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Synthesis and structural study of bis-perfluoropyridyl bridged by 1,4 and 1,2 dihydropyridine

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ABSTRACT

Bis-perfluoropyridyl bridged by 1,4 and 1,2 dihydropyridine compounds was synthesized by reaction of 2 and 4 aminopridine derivatives with pentafluoropyridine. The structures were determined by X-ray crystallography. Compound **3a** comprises two crystallographically independent molecules in the asymmetric unit in which one of them shows 1-D infinite chains along [0 1 0] direction due to the intermolecular C–H···N hydrogen bonds. In compound **5** intermolecular C–H···F and C–H···N hydrogen bonds link neighbouring molecules to each other. In addition, in both structures a series of C–F···π interactions stabilize the crystal packing.

1. Introduction

Pentafluoropyridine has attracted considerable interest due to its synthetic utility. Various multi-functional pyridine derivatives and construction of new heterocyclic and macrocyclic systems could be accessed from simple reaction conditions [1–3]. Highly fluorinated organic molecules do form a group of compounds distinctively different from their hydrocarbon counterparts [4]. Due to their high electronegativity fluorine atoms are able to alter the physico-chemical properties of molecules drastically. Fluorine is less polarizable and heavier than hydrogen, leading to more tightly packed denser materials. At the same time attractive interactions between fluoro moieties are weaker than in hydrocarbons, which cause the compounds to have lower surface energies and boiling points as liquids as well as weaker intermolecular interactions in the solid state. These and other related properties of perfluorinated compounds makes them a distinct class of compounds set apart from both polar and classical unpolar materials [5].

Synthesis of organic–inorganic hybrid polymeric complexes has attracted considerable interest due to their numerous potential applications with properties such as photochemical, catalytic, magnetic and crystal engineering [6]. Amongst the synthetic strategies to achieve the polymers, one of the choices is to use organic ligands such as N-donor bispyridyl-derived bridging organic ligands that can act as bridges in combination with transition metal ions [7].

In previous papers [8,9] we described synthesis of some bis-pyridyl bridged compounds from reaction of pentafluoropyridine with some equal diamines (primary or secondary). Initial substitution of the fluorine atom located at the most activated 4-position of one pentafluoropyridine molecule is followed by displacement of the 4-position of another pentafluoropyridine molecule leading to bis-perfluoropyridyl bridged products. In this context, we have been developing a general approach to the synthesis of new perfluoroheterocyclic compounds involving reactions between pentafluoropyridine **1** and unequal diamines [8–10]. Synthesis of 1D organic–inorganic coordination polymers from these bis-perfluoropyridyl bridged compounds is our next goal.

Recent publication describes syntheses of pyridoimidazole systems via intramolecular cyclization reactions between 2-amino-pyridine derivatives with pentafluoropyridine and various tetrafluoropyridine derivatives [11]. Also new nucleophilic fluorinating reagent, derived from reaction of dimethylaminopyridine with pentafluoropyridine has been prepared by those researchers [12].

In this paper, we describe very efficient methodology for the synthesis of bis-perfluoropyridyl bridged by 1,4 and 1,2 dihydropyridine systems by reaction of pentafluoropyridine with appropriate 2-amino-pyridine derivatives via intermolecular reactions.

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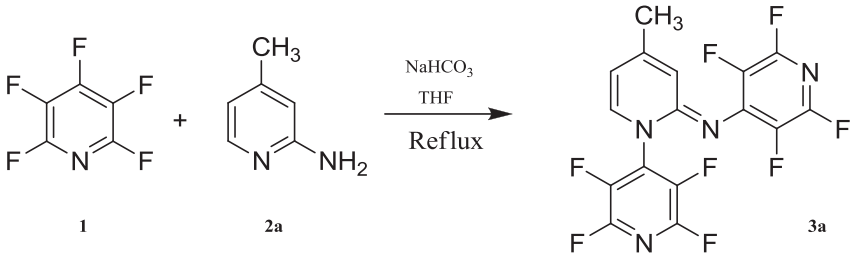
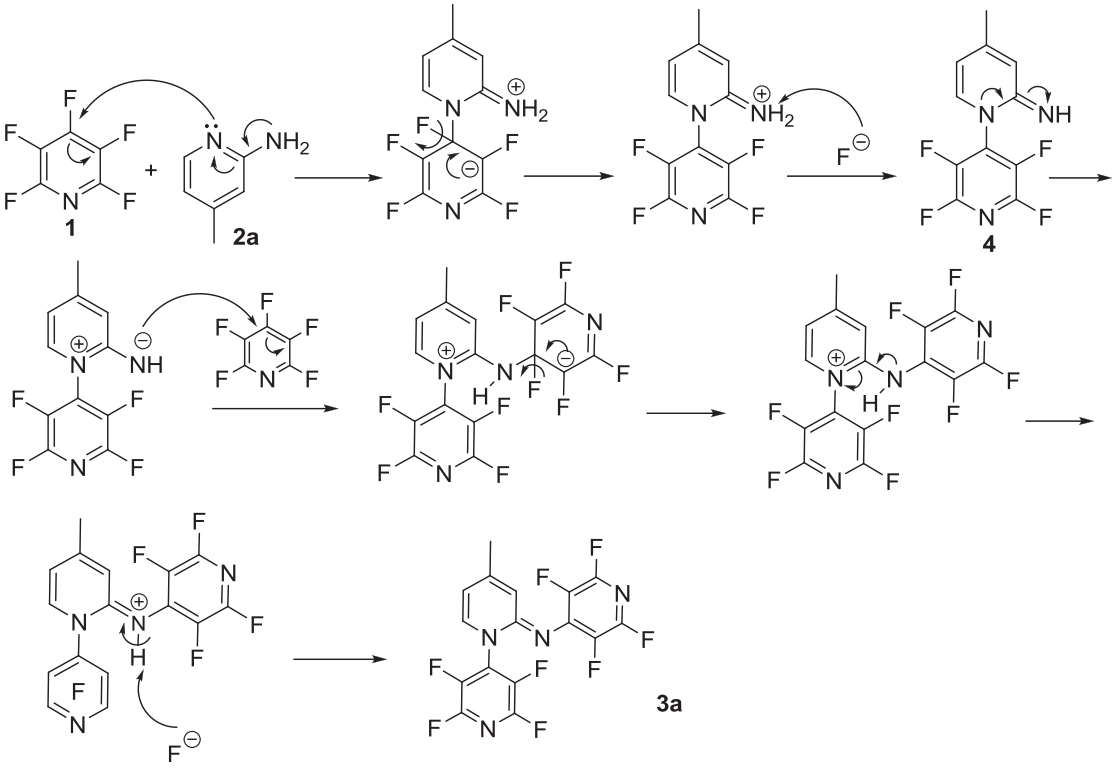


Scheme 1. Reaction of pentafluoropyridine with 2-amino-4-picoline.



Scheme 2. Mechanism of **3a**.

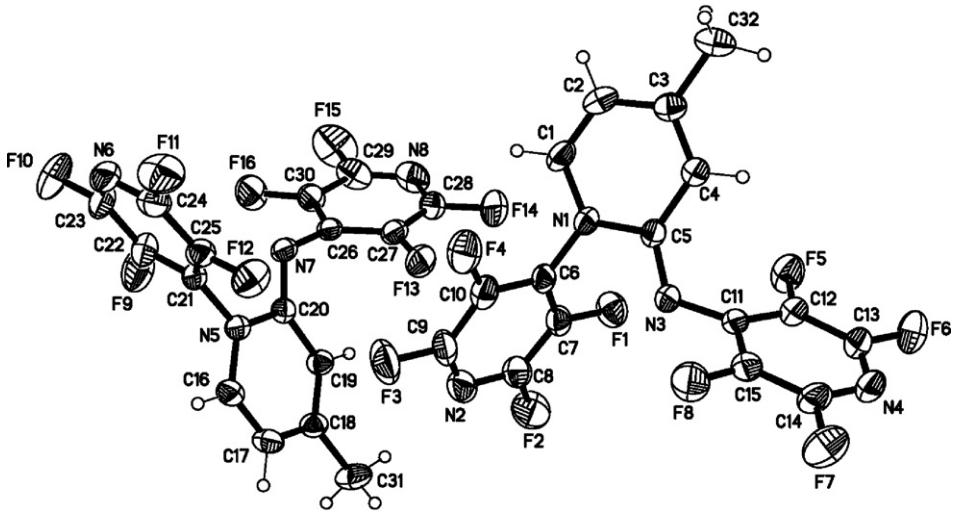
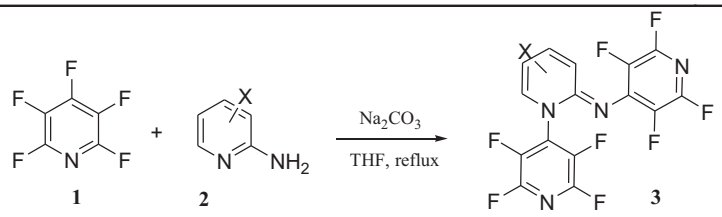
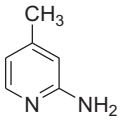
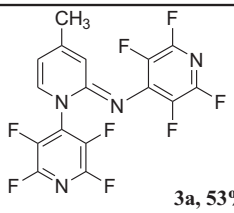
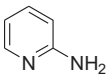
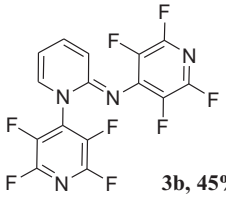
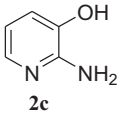
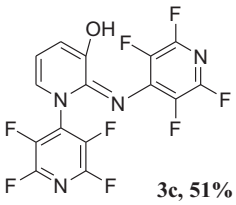


Fig. 1. The molecular structure of **3a**, showing 40% probability displacement ellipsoids and the atomic numbering.

Table 1
Reaction of aminopyridine derivatives with pentafluoropyridine.



Entry	binucleophile	product
1	 **2a**	 **3a, 53%**
2	 **2b**	 **3b, 45%**
3	 **2c**	 **3c, 51%**

2. Results and discussion

Reaction of pentafluoropyridine **1** with 2-amino-4-picoline **2a** in the presence of sodium bicarbonate and also in concentrated acetonitrile solution to maximize intermolecular reaction at reflux gave a single product, bis-perfluoropyridyl bridged by 1,2 dihydropyridine **3a**, in good yield. This processes could also be affected by ultrasonic irradiation, and in a much shorter reaction time, a similar yield of **3a** was obtained from **1** and **2a**. Four resonances by ^{19}F NMR (−87.0, −93.1, −144.1 and −153.2 ppm), indicate displacement of fluorine atoms attached to 4-positions of two pyridine ring (Scheme 1). Purification of **3a** was achieved by column chromatography. Identification of **3a** was done by ^{19}F NMR analysis, in which the resonance attributed to fluorines located *ortho* to ring nitrogen have a chemical shifts of −87.0 and −93.1 ppm similar to the shift observed for the **3b** and **3c**. The corresponding resonance for fluorines located *meta* to ring nitrogen in 3a occurs at −144.1 and −153.2 ppm similar to the analogous system **3b** and **3c**.

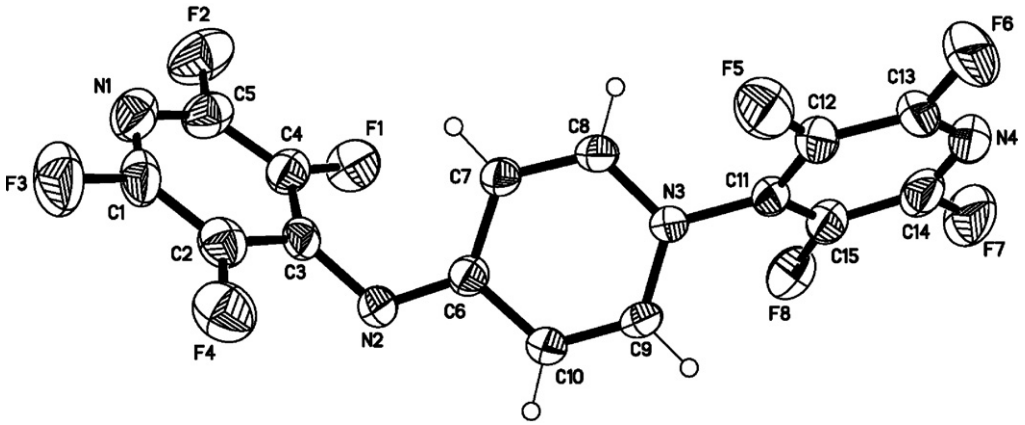
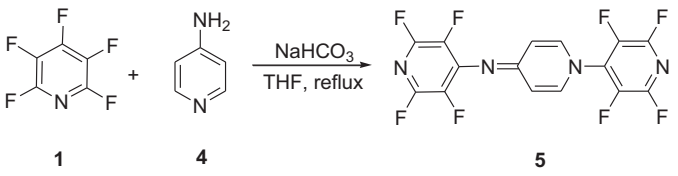


Fig. 2. The molecular structure of **5**, showing 40% probability displacement ellipsoids and the atomic numbering.



Scheme 3. Reaction of pentafluoropyridine with 4-aminopyridine.

Initial attack of 2-amino-4-picoline **2a** occurs through the more nucleophilic pyridine ring nitrogen to give the imine intermediate 4, and subsequent second attack involving nucleophilic substitution at the 4-position of another pyridine ring gives rise to the product obtained **3a** (Scheme 2).

The related 2-aminopyridine and 2-amino-3-hydroxypyridine **2b** and **2c** gave the analogous products **3b** and **3c**, respectively, by similar procedures (Table 1). The progress of all these reactions was monitored by ^{19}F NMR, and the disappearance of resonances attributed to fluorine atoms located at the 4-position of pentafluoropyridine allowed simple identification of bispyridyl products **3**.

With this encouraging result in hand, we found that 4-aminopyridine **4** react efficiently with **1** to give 2,3,5,6-tetrafluoro-4-[1-(2,3,5,6-tetrafluoro-4-pyridyl)-1,4-dihydro-4-pyridinylidenamino]-pyridine **5** (Scheme 3). Bifunctional nucleophiles **4**, reacted efficiently with 2 mol of **1** to give good yields of bis-pyridyl system **5**. Purification of **5** was achieved by recrystallization of the crude product mixture from dichloromethane.

Identification of **5** was done by ^{19}F NMR analysis, in which the resonance attributed to fluorines located *ortho* to ring nitrogen have a chemical shifts of −88.2 and −93.7 ppm similar to the shift observed for the **3**. The corresponding resonance for fluorines located *meta* to ring nitrogen in **5** occurs at −145.6 and −152.0 ppm similar to the analogous system **3**.

The regioselectivity of nucleophilic substitution of **1** with **2** and **4** may be explained by the high nucleopilicity of the secondary or primary amino groups and also the activating influences of the pyridine ring's nitrogen that significantly activates the *ortho* and *para* sites to itself.

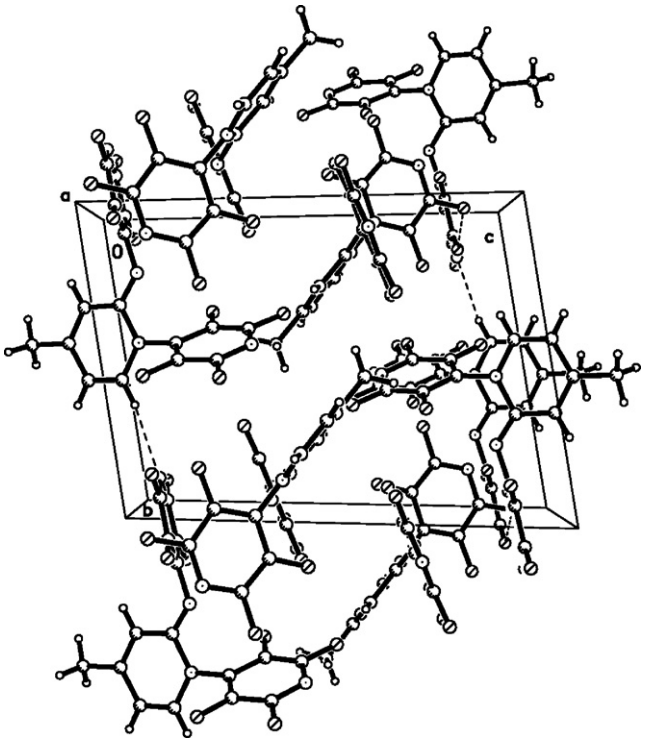


Fig. 3. The crystal packing of **3a** viewed down the *a* axis.

The structures of compounds **3a** and **5** were confirmed by X-ray crystallography. The ORTEP plots of compounds **3a** and **5** are shown in Figs. 1 and 2, respectively. The crystal packing of **3a** and **5** are shown in Figs. 3 and 4, respectively. The asymmetric unit of the compound **3a** contains two crystallographically independent molecules with different conformations in which one of them forms 1-D extended chain along the [0 1 0] direction through intermolecular C–H···N hydrogen bonds (Table 1). The interesting feature of the crystal structure of **3a** is weak intermolecular F6···F11 [2.818(3) Å]

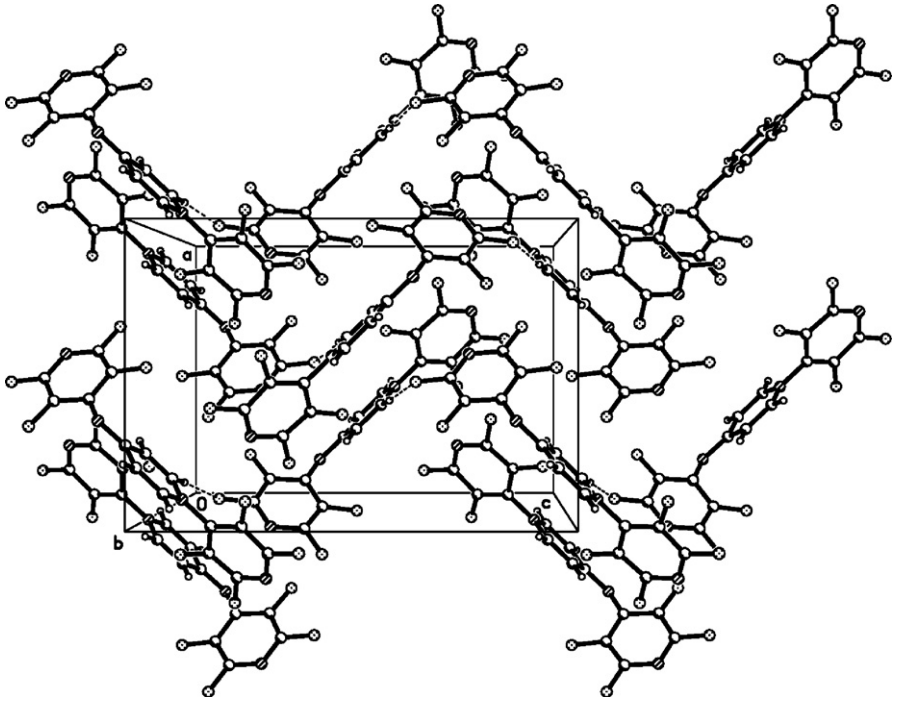


Fig. 4. The crystal packing of **5** viewed down the *b* axis.

interaction which is shorter than the sum of the van der Waals radius [2.94 Å] of F atoms [17]. Also, there is a π–π interaction between the fluorinated benzene ring which to some extent controls the crystal packing of **3a**. The structure of compound **5** shows intermolecular C–H···F and C–H···N hydrogen bonds (Table 1). In both structures **3a** and **5**, the crystal packing is further stabilized by a series of C–F···π interactions and short intermolecular F···N and F···C contacts.

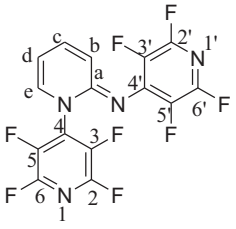
3. Conclusions

Bis-perfluoropyridyl bridged by 1,4 and 1,2 dihydropyridine compounds may be accessed very readily in one pot processes by reaction of 2- and 4-aminopyridine derivatives with pentafluoropyridine. The regioselectivity of nucleophilic substitution in this process may be explained by the high nucleoplilicity of the secondary or primary amino groups and by the activating influence of pyridine ring nitrogen that significantly activates the *ortho* and *para* sites to itself. Compound **3a** comprises two crystallographically independent molecules in the asymmetric unit in which one of them shows 1-D infinite chains along [0 1 0] direction due to the intermolecular C–H···N hydrogen bonds. In compound **5** intermolecular C–H···F and C–H···N hydrogen bonds link neighbouring molecules to each other. In addition, in both structures a series of C–F···π interactions stabilize the crystal packing. Synthesis of 1D organic–inorganic coordination polymers from these bis-perfluoropyridyl bridged compounds is our next goal.

4. Experimental

All solvents were dried using the literature procedures and distilled before use. The reactions were carried out under an atmosphere of argon unless otherwise specified. The elemental analyses for C, H, and N were performed using Heraeus CHN-O-Rapid analyzer. The ^{13}C NMR spectra were recorded at 125 MHz. The ^{19}F NMR spectra were recorded at 470 MHz. In the ^{19}F NMR spectra, upfield shifts were quoted as negative and referenced to $CFCl_3$. Mass spectra were taken by a Micromass Platform II: EI mode (70 eV). Silica plates (Merck) were used for TLC analysis.

The following numbering scheme is used for indicating NMR assignments for Bis-perfluoropyridyl bridged systems.



4.1. General procedure

Sodium bicarbonate (3 mmol) was added to the mixture of **2** or **4** (1 mmol) in THF (6 mL). Then **1** (2 mmol) was added and the resulting solution was refluxed at 87 °C for 2 days. The reaction mixture was cooled to room temperature and the solvent was evaporated. The reaction mixture was extracted with dichloromethane. The solvent was evaporated to yield the crude product, which was then purified by recrystallization or column chromatography on silica gel.

4.1.1. 2,3,5,6-Tetrafluoro-4-[4-methyl-1-(2,3,5,6-tetrafluoro-4-pyridyl)-1,2-dihydro-2-pyridinylidenamino]pyridine 3a

Sodium bicarbonate (0.25 g, 3 mmol), 2-amino-4-methylpyridine **2a** (0.11 g, 1 mmol), pentafluoropyridine **1** (0.34 g, 2 mmol) and THF (6 mL) gave the product that was purified by column chromatography on silica gel (ethyl acetate/hexane, 1:3) gave 2,3,5,6-tetrafluoro-4-[4-methyl-1-(2,3,5,6-tetrafluoro-4-pyridyl)-1,2-dihydro-2-pyridinylidenamino]pyridine **3a**, 0.21 g (53%), yellow solid; decomposed at 160 °C. ^{13}C NMR ($CDCl_3$): δ (ppm) 22.12 (s, CH_3), 115.87 (s, Ar-C), 116.84 (s, Ar-C), 123.48 (dm, $^1J_{CF}$ = 240.2 Hz, C-3), 126.15 (s, Ar-C), 129.19 (dm, $^1J_{CF}$ = 232.6 Hz, C-3′), 132.19 (m, C-4), 134.01 (m, C-4′), 140.75 (dm, $^1J_{CF}$ = 233.7 Hz, C-2), 141.67 (s, Ar-C), 147.02 (dm, $^1J_{CF}$ = 268.0 Hz, C-2′), 148.24 (s, Ar-C). ^{19}F NMR ($CDCl_3$): δ (ppm) −153.40 (m, 2F, F-3, F-5), −144.16 (m, 2F, F-3′, F-5′), −93.47 (m, 2F, F-2, F-4), −87.35 (m, 2F, F-2′, F-4′) ppm. ^1H NMR (500 MHz, $CDCl_3$): δ_H 2.51 (3H, s), 6.84 (1H, d, $^3J_{HH}$ = 7 Hz, Ar H), 7.48 (1H, s, Ar H), 8.49 (1H, d, $^3J_{HH}$ = 7 Hz, Ar H). MS (EI), *m/z* (%) = 406 (M^+, 100), 391 (25), 256 (80), 149 (35). Anal. Calcd for $C_{16}H_6N_4F_8$: C, 47.3; H, 1.5; N; 13.8. Found: C, 47.4; H, 1.5; N, 13.7.

4.1.2. 2,3,5,6-Tetrafluoro-4-[1-(2,3,5,6-tetrafluoro-4-pyridyl)-1,2-dihydro-2-pyridinylidenamino]pyridine 3b

Sodium bicarbonate (0.25 g, 3 mmol), 2-aminopyridine **2b** (0.09 g, 1 mmol), pentafluoropyridine **1** (0.34 g, 2 mmol) and THF (6 mL) gave the product that was purified by column chromatography on silica gel (ethyl acetate/hexane, 1:3) gave 2,3,5,6-tetrafluoro-4-[1-(2,3,5,6-tetrafluoro-4-pyridyl)-1,2-dihydro-2-pyridinylidenamino]pyridine **3b**, 0.18 g (45%), orange solid; decomposed at 185 °C. ^{13}C NMR ($CDCl_3$): δ (ppm) 115.63 (s, Ar-C), 118.82 (dm, $^1J_{CF}$ = 130.5 Hz,C-3), 120.16 (m, C-4), 120.82 (dm, $^1J_{CF}$ = 134.4 Hz,C-3′), 121.56 (s, Ar-C), 136.26 (dm, $^1J_{CF}$ = 252.0 Hz, C-2), 137.83 (d, $^2J_{CF}$ = 14.40 Hz, Ar-C), 141.57 (m, C-4′), 144.00 (Ar-C), 145.09 (dm, $^1J_{CF}$ = 241.3 Hz, C-2′), 148.55 (s, Ar-C). ^{19}F NMR ($CDCl_3$): δ (ppm) −153.18 (m, 2F, F-3′, F-4′), −144.07 (m, 2F, F-3, F-4), −93.11 (m, 2F, F-2′, F-6′), −87.03 (m, 2F, F-2, F-6) ppm. ^1H NMR (500 MHz, $CDCl_3$): δ_H 6.37 (1H, Ar-H), 6.43 (1H, Ar-H), 7.11 (1H, Ar-H), 7.34 (1H, Ar-H). MS (EI), *m/z* (%) = 392 (M^+, 100), 242 (80), 149 (40). Anal. Calcd for $C_{15}H_4N_4F_8$: C, 45.9; H, 1.0; N; 14.3. Found: C, 45.8; H, 1.0; N, 14.4.

4.1.3. 1-(2,3,5,6-Tetrafluoro-4-pyridyl)-2-(2,3,5,6-tetrafluoro-4-pyridylimino)-1,2-dihydro-3-pyridinol 3c

Sodium bicarbonate (0.25 g, 3 mmol), 2-amino-3-hydroxypyridine **2c** (0.11 g, 1 mmol), pentafluoropyridine **1** (0.34 g, 2 mmol) and THF (6 mL) gave the product that was purified by column chromatography on silica gel (ethyl acetate/hexane, 1:3) gave 1-(2,3,5,6-tetrafluoro-4-pyridyl)-2-(2,3,5,6-tetrafluoro-4-pyridylimino)-1,2-dihydro-3-pyridinol **3c**. 0.11 g (51%), cream solid, decomposed at 119 °C; ^{13}C NMR ($CDCl_3$): δ (ppm) δ_C 113.87 (s, Ar-C), 117.98 (dm, $^1J_{CF}$ = 131.1 Hz,C-3), 119.19 (m, C-4), 121.06 (dm, $^1J_{CF}$ = 136.9 Hz,C-3′), 122.7 (s, Ar-C), 135.98 (dm, $^1J_{CF}$ = 250.3 Hz, C-2), 138.75 (d, $^2J_{CF}$ = 16.44 Hz, Ar-C), 142.03 (m, C-4′), 144.16 (Ar-C), 144.18 (dm, $^1J_{CF}$ = 246.8 Hz, C-2′), 149.91 (s, Ar-C). ^{19}F NMR ($CDCl_3$): δ (ppm) −154.87 (2F, F-3′,5′), −149.86 (2F, m, F-3,5), −92.84 (2F, m, F-2′,6′), −88.16 (2F, m, F-2,6). MS (EI), *m/z* (%) = 408 (M^+, 70), 407 (100), 391 (55), 258 (70), 149 (30). Anal. Calcd for $C_{15}H_4N_4F_8O$: C, 44.1; H, 0.9; N; 13.7. Found: C, 44.2; H, 1.0; N, 13.8.

4.1.4. 2,3,5,6-Tetrafluoro-4-[1-(2,3,5,6-tetrafluoro-4-pyridyl)-1,4-dihydro-4-pyridinylidenamino]pyridine 5

Sodium bicarbonate (0.25 g, 3 mmol), 4-aminopyridine **4** (0.09 g, 1 mmol), pentafluoropyridine **1** (0.34 g, 2 mmol) and THF (6 mL) gave the product that was purified by recrystalization (ethyl acetate/n-hexane, 1:4) gave 2,3,5,6-tetrafluoro-4-[1-(2,3,5,6-tetrafluoro-4-pyridyl)-1,4-dihydro-4-pyridinylidenamino]pyridine **5**, 0.24 g (85%), orange solid; mp 203–207 °C, ^{13}C NMR ($CDCl_3$): δ (ppm) 114.23 (s, Ar-C), 122.42 (dm, $^1J_{CF}$ = 238.3 Hz, C-3), 127.25 (s, Ar-C), 130.08 (dm, $^1J_{CF}$ = 230.4 Hz, C-3′), 131.71 (m, C-4), 134.76 (m, C-4′), 141.49 (dm, $^1J_{CF}$ = 232.1 Hz, C-2), 148.93 (dm,

Table 2
The hydrogen bonds parameters in **3a** and **5**.

	D–H···A	H···A (Å)	D···A (Å)	D – H···A (°)
1	C(1)–H(1A)···N(4)[a]	2.5000	3.415(3)	167
2	C(8)–H(8)···F(3)[b]	2.78	3.387(4)	173
	C(9)–H(9)···N(2)[c]	2.75	3.475()	152

[a] Symmetry code: x, $1+y$, z.
[b] Symmetry code: $3/2-x$, $1-y$, $-1/2+z$.
[c] Symmetry code: $-1/2+x$, $3/2-y$, $1-z$.

$^1J_{CF}$ = 261.6 Hz, C-2′), 151.33 (s, Ar-C). ^{19}F NMR ($CDCl_3$): δ (ppm) −152.01 (m, 2F, F-3, F-5), −145.62 (m, 2F, F-3′, F-5′), −93.75 (m, 2F, F-2, F-4), −88.28 (m, 2F, F-2′, F-4′). MS (EI), *m/z* (%) = 392 (M^+, 100), 242 (80), 149 (40). Anal. Calcd for $C_{15}H_4N_4F_8$: C, 45.9; H, 1.0; N; 14.3. Found: C, 45.8; H, 1.1; N, 14.3.

4.2. X-ray crystal structures determination of 3a and 5[1]

The X-ray single crystal data for **3a** and **5** were collected at 296(1) K on STOE IPDS II diffractometers (Mo $K\alpha$ = 0.71073 Å). Cell parameters were retrieved using X-AREA [13] software and refined using X-AREA on all observed reflections. Data reduction and correction for Lp (Lorentz-polarization) and decay were performed using X-AREA software. Absorption corrections were applied using MULABS [14] in PLATON [15]. All structures were solved by direct methods and refined by full-matrix least squares on F^2 for all data using SHELXTL [16] software. All calculations were performed by PLATON. All non-hydrogen atoms were refined anisotropically. Hydrogen atoms were positioned geometrically and refined with a riding model approximation with their parameters constrained to the parent atom with U_{iso} (H) = 1.2 or 1.5 U_{eq} (C) (Table 2).

Crystal data for **3a**: $C_{16}H_6F_8N_4$, M = 406.25, triclinic, space group *P-1*, a = 10.4855(15), b = 10.7789(13), c = 15.453 (2) Å, α = 81.021(10), β = 71.652(10)°, γ = 89.001(11)°, V = 1636.5 (4) Å^3, $F(000)$ = 808, Z = 4, D_c = 1.649 Mg m^{-3}, μ = 0.166 mm^{-1}, 15,839 reflections collected; 7119 unique data (R_{int} = 0.046). Final $wR_2(F^2) = 0.1311$ for all data (507 refined parameters), $w = 1/[\sigma^2(F_o^2) + (0.0659P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$, conventional $R(F)$ = 0.0488 for 4284 reflections with $I > 2\sigma(I)$, GOF = 1.00.

Crystal data for **5**: $C_{15}H_4F_8N_4$, M = 392.22, orthorhombic, space group *Pbca*, a = 10.322(5), b = 19.663(5), c = 14.927(5) Å, V = 3029.6(19) Å^3, $F(000)$ = 1552, Z = 8, D_c = 1.720 Mg m^{-3}, μ = 0.176 mm^{-1}, 18,063 reflections collected; 2943 unique data (R_{int} = 0.087), Final $wR_2(F^2) = 0.0918$ for all data (244 refined parameters); $w = 1/[\sigma^2(F_o^2) + (0.0262P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$, conventional $R(F)$ = 0.0552 for 1346 reflections with $I > 2\sigma(I)$, GOF = 0.97.

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[1] CCDC 785354–785355 contain the supplementary crystallographic data for this paper. These data can be obtained free of charge via http://www.ccdc.cam.ac.uk/conts/retrieving.html (or from the CCDC, 12 Union Road, Cambridge CB2 1EZ, UK. Fax: +44 1223 336033; e-mail: deposit@ccdc.cam.ac.uk).