Electronic Supplementary Material (ESI) for CrystEngComm

Supporting Information

Anti-Parallel Sheet Structures of Side-Chain-Free γ-, δ-, and ε-Dipeptides Stabilized by Benzene-Pentafluorobenzene Stacking

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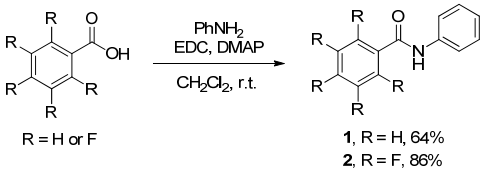
Contents

I. Synthesis and characterization of Compounds 1, 2, 3a–7a, and 5b–7b

General Considerations. All reactants were used as purchased. The ^{1}H, ^{19}F, and ^{13}C NMR spectra were recorded on a Varian Mercury 300, Jeol ECA 400 or Bruker Avance DPX 400 or 500 spectrometer; the chemical shifts are reported in ppm and were referenced to TMS as an internal standard for ^{1}H and ^{13}C NMR and were referenced to $CFCl_3$ as external standard for ^{19}F NMR. High resolution mass spectrometric measurements were obtained on a Bruker microTOF II instrument.

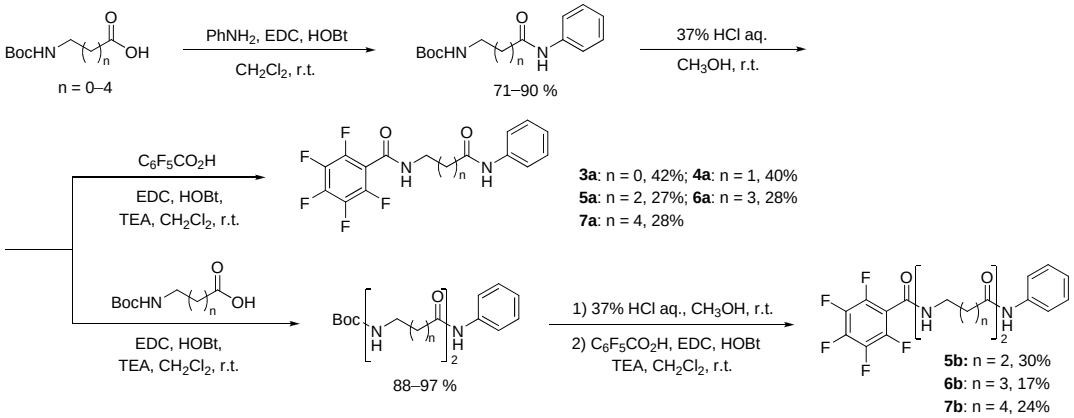
Scheme S1. Synthesis of compounds **1** and **2**.



Compound 1. The compound was prepared according to reported method.[1] ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.87 (d, J = 7.2 Hz, 2H), 7.82 (brs, 1H), 7.64 (d, J = 8.0 Hz, 2H), 7.56 (t, J = 7.6 Hz, 1H), 7.49 (t, J = 7.6 Hz, 2H), 7.38 (t, J = 7.6 Hz, 2H), 7.16 (t, J = 7.6 Hz, 1H).

$C_6F_5CONHPh$ (2). The compound was prepared as a white solid in 86% yield according to reported method.[2] ^{1}H NMR (400 MHz, DMSO-d_6): δ 10.99 (s, 1H), 7.66 (d, J = 7.2 Hz, 2H), 7.40 (t, J = 7.6 Hz, 2H), 7.18 (t, J = 7.2 Hz, 1H).

Scheme S2. Synthesis of compounds **3**–**7**.



C_6F_5CO-Gly-NHPh (3a). To the mixture of Boc-NHGly-OH (1.59 g, 10 mmol), EDC·HCl (3.83 g, 20 mmol), and HOBt (1.35 g, 10 mmol) in 30 mL of dichloromethane was added aniline (1.82 mL, 20 mmol). Then the solution was

stirred at room temperature for 27 h. The solvent was then removed in vacuo. To the pale brown residue, 25 mL of saturated sodium bicarbonate solution was added. After shaking vigorously a white precipitate was formed. The precipitate was filtrated and dissolved in 100 mL of dichloromethane. The solution was washed with saturated sodium bicarbonate solution, hydrochloric acid (1N), and brine, and then dried with sodium sulfate. The solvent was then removed in vacuo. The pale yellow crude product was subjected to column chromatography (ethyl acetate/hexane 1:2) to give Boc-NHGly-NHPh (1.86 g, 79%) as a white powder. The product was then dissolved in methanol (15 mL) and concentrated hydrochloric acid (37%, 16 mL). The solution was stirred at room temperature for 5 h and then concentrated in vacuo, the resulting crude product, NH_2-Gly-NHPh·HCl salt, was used directly for the next step without further purification. A mixture of crude Gly-NHPh·HCl (1.47 g), pentafluorobenzoic acid (0.85 g, 4.0 mmol), EDC·HCl (1.53 g, 8.0 mmol), HOBt (0.54 g, 4.0 mmol), and triethylamine (2.22 mL, 16 mmol) in dichloromethane (20 mL) was stirred at room temperature for 22 h. The solvent was then removed in vacuo. To the resulting pale yellow residue was added saturated sodium bicarbonate solution (30 mL). After shaking vigorously a white precipitate was formed. The precipitate was filtrated and washed with water, and dried in vacuo and then recrystallized from methanol to afford **3a** as a colorless needle solid (0.53 g, 42%). ^{1}H NMR (400 MHz, DMSO-d_6): δ 10.17 (s, 1H), 9.30 (brs, 1H), 7.60 (d, J = 8.0 Hz, 2H), 7.32 (t, J = 7.6 Hz, 2H), 7.06 (t, J = 7.2 Hz, 1H), 4.14 (d, J = 5.2 Hz, 2H). ^{13}C NMR (125 MHz, DMSO-d_6): δ 166.9, 157.6, 143.6 (d, J = 242.8 Hz), 141.6 (d, J = 258.9 Hz), 139.2, 137.4 (d, J = 251.4 Hz), 129.2, 123.8, 119.5, 112.8, 43.6. ^{19}F NMR (376 MHz, DMSO-d_6) δ –141.5 (dd, J = 22.9, 5.3 Hz, 2F), –153.0 (t, J = 21.8 Hz, 1F), –161.6 (td, J = 22.9, 5.6 Hz, 2F). HRMS (ESI): Calcd. for $C_{15}H_9F_5N_2O_2Na$: 367.0482. Found: 367.0489 $[M + Na]^+$, 711.1058 $[2M + Na]^+$.

The following compounds were prepared according to the similar procedure.

C_6F_5CO-*h*Gly-NHPh (4a). White solid in 33% yield for three steps. ^{1}H NMR (400 MHz, DMSO-d_6): δ 10.00 (s, 1H), 9.07 (brs, 1H), 7.60 (d, J = 8.0 Hz, 2H), 7.29 (t, J = 7.6 Hz, 2H), 7.03 (t, J = 7.6 Hz, 1H), 3.55 (q, J =6.0 Hz, 2H), 2.63 (t, J = 6.8 Hz, 2H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 168.9, 156.3, 143.0 (d, J = 243.5 Hz), 141.0 (d, J = 246.4 Hz), 139.1, 136.8 (d, J = 240.6 Hz), 128.6, 123.1, 119.1, 112.6 (t, J = 21.1 Hz), 35.8, 35.7. ^{19}F NMR (376 MHz, DMSO-d_6) δ –142.2 (dd, J = 24.4, 8.3 Hz, 2F),

–153.2 (t, J = 21.8 Hz, 1F), –161.4 (td, J = 22.9, 6.8 Hz, 2F). HRMS (ESI): Calcd. for $C_{16}H_{11}F_5N_2O_2Na$ $[M + Na]^+$: 381.0638. Found: 381.0629.

C_6F_5CO-GABA-NHPh (5a). White solid in 19% yield for three steps. ^1H NMR (400 MHz, DMSO-d_6): δ 9.91 (s, 1H), 8.98 (t, J = 4.8 Hz, 1H), 7.59 (d, J = 8.0 Hz, 2H), 7.28 (t, J = 8.0 Hz, 2H), 7.02 (t, J = 7.8 Hz, 1H), 3.35–3.30 (m, 2H), 2.39 (t, J = 7.6 Hz, 2H), 1.86–1.79 (m, 2H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 170.6, 156.5, 143.1 (d, J = 247.3 Hz), 141.0 (d, J = 250.9 Hz), 139.2, 136.9 (d, J = 250.8 Hz), 128.4, 122.8, 119.0, 112.7 (t, J = 21.2 Hz), 39.0, 33.4, 24.7. ^{19}F NMR (376 MHz, $CDCl_3$) δ –140.4 (d, J = 20.3 Hz, 2F), –150.6 (t, J = 21.8 Hz, 1F), –159.8 (t, J = 21.8 Hz, 2F). HRMS (ESI): Calcd. for $C_{17}H_{13}F_5N_2O_2Na$ $[M+Na]^+$: 395.0795. Found: 395.0805.

C_6F_5CO-$(GABA)_2$-NHPh (5b). The intermediate GABA-NHPh·HCl was synthesized following the procedure described above for the preparation of NH_2-Gly-NHPh·HCl. Then a mixture of GABA-NHPh·HCl (5.04 g, 27 mmol), Boc-GABA-OH (4.06 g, 20 mmol), EDC·HCl (6.20 g, 32 mmol), HOBt (2.70 g, 20 mmol), and triethylamine (12.5 mL, 90 mmol) in 80 mL of dichloromethane was stirred at room temperature for 20 h. The solvent was then removed in vacuo to give a brown residue, which wan further suspended in saturated sodium bicarbonate solution (80 ml). The mixture was extracted by chloroform (80 mL × 3) and the organic phases were combined and washed with hydrochloric acid (1N, 80 mL), water (80 mL), and brine (80 mL), and dried anhydride sodium sulfate. Evaporation of the solvent afforded Boc-$(GABA)_2$-NHPh as a white solid (6.80 g, 94%). To the mixture of Boc-$(GABA)_2$-NHPh (5.08 g, 14.0 mmol) in methanol (mL) was added concentrated hydrochloric acid (25 mL). The solution was stirred at room temperature for 3 h and then concentrated with a rotavapor to afford $(GABA)_2$-NHPh·HCl as a white solid. A mixture of $(GABA)_2$-NHPh·HCl (4.11 g, 8.0 mmol), pentafluorobenzoic acid (1.70 g, 8.0 mmol), EDC·HCl (3.06 g, 16 mmol), HOBt (1.08 g, 8.0 mmol), and triethylamine (7 mL, 50 mmol) in dichloromethane (100 mL) was stirred at room temperature for 20 h. The solvent was then removed in vacuo. After workup, the crude product was separated by flash column chromatography (CH_2Cl_2/MeOH 15:1) to give **5b** as a white solid (1.08 g, 30%). ^1H NMR (500 MHz, DMSO-d_6): δ 9.87 (s, 1H), 8.93 (t, J = 5.0 Hz, 1H), 7.86 (t, J = 5.0 Hz, 1H), 7.58 (d, J = 8.0 Hz, 2H), 7.27 (t, J = 7.5 Hz, 2H), 7.01 (t, J = 7.5 Hz, 1H), 3.25 (q, J = 6.5 Hz, 2H), 3.08 (q, J = 6.5 Hz, 2H), 2.30 (t, J = 7.5 Hz, 2H), 2.14 (t, J = 7.5 Hz, 2H), 1.75–1.68 (m, 4H). ^{13}C NMR (125 MHz,

DMSO-d_6): δ 172.0, 171.4, 156.9, 143.5 (d, J = 258.0 Hz), 141.5 (d, J = 242.8 Hz), 139.8, 137.4 (d, J = 252.2 Hz), 129.1, 123.4, 119.5, 113.2, 39.6, 38.6, 34.4, 33.1, 25.7, 25.5. ^{19}F NMR (376 MHz, DMSO-d_6): δ –142.1 (dd, J = 23.3, 6.8 Hz, 2F), –153.3 (t, J = 21.4 Hz, 1F), –161.4 (td, J = 22.9, 6.8 Hz, 2F). HRMS (ESI): Calcd. for $C_{21}H_{20}F_5N_3O_3Na$ $[M+Na]^+$: 480.1322; Found: 480.1314.

***F*$_5$-PhCO-AVA-NHPh (6a).** (AVA, 5-aminovaleric acid) The synthesis was followed the representative procedure which was described as the synthesis of compound **3a**. White solid in 25% yield for three steps. ^{1}H NMR (400 MHz, DMSO-d_6) δ 9.88 (s, 1H), 8.93 (t, J = 5.2 Hz, 1H), 7.59 (d, J = 8.0 Hz, 2H), 7.28 (t, J = 8.0 Hz, 2H), 7.02 (t, J = 7.2 Hz, 1H), 3.31–3.28 (m, 2H), 2.35 (t, J = 7.2 Hz, 2H), 1.67–1.61 (m, 2H), 1.58–1.53 (m, 2H); ^{13}C NMR (125 MHz, DMSO-d_6) δ 171.5, 156.9, 143.5 (d, J = 249.4 Hz), 141.5 (d, J = 255.1 Hz), 139.8, 137.3 (d, J = 243.8 Hz), 129.0, 123.3, 119.5, 113.3, 39.6, 36.3, 28.8, 22.9; ^{19}F NMR (376 MHz, DMSO-d_6) δ –142.2 (dd, J = 23.3, 5.6 Hz, 2F), –153.3 (t, J = 23.3 Hz, 1F), –161.3 (td, J = 21.8, 6.8 Hz, 2F); HRMS (ESI): Calcd. For $C_{18}H_{16}F_5N_2O_2$ $[M+H]^+$: 387.1132; Found: 387.1130.

The following compounds were prepared according to procedures similar to those described for the preparation of **6a**.

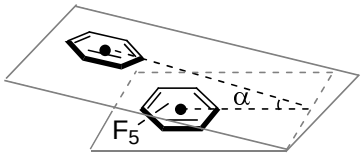
C$_6$F$_5$CO-(AVA)$_2$-NHPh (6b). White solid in 15% yield for five steps. ^{1}H NMR (400 MHz, DMSO-d_6): δ 9.85 (s, 1H), 8.89 (t, J = 5.2 Hz, 1H), 7.80 (t, J = 5.2 Hz, 1H), 7.57 (d, J = 8.0 Hz, 2H), 7.27 (t, J = 8.0 Hz, 2H), 7.01 (t, J = 7.6 Hz, 1H), 3.26 (q, J = 6.4 Hz, 2H), 3.06 (q, J = 6.4 Hz, 2H), 2.30 (t, J = 7.2 Hz, 2H), 2.08 (t, J = 6.8 Hz, 2H), 1.59–1.40 (m, 8H). ^{13}C NMR (125 MHz, DMSO-d_6) δ 172.2, 171.6, 156.8, 143.5 (d), 141.5 (d), 139.8, 137.3 (d), 129.0, 123.3, 119.4, 113.3, 39.6, 38.6, 36.5, 35.4, 29.3, 28.8, 23.1, 23.0. ^{19}F NMR (376 MHz, DMSO-d_6): δ –142.2 (dd, J = 23.3, 5.6 Hz, 2F), –153.3 (t, J = 21.8 Hz, 1F), –161.3 (td, J = 22.2, 5.6 Hz, 2F). HRMS (ESI): Calcd. for $C_{23}H_{25}F_5N_3O_3$ $[M+H]^+$: 486.1816. Found: 486.1824.

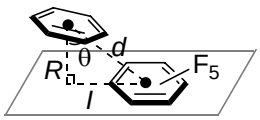
C$_6$F$_5$CO-EACA-NHPh (7a). White solid in 22% yield for three steps. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.49–7.42 (m, 3H), 7.32–7.30 (m, 2H), 7.09 (t, J = 7.2 Hz, 1H), 6.71 (br, 1H), 3.52 (q, J = 6.4 Hz, 2H), 2.39 (t, J = 7.2 Hz, 2H), 1.79–1.65 (m, 4H), 1.49–1.45 (m, 2H). ^{13}C NMR (100 MHz, DMSO-d_6): δ 171.1, 156.4, 143.0 (d), 141.0 (d), 139.3, 136.9 (d), 128.4, 122.8, 119.0, 112.8 (t), 39.1, 36.3, 28.5, 25.9, 24.7. ^{19}F NMR (282 MHz, $CDCl_3$): δ –141.0 (dd, J = 21.7, 5.9 Hz, 2F), –151.5 (t, J = 21.7 Hz, 1F), –160.5 (td, J = 21.9, 8.2 Hz, 2F). HRMS (ESI): Calcd. for $C_{19}H_{17}F_5N_2O_2Na$ $[M+Na]^+$: 423.1108. Found: 423.1116.

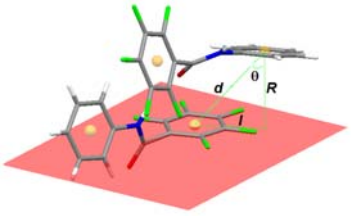
***F*$_5$-PhCO-(EACA)$_2$-NHPh (7b).** White solid in 16% yield for five steps. ^{1}H NMR (400 MHz, DMSO-d_6): δ 9.83 (s, 1H), 8.89 (t, J = 5.2 Hz, 1H), 7.75 (t, J = 5.2 Hz, 1H), 7.58 (d, J = 8.4 Hz, 2H), 7.27 (t, J = 8.0 Hz, 2H), 7.01 (t, J = 7.2 Hz, 1H), 3.25 (q, J = 6.4 Hz, 2H), 3.02 (q, J = 6.4 Hz, 2H), 2.28 (t, J = 7.2 Hz, 2H), 2.04 (t, J = 6.8 Hz, 2H), 1.59–1.51 (m, 2H), 1.51–1.46 (m, 4H), 1.42–1.37 (m, 2H), 1.30–1.26 (m, 4H). ^{13}C NMR (125 MHz, DMSO-d_6): δ 172.3, 171.6, 156.8, 143.4 (d, J = 247.5 Hz), 141.4 (d, J = 256.6 Hz), 139.8, 137.3 (d, J = 244.6 Hz), 129.0, 123.3, 119.5, 113.3, 39.7, 38.8, 36.8, 35.8, 29.5, 28.9, 26.6, 26.4, 25.4, 25.3. ^{19}F NMR (376 MHz, DMSO-d_6): δ –142.3 (dd, J = 23.3, 6.8 Hz, 2F), –153.3 (t, J = 21.8 Hz, 1F), –161.3 (td, J = 23.3, 5.3 Hz, 2F). HRMS (ESI): Calcd. for $C_{25}H_{29}F_5N_3O_3$ $[M+H]^+$: 514.2129. Found: 514.2116.

II. Geometric parameters and intermolecular hydrogen bonding

Table S1. Geometric Parameters and Intermolecular Hydrogen Bonding of Compounds **1** and **2** in Crystal[a)]







compound	α [°]	d [Å]	R[b)] [Å]	I[c)] [Å]	θ[d)] [°]	$d_{NH\cdots O}$ [Å]	∠NH···O [°]
1	–	5.35	2.75	4.59	59.1	2.41	144.3
2	15.8	4.78	3.64	3.10	40.4	2.02	167.5
		4.54	3.79	2.50	33.4		

a) Geometric parameters used to define the orientation of the two interacting aromatic rings are depicted in the figures; b) the distance of the benzene ring centroid to the plane defined by the opposite pentafluorobenzene ring; c) the horizontal displacement between two ring centroids, calculated by Pythagorean theorem $\{I = (d^2 - R^2)^{1/2}\}$; d) the displacement angle, calculated by Law of cosines $\{\theta = \cos^{-1}[(d^2 + R^2 - I^2) / (2 \cdot d \cdot R)]\}$.

Table S2. Geometric Parameters and Intermolecular Hydrogen Bonding of Compounds **3a**, **4a**, **5a**, and **7a** in Crystal

Compound	α [°]	*d* [Å]	$R^{a)}$ [Å]	$I^{a)}$ [Å]	$\theta^{a)}$ [°]	$d_{NH\cdots O}$ [Å]	∠NH···O[°]
3a	5.8	3.66	3.36	1.45	23.3	2.08	167.6
						2.16	171.2
4a	6.1	3.65	3.39	1.35	21.7	2.00	160.6
						2.14	170.9
5a	6.0	3.72	3.49	1.29	20.3	1.98	169.3
						2.22	170.8
7a	2.1	3.64	3.58	0.66	10.4	2.06	166.5
						2.00	172.3

a) see the note of Table S1.

Table S3. Geometric Parameters and Intermolecular Hydrogen Bonding Values of Triamides **5b–7b** in the Solid State

compound	α [°]	*d* [Å]	$R^{a)}$ [Å]	$I^{a)}$ [Å]	$\theta^{a)}$ [°]	$d_{NH\cdots O}$ [Å]	∠NH···O [°]
5b-1	3.0	3.56	3.34	1.23	20.2	2.02	175.4
						2.01	170.7
						2.13	173.1
5b-2	6.8	3.64	3.36	1.40	22.6	2.06	167.5
						2.10	163.0
						2.24	167.8
6b	19.5	4.08	3.68	1.76	25.6	2.16	168.6
						2.05	172.6
						2.12	158.2
7b	18.6	3.87	3.56	1.52	23.1	2.03	170.2
						2.00	173.5
						2.06	168.6

a) see the note of Table S1.

III. Views of the supramolecular packing of compounds 1, 2, 3a–5a, 7a, and 5b–7b

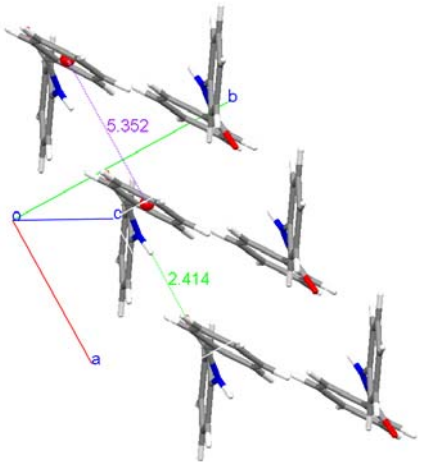


Fig. S1 Views of the supramolecular packing of compound **1**.

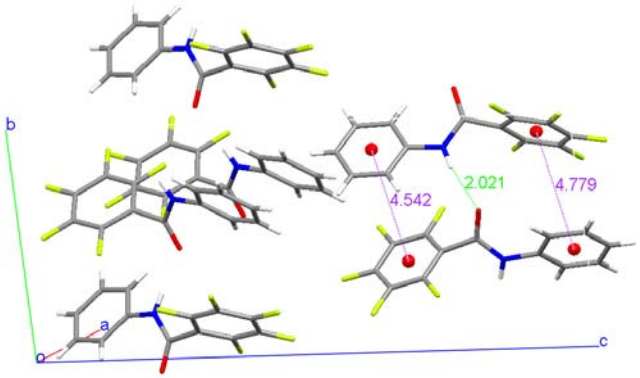


Fig. S2 Views of the supramolecular packing of compound **2**.

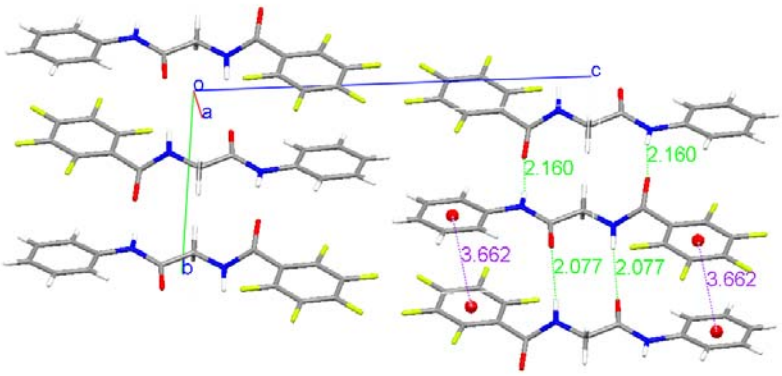


Fig. S3 Views of the supramolecular packing of diamide **3a**.

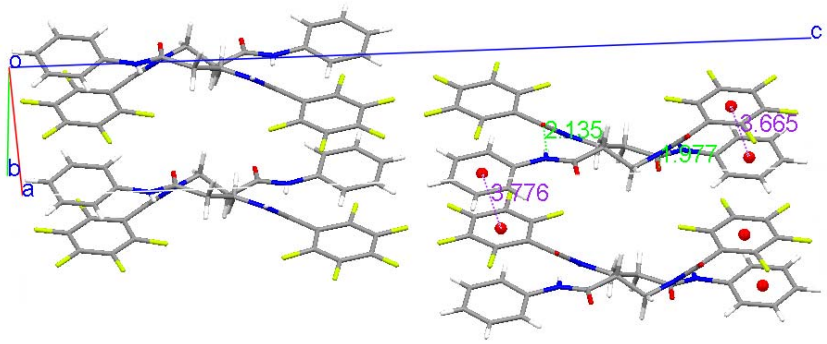


Fig. S4 Views of the supramolecular packing of diamide **4a**.

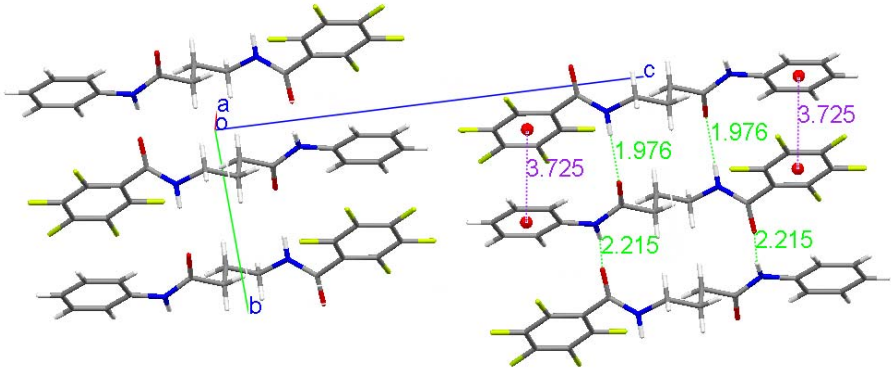


Fig. S5 Views of the supramolecular packing of diamide **5a**.

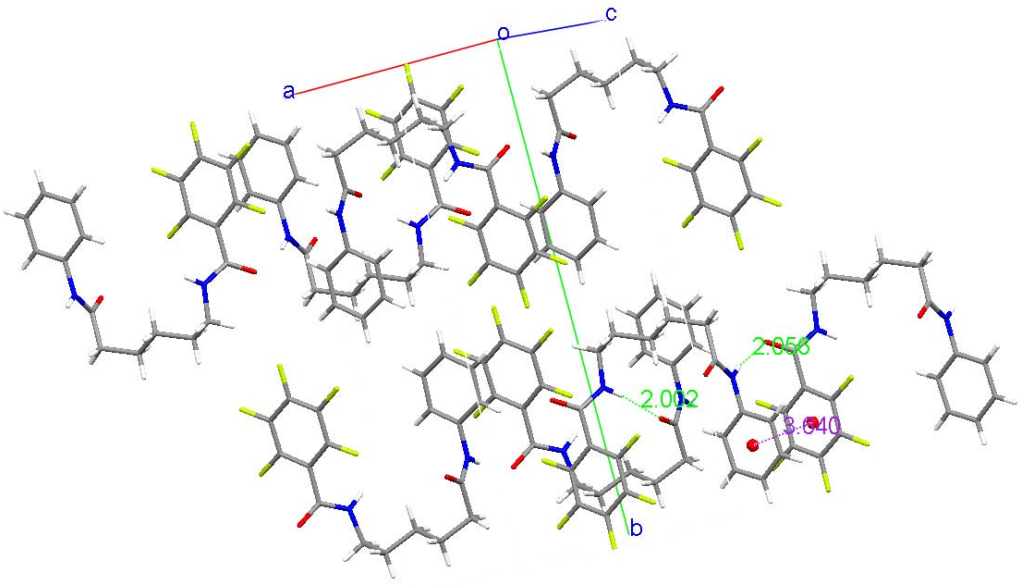


Fig. S6 Views of the supramolecular packing of diamide **7a**.

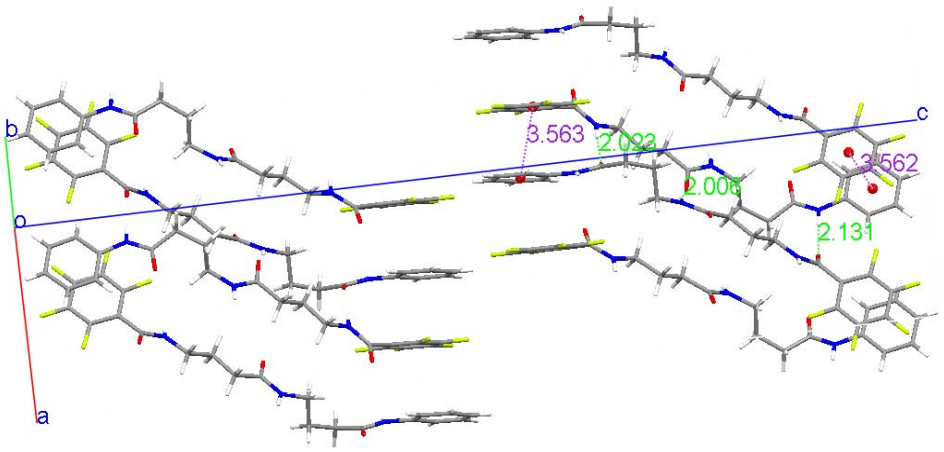


Fig. S7 Views of the supramolecular packing of triamide **5b**.

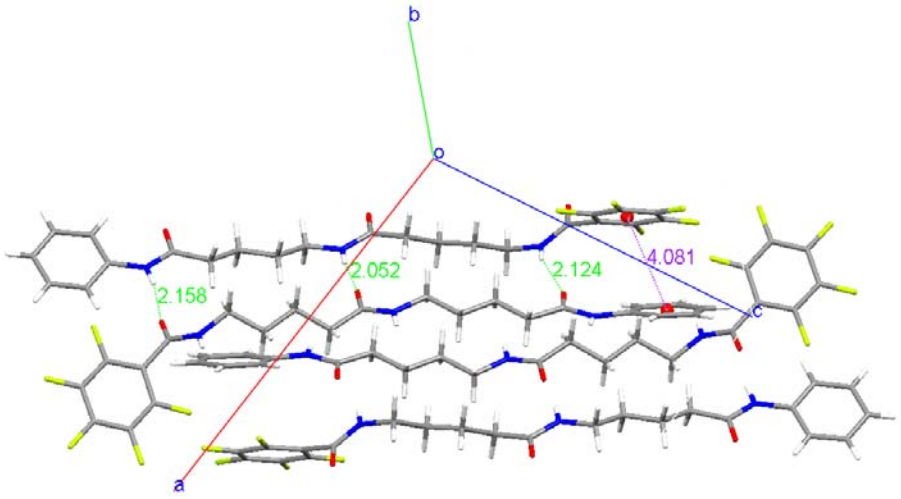


Fig. S8 Views of the supramolecular packing of triamide **6b**.

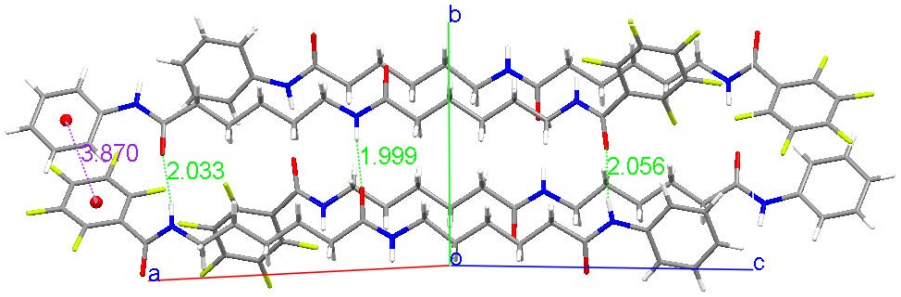


Fig. S9 Views of the supramolecular packing of triamide **7b**.

IV. X-ray analysis of compounds 1, 2, 3a–5a, 7a, and 5b–7b

Crystal Growth. Crystals of compounds **1**, **2**, **3a**–**5a**, **7a**, and **5b**–**7b** suitable for single-crystal X-ray diffraction were obtained as follows. Crystals of **1** were grown by slow evaporation of its dichloromethane solution. Crystals of **2**, **4a**, and **5b**–**7b** were

grown by slow evaporation of their methanol solution. Crystals of **3a** were obtained from recrystallization from methanol. Crystals of **5a** were grown by slow evaporation from its acetone solution. Crystals of **7a** were grown by slow evaporation from its ethyl acetate solution. The crystal data and structure refinements for these compounds were summarized in Table S4.

Crystallographic Studies. X-ray diffraction intensity data for each compound were measured on a graphite-monochromated Bruker SMART APEX CCD-based diffractometer system (Mo K radiation, λ = 0.71073 Å). All the structures were solved by Direct Method of SHELXS-97 and refined by full-matrix leastsquares techniques using the SHELXL-97 program within WINGX.[3] Non-hydrogen atoms of the crystallized compounds were refined with anisotropic temperature parameters. The hydrogen atoms attached to carbons were generated geometrically. Other hydrogen atoms were located from difference Fourier maps and refined with isotropic displacement parameters.

Table S4. X-ray Data Collection and Structure Analysis Details for Compounds **1**, **2**, **3a**, **4a**, **5a**, **7a**, **5b**, **6b**, and **7b**

	1	**2**	**3a**	**4a**	**5a**	**7a**	**5b**	**6b**	**7b**
CCDC number	965773	965775	965769	965772	965768	965771	965767	965774	965770
formula	$C_{13}H_{11}NO$	$C_{13}H_6F_5NO$	$C_{15}H_9F_5N_2O_2$	$C_{16}H_{11}F_5N_2O_2$	$C_{17}H_{13}F_5N_2O_2$	$C_{19}H_{17}F_5N_2O_2$	$C_{21}H_{20}F_5N_3O_3$	$C_{23}H_{24}F_5N_3O_3$	$C_{25}H_{28}F_5N_3O_3$
fw [$gmol^{-1}$]	197.23	287.18	344.24	358.27	372.29	400.34	457.39	485.45	513.50
cryst system	triclinic	orthorhombic	triclinic	triclinic	triclinic	monoclinic	monoclinic	monoclinic	monoclinic
space group	*P*–1	$P2_12_12_1$	*P*–1	*P*–1	*P*–1	$P2_1/c$	$P2_1/c$	$P2_1/c$	$P2_1/c$
density [gcm^{-3}]	1.311	1.578	1.657	1.608	1.560	1.442	1.487	1.429	1.421
T [K]	293(2)	293(2)	293(2)	293(2)	293(2)	293(2)	293(2)	293(2)	140(2)
a [Å]	5.352(6)	5.434(4)	6.214(6)	6.235(4)	6.303(6)	8.7960(9)	10.4352(16)	16.8272(18)	18.850(3)
b [Å]	7.971(8)	9.157(7)	7.246(7)	7.257(5)	7.483(7)	20.174(2)	9.1374(13)	9.5857(10)	9.5190(13)
c [Å]	12.471(13)	24.300(19)	15.518(14)	32.88(2)	17.132(15)	10.7947(12)	42.862(7)	13.9962(14)	13.5917(19)
α [deg]	73.23(2)	90.00	93.01(1)	87.83(1)	85.19(2)	90.00	90.00	90.00	90.00
β [deg]	79.20(2)	90.00	96.52(1)	89.41(1)	81.42(2)	105.70(3)	90.359(3)	91.955(2)	100.231(2)
γ [deg]	89.85(2)	90.00	95.24(1)	84.55(1)	83.94(2)	90.00	90.00	90.00	90.00
V [$Å^3$]	499.6(9)	1209.1(16)	689.9(11)	1480.1(1)	792.6(12)	1844.0(3)	4086.8(11)	2256.3(4)	2400.1(6)
Z	2	4	2	4	2	4	8	4	4

μ [mm^{-1}]	0.083	0.153	0.157	0.149	0.143	0.128	0.131	0.123	0.120
θ_{min} [deg]	2.73	2.38	1.32	0.62	1.20	2.02	1.90	2.42	2.40
θ_{max} [deg]	25.50	26.00	25.01	25.50	25.50	26.0	26.00	25.50	26.00
no. of reflns collected	1847	7275	2884	7991	4193	11065	23870	12875	16414
no. of unique reflns	1847	2383	2396	5441	2882	3616	8018	4208	4702
no. of reflns observed	1254	2014	1852	4273	1989	2174	8018	4208	4702
threshold exp	> 2σ(I)	> 2σ(I)	> 2σ(I)	> 2σ(I)	> 2σ(I)	> 2σ(I)	> 2σ(I)	> 2σ(I)	> 2σ(I)
no. of params	141	185	225	452	244	261	601	320	326
no. of restraints	2	2	2	1	0	0	6	0	0
R_1, I > 2σ(I)	0.0646	0.0377	0.089	0.0749	0.0565	0.0463	0.0638	0.0442	0.0811
wR_2 (All)	0.1886	0.1093	0.2584	0.2331	0.1750	0.1318	0.1862	0.1439	0.3173

Reference

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