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Macrocycles from pentafluoropyridine and tetrafluoropyrimidine

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Abstract

Macrocycles were synthesised by sequential nucleophilic aromatic substitution processes involving pentafluoropyridine and tetrafluoropyrimidine and various diamines as the structural components.

Keywords: Macrocycles; Heterocyclic; Perfluoroheterocycle; Pentafluoropyridine

1. Introduction

The field of macrocyclic synthesis [1–3] and, more generally, supramolecular chemistry [4] has attracted great interest over recent years and as a result macrocyclic systems are now used in various applications [3] as, for example, sensors and contrast imaging agents [4]. The synthesis of many macrocyclic derivatives relies upon nucleophilic substitution processes at saturated sites (e.g. many crown ether and cryptand syntheses [5]) or addition–elimination reactions involving carbonyl systems (e.g. calixarenes [6]) in the ring closing steps. In contrast, synthetic approaches that involve nucleophilic aromatic substitution processes in the macrocyclic ring forming step are far less common [7] because, in part, only a limited number of suitably activated aromatic building blocks, such as chlorinated triazines [8,9], tetrachloropyridine [10], chloropyrimidine derivatives [11] or difluorodinitrobenzene [12,13] are available for successful macrocyclic ring closure to occur. In this context, we have recently described [14–16] the synthesis of novel macrocyclic systems starting from pentafluoropyridine **1** via various tetrafluoropyridine systems **2** by a strategy outlined in Scheme 1 (*Route a*).

This strategy involves firstly 'blocking' the most reactive 4-position of pentafluoropyridine to give tetrafluoropyridine derivatives **2** that react with suitable difunctional 'bridging groups' at the 2- and 6-positions of the pyridine unit to give the corresponding 'bridged compounds' **3** and, ultimately, macrocycles **4**. This versatile sequential methodology allows a variety of bridging units (Nuc_2–Nuc_2) to be used for a range of cryptand and crown ether syntheses and, in all preparations carried out following *Route a*, the nitrogen atoms of the pyridine ring form part of the macrocyclic ring.

In this paper, we adapt our synthetic methodology to the synthesis of macrocyclic systems **6** that are formed from reaction of the 2- and 4-positions of the pyridine sub-units (*Route b*, Scheme 1) via a different structural class of bridging unit **5**. This strategy will enable access to macrocycles with different structural features such as macrocycles in which the pyridine nitrogen atoms are not part of the macrocyclic ring and fluorine atoms that are located at active sites that are outside the ring cavity and may be further functionalised if required, for example, for the attachment of a porphyrin-based sensor unit [12]. We also extend this methodology to the incorporation of pyrimidine units by utilising tetrafluoropyrimidine **7** as the starting material in macrocyclic synthesis for the first time.

2. Results and discussion

Reaction of an excess of pentafluoropyridine **1** and diamines **8a** and **8b** gave the corresponding bis-pyridyl bridged compounds **9a** and **9b** in 76% and 88% yield, respectively after heating together at reflux temperature in THF (Scheme 2).

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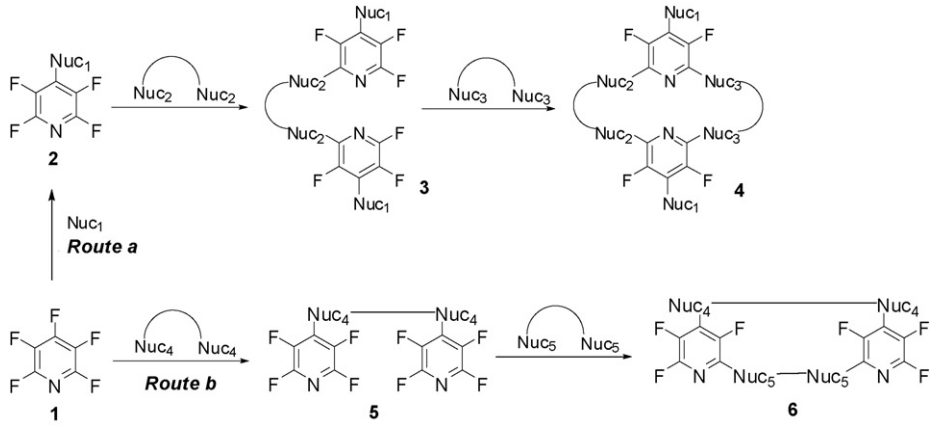
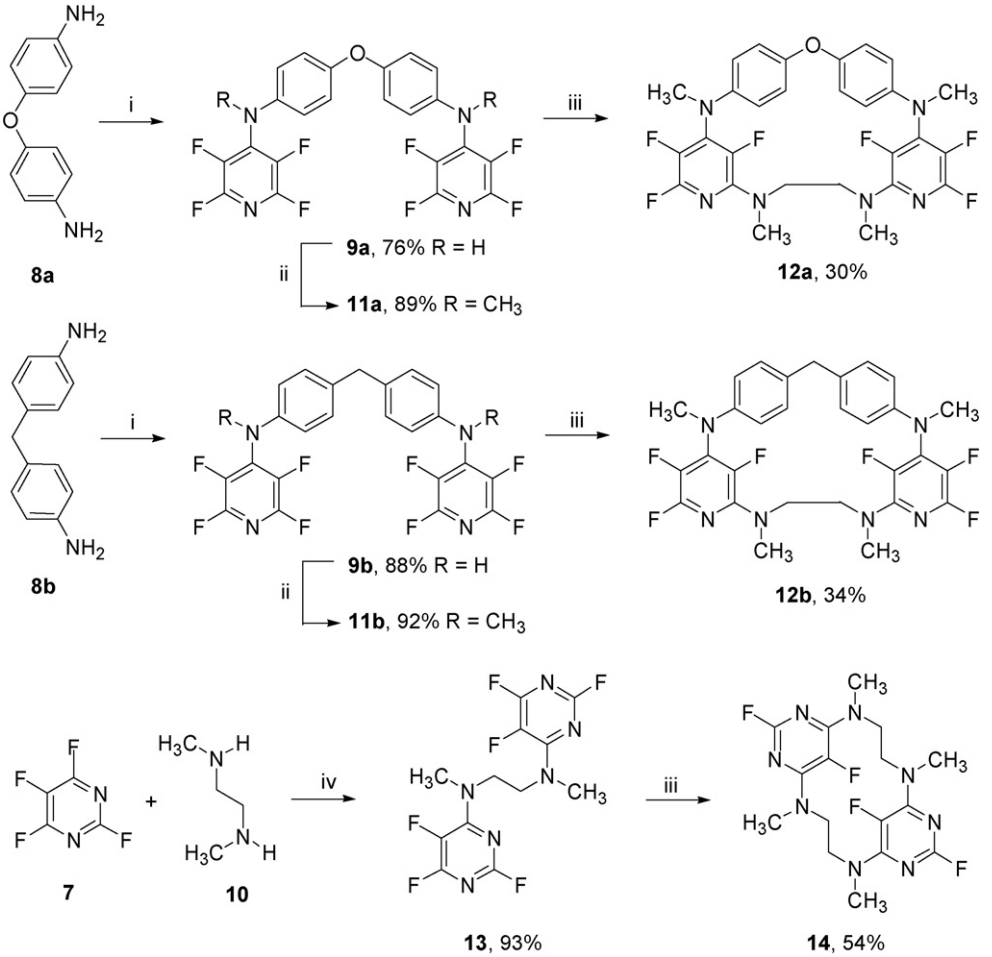


Scheme 1. Synthetic strategies for the construction of macrocycles from pentafluoropyridine.

Subsequent attempts to form macrocycles from **9a** and **9b** upon reaction with *N*,*N*′-dimethylethylene diamine **10** were unsuccessful due to competing side product formation. However, we found that methylation of the NH groups by reaction of **9a** and **9b** with methyl iodide to give **11a** and **11b**, respectively was necessary before cyclization to macrocycles **12a** and **12b** could be effected using diamine **10** in high dilution conditions. By a similar process tetrafluoropyrimidine **7** gave bridged compound **13** upon reaction with diamine **10** and this subsequently formed macrocycle **14** upon reaction with a further equivalent of **10** in high dilution conditions in acetonitrile.

In all reactions, the nucleophilic aromatic substitution processes are regioselective, following established principles [17,18]. For example, reaction of pentafluorinated aromatic



Scheme 2. Synthesis of macrocycles. Reagents and conditions: (i) C_5F_5N **1**, THF, reflux, 12 h; (ii) MeI, K_2CO_3, MeCN, reflux, 12 h; (iii) **10**, MeCN, reflux 3 d; (iv) THF, reflux 12 h.

systems such as pentafluoroiodobenzene occur selectively at sites *para* to the substituent [18], reaction of pentafluoropyridine with nucleophiles occurs at the most activated 4-position [17,18] and reaction of 4-amino tetrafluoropyridine systems with nucleophiles was established recently [19] to occur at the 2-position.

The structures of compounds **9b**, **11a**, **13** and **14** were confirmed by X-ray crystallography (Fig. 1a–d) and by comparison of NMR spectroscopic data. Structure **9b** contains two independent molecules with different conformations which are quite different from the conformation of the closely related molecule **11a**, reflecting the conformational lability of these molecules.

In previous papers [19–21] we noted the importance of stacking interaction between aromatic rings in the crystal packing of compounds containing fluorinated heterocycles. In these cases, the compounds also exhibit π···π interactions which determine not only the packing of the molecules in the crystals but also molecular conformation. The comparison of the molecular conformation of **13** with its Ph-analogues [22] shows the drastic change of shape of the molecule from extended to angular. There is a number of C–H···N and C–H···π interactions in the structure of the Ph-analogue, but no stacking interactions were observed. At the same time the packing of the molecules in the structure **13** is determined by interactions between planar aromatic fragments (Fig. 2). Similar overlapping of the aromatic systems is also present in the crystal structures of **9b**, **11a** and **14**. The hydrogen bonds of the N–H···O type link molecules in zigzag chains in the structure of **9b**. The significance of π···π interactions between aromatic systems for crystal packing and their influence on the conformation of molecules is well known and has been a subject of several reviews [23].

In summary, macrocycles may be synthesised by reaction of pentafluoropyridine and tetrafluoropyrimidine by linking positions most reactive towards nucleophilic attack by various diamine-bridging units. The range of macrocyclic structures with differing architectures that may be accessed using this S_NAr ring-closing methodology is, therefore, expanded considerably.

3. Experimental

All starting materials were obtained commercially. All solvents were dried using literature procedures. NMR spectra were recorded in deuteriochloroform, unless otherwise stated, on a spectrometer operating at 500 MHz (^{1}H NMR), 376 MHz (^{19}F NMR) and 100 MHz (^{13}C NMR) with tetramethylsilane and trichlorofluoromethane as internal standards. Mass spectra were recorded on a VG 7070E spectrometer coupled with a Hewlett Packard 5890 series II gas chromatograph. Elemental analyses were obtained on an Exeter Analytical CE-440 elemental analyser. Melting points and boiling points were recorded at atmospheric pressure unless otherwise stated and are uncorrected. The progress of reactions was monitored by ^{19}F NMR and column chromatography was carried out on silica gel.

4. Reactions of diamines with perfluoroheteroaromatic systems

4.1. General procedure

Diamine was added to a solution of the perfluoroheterocycle in dry THF (70 mL) and heated to reflux temperature for 12 h under an atmosphere of dry nitrogen. After cooling to room temperature, dil. sodium hydrogen carbonate solution (20 mL) was added. The organic products were extracted into dichloromethane (2× 30 mL), dried ($MgSO_4$) and evaporated to give the product which was purified by recrystalisation from ethyl acetate/*n*-hexane.

4.1.1. *N,N′-Bis-(2,3,5,6-tetrafluoro-pyridin-4-yl)-4,4′-diaminodiphenyl-ether* ***9a***

Pentafluoropyridine **1** (1.60 g, 9.3 mmol) and 4,4′-diaminodiphenyl ether **8a** (0.94 g, 4.7 mmol) gave *N,N′*-bis-(2,3,5,6-tetrafluoro-pyridin-4-yl)-4,4′-diaminodiphenyl ether **9a** (1.77 g, 76%) as a white crystalline solid; mp 117–119 °C (found: C, 52.9; H, 2.3; N, 10.9. $C_{22}H_{10}F_8N_4O$ requires C, 53.0; H, 2.0; N, 11.2%); δ_H 6.29 (2H, br s, NH), 7.07 (8H, m, aryl-H); δ_C 119.5 (s, C-2′), 124.7 (s, C-3′), 132.4 (dm, $^1J_{CF}$ 253.6, C-3), 133.8 (s, C–NH), 134.9 (m, C-4), 144.4 (dm, $^1J_{CF}$ 241.1, C-2), 155.2 (s, C–O); δ_F −92. 9 (2F, m, F-2), −157.2 (2F, m, F-3); *m/z* (EI$^+$) 498 ([M]$^+$, 38%), 257 (52), 229 (20).

4.1.2. *N,N′-Bis-(2,3,5,6-tetrafluoro-pyridin-4-yl)-diphenylmethane 4,4′-diamine* ***9b***

Pentafluoropyridine **1** (1.42 g, 8.4 mmol) and 4,4′-diaminodiphenylmethane **8b** (0.83 g, 4.2 mmol) gave *N,N′*-bis-(2,3,5,6-tetrafluoro-pyridin-4-yl)-diphenylmethane 4,4′-diamine **9b** (1.83 g, 88%); mp 89–92 °C (found: C, 55.4; H, 2.4; N, 11.1. $C_{23}H_{12}F_8N_4$ requires C, 55.65; H, 2.4; N, 11.3%); δ_H 3.98 (2H, s, CH_2), 6.45 (2H, br s, NH), 7.04–7.18 (8H, m, aryl-H); δ_C 40.9 (s, CH_2), 122.5 (s, C-2′), 129.7 (s, C-3′), 132.6 (dm, $^1J_{CF}$ 253.8, C-3), 134.6 (m, C-4), 136.6 (s, C–CH_2), 138.5 (s, C–N), 144.4 (dm, $^1J_{CF}$ 239.4, C-2); δ_F −93.1 (2F, m, F-2), −156.3 (2F, m, F-3); *m/z* (EI$^+$) 496 ([M]$^+$, 88%), 330 (93), 254 (64), 164 (100).

4.1.3. *N,N′-Dimethyl-N,N′-bis-(2,5,6-trifluoro-pyrimidin-4-yl)-ethane-1,2-diamine* ***13***

Tetrafluoropyrimidine **7** (1.32 g, 8.5 mmol) and *N,N′*-dimethylethylene diamine **10** (0.38 g, 4.3 mmol) gave *N,N′*-dimethyl-*N,N′*-bis-(2,5,6-trifluoro-pyrimidin-4-yl)-ethane-1,2-diamine **13** (1.4 g, 93%); mp 100–102 °C (found: C, 40.7; H, 2.8; N, 23.6. $C_{12}H_{10}F_6N_6$ requires C, 40.9; H, 2.9; N, 23.9); δ_H 3.24 (3H, s, CH_3), 3.87 (2H, m, CH_2); δ_C 38.6 (s, CH_3), 49.7 (s, CH_2), 130.7 (ddd, $^1J_{CF}$ 234, $^2J_{CF}$ 15, $^4J_{CF}$ 9, C-5), 154.7 (ddd, $^1J_{CF}$ 178, $^3J_{CF}$ 21, $^3J_{CF}$ 3, C-6), 155.6 (dt, $^2J_{CF}$ 19, $^3J_{CF}$ 5, C-4), 160.1 (ddd, $^1J_{CF}$ 241, $^3J_{CF}$ 21, $^4J_{CF}$ 9, C-2); δ_F −48.8 (1F, d, $^3J_{FF}$ 25, F-5), −87.7 (1F, d, $^4J_{FF}$ 16, F-2), −174.1 (1F, dd, $^3J_{FF}$ 25, $^4J_{FF}$ 16, F-6); *m/z* (EI$^+$) 352 ([M]$^+$, 2%), 180 (5), 178 (100), 165 (12), 131 (12), 40 (22).

5. Methylation of diamines 9

5.1. General procedure

Potassium carbonate was added to a solution of bridged compound **9** in dry acetonitrile (70 mL) and heated to reflux temperature for 1 h under an atmosphere of dry nitrogen. Methyl iodide was added to the reaction mixture which was heated to reflux temperature for a further 12 h. After cooling to RT, the organic products were extracted into dichloromethane (2× 30 mL), dried ($MgSO_4$) and the solvent evaporated. Recrystalisation from dichloromethane/light petroleum gave pure product **11**.

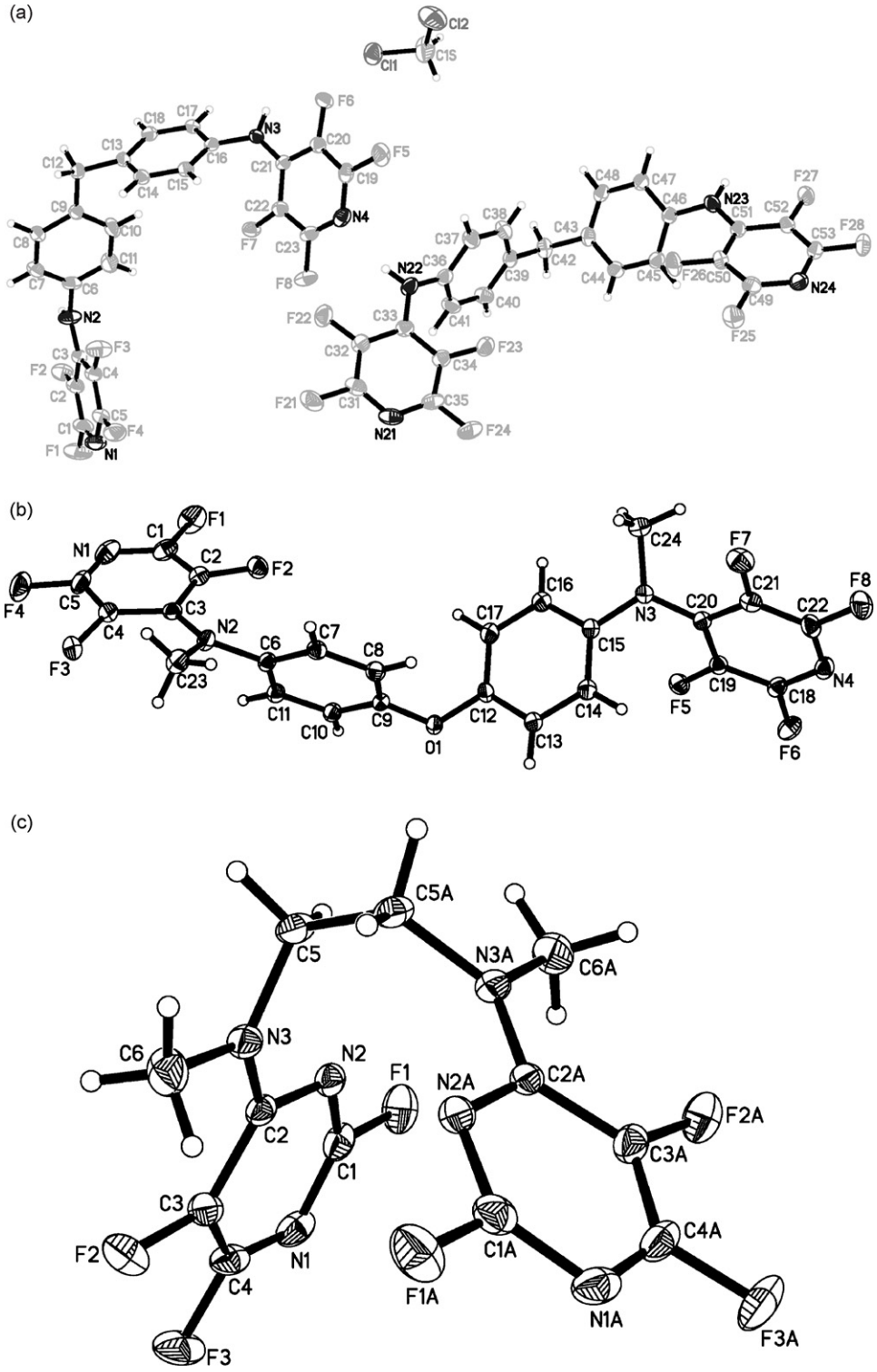


Fig. 1. (a) Molecular structure of **9b**, (b) molecular structure of **11a**, (c) molecular structure of **13**, and (d) molecular structure of **14**.

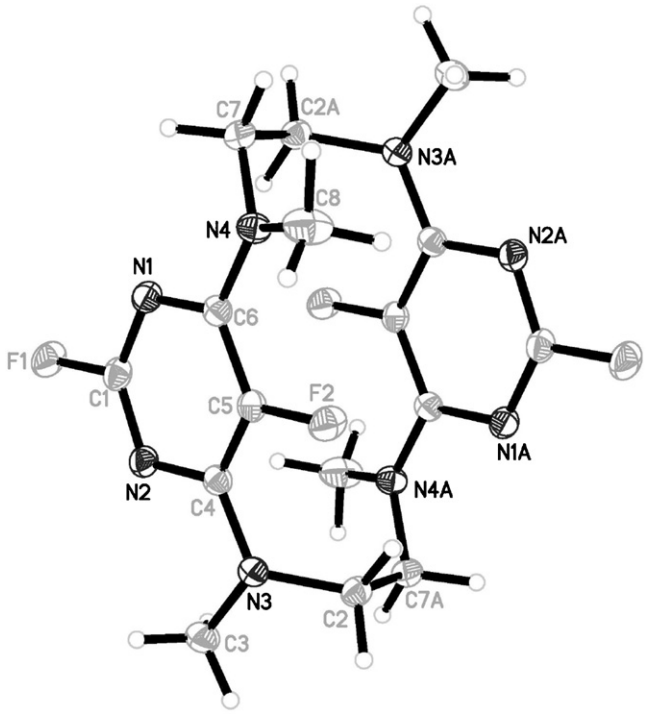


Fig. 1. (*Continued*).

*5.1.1. N,N′-Dimethyl-N,N′-bis-(2,3,5,6-tetrafluoro-pyridin-4-yl)-4,4′-diaminodiphenyl ether **11a***

Methyl iodide (0.84 g, 5.8 mmol) and **9a** (1.47 g, 2.9 mmol) gave *N*,*N*′-dimethyl-*N*,*N*′-bis-(2,3,5,6-tetrafluoro-pyridin-4-yl)-4,4′-diaminodiphenyl ether **11a** (1.38 g, 89%); mp 87–89 °C (found: C, 54.6; H, 2.4; N, 10.5. $C_{24}H_{14}F_8N_4O$ requires C, 54.8; H, 2.7; N, 10.6%); δ_H 3.50 (6H, m, CH_3), 6.98 (8H, m, aryl-H); δ_C 41.3 (t, $^4J_{CF}$ 4.4, CH_3), 119.8 (s, C-2′), 121.2 (s, C-3′), 136.8 (ddd, $^1J_{CF}$ 256.9, $^2J_{CF}$ 22.7, $^3J_{CF}$ 4.7, C-3) 138.3 (m, C-4), 142.0 (s, C–N), 144.9 (ddd, $^1J_{CF}$ 241.3, $^2J_{CF}$ 16.3, $^3J_{CF}$ 3.2, C-2), 153.7 (s, C–O); δ_F −92.3 (2F, m, F-2), −149.2 (2F, m, F-3); *m*/*z* (EI$^+$) 526 ([M]$^+$, 100%), 497 (100), 255 (62).

*5.1.2. N,N′-Dimethyl-N,N′-bis-(2,3,5,6-tetrafluoro-pyridin-4-yl)-diphenylmethane 4,4′-diamine **11b***

Methyl iodide (0.84 g, 5.8 mmol) and **9b** (1.45 g, 2.9 mmol) gave *N*,*N*′-dimethyl-*N*,*N*′-bis-(2,3,5,6-tetrafluoro-pyridin-4-yl)-diphenylmethane 4,4′-diamine **11b** (1.43 g, 92%); mp 93–96 °C (found: C, 57.0; H, 2.9; N, 10.6. $C_{25}H_{16}F_8N_4$ requires C, 57.3; H, 3.1; N, 10.7%); δ_H 3.47 (6H, m, CH_3), 3.93 (2H, s, CH_2), 6.92 (4H, m, aryl-H), 7.12 (4H, m, aryl-H); δ_C 40.5 (s, CH_2), 40.7 (t, $^4J_{CF}$ 4.1, CH_3), 119.0 (s, C-2′), 130.0 (s, C-3′), 137.2 (ddd, $^1J_{CF}$ 256, $^2J_{CF}$ 22, $^3J_{CF}$ 5.76, C-3), 136.3 (s, C–CH_2), 138.1 (m, C-4), 144.3 (s, C–N), 144.9 (ddd, $^1J_{CF}$ 243, $^2J_{CF}$ 16.6, $^3J_{CF}$ 3.1, C-2); δ_F −93.2 (2F, m, F-2), −148.1 (2F, m, F-3); *m*/*z* (EI$^+$) 524 ([M]$^+$, 100%), 345 (70), 269 (62), 164 (60).

6. Synthesis of macrocycles

6.1. General procedure

Sodium hydrogen carbonate was added to a solution of bridged compounds **11** or **13** and diamine in dry acetonitrile (200 mL) under an atmosphere of dry nitrogen. The reaction mixture was heated to reflux temperature for 72 h. After cooling, solvent was evaporated, the organic products were extracted into dichloromethane (3× 30 mL), dried ($MgSO_4$) and the solvent evaporated. The crude product was purified by column chromatography using ethyl acetate/*n*-hexane (1:3) and recrystalisation.

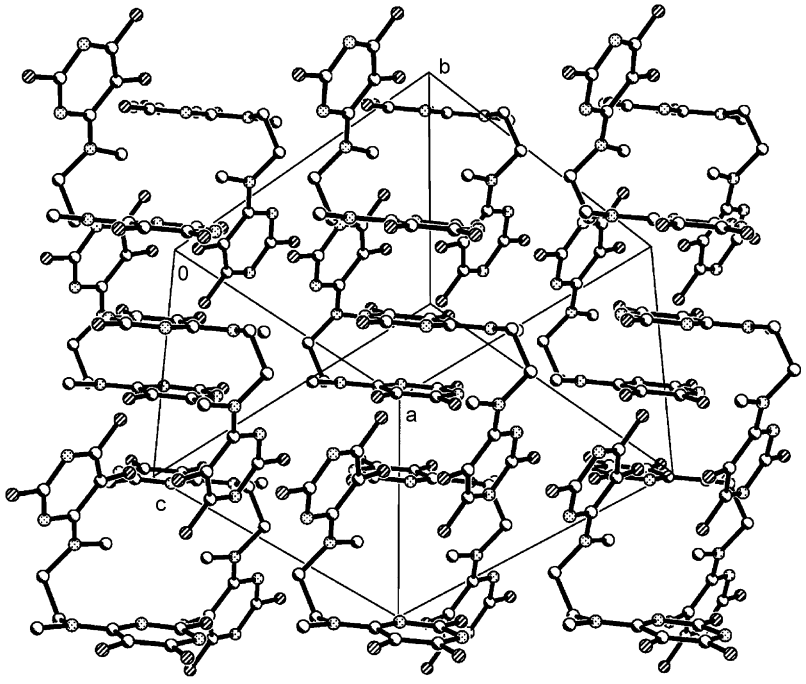


Fig. 2. Packing in a crystal of **13**.

6.2. Macrocycle **12a**

Sodium hydrogen carbonate (0.35 g, 5 mmol), **11a** (1.31 g, 2.5 mmol) and *N*,*N*′-dimethylethylene diamine **10** (0.22 g, 2.5 mmol) gave macrocycle **12a** (0.43 g, 30%) as a white solid; mp 161–163 °C (found: C, 58.4; H, 4.2; N, 14.5%. $C_{28}H_{24}F_6N_6O$ requires C, 58.5; H, 4.2; N 14.6%); δ_H 3.29 (6H, s, CH_3), 3.40 (4H, s, CH_2), 3.45 (6H, s, CH_3), 6.82–6.92 (8H, m, aryl-H); δ_F −93.0 (1F, m, F-2), −141.0 (1F, m, F-5), −158.6 (1F, m, F-3); *m*/*z* (EI^+) 574 ($[M]^+$, 10%), 489 (37), 349 (100), 226 (21).

6.3. Macrocycle **12b**

Sodium hydrogen carbonate (0.35 g, 5 mmol), **11b** (1.31 g, 2.5 mmol) and *N*,*N*′-dimethylethylene diamine **10** (0.22 g, 2.5 mmol) gave macrocycle **12b** (0.48 g, 34%) as a white solid; mp 152–154 °C (found: C, 60.7; H, 4.5; N, 14.6. $C_{29}H_{26}F_6N_6$ requires C, 60.9; H, 4.6; N 14.7%); δ_H 3.06 (6H, s, CH_3), 3.31 (6H, s, CH_3), 3.51 (4H, s, CH_2), 3.87 (2H, s, CH_2), 6.77 (4H, m, aryl-H), 7.02 (4H, m, aryl-H); δ_F −94.1 (1F, m, F-2), −141.5 (1F, m, F-5), −161.4 (1F, m, F-3); *m*/*z* (EI^+) 572 ($[M]^+$, 5%), 331 (17), 330 (100).

6.4. Macrocycle **14**

Sodium hydrogen carbonate (0.35 g, 5 mmol), **13** (0.6 g, 1.7 mmol) and *N*,*N*′-dimethylethylene diamine **10** (0.14 g, 1.7 mmol) gave macrocycle **14** (0.35 g, 54%); mp 134–136 °C (found: C, 47.9; H, 5.0; N, 27. 9%. $C_{16}H_{20}F_4N_8$ requires C, 48.0; H, 5.0; N 28.0%); δ_H 3.09 (6H, s, CH_3), 3.12 (4H, m, CH_2); δ_C 38.6 (s, CH_3), 49.7 (s, CH_2), 130.8 (dd, $^1J_{CF}$ 236.6, $^4J_{CF}$ 4.5, C-5), 153.84 (m, C-4), 155.2 (dd, $^1J_{CF}$ 204.3, $^4J_{CF}$ 4.1, C-2); δ_F −51.3 (2F, d, $^5J_{FF}$ 11.8, F-2), −89.65 (2F, d, $^5J_{FF}$ 11.8, F-5); *m*/*z* (EI^+) 400 ($[M]^+$, 20%), 226 (30), 200 (75), 186 (100).

6.5. X-ray crystal structure determination of **9b**, **11a**, **13** and **14**[1]

The X-ray single crystal data for the compounds were collected at 120K on Bruker SMART CCD 6000 (**11a**, **13**, **14**) and APEX Proteum-M (**9b**) diffractometers (Mo K(, (= 0.71073 Å, (-scan, 0.3°/frame) equipped with Oxford Cryostream cooling devices. All structures were solved by direct methods and refined by full-matrix least squares on F^2 for all data using SHELXTL software. All non-hydrogen atoms were refined with anisotropic displacement parameters, all H-atoms (except those of the disordered solvent in the structure of **9b**, which were placed in calculated positions and refined in the riding mode) were located on the difference map and refined isotropically.

Crystal data for **9b**: $2(C_{23}H_{12}F_8N_4)\cdot CH_2Cl_2$, M = 1077.66, monoclinic, space group $P2_1/c$, a = 25.9093(7), b = 7.4516(2), c = 26.3977(7) Å, (= 116.29(1)°, U = 4569.3(2) Å^3, F(0 0 0) = 2168, Z = 4, D_c = 1.567 mg m^{-3}, (= 0.253 mm^{-1}. 50402 reflections collected; 12727 unique data (R_{merg} = 0.046). Final w$R_2(F^2)$ = 0.1395 for all data (758 refined parameters), conventional $R(F)$ = 0.0469 for 10,075 reflections with $I \geq 2($, GOF = 1.032.

Crystal data for **11a**: $C_{24}H_{14}F_8N_4O$, M = 526.39, monoclinic, space group $P2_1/c$, a = 14.5346(4), b = 7.8419(2), c = 19.3663(6) Å, (= 102.15(1)°, U = 2157.9(1) Å^3, F(0 0 0) = 1064, Z = 4, D_c = 1.620 mg m^{-3}, (= 0.150 mm^{-1}, 27,215 reflections collected; 5998 unique data (R_{merg} = 0.095). Final w$R_2(F^2)$ = 0.1355 for all data (390 refined parameters), conventional $R(F)$ = 0.0394 for 5007 reflections with $I \geq 2($, GOF = 1.076.

Crystal data for **13**: $C_{12}H_{10}F_6N_6$, M = 352.26, monoclinic, space group $C2/c$, a = 11.4620(3), b = 12.2442(4), c = 9.9728(3) Å, (= 90.45(1)°, U = 1399.69(7) Å^3, F(0 0 0) = 712, Z = 4, D_c = 1.672 mg m^{-3}, (= 0.163 mm^{-1}, 8624 reflections collected; 1871 unique data (R_{merg} = 0.067). Final w$R_2(F^2)$ = 0.1350 for all data (133 refined parameters), conventional $R(F)$ = 0.0429 for 1514 reflections with $I \geq 2($, GOF = 1.079. Molecule is located on the twofold axis.

Crystal data for **14**: $C_{16}H_{20}F_4N_8$, M = 400.40, monoclinic, space group $P2_1/c$, a = 8.7586(2), b = 11.4815(3), c = 8.4589(2) Å, (= 93.07(1)°, U = 849.42(4) Å^3, F(0 0 0) = 416, Z = 2, D_c = 1.565 mg m^{-3}, (= 0.132 mm^{-1}, 10,668 reflections collected; 2360 unique data (R_{merg} = 0.044). Final w$R_2(F^2)$ = 0.1366 for all data (167 refined parameters), conventional $R(F)$ = 0.0381 for 2133 reflections with $I \geq 2($, GOF = 1.185.

[1] CCDC 667916–667919 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).

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