quinoline N-oxide (0.290 g, 2 mmol) solution in toluene yielded pure crystalline cocrystals (B) (yield, 26%). IR (KBr, cm⁻¹): 3303 (b), 1638 (m), 1577 (m), 1514 (m), 1430 (m), 1399 (s), 1380 (s), 1329 (s) 1311 (s), 1263 (s), 1229(m), 1211(m), 1162 (s), 1135 (s), 1084 (m), 1043 (s), 1008 (s), 880 (m), 848 (s), 804 (s), 776 (s). ¹H NMR (DMSO-*d*₆, 400 MHz): 8.6 (1H, d, J = 8.0 Hz), 8.5 (1H, d, J = 8.0 Hz), 8.1 (1H, d, J = 8.0 Hz), 8.0 (4H, s), 7.9 (1H, d, J = 8.0 Hz), 7.8 (2H, m), 7.7 (4H, s), 7.4 (1H, q, J = 8.0 Hz). Similarly, the co-crystals of isoquinoline N-oxide with *p*-phenylenediboronic acid (2:1)(C) were obtained; but in very low vield of crystalline pure product (2%). IR of the sample and NMR of solution shows identical peak to that of the parent compounds, so these are not listed. A dimethyl sulfoxide solution of p-phenylenediboronic acid (0.165 g, 1 mmol) and 4,4'-bipyridine N, N'-dioxide (0.376 g, 2 mmol) led to the formation of co-crystals (1:1). Yield of the pure crystalline complex was found to be 82%. IR (KBr, cm^{-1}): 3400 (b), 1633 (s), 1508 (m), 1480 (s), 1437 (m), 1398 (m), 1361 (m), 1328 (s), 1257 (m), 1237 (s), 1195 (m), 1155 (s), 1116 (m), 1097 (m), 1051 (m), 1026 (m), 820 (s), 730 (m). ¹H NMR (DMSO-*d*₆, 400 MHz): 8.3 (4H, d, J = 8.0 Hz), 7.6 (4H, d, J = 8.0 Hz), 7.3 (8H, s).

3. Results and discussion

3.1. Structural study

The co-crystals of *p*-phenylenediboronic acid with different aromatic *N*-oxides namely pyridine *N*-oxide, quinoline *N*-oxide, isoquinoline *N*-oxide, and 4,4'-bipyridine *N*, *N*'-dioxide are easily prepared by mixing respective solution of the corresponding *N*-oxide with the *p*-phenylenediboronic acid. The co-crystals have characteristic IR absorptions in the range 1211-1253 cm⁻¹ due to N–O stretching frequency. The structures of each of them are determined and their packing patterns are shown in Fig. 1A–D. Each of these adduct has the N–O…H hydrogen bond interaction as the primary interactions to form a network structure; these structures are further held by weak interactions.

The common point in each of these structures is that, the strong hydrogen bonding interactions between the two oxygen atoms of N-oxide from two independent N-oxide molecules with the two B-OH groups of the *p*-phenylenediboronic acid molecules. This type of interactions over-powers the original hydrogen bonding interactions between the B-OH groups of parent p-phenylenediboronic acid molecules [26-30]. The hydrogen bond parameters for these interactions are listed in Table 2. The packing patterns of these co-crystals show that each structure is associated with different types of weak interactions. The 1:1 co-crystal of pyridine Noxide has the B(OH)₂ groups anchored to N-oxides through C-H...O, and B- π interactions. The B- π interactions in the co-crystals sandwich a pyridine N-oxide ring between two B(OH)₂ units through η^2 and η^3 -type of B- π interactions. The distance of separation between the N-oxide containing rings and B(OH)₂ units are 3.40 Å and 3.52 Å, respectively. On co-crystal formation with Noxide, the complementing properties among the parent boronic acids are lost. The C-H-O interactions become dominating among the weak interactions; the interactions are listed in Table 2.

The quinoline *N*-oxide forms a 1:1 co-crystal whereas isoquinoline *N*-oxide forms 2:1 co-crystals with *p*-phenylenediboronic acid. These co-crystals self-assembles through strong hydrogen bond interactions between the N⁺–O⁻ of *N*-oxide and the O–H of the boronic acid groups. In both these cases extensive C–H…O and O–H…O hydrogen bond interactions are observed (Fig. 1B and C). However, significantly different π -interactions in the two co-crystals are observed. The co-crystal of quinoline *N*-oxide is devoid of π -interactions but in the case of the co-crystal of isoquinoline *N*oxide π -stacking effect is predominant. In the lattice of the co-crys-



Fig. 1. The weak interactions in the structures of co-crystals of *p*-phenylenediboronic acid with (A) pyridine *N*-oxide, (B) quinoline *N*-oxide, (C) isoquinoline *N*-oxide, (D) 4,4'-bipyridine *N*, *N*'-dioxide. In each case inset are the structures of the corresponding co-crystals.

Table 2

Hydrogen bond parameters in co-crystals of p-phenylenediboronic acid.

D-H…A	dD–H (Å)	<i>d</i> H… A (Å)	dD … A (Å)	<d-h…a (°)<="" th=""></d-h…a>
With pyridine N-oxide				
O2-H2AO1 [-x, -1/2 + y, 1/2 - z]	0.82	1.92	2.69 (6)	158
03-H3A01[-x, 1-y, -z]	0.99	1.71	2.70 (6)	176
C1-H1O3 [-x, 1-y, -z]	0.93	2.60	3.29 (7)	131
C5-H5O3 $[-x, 1/2 + y, 1/2 - z]$	0.93	2.35	3.26 (7)	165
C6-H6O1 $[-x, 1 - y, -z]$	0.93	2.47	3.37 (7)	161
With quinoline N-oxide				
$02-H2\cdots01[-x, 1-y, 1-z]$	0.82	2.03	2.81 (2)	157
$03-H3\cdots01 [1 - x, 1 - y, 1 - z]$	0.82	2.11	2.81 (1)	144
C1–H1····O3 [x, y, 1 + z]	0.93	2.59	3.50 (2)	168
С5-Н5…02	0.93	2.42	3.26 (3)	150
$C12-H12\cdots O1 [-x, 1-y, 1-z]$	0.93	2.49	3.34 (2)	152
With isoquinoline N-oxide				
$03-H3\cdots01 [1-x, 1-y, -z]$	0.82	1.98	2.71 (4)	149
O4-H4O2[x, y, -1+z]	0.82	2.01	2.76 (3)	152
C10-H10O1 [-1 + x, y, 1 + z]	0.93	1.93	2.85 (4)	171
C1-H101	0.93	1.89	2.82	174
With 4,4'-bipyridine N, N'-dioxide				
02-H201	0.82	2.09	2.87 (3)	157
03–H3····01 [1 + x, y, z]	0.82	2.03	2.79 (2)	152
C5-H5O2 [-1 + x, y, z]	0.93	2.56	3.45 (3)	161

tal of quinoline N-oxide and p-phenylenediboronic acid, the quinoline *N*-oxide molecules are placed in a head to tail arrangement, whereas in the later case the isoquinoline N-oxide molecules are positioned in head to head arrangement. The head to head sandwiched arrangement is electro-statistically unfavorable; but due to the complementarities for hydrogen bond formation they prefer to have the dimeric arrangement adopting a non-sandwiched arrangement as shown in Fig. 1C. These dimers are formed through C10-H-O1 and C1-H-O2 interactions. These dimeric pairs constitute a layered structure that are held by the C-H…O and O-H…O interactions of boronic acid and π - π interactions between two lavers, one above another, and placed offset to each other. The π - π separation between the layers is 3.34 Å within the generally referred limit of π -stacks [37]. In the co-crystals of guinoline N-oxide and *p*-phenylenediboronic acid, the boronic acid groups are placed parallel to each other. Two groups from two opposite ends of the parent diboronic acid are parallel with two boron atoms placed one above the other. The distance of separation between these two parallel boron atoms is 3.98 Å. The hydrogen bond parameters of the weak C-H-O interactions in the lattices of the co-crystals are given in Table 2.

The co-crystals of the 4,4'-bipyridyl N, N'-dioxide and p-phenylenediboronic acid posses conventional O-H--O, C-H--O as well as less conventional C–H…B [38] and B… π interactions. The strong N...OH interactions between the N-oxide and boronic acid O-H namely 02-H2....01 and 03-H3....01 makes a sheet like structures (Table 2). Generally, a B–O interaction is expected between the lone pair of oxygen with a vacant p-orbital of boron atom. However, the aromatic rings in the co-crystal are oriented in such a way that the oxygen atom of N-oxide is involved in O-H...O, C-H…O to form layered structure. Among the layers the oxygen atom of N-oxide occupies a space just above the boron atom with a distance of separation 3.10 Å, based on this distance of separation and projection of the lone pair of oxygen of N-oxide (Fig. 1D) it is proposed that there is a $B \cdots \pi$ interaction among the layers. It may be mentioned that the B $\cdots\pi$ interactions are rarely found interactions [39-43].

3.2. Solution study

The ultraviolet spectra of solutions of *p*-phenylenediboronic acid with pyridine *N*-oxide as well as with 4,4'-bipyridyl *N*-oxide

shows the interaction between the respective pairs. Fig. 2(a)shows the effect of pyridine N-oxide on the absorption spectrum of *p*-phenylenediboronic acid. The *p*-phenylenediboronic acid absorbs at 274 nm and 229 nm, but with the addition of pyridine Noxide solution the peak at 274 nm undergoes a blue shift to 263 nm and the peak at 229 nm shifts to 231 nm. Both the peaks are accompanied by a change in the absorption with a hyperchromic shift. The peak at 263 nm is characteristic of pyridine N-oxide so increase in the intensity with concentration is obvious. However, the increase in the absorption coefficient of the peak at 231 nm may be attributed to the formation of the co-crystal. Also the presence of the isobestic point at 222 nm shows the presence of two species at equilibrium. To check the origin of hyperchromic shift of the absorption peak at \sim 231 nm we have checked the absorption spectra of a mixture of components viz. p-phenylenediboronic acid and ethylene glycol followed by the addition of pyridine N-oxide solution (Fig. 2b). Interestingly no change in the absorption coefficient of the peak at ~231 nm is observed in this case. From these observation it can be interpreted that ethylene glycol interact with the diboronic acid first [18–19] and it renders the pyridine N-oxide to bind with the acid; further evidence comes from the disappearance of the isobestic point in this set of experiments. Fig. 2c shows the absorption spectra of the 4,4'bipyridyl N, N'-dioxide upon addition of p-phenylenediboronic acid. In this case also the presence of the isobestic point at 288 nm shows the presence of two different species at equilibrium. The 4, 4'-bipyridyl N, N'-dioxide absorbs at 223 nm and 330 nm. Upon addition of the *p*-phenylenediboronic acid the value of the absorption coefficient of the peaks changes, the intensity of the peak at 223 nm increases, which is due to gradual increase in the concentration of the *p*-phenylenediboronic acid. However, the absorption coefficient of the peak at 330 nm decreases; which may be attributed to the formation of the cocrvstals.

4. Conclusions

In conclusion, structural aspects of few co-crystals of boronic acids with *N*-oxides are studied and observed that the interactions in each case is different and some rare interactions such as $B \cdots \pi$ interactions and C-H \cdots B interactions are present in this class of



Fig. 2. (a) UV-spectra of *p*-phenylenediboronic acid (10^{-3} M) upon addition of pyridine N-oxide solution (10^{-3} M) (inset is the absorption spectra of p-phenylenediboronic acid). (b) UV-spectra of *p*-phenylenediboronic acid (10⁻³ M), ethylene glycol (10^{-3} M) upon addition of pyridine *N*-oxide solution (10^{-3} M). (c) UV-spectra of 4,4'-bipyridyl N-oxide (10^{-3} M) upon addition of *p*-phenylenediboronic acid solution (10^{-3} M) .

molecules and the study suggests about ample avenues for looking at the new interactions in the co-crystals of boronic acids.

Supplementary data

The CIF files of the co-crystals are deposited to Cambridge Crystallographic Database and have the CCDC Nos. 703292-703295.

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