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HETEROCYCLES IN ORGANIC MATERIALS CHEMISTRY. SYNTHESIS OF DI-, TRI-, AND TETRAIMIDE POLYCARBOXYLIC ACIDS FOR USE IN ORGANIC NETWORK ASSEMBLY

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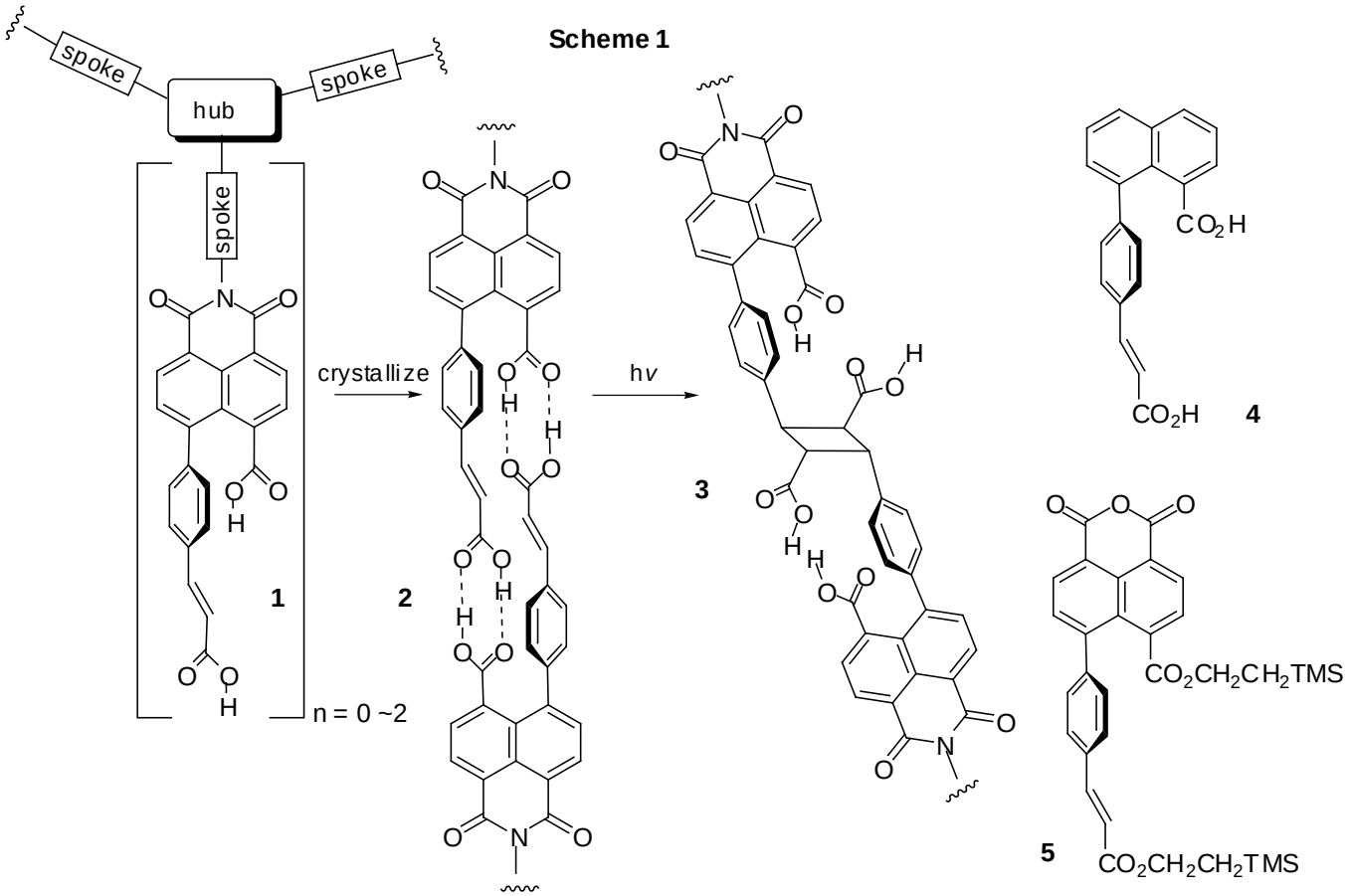
Abstract - Seven linear divalent monomers, three bent divalent amide-containing monomers, 11 bent divalent ester-containing monomers, one planar trivalent monomer, and two tetrahedral tetravalent monomers were prepared by imide-forming condensation of the appropriate polyamine hub with a specially designed anhydride connector unit. These polyvalent polycarboxylic acid monomers were subjected to crystallization/photochemical crosslinking studies in an attempt to prepare periodic, covalent organic solids. Difficulties in achieving suitable crystals have so far frustrated efforts to reach this goal.

The multi-dimensional polymerization of polyvalent monomers to furnish periodic, covalently linked solids remains an enduring challenge in materials chemistry. Problems with orienting monomer units to facilitate bond formation between divergent functionality, and the difficulty of bringing reactive ends together in increasingly constrained lattices as polymerization proceeds, among other factors, have conspired to thwart direct attempts at polyvalent monomer polymerization.[1] An alternative indirect strategy for accomplishing this goal has been under development in our laboratory.[2] This strategy relies on a two-stage sequence featuring initial monomer pre-organization into a periodic lattice, and subsequent *in situ* covalent connection of now proximate functionality on the monomer units.[3] The key connector unit was chosen to provide organizational elements compatible with the bond-forming chemistry, and to present these elements in a disposition that minimizes molecular motion, and therefore lattice distortion, upon covalent union. The generic hub-spoke-connector architecture (**1**) (Scheme 1) was designed to meet

these criteria, where the crucial naphthalene-based connector unit bears nearly parallel carboxylic acid and cinnamic acid moieties extending from the peri positions. Hydrogen bond-mediated dimerization of the acidic termini of two connectors would then position the two alkenes for photochemical crosslinking, **1**→**2**→**3**. This two-step procedure would thus enable monomer pre-organization through reversible and self-correcting H-bond directed crystallization, and then replace the labile H-bond linkage with approximately coincident, robust covalent C-C bonds in the second step. Previous reports from our laboratory have documented the feasibility of this procedure in a simple model system (**4**), and have described the synthesis of the key coupling connector, anhydride (**5**).[2] This account details the synthesis of families of linear divalent, bent divalent, planar trivalent, and tetrahedral tetravalent monomer units based on the hub-spoke-connector motif (**1**). In addition, the results of preliminary crystallization/photochemical crosslinking studies with these polyacid species are disclosed.

Scheme 1



RESULTS AND DISCUSSION

Polyimide Polyacid Synthesis. The imidation chemistry of anhydride (**5**) utilized in the synthesis of the polyfunctional monomers was developed earlier in our laboratory.[4] Simple condensation of the appropriate di-, tri-, or tetraamine core with **5** in THF or a high-boiling aromatic solvent at reflux sufficed

to provide a moderate yield of polyimide in most instances. However, the incorporation of stoichiometric amounts of the mild Lewis acid $Zn(OAc)_2$ in the reaction mixture often led to increased yields. The diamine-derived hubs (**7a**-**7g**) constituted the initial attempts at constructing linear divalent monomers. These species were conveniently prepared by addition of two equivalents of anhydride (**5**) to the appropriate commercially available diamine (**6a**-**6g**) under the influence of $Zn(OAc)_2$, eq. (1) and Table 1. The yields of diimide product (**7**) were variable. The simple aryl- and alkylamine precursors (**6a**-**6c**, **6f**-**6g**) performing acceptably, whereas the perfluoro species (**6d**) and the cyclohexyl-derived diamine (**6e**) gave disappointingly low yields. Electronic effects in the former case and steric effects in the latter example may have contributed to the relatively poor reactivity of these diamines, even at higher temperatures (mesitylene vs. toluene at reflux). Deprotection of the tetraesters (**7a**-**7g**) with Bu_4NF proceeded smoothly with most substrates to yield the desired tetraacids in pure form following precipitation from the reaction medium upon aqueous H_3PO_4 addition. As with the imidation reaction, the octafluoro- and cyclohexyl-based substrates (**7d**) and (**7e**), respectively, afforded the tetraacid products in much diminished yields. It is unclear that the now remote steric and electronic factors cited above should influence these desilylation transformations.

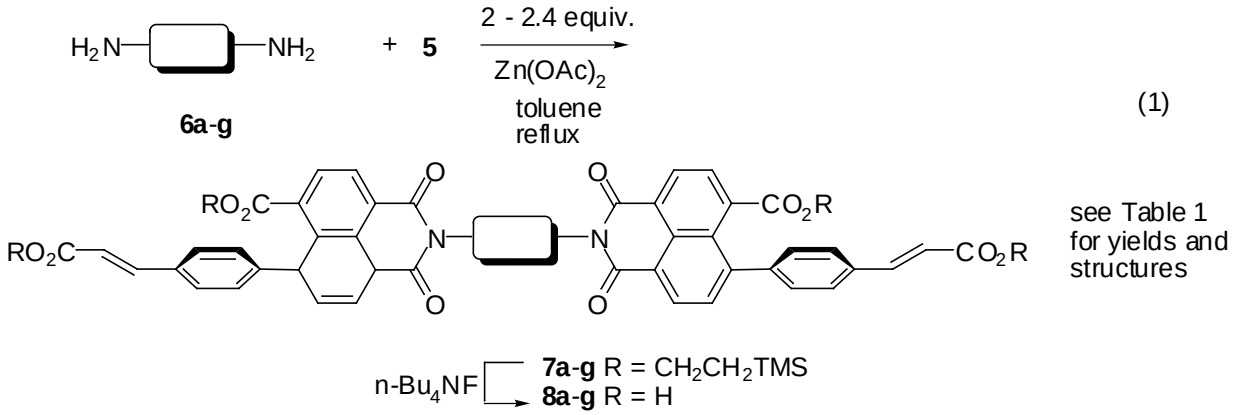
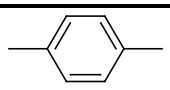
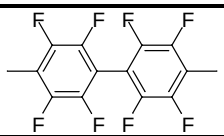
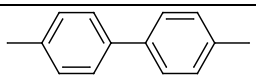
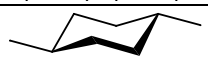
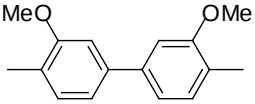
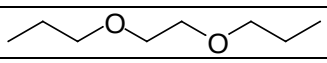


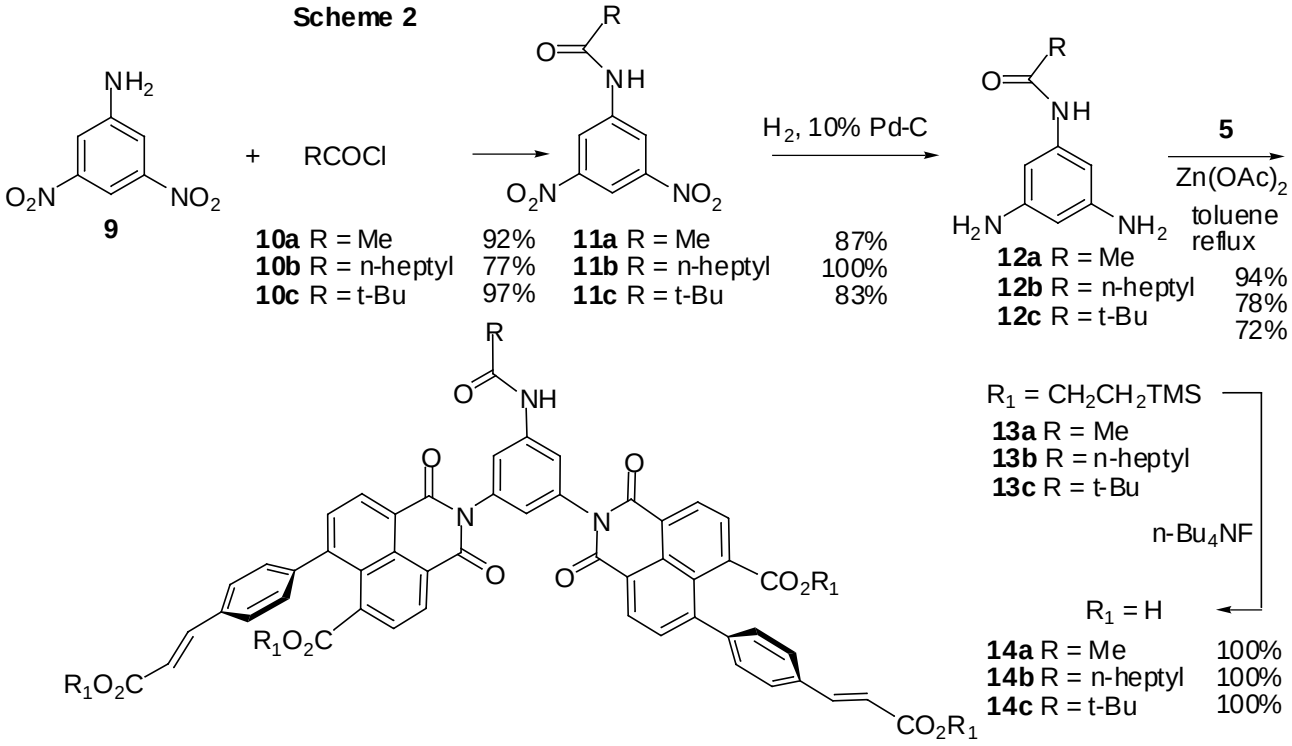
Table 1. Yields of tetraesters (**7**) and tetraacids (**8**) from diamines (**6a**-**6g**) (cf. Eq. (1)).

entry	**6**	**7** %	**8** %	entry	**6**	**7** %	**8** %
a		68	98	*d*		39[a]	34
b		38	98	*e*		38[a]	45
c		59	95	*f*	-$(CH_2)_8$-	69	90
				g		81	85

[a] refluxing mesitylene solvent.

The synthesis of the bent, divalent monomer family commenced with the diamine amide species (**12a**-**12c**), Scheme 2. Variation in the amide's alkyl substituent "R" was introduced in an effort to increase the prospects for obtaining suitably crystalline material. These hubs were readily prepared from the dinitro precursor (**9**) and the appropriate acyl chlorides (**10a**-**10c**). The derived diimides (**13a**-**13c**) then were available by application of the standard $Zn(OAc)_2$-mediated coupling procedure used successfully with the linear divalent monomers (**7a**-**7g**). Ester deprotection within these diimides with n-Bu_4NF furnished the pure tetra acids (**14a**-**14c**) as yellow powders in excellent yield following aqueous H_3PO_4 workup and extraction into EtOAc.

Scheme 2



Concerns with complications that might arise during crystallization of **14a**-**14c** as a consequence of the secondary amide functionality prompted synthesis of analogous ester-substituted bent divalent monomers (**20a**-**20h**), Scheme 3 and Table 2. Acylation of 3,5-dinitrobenzoyl chloride (**15**) with a variety of alcohols (**16a**-**16h**), followed by nitro-to-amine reduction, provided the diamine hubs (**18a**-**18h**) ready for imidation. Unlike the prior series, the benzoyl esters (**19a**-**19h**) did not respond favorably to $Zn(OAc)_2$ catalysis of the condensation with **5**. Low and variable yields resulted with this Lewis acid in refluxing aromatic solvents, and many uncharacterized byproducts accompanied these trials. However, omission of the Lewis acid promoter and inclusion of excess Et_3N furnished the desired diimides (**19a**-**19h**) when the diamines (**18a**-**18h**) were combined with two equivalents of the anhydride (**5**) in the solvent indicated (reflux). The yields of diimide were modest in all cases, but further optimization studies were deferred pending the results of the crystallization/photocrosslinking experiments. Deprotection of the

trimethylsilylethyl ester moieties in **19a-19h** proceeded uneventfully to furnish the desired tetra acids (**20a**-**20h**) as yellow powders.

Scheme 3

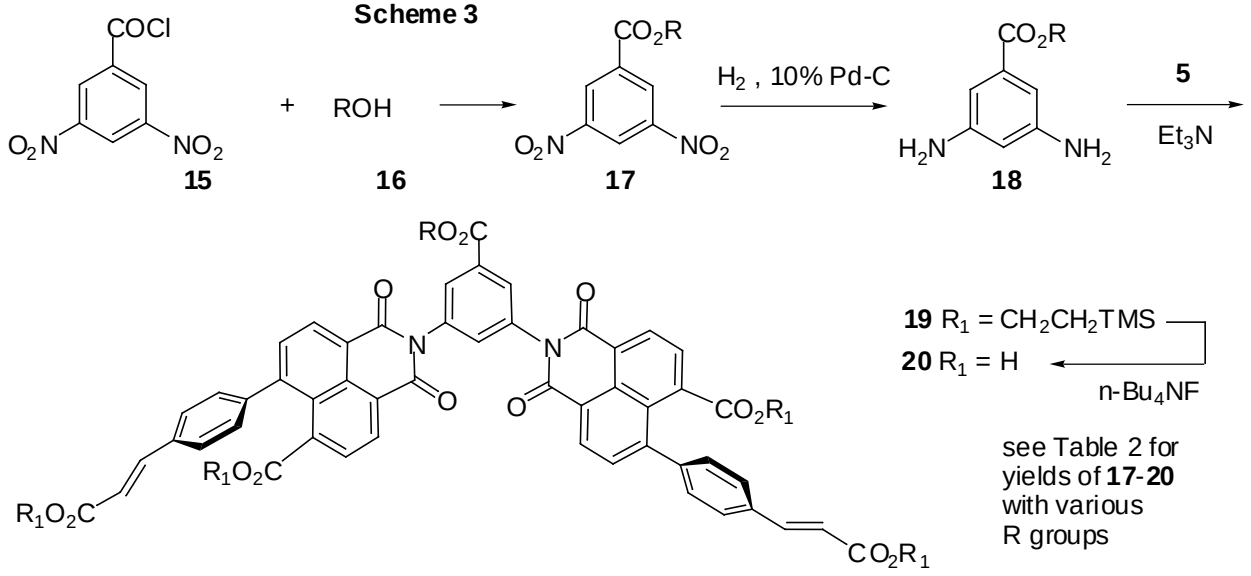
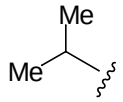
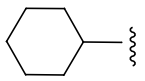
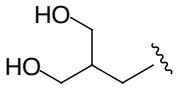
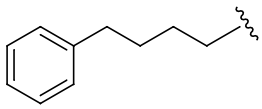
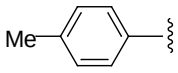
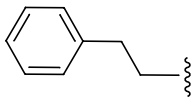
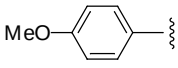


Table 2. Yields of species en route to the bent divalent ester-containing diimides (**20**) (cf. Scheme 3).

entry	R	**17** %	**18** %	**19** %	**20** %	entry	R	**17** %	**18** %	**19** %	**20** %
a		100	94	44[a]	88	*e*	$HO(CH_2)_6$-	50	80	25[b]	57
b		91	70	24[b]	52	*f*		39	88	30[b]	100
c		87	95	34[b]	100	*g*		100	86	51[a]	61
d		87	80	48[a]	97	*h*		98	90	37[b]	90

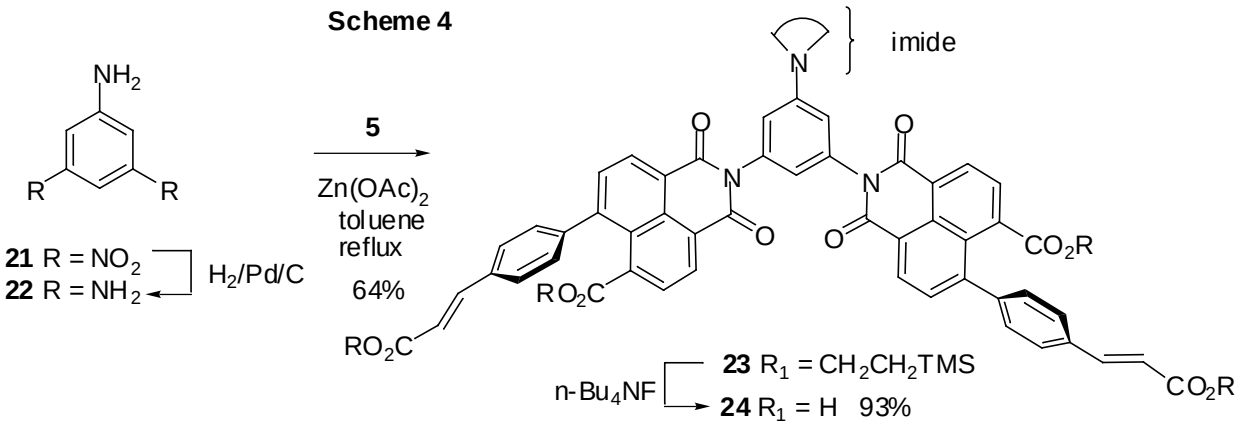
[a] THF solvent. [b] xylene solvent.

A single trivalent hub (**24**) was prepared from the sensitive triamine (**22**)[5] and three equivalents of anhydride (**5**) under $Zn(OAc)_2$ mediation, Scheme 4. Desilylethylation of the six esters within **23** afforded the trimesic acid homologue (**24**) as a yellow solid. As before, even this hexaacid could be isolated in pure form after simple acidification of the crude reaction mixture and extraction into EtOAc.

The preparation of tetrahedral tetravalent analogues of these polyimide constructs required a tetrahedrally divergent tetraamine core. Candidate alkyl- and arylamines include the known species (**25-2**)[6] and the ethyl and propyl chain higher homologues of **27**, **28a** and **28b**, respectively, which were synthesized in-

house (Scheme 5).[7] Attempted tetraimidation of the three tetraamines (**25**, **26**, and **27**) with excess anhydride (**5**) under a variety of experimental conditions led, at best, to only partially imidated products. The rigid tetraamines (**25**) and (**26**) were too insoluble in any of the solvents examined to enter into a productive imidation process at all. The more soluble pentaerythitol derivative (**27**) proceeded as far as a diimide product (15%) upon reaction with excess **5** in refluxing mesitylene. Resubmission of this partially imidated product to more anhydride did not afford further imide formation. Apparently, unfavorable steric constraints hinder attachment of the third or fourth anhydride unit to the now densely packed core. The tetraamines (**28a**) and (**28b**) were designed to alleviate this steric crowding problem. Application of the $Zn(OAc)_2$-mediated condensation conditions to these tetraamines and excess **5** did indeed afford modest yields of the desired tetraimide products (**29a**) and (**29b**). Deprotection of these octaesters with Bu_4NF and extraction of the crude, acidified reaction mixture with EtOAc did not in these cases provide the octaacid products sufficiently pure for the subsequent crystallization studies. Pure yellow, powdery octaacids (**30a**) and (**30b**) could be obtained by dissolution of the impure materials in aqueous base, removal of impurities with an EtOAc wash, reacidification and extraction into EtOAc/THF.

Scheme 4



Crystallization/Photochemistry Studies. The attempts at growing suitable crystals from the MeCN, THF, DME, dioxane, CH_2Cl_2, EtOAc, toluene, benzene, hexane, heptane, and octane) in various combinations within the context of several different crystallization techniques (slow evaporation, slow cooling, vapor diffusion, liquid diffusion). The results were quite variable and no trends emerged. The model system diacid (**4**) furnished high quality 1 mm x 3 mm rods upon vapor diffusion of heptane into a DME solution containing a trace of MeOH at 4 ^{o}C.[2b] The tetra-, hexa- and octaacids described herein had, in general, only limited solubility in ethereal (or less polar) solvents, which rendered many crystallization attempts problematical. Eventually, through extensive trial-and-error, crystals of variable sizes/qualities were deposited from several substrates as indicated in Table 3. One of these crystalline solids proved suitable for X-Ray crystallographic analysis, the needles deposited from tetraacid (**8f**) (Figure 1).

Unfortunately, the disruptive role of DMSO on the intended carboxylic acid H-bond dimerization scheme is evident. The alkene moieties are held far from the critical bonding distance (< 4.2 Å),[8] and not surprisingly, irradiation of a solid crystalline sample of **8f** did not lead to any observed chemistry. Furthermore, in no instance did irradiation of any of the crystalline samples listed in Table 3 furnish any new compounds or any evidence of photooligomerization.

Scheme 5

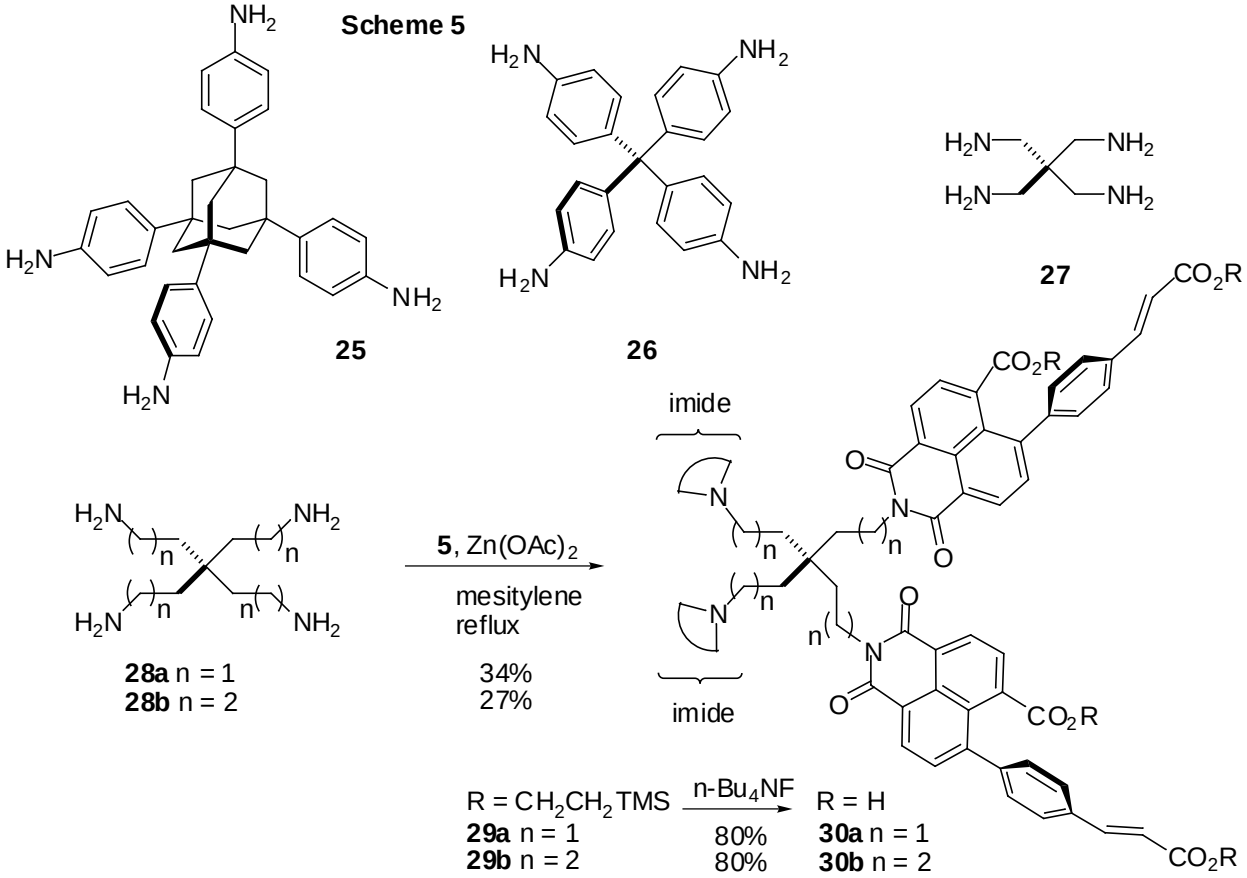


Table 3. Crystals obtained from the polyacid polyimides described herein.

entry	poly-acid	recrystalization solvent; technique	crystal description	entry	poly-acid	recrystalization solvent; technique	crystal description
a	**8f**	DMSO, EtOH; liquid diffusion	needles	*e*	**20d**	THF, DME; slow evaporation	needles
b	**14c**	DME, MeOH, heptane; liquid diffusion	irregular clusters	*f*	**20h**	THF, DMSO; slow evaporation	plates
c	**20a**	THF/i-PrOH, heptane; vapor diffusion	needles	*g*	**30a**	DME; slow cooling	needles
d	**20b**	THF, H_2O; slow evaporation	needles				

In summary, a variety of polyvalent monomers, characterized by divergent poly(carboxylic acid/cinnamic acid)-tipped spokes extending from a central core, were synthesized by dehydrative condensation of polyamine hubs with a key naphthalic anhydride. Crystallization/photochemical crosslinking studies with these species were disappointing in that no evidence for covalent network formation was forthcoming. Future studies will focus on modifications of the monomer design to facilitate crystallization from non-participating solvents.

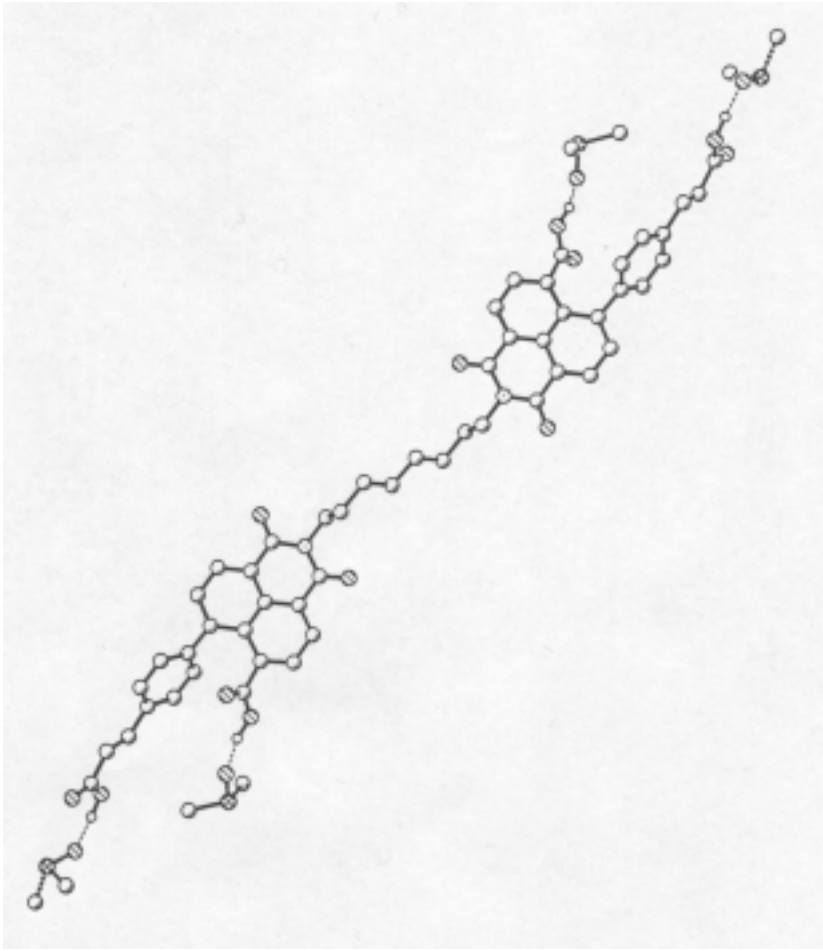


Figure 1. ORTEP of the crystal structure of tetraacid (**8f**) (crystallized from DMSO/ethanol).

ACKNOWLEDGMENT

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EXPERIMENTAL

THF and ether were purified by distillation from sodium/benzophenone under Ar immediately before use. Moisture and oxygen sensitive reactions were carried out in flame-dried glassware under Ar. Solvents for chromatography (Et_2O, EtOAc, CH_2Cl_2, hexane) were distilled from CaH_2 prior to use. Flash column

chromatography was carried out under positive pressure using 32-63 μm silica gel and the indicated solvents.[9] Melting points are uncorrected. CIMS were obtained with isobutane as the reagent gas, and EIMS were obtained at 50-70 eV. FAB HRMS were obtained from the mass spectrometry laboratory at the University of Texas at Austin. Combustion analyses were performed by either Galbraith Laboratories, Knoxville, TN or Midwest Microlab, Indianapolis, IN.

General Procedure A. The polyamine core and 1 – 1.1 equiv of anhydride (**5**) per $-NH_2$ were dissolved in the indicated solvent (~ 0.02 – 0.05 M). $Zn(OAc)_2 \bullet 2H_2O$ was added (~ 1 - 2 equiv per equiv of **5**) and the solution was purged with Ar and brought to reflux. After TLC indicated consumption of anhydride (hours to days), the reaction mixture was cooled to rt and diluted with CH_2Cl_2. The solution was washed with an equal volume of 1M H_3PO_4 and brine, dried over Na_2SO_4, filtered, and concentrated *in vacuo*. Silica gel flash chromatography of the residue in the indicated solvent system furnished the pure polyimide.

General Procedure B. The polyiimide poly(trimethylsilylethyl) ester was dissolved/suspended in THF (~ 0.2 - 0.5 M). A solution of 1M n-Bu_4NF in THF containing ~ 1.5 – 2.0 equiv F^- per ester was added and the reaction mixture was stirred at rt. When TLC analysis indicated consumption of starting material (hours to ~ 1 day), the solution was diluted with 2 volumes of THF and added to an equal volume of 1M H_3PO_4. If a precipitate was observed, simple filtration afforded the pure polyacid following washing with water, THF, and then Et_2O, and drying *in vacuo*. If no precipitate was observed, the mixture was extracted with several volumes of EtOAc, and the combined organic phases were washed with brine, dried with Na_2SO_4, filtered, and concentrated *in vacuo* to deliver pure polyacid product.

General Procedure C. A solution of 3,5-dinitrobenzoyl chloride in toluene or THF (~ 0.4 M) was added *via* syringe to a stirring solution of alcohol (~ 0.3 M) and 6 equiv of pyridine in the same solvent. The solution was brought to reflux for 20 min, cooled to rt, and poured into an equal volume of ice cold 1N HCl. The mixture was extracted with toluene, and the combined organic phases were washed in sequence with water, 1M potassium carbonate, and brine, and then dried over Na_2SO_4. The toluene solution was concentrated *in vacuo*. The crude powder or oil was purified by silica gel flash column chromatography with the indicated solvent system.

General Procedure D. A solution of the dinitro compounds (**17a-h**) in THF, EtOAc, or MeOH as indicated (~ 0.05 M) was treated with ~ 190 mg of 10% Pd/C per mmol substrate. The flask was purged with Ar, and then charged with 1 atm of H_2 gas. The solution was stirred at rt with TLC monitoring.

After starting material was consumed, the mixture was purged with Ar, filtered through Celite, and concentrated in vacuo to provide the pure diamino products (**18a**-**h**).

Diimide (7a). Following General Procedure A, 1,4-diaminobenzene and anhydride (**5**) were combined to afford **7a** as a tan solid following silica gel flash chromatography with 10% EtOAc/CH_2Cl_2 as eluent. mp 301-304 °C: IR ($CHCl_3$) 1712, 1666, 1634, 1588 cm^{-1}; ^1H NMR (360 MHz, $CDCl_3$) δ 8.77 (d, J = 7.7 Hz, 2H), 8.75 (d, J = 8.3 Hz, 2H), 7.98 (d, J = 7.5 Hz, 2H), 7.82 (d, J = 7.6 Hz, 2H), 7.73 (d, J = 16.0 Hz, 2H), 7.66 (d, J = 8.3 Hz, 4H), 7.55 (s, 4H), 1.10 (m, 4H), 0.83 (m, 4H), 0.09 (s, 18H), -0.02 (s, 18H); ^{13}C NMR (90 MHz, $CDCl_3$) δ 168.3, 166.8, 163.6, 163.4, 145.8, 143.2, 142.8, 137.9, 135.1, 134.5, 131.5, 130.95, 130.94, 130.0, 129.6, 129.5, 129.0, 128.2, 127.1, 125.0, 122.2, 119.5, 64.4, 62.9, 17.4, 17.0, -1.4, -1.6; MS m/z (relative intensity) 1250 (M^+, 0.8). Anal. Calcd for $C_{70}H_{76}N_2O_{12}Si_4$: C, 67.28 ; H, 6.13. Found: C, 66.87; H, 6.12.

Diimide (7b). Following General Procedure A, benzidene and anhydride (**5**) were combined to afford **7b** as a light yellow solid following silica gel flash chromatography with 30% EtOAc/hexane as eluent. mp 279-282 °C: IR ($CHCl_3$) 1711, 1671, 1638, 1590 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 8.77 (d, J = 7.3 Hz, 2H), 8.74 (d, J = 7.0 Hz, 2H), 7.98 (d, J = 7.5 Hz, 2H), 7.84 (d, J = 8.3 Hz, 4H), 7.81 (d, J = 7.5 Hz, 2H), 7.73 (d, J = 16.0 Hz, 2H), 7.66 (d, J = 8.3 Hz, 4H), 7.50 (d, J = 8.2 Hz, 4H), 7.46 (d, J = 8.4 Hz, 4H), 6.52 (d, J = 16.0 Hz, 2H), 4.34 (m, 4H), 3.56 (m, 4H), 1.09 (m, 4H), 0.83 (m, 4H), 0.09 (s, 18H), -0.02 (s, 18H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.2, 166.8, 163.9, 163.7, 145.8, 143.1, 142.7, 141.1, 137.9, 134.5, 134.4,131.5, 130.93, 130.86, 130.0, 129.5, 129.0, 128.9, 128.5,128.2, 127.0, 124.9, 122.1, 119.5, 64.4, 62.9, 17.3, 17.0, -1.4, -1.6; MS m/z (relative intensity) 1325 (M^+, 3.1) 1225 (2.7). HRMS calcd for $C_{76}H_{80}N_2O_{12}Si_4$ 1325.4901, found 1325.4867. Anal. Calcd for $C_{76}H_{80}N_2O_{12}Si_4$: C, 68.85; H, 6.08. Found: C, 68.61; H, 6.11.

Diimide (7c). Following General Procedure A, 4,4'-diamino-3,3'-dimethoxybiphenyl and anhydride (**5**) were combined to afford **7c** as a light yellow solid following silica gel flash chromatography with 1-6%EtOAc/benzene as eluent. mp 285-288 °C: IR ($CHCl_3$) 1714, 1673, 1635, 1591 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 8.76 (d, J = 7.5 Hz, 2H), 8.74 (d, J = 7.5 Hz, 2H), 7.97 (d, J = 7.5 Hz, 2H), 7.80 (d, J = 7.6 Hz, 2H), 7.73 (d, J = 16.0 Hz, 2H), 7.66 (d, J = 8.1 Hz, 4H), 7.50 (d, J = 7.4 Hz, 4H), 7.38 (s, 4H), 7.33 (s, 2H), 6.51 (d, J = 16.0 Hz, 2H), 4.34 (m, 4H), 3.88 (s, 6H), 3.56 (m, 4H), 1.09 (m, 4H), 0.83 (m, 4H), 0.09 (s, 18H), -0.02 (s, 18H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.3, 166.8, 163.5, 163.3, 155.0, 145.6, 143.7, 143.2, 142.8, 137.7, 134.4, 131.3, 130.8, 130.2, 130.0, 129.5, 129.0, 128.18, 128.17, 127.0, 125.0,

123.2, 122.3, 120.3, 119.4, 111.5, 64.4, 62.9, 55.9, 17.3, 17.0, -1.5, -1.6; MS m/z (relative intensity) 1384 (M^+, 100) 1239 (40). HRMS calcd for $C_{78}H_{84}N_2O_{14}Si_4$ 1385.5087, found 1385.5078. Anal. Calcd for $C_{78}H_{84}N_2O_{14}Si_4$: C, 67.60; H, 6.11. Found: C, 67.50; H, 6.14.

Diimide (7d). Following General Procedure A, 4,4'-diamino-2,2',3,3',5,5',6,6'-octafluorobiphenyl and anhydride (**5**) were combined to afford **7d** as a light yellow solid following silica gel flash chromatography with 30%Et_2O/hexane → 4:11:35 EtOAc/CH_2Cl_2/hexane as eluent. mp > 400°C (decomp): IR (KBr) 1722, 1690, 1638, 1590 cm^{-1}; ^1H NMR (360 MHz, $CDCl_3$) δ 8.81 (d, J = 7.7 Hz, 2H), 8.79 (d, J = 7.6 Hz, 2H), 8.01 (d, J = 7.5 Hz, 2H), 7.86 (d, J = 7.6 Hz, 2H), 7.73 (d, J = 16.0 Hz, 2H), 7.67 (d, J = 8.2 Hz, 4H), 7.50 (d, J = 8.2 Hz, 4H), 6.52 (d, J = 16.0 Hz, 2H), 4.34 (m, 4H), 3.57 (m, 4H), 1.09 (m, 4H), 0.83 (m, 4H), 0.09 (s, 18H), -0.02 (s, 18H); ^{13}C NMR (90 MHz, $CDCl_3$) δ 167.9, 166.7, 162.0, 161.8, 146.9, 145.6 (m), 143.0, 142.7 (m), 142.4, 138.9, 134.7, 132.1, 131.6, 131.0, 130.3, 129.5, 129.1, 128.2, 127.4, 123.8, 121.1, 119.7, 116.3 (m), 108.0 (m), 64.6, 62.9, 17.4, 17.0, -1.4, -1.6; MS m/z (relative intensity) 1468 (M^+, 100) 1323 (58). Anal. Calcd for $C_{76}H_{72}N_2O_{12}F_8Si_4$: C, 62.11; H, 4.94; F, 10.34. Found: C, 61.96; H, 5.11; F, 10.90.

Diimide (7e). Following General Procedure A, *trans*-1,4-diaminocyclohexane and anhydride (**5**) were combined to afford **7e** as a light yellow solid following silica gel flash chromatography with CH_2Cl_2 and then 5%EtOAc/CH_2Cl_2 as eluent. mp > 305 °C (decomp, recrystallized from hexane/$CHCl_3$): IR ($CHCl_3$) 1702, 1661, 1638, 1590 cm^{-1}; ^1H NMR (360 MHz, $CDCl_3$) δ 8.67 (d, J = 7.6 Hz, 2H), 8.65 (d, J = 7.5 Hz, 2H), 7.91 (d, J = 7.5 Hz, 2H), 7.73 (d, J = 7.6 Hz, 2H), 7.69 (d, J = 16.0 Hz, 2H), 7.61 (d, J = 8.3 Hz, 4H), 7.44 (d, J = 8.2 Hz, 4H), 6.48 (d, J = 16.0 Hz, 2H), 5.27 (br s, 2H), 4.31 (m, 4H), 3.51 (m, 4H), 2.87 (m, 4H), 1.91 (m, 4H), 1.06 (m, 4H), 0.78 (m, 4H), 0.07 (s, 18H), -0.06 (s, 18H); ^{13}C NMR (90 MHz, $CDCl_3$) δ 168.3, 166.8, 164.0, 163.2, 145.0, 143.2, 142.9, 137.2, 134.4, 130.9, 130.8, 130.4, 129.6, 129.4, 128.9, 128.1, 126.7, 125.4, 122.7, 119.5, 64.2, 62.8, 52.8, 28.4, 17.4, 17.0, -1.4, -1.6; MS m/z (relative intensity) 1254 (M^+, 3.6) 1154 (0.44). HRMS calcd for $C_{50}H_{28}N_2O_{12}$ 1254.4948, found 1254.4945. Anal. Calcd for $C_{70}H_{82}N_2O_{12}Si_4$: C, 66.95; H, 6.58. Found: C, 67.14; H, 6.85.

Diimide (7f). Following General Procedure A, 1,8-diaminooctane and anhydride (**5**) were combined to afford **7f** as a light yellow solid following silica gel flash chromatography with 40%Et_2O/hexane and then 4% EtOAc/CH_2Cl_2 as eluent. mp 203-206 °C: IR ($CHCl_3$) 1704, 1660, 1637, 1591 cm^{-1}; ^1H NMR (360 MHz, $CDCl_3$) δ 8.67 (d, J = 7.7 Hz, 2H), 8.65 (d, J = 8.3 Hz, 2H), 7.91 (d, J = 7.5 Hz, 2H), 7.74 (d, J = 7.7 Hz, 2H), 7.70 (d, J = 16.2 Hz, 2H), 7.62 (d, J = 8.2 Hz, 4H), 7.45 (d, J = 8.2 Hz, 4H), 6.49 (d, J = 16.0

Hz, 2H), 4.32 (m, 4H), 4.18 (m, 4H), 3.52 (m, 4H), 1.74 (m, 4H), 1.40 (br s, 8H), 1.08 (m, 4H), 0.80 (m, 4H), 0.08 (s, 18H), -0.05 (s, 18H); ^{13}C NMR (90 MHz, $CDCl_3$) δ 168.3, 166.8, 163.7, 163.4, 145.2, 143.2, 142.9, 137.4, 134.4, 131.0, 130.7, 130.4, 129.6, 129.4, 128.9, 128.1, 126.8, 124.9, 122.2, 119.4, 64.3, 62.9, 40.6, 29.2, 28.0, 27.0, 17.4, 17.0, -1.4, -1.6; MS m/z (relative intensity) 1284 (M^+, 100) 994 (26). HRMS calcd for $C_{72}H_{88}N_2O_{12}Si_4$ 1284.5440, found 1284.5414.

Diimide (7g). Following General Procedure A, 2,2'-(ethylenedioxy)bis(ethylamine) and anhydride (**5**) were combined to afford **7g** as a glassy yellow solid following silica gel flash chromatography with 16% EtOAc/CH_2Cl_2 as eluent. mp 149-152 °C: IR ($CHCl_3$) 1705, 1663, 1638, 1592, 1170, 862, 838 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.64 (d, J = 8.6 Hz, 2H), 8.60 (d, J = 7.5 Hz, 2H), 7.90 (d, J = 7.5 Hz, 2H), 7.72 (d, J = 8.6 Hz, 2H), 7.70 (d, J = 16.0 Hz, 2H), 7.62 (d, J = 7.9 Hz, 4H), 7.44 (d, J = 7.9 Hz, 4H), 6.49 (d, J = 16.0 Hz, 2H), 4.39 (br s, 4H), 4.34 (m, 4H), 3.80 (br s, 4H), 3.69 (br s, 4H), 3.54 (m, 4H), 1.09 (m, 4H), 0.81 (m, 4H), 0.10 (s, 18H), -0.04 (s, 18H); ^{13}C NMR (90 MHz, $CDCl_3$) δ 168.5, 167.0, 163.8, 163.6, 145.5, 143.3, 143.0, 137.6, 134.5, 131.2, 130.9, 130.6, 129.8, 129.6, 129.1, 128.3, 127.0, 124.9, 122.2, 119.6, 70.3, 68.0, 64.5, 63.0, 39.4, 17.5, 17.2, -1.2, -1.4; MS m/z (relative intensity) 1328.3 ($M+K^+$, 48) 1311.5 ($M+Na^+$, 100). HRMS calcd for $C_{70}H_{84}N_2O_{14}NaSi_4$ 1311.4897, found 1311.4797. Anal. Calcd for $C_{70}H_{84}N_2O_{14}Si_4$: C, 65.19; H, 6.56; N, 2.17. Found: C, 64.88; H, 6.53; N, 2.22.

Tetraacid (8a). Following General Procedure B, the diimide (**7a**) was deprotected to furnish the tetraacid (**8a**). mp >360 °C: IR (KBr) 1670, 1589, 1510 cm^{-1}; ^{1}H NMR (300 MHz, DMSO-d_6) δ 13.2 (br s, 2H), 12.4 (br s, 2H), 8.63 (d, J = 7.4 Hz, 2H), 8.62 (d, J = 7.4 Hz, 2H), 8.06 (d, J = 7.5 Hz, 2H), 7.91 (d, J = 7.6 Hz, 2H), 7.81 (d J = 8.1 Hz, 4H), 7.68 (d, J = 15.9 Hz, 2H), 7.60 (s, 4H), 7.74 (d, J = 8.0 Hz, 4H), 6.64 (d, J = 16.0 Hz, 2H); ^{13}C NMR (75 MHz, DMSO-d_6) δ 168.8, 167.6, 163.5, 163.3, 145.4, 143.4, 142.7, 138.6, 135.7, 134.1, 130.8, 130.5, 130.3, 129.6, 129.3, 128.7, 128.4, 126.2, 124.7, 122.8, 122.2, 119.7; MS m/z (relative intensity) 849 (M^+, 0.08) 769 (1.5). HRMS calcd for $C_{50}H_{28}N_2O_{12}$ 849.1720, found 849.1734.

Tetraacid (8b). Following General Procedure B, the diimide (**7b**) was deprotected to furnish the tetraacid (**8b**). mp >360 °C: IR (KBr) cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6) δ 12.6 (br s, 4H), 8.58 (d, J = 7.0 Hz, 4H), 8.03 (d, J = 7.1 Hz, 2H), 7.91 (d, J = 8.0 Hz, 4H), 7.88 (d, J = 7.0 Hz, 2H), 7.79 (d, J = 7.4 Hz, 4H), 7.67 (d, J = 15.9 Hz, 2H), 7.55 (d, J = 7.6 Hz, 4H), 7.45 (d, J = 7.3 Hz, 4H), 6.63 (d, J = 16.0 Hz, 2H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 168.9, 167.6, 163.4, 163.3, 145.4, 143.40, 143.36, 142.7, 139.7, 135.4, 134.1, 130.7, 130.4, 130.3, 129.7, 129.5, 129.3, 128.7, 128.4, 127.5, 126.2, 124.6, 122.2, 119.8; MS m/z

(relative intensity) 925 (MH^+, 100) 881 ($M-CO_2$, 87). HRMS calcd for $C_{56}H_{33}N_2O_{12}$ 925.2034, found 925.2078.

Tetraacid (8c). Following General Procedure B, the diimide (**7c**) was deprotected to furnish the tetraacid (**8c**). mp >360 °C: IR (KBr) 3460, 1710, 1672, 1633, 1590, 1244 cm^{-1}; ^1H NMR (360 MHz, DMSO- d_6) δ 12.7 (br s, 4H), 8.63 (d, J = 7.7 Hz, 2H), 8.62 (d, J = 7.3 Hz, 2H), 8.06 (d, J = 7.3 Hz, 2H), 7.91 (d, J = 7.7 Hz, 2H), 7.81 (d, J = 7.7 Hz, 4H), 7.68 (d, J = 15.9 Hz, 2H), 7.55 (m, 6H), 7.49 (d, J = 8.2 Hz, 4H), 6.64 (d, J = 15.9 Hz, 2H), 3.88 (s, 6H); ^{13}C NMR (90 MHz, DMSO-d_6) δ 168.8, 167.6, 162.9, 162.8, 155.2, 145.7, 143.5, 142.7, 142.2, 139.2, 138.9, 134.2, 130.9, 130.8, 130.7, 129.5, 129.4, 128.8, 128.4, 126.4, 124.2, 123.8, 121.7, 119.8, 119.5, 111.2, 56.0; MS m/z (relative intensity) 985 (M^+, 47) 941 (44). HRMS calcd for $C_{58}H_{37}N_2O_{14}$ 985.2245, found 985.2244.

Tetraacid (8d). Following General Procedure B, the diimide (**7d**) was deprotected to furnish the tetraacid (**8d**). mp >360 °C: IR (KBr) 1719, 1686, 1655, 1637, 1590 cm^{-1}; ^1H NMR (300 MHz, DMSO-d_6) δ 12.7 (br s, 4H), 8.69 (d, J = 7.3 Hz, 2H), 8.67 (d, J = 7.4 Hz, 2H), 8.07 (d, J = 7.5 Hz, 2H), 7.93 (d, J = 7.6 Hz, 2H), 7.79 (d, J = 8.1 Hz, 4H), 7.67 (d, J = 15.9 Hz, 2H), 7.45 (d, J = 7.9 Hz, 4H), 6.64 (d, J = 16.0 Hz, 2H); ^{13}C NMR (75 MHz, DMSO-d_6) δ 168.6, 167.6, 161.6, 161.5, 147.0, 145.2 (m), 143.3, 142.4, 141.9 (m), 140.3, 134.3, 131.6, 131.0, 129.8, 129.4, 128.8, 128.5 (m), 128.41, 128.39, 126.6, 122.7, 120.4, 119.9, 116.5 (m); MS m/z (relative intensity) 1069 (M^+, 100) 1051 (24). HRMS calcd for $C_{56}H_{24}N_2O_{12}F_8$ 1069.1283, found 1069.1280.

Tetraacid (8e). Following General Procedure B, the diimide (**7e**) was deprotected to furnish the tetraacid (**8e**). mp >370 °C: IR (KBr) 3448, 1701, 1663 cm^{-1}; ^1H NMR (400 MHz, DMSO- d_6) δ 13.06 (br s, 2H), 12.50 (br s, 2H), 8.60 (d, J = 7.0 Hz, 2H), 8.58 (d, J = 7.0 Hz, 2H), 7.99 (d, J = 7.0 Hz, 2H), 7.84 (d, J = 7.0 Hz, 2H), 7.77 (d, J = 7.4 Hz, 4H), 7.65 (d, J = 15.8 Hz, 2H), 7.42 (d, J = 7.4 Hz, 4H), 6.62 (d, J = 15.8 Hz, 2H), 5.08 (br s, 2H), 2.69 (br s, 4H), 1.90 (br s, 4H); Tetraacid (**8e**) was too insoluble to acquire a ^{13}C NMR spectrum; MS m/z (relative intensity) 855 (M^-, 9) 811 (18) 388 (100). HRMS calcd for $C_{50}H_{34}N_2O_{12}$ 854.2112, found 884.2110.

Tetraacid (8f). Following General Procedure B, the diimide (**7f**) was deprotected to furnish the tetraacid (**8f**). mp >360 °C: IR (KBr) 1700, 1656, 1590 cm^{-1}; ^1H NMR (360 MHz, DMSO- d_6) δ 12.7 (br s, 4H), 8.54 (d, J = 7.6 Hz, 2H), 8.54 (d, J = 7.5 Hz, 2H), 7.98 (d, J = 7.5 Hz, 2H), 7.81 (d, J = 7.6 Hz, 2H), 7.76 (d, J = 8.3 Hz, 4H), 7.66 (d, J = 16.0 Hz, 2H), 7.40 (d, J = 8.2 Hz, 4H), 6.61 (d, J = 16.0 Hz, 2H), 4.03 (m,

4H), 1.64 (br s, 4H), 1.34 (br s, 8H); ^{13}C NMR (90 MHz, DMSO-d_6) δ 168.7, 167.5, 163.0, 162.9, 145.1, 143.4, 142.6, 138.2, 134.0, 130.7, 130.4, 130.2, 129.2, 129.0, 128.6, 128.3, 126.0, 124.0, 121.6, 120.0, 40.2, 28.6, 27.3, 26.4; MS m/z (relative intensity) 885 (M^+, 100) 842 (8). HRMS calcd for $C_{52}H_{40}N_2O_{12}$ 885.2676, found 885.2660.

Tetraacid (8g). Following General Procedure B, the diimide (**7g**) was deprotected to furnish the tetraacid (**8g**). mp >360 °C: IR (KBr) 3448, 1702, 1663, 1590 cm^{-1}; ^{1}H NMR (400 MHz, DMSO- d_6) δ 12.7 (br s, 4H), 8.50 (d, J = 7.5 Hz, 2H), 8.49 (d, J = 7.5 Hz, 2H), 7.94 (d, J = 7.5 Hz, 2H), 7.75 (d, J = 7.5 Hz, 2H), 7.74 (d, J = 8.3 Hz, 4H), 7.64 (d, J = 16.1 Hz, 2H), 7.35 (d, J = 8.3 Hz, 4H), 6.60 (d, J = 16.1 Hz, 2H), 4.14 (m, 4H), 3.61 (m, 4H), 3.53 (m, 4H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 168.7, 167.6, 163.0, 162.8, 145.2, 143.4, 142.6, 138.3, 134.0, 130.7, 130.4, 130.3, 129.2, 129.0, 128.6, 128.3, 126.0, 123.9, 121.4, 119.7, 69.6, 66.8, 38.8; MS m/z (relative intensity) 889 (MH^+, 8.4) 845 (15) 476 (100). HRMS calcd for $C_{50}H_{37}N_2O_{14}$ 889.2245, found 889.2237.

***N*-(3,5-Dinitrophenyl)-n-octanamide (11b)**. 3,5-Dinitroaniline (300 mg, 1.6 mmol) was dissolved in CH_2Cl_2 (5 mL). Et_3N (223 μL, 1.6 mmol) was added *via* syringe and the solution cooled to 0º C at which time octanoyl chloride (294 mg, 1.8 mmol) was added *via* syringe and the solution was allowed to warm to rt. After 1 h, TLC indicated complete consumption of starting material, so the solution was diluted with CH_2Cl_2 (15 mL) and washed with water (20 mL). The organic phase was dried with Na_2SO_4, filtered, and concentrated *in vacuo*. The resulting solid was purified by silica gel chromatography with 30% EtOAc/hexane as eluent to afford 382 mg (77%) of **11b** as a white solid. mp 86-87 ºC. IR ($CHCl_3$) 3428, 1711 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.82 (d, *J* = 2.0 Hz, 2 H), 8.75 (t, *J* = 2.0 Hz, 1 H), 7.86 (s, 1 H), 2.49 (t, *J* = 7.4 Hz, 2 H), 1.82 - 1.70 (m, 2 H), 1.46 - 1.42 (m, 8 H), 0.88 (t, *J* = 6.8 Hz, 3 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 172.1, 148.8, 140.2, 119.1, 113.5, 37.6, 31.6, 29.1, 28.9, 25.1, 22.6, 14.0; MS *m/z* (relative intensity) 310 (M^+, 2); HRMS calcd for $C_{14}H_{19}N_3O_5$ 310.1403, found 310.1409.

***N*-(3,5-Dinitrophenyl)-2,2-dimethylpropionamide**. **(11c).** Following the procedure used for **11a**, 3,5-dinitroaniline (50 mg, 0.27 mmol) was combined with 2,2-dimethylproprioyl chloride (41 μL 0.33 mmol) to afford 70 mg (97%) of **11c** as a white solid. An analytical sample was prepared by recrystallization from EtOAc/hexane. mp 206-210 ºC. IR ($CHCl_3$) 3446, 1700 cm^{-1}; ^{1}H NMR (200 MHz, $CDCl_3$) δ8.84 (s, 2 H), 8.77 (s, 1H), 7.79 (s, 1 H), 1.38 (s, 9 H); ^{13}C NMR (50 MHz, $CDCl_3$) δ 177.2, 148.7, 140.4,

119.4, 113.5, 40.1, 27.4; MS *m/z* (relative intensity) 267 (M^+, 3), 57 (100); Anal. Calcd for $C_{11}H_{13}N_3O_5$; C, 49.44; H, 4.90; N, 15.72. Found: C, 49.50; H, 4.99; N, 15.59.

***N*-(3,5-Diaminophenyl)-n-heptanamide (12b)**. Amide (**11b**) (100 mg. 0.32 mmol) and 10% Pd/C (10 mg) were mixed and EtOAc (5 mL) was added. The mixture was degassed (freeze, pump, thaw) and the resulting suspension was heated under a hydrogen atmosphere at 80ºC for 1 h, at which time TLC indicated complete consumption of starting material. The mixture was cooled, filtered through Celite, and the Celite was washed with EtOAc (50 mL). The solution was concentrated *in vacuo* to afford 80 mg (100%) of **12b** as a white solid. mp 76-78 ºC. IR ($CHCl_3$) 3400, 1682 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.44 (s, 1 H), 6.33 (d, *J* = 1.3 Hz, 2 H), 5.71 (s, 1 H), 3.54 (s, 4 H), 2.26 (t, *J* = 7.3, 7.9 Hz, 2 H), 1.67 - 1.62 (m, 2 H), 1.28 - 1.26 (m, 8 H), 0.86 (t, *J* = 6.6, 6.8 Hz, 3 H); ^{13}C NMR (90 MHz, $CDCl_3$) δ 171.5, 148.0, 140.0, 97.9, 97.4, 37.9, 31.7, 29.2, 29.0, 25.6, 22.6, 14.0; MS *m/z* (relative intensity) 250 (MH^+, 23); HRMS calcd for $C_{14}H_{24}N_3O$ 250.1919, found 250.1915.

***N*-(3,5-Diaminophenyl)-2,2-dimethylpropionamide (12c).** Following the procedure used for **12b**, amide (**11c**) (587 mg, 2.2 mmol) was hydrogenated to produce 380 mg (83%) of **12c** as white crystals. mp 214-215 ºC. IR ($CHCl_3$) 3455, 3400 cm^{-1}; ^1H NMR (300 MHz, $CHCl_3$) δ 6.38 (d, *J* = 1.9 Hz, 2 H), 5.80 (t, *J* = 1.9 Hz, 1 H), 3.57 (s, 4 H), 1.28 (s, 9H); ^{13}C NMR (50 MHz, DMSO-d_6) δ 175.6, 148.8, 140.1, 96.3, 95.9, 38.9, 27.2; MS *m/z* (relative intensity) 207 (M^+, 58), 123 (100); HRMS calcd for $C_{11}H_{17}N_3O$ 207.1372, found 207.1367.

Diimide (13a). Following General Procedure A, diamine(**12a**)[10] and anhydride (**5**) were combined to furnish diimide (**13a**) as a brown solid. mp 138-144 ºC. IR ($CHCl_3$) 1712, 1676, 1638, cm^{-1}; ^1H NMR (200 MHz, $CDCl_3$), δ 8.70 (m, 4 H), 7.94 (d, *J* = 7.4 Hz, 1 H), 7.78 - 7.62 (m, 12 H), 7.47 (d, *J* = 8.0 Hz, 4 H), 7.11 (s, 1 H), 6.50 (d, *J* = 15.9 Hz, 2 H), 4.33 (t, *J* = 8.4 Hz, 4 H), 3.55 (t, *J* = 8.8 Hz, 4 H), 2.16 (s, 3 H), 1.09 (t, *J* = 8.7 Hz, 4 H), 0.82 (t, *J* = 8.7 Hz, 4 H), 0.09 (s, 18 H), -0.04 (s, 18 H); ^{13}C NMR (50 MHz, $CDCl_3$) δ 169.3, 168.1, 166.7, 163.4, 163.2, 145.6, 143.1, 142.7, 140.6, 137.8, 135.6, 134.4, 131.4, 130.90, 130.85, 129.7, 129.4, 129.0, 128.1, 126.8, 124.6, 123.7, 121.9, 119.9, 119.5, 64.3, 62.8, 24.4, 17.3, 17.0, -1.5, -1.6; MS *m/z* (relative intensity) 1306 (MH^+, 3); HRMS calcd for $C_{72}H_{80}N_3O_{13}Si_4$ 1306.4768, found 1306.4776.

Diimide (13b). Following General Procedure A, diamine (**12b**) and anhydride (**5**) were combined to furnish diimide (**13b**) as a brown solid following silica gel chromatography with 30% EtOAc/hexane as

eluent. mp 148-152 ºC. IR ($CHCl_3$) 3435, 1713, 1676 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 8.65 (dd, *J* = 7.5, 7.6 Hz, 4 H), 7.93 (m, 3 H), 7.81 - 7.69 (m, 6 H), 7.64 (d, *J* = 8.2 Hz, 4 H), 7.46 (d, *J* = 8.1 Hz, 4 H), 7.08 (s, 1 H), 6.51 (d, *J* = 16.0 Hz, 2 H), 4.34 (t, *J* = 8.3 Hz, 4 H), 3.55 (t, *J* = 8.9 Hz, 4 H), 2.35 (t, *J* = 7.5 Hz), 1.87 - 1.67 (m, 2 H), 1.29 - 1.24 (m, 8 H), 1.09 (t, *J* = 8.2 Hz, 4 H), 0.85 - 0.79 (m, 7 H), 0.09 (s, 18 H), -0.03 (s, 18 H); ^{13}C NMR (75 MHz, $CDCl_3$)δ 171.6, 168.2, 166.8, 163.4, 163.2, 145.7, 143.2, 142.7, 140.2, 137.8, 135.7, 134.5, 131.4, 130.9, 130.8, 129.9, 129.5, 129.0, 128.2, 126.9, 124.8, 123.5, 122.0, 120.0, 119.5, 64.4, 62.9, 37.7, 31.0, 29.2, 29.0, 25.4, 22.6, 17.4, 17.0, 14.0, -1.5, -1.6; *m/z* (relative intensity) 1390 (MH^+, 12); HRMS calcd for $C_{78}H_{92}N_3O_{13}Si_4$ 1390.5707, found 1390.5688.

Diimide (13c). Following General Procedure A, diamine (**12c**) and anhydride (**5**) were combined to furnish diimide (**13c**) as a brown solid following silica gel chromatography with 30% EtOAc/hexane as eluent. mp 175-180 ºC. IR ($CHCl_3$) 1713, 1676, 1638 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 8.74 (dd, *J* = 7.6, 7.4 Hz, 4 H), 7.98 (d, *J* = 7.5 Hz, 2 H), 7.88-7.66 (m, 11 H), 7.52 (d, *J* = 8.6 Hz, 4 H), 7.17 (s, 1 H), 6.54 (d, *J* = 15.6 Hz, 2 H), 4.37 (t, *J* = 8.4 Hz, 4 H), 3.59 (t, *J* = 8.7 Hz, 4 H), 1.34 (s, 9 H), 1.12 (t, *J* = 8.4 Hz, 4 H), 0.86 (t, *J* = 8.6 Hz, 4 H), 0.15 (s, 18 H), 0.00 (s, 18 H); ^{13}C NMR (90 MHz, $CDCl_3$) δ 176.4, 168.2, 166.8, 163.3, 163.1, 145.7, 143.2, 142.8, 139.9, 137.9, 135.9, 134.5, 131.4, 130.89, 130.80, 129.9, 129.5, 129.0, 128.2, 127.0, 124.8, 124.7, 122.1, 120.3, 119.5, 64.4, 62.9, 39.7, 27.5, 17.4, 17.1, -1.4, -1.6; MS *m/z* (relative intensity) 1348 (M^+, 100%); HRMS calcd for $C_{75}H_{85}N_3O_{13}Si_4$ 1348.5238, found 1348.5251.

Tetraacid (14a). Following General Procedure B, tetraester (**13a**) was deprotected to furnish the tetraacid (**14a**) as a yellow solid. mp ≥ 285 ºC (decomp). IR (KBr) 3568, 1702, 1670 cm^{-1}; ^1H NMR (200 MHz, THF-d_8) 9.40 (s, 1 H), 8.61 (d, J = 7.5 Hz, 4 H), 7.99 (d, J = 7.5 Hz, 2 H), 7.81-7.66 (m, 10 H), 7.46 (d, *J* = 7.3 Hz, 4 H), 7.06 (s, 1 H), 6.56 (d, *J* = 16.1 Hz, 2 H), 2.05 (s, 3 H); ^{13}C NMR (50 MHz, THF-d_8) δ 173.0, 169.3, 167.7, 163.8, 163.6, 146.7, 144.5, 144.3, 141.5, 139.2, 137.1, 135.5, 133.1, 132.9, 131.4, 131.2, 131.0, 130.5, 129.5, 128.9, 127.9, 126.3, 125.5, 123.7, 120.2; MS *m/z* (relative intensity) 906 (M^+, 16); HRMS calcd for $C_{52}H_{32}N_3O_{13}$ 906.1935, found 906.1921.

Tetraacid (14b). Following General Procedure B, tetraester (**13b**) was deprotected to furnish the tetraacid (**14b**) as a yellow solid following a final trituration with Et_2O. mp ≥ 260°C (decomp). IR (KBr) 3448, 1708, 1678 cm^{-1}; ^1H NMR (200 MHz, THF-d_8) δ 9.28 (s, 1 H), 8.66-8.61 (m, 4 H), 8.00 (d, *J* = 7.5 Hz, 2 H), 7.83-7.66 (m, 10 H), 7.48 (d, *J* = 8.1 Hz, 4 H), 7.07 (s, 1 H), 6.57 (d, *J* = 16.0 Hz, 2 H), 3.58 (s,

2 H), 2.33 (m, 2H), 1.33 (m, 8 H), 0.85 (m, 3 H_3); ^{13}C NMR (100 MHz, THF-d_8) δ 171.6, 169.3, 167.7, 163.8, 163.6, 146.6, 144.5, 144.4, 141.5, 139.1, 137.1, 135.5, 131.4, 131.2, 130.9, 130.8, 130.5, 129.6, 128.9, 127.9, 126.3, 125.1, 123.7, 120.4, 120.1, 37.8, 32.8, 30.3, 30.2, 26.4, 23.6, 14.5; MS *m/z* (relative intensity) 990 (M^+, 100); HRMS calcd for $C_{58}H_{44}N_3O_{13}$ 990.2874, found 990.2878.

Tetraacid (14c). Following General Procedure B, tetraester (**13c**) was deprotected to furnish the tetraacid (**14c**) as a yellow solid following a final trituration with Et_2O. mp ≥ 325 ºC (decomp). IR (KBr) 3472, 1708, 1669, 1636, 1589 cm^{-1}; ^{1}H NMR (200 MHz, THF-d_8) δ 8.73 (s, 1H), 8.61 (d *J* = 7.5 Hz, 4H), 7.99 (d, *J* = 7.5 Hz, 2 H), 7.87 (s, 2 H), 7.79 (d, *J* = 7.6 Hz, 2 H), 7.73 - 7.66 (m, 6 H), 7.45 (d, *J* = 8.0 Hz, 4 H), 7.0 (s, 1H), 6.56 (d, *J* = 16.0 Hz, 2 H), 1.29 (s, 9H); ^{13}C NMR (90 MHz, THF-d_8) δ 176.7, 169.3, 167.7, 163.8, 163.3, 146.7, 144.4, 144.2, 141.4, 139.2, 137.0, 135.5, 131.4, 131.2, 130.9, 130.8, 130.5, 129.6, 128.9, 127.9, 126.2, 125.3, 123.6, 121.2, 120.2, 40.3, 27.7; MS *m/z* (relative intensity) 948 (MH^+,100); HRMS calcd for $C_{55}H_{38}N_3O_{13}$ 948.2405, found 948.2412.

4-Phenylbutyl 3,5-Dinitrobenzoate (17c). Following General Procedure C, 3,5-dinitrobenzoyl chloride was combined with 4-phenylbutyl alcohol in toluene to furnish ester (**17c**) as a yellowish powder following chromatography with 7:3 hexane/EtOAc (87%). mp 54-55 ºC. IR (KBr) 1725 cm^{-1}; ^{1}H NMR (360 MHz, $CDCl_3$) δ 9.09 (t, *J*=2.0 Hz, 1H), 9.15 (d, *J*=2.0 Hz, 2H), 7.2~7.0 (m, 5H), 4.36 (t, *J*=6.5 Hz, 2H), 2.60 (t, *J*=7.0 Hz, 2H), 1.80~1.60 (m, 4H); ^{13}C NMR (75 MHz, acetone-d_6) δ 163.9, 150.1, 143.3, 135.0, 130.6, 129.60, 129.56, 127.0, 123.9, 67.7, 36.4, 29.2, 28.9; MS (APCI) m/z (relative intensity) 285 (M^+ + 1, 100).

6-Hydroxyhexyl 3,5-Dinitrobenzoate (17e). Following General Procedure C, 3,5-dinitrobenzoyl chloride was combined with n-hexanol in THF to furnish ester (**17e**) as a white powder following chromatography with 6:4 hexane/EtOAc (50%). mp 61-62 ºC. IR (KBr) 1735 cm^{-1}; ^{1}H NMR (360 MHz, $CDCl_3$) δ 9.23 (t, *J*=2.2 Hz, 1H), 9.16 (d, *J*=2.2 Hz, 2H), 4.47 (t, *J*=7.7 Hz, 2H), 3.68 (m, 2H), 1.87 (m, 2H), 1.63 (m, 2H), 1.49 (m, 4H); ^{13}C NMR (75 MHz, acetone-d_6) δ 163.9, 150.1, 135.1, 130.2, 123.6, 67.8, 62.7, 34.0, 29.7, 26.9, 26.7; MS (APCI) m/z (relative intensity) 313 (M^++1, 22).

2-Hydroxymethyl-3-hydroxypropyl 3,5-Dinitrobenzoate (17f). Following General Procedure C, 3,5-dinitrobenzoyl chloride was combined with 2-hydroxymethyl-1,3-propanediol in THF to furnish ester (**17f**) as a yellowish solid following chromatography with 1:1 hexane/EtOAc (39%). mp 83-84 ºC. IR

(KBr) 3318, 1791 cm^{-1}; ^{1}H NMR (360 MHz, acetone-d_6) δ 9.15 (t, *J*=2.0 Hz, 1H), 9.07 (d, *J*=2.0 Hz, 2H), 4.56 (d, *J*=6.2 Hz, 2H), 4.00~3.70 (m, 6H), 2.22 (m, 1H); ^{13}C NMR (90 MHz, acetone-d_6) δ 164.0, 150.2, 135.1, 130.1, 124.6, 66.3, 61.5, 44.6; MS (APCI) m/z (relative intensity) 301 (M^++1, 9), 283 (100).

Isopropyl 3,5-Diaminobenzoate (18a). Following General Procedure D, **17**[11] in THF was converted into the colorless oil (**18a**) (94%). A sample for spectral characterization was prepared by chromatography with EtOAc as eluent. IR (neat) 3448, 3354, 1696 cm^{-1}; ^{1}H NMR (300 MHz, acetone-d_6) δ 6.61 (d, *J*=2.0 Hz, 2H), 6.19 (t, *J*=2.1 Hz, 1H), 5.09 (hept, *J*=6.3 Hz, 1H), 1.27 (t, *J*=6.2 Hz, 6H); ^{13}C NMR (75 MHz, acetone-d_6) δ 167.6, 150.4, 133.6, 106.2, 105.7, 68.5, 22.6; MS (APCI) m/z (relative intensity) 195 (M^++1, 100).

Cyclohexyl 3,5-Diaminobenzoate (18b). Following General Procedure D, **17b**[12] in EtOAc was converted into the colorless oil (**18b**) (70%). A sample for spectral characterization was prepared by chromatography with EtOAc as eluent. IR (neat) 3436, 3354, 1696 cm^{-1}; ^{1}H NMR (300 MHz, acetone-d_6) δ 6.63 (d, *J*=2.1 Hz, 2H), 6.19 (t, *J*=2.1 Hz, 1H), 4.87 (m, 1H), 1.86 (m, 2H), 1.75 (m, 2H), 1.60~1.30 (m, 6H); ^{13}C NMR (75 MHz, acetone-d_6) δ 167.4, 150.4, 133.6, 106.6, 105.7, 73.2, 32.7, 26.6, 24.7; MS (EI) m/z (relative intensity) 234 (M^+, 32), 152 (100).

4-Phenylbutyl 3,5-Diaminobenzoate (18c). Following General Procedure D, **17c** in EtOAc was converted into the gray powder (**18c**) (95%). A sample for spectral characterization was prepared by chromatography with 9:1 EtOAc/MeOH as eluent. mp 84-85 °C. IR (KBr) 3448, 3342, 1707 cm^{-1}; ^{1}H NMR (360 MHz, $CDCl_3$) δ 7.30~7.10 (m, 5H), 6.79 (d, *J*=2.0 Hz, 2H), 6.29 (br s, 1H), 4.23 (m, 2H), 2.64 (m, 2H), 1.73 (m, 4H); ^{13}C NMR (75 MHz, acetone-d_6) δ 168.1, 150.5, 143.5, 133.1, 129.7, 129.6, 127.0, 106.2, 105.7, 65.2, 36.5, 29.6, 29.1; MS (MALDI) m/z (relative intensity) 285 (M^++1, 100).

2-Phenylethyl 3,5-Diaminobenzoate (18d). Following General Procedure D, **17d**[13] in EtOAc was converted into the yellow powder (**18d**) (80%). A sample for spectral characterization was prepared by chromatography with 9:1 EtOAc/CH_3OH as eluent. mp 66-68 °C. IR (KBr) 3448, 3366, 1702 cm^{-1}; ^{1}H NMR (300 MHz, acetone-d_6) δ 7.35~7.15 (m, 5H), 6.62 (d, *J*=2.1 Hz, 2H), 6.20 (t, *J*=2.1 Hz, 1H), 4.39 (t, *J*=7.0 Hz, 2H), 4.02 (t, *J*=7.0 Hz, 2H); ^{13}C HNMR (75 MHz, acetone-d_6) δ 170.4, 150.5, 139.7, 133.0, 130.3, 129.7, 127.7, 106.2, 105.8, 66.2, 36.3; MS (MALDI) m/z (relative intensity) 257 (M^++1).

6-Hydroxylhexyl 3,5-Diaminobenzoate (18e). Following General Procedure D, **17e** in MeOH was converted into **18e** (87%, gray needles). A sample for spectral characterization was prepared by chromatography with EtOAc as eluent. mp 70-71 °C. IR (KBr) 3413, 1702 cm^{-1}; ^{1}H NMR (360 MHz, acetone-d_6 +D_2O) δ 6.63 (d, *J*=2.0 Hz, 2H), 6.22 (t, *J*=2.0 Hz, 1H), 4.18 (t, *J*=6.6 Hz, 2H), 3.51 (t, *J*=6.4 Hz, 2H), 2.04 (m, 2H), 1.80~1.30 (m, 6H); ^{13}C NMR (75 MHz, acetone-d_6) δ 168.1, 150.5, 133.2, 106.2, 105.7, 65.3, 62.8, 34.0, 29.5, 27.1, 26.8; MS (MALDI) m/z (relative intensity) 253 (M^++1, 100).

2-(Hydroxymethyl)-3-hydroxypropyl 3,5-Diaminobenzoate (18f). Following General Procedure D, **17f** in MeOH was converted into the off-white powder (**18f**) (88%). A sample for spectral characterization was prepared by chromatography with 6:4 EtOAc/MeOH as eluent. mp 104-106 °C. IR (KBr) 3392, 3345, 1706 cm^{-1}; ^{1}H NMR (360 MHz, acetone-d_6+D_2O) δ 6.62 (d, *J*=2.2 Hz, 2H), 6.22 (t, *J*=2.0 Hz, 1H), 4.26 (d, *J*=5.9 Hz, 2H), 3.66 (d, *J*=5.5 Hz, 4H), 2.08 (m, 1H); ^{13}C NMR (75 MHz, acetone-d_6) δ 168.9, 150.2, 150.1, 132.7, 106.9, 64.1, 61.0, 44.4; MS (APCI) m/z (relative intensity) 241 (M^++1, 100).

4-Methylphenyl 3,5-Diaminobenzoate (18g). Following General Procedure D, **17g**[14] in THF was converted into the off-white powder (**18g**) (86%). A sample for spectral characterization was prepared by chromatography with 4:1 EtOAc/hexane as eluent. mp 100-101 °C. IR (KBr) 3399, 1735 cm^{-1}; ^{1}H NMR (360 MHz, acetone-d_6) δ 7.22 (d, *J*=8.6 Hz, 2H), 7.06 (d, *J*=8.6 Hz, 2H), 6.76 (d, *J*=1.8 Hz, 2H), 6.29 (t, *J*=1.8 Hz, 1H), 4.65 (br s, 4H), 2.33 (s, 3H); ^{13}C NMR (75 MHz, acetone-d_6) δ 166.9, 150.7, 150.6, 136.3, 132.2, 131.1, 122.9, 106.6, 106.2, 21.3; MS (APCI) m/z (relative intensity) 243 (M^++1, 100).

4-Methoxyphenyl 3,5-Diaminobenzoate (18h). Following General Procedure D, **17h**[15] in THF was converted into the yellow powder (**18h**) (98%). A sample for spectral characterization was prepared by chromatography with 4:1 EtOAc/hexane as eluent. mp 80-81 °C. IR (KBr) 3378, 1719 cm^{-1}; ^{1}H NMR (360 MHz, acetone-d_6) δ 7.10 (d, *J*=9.1 Hz, 2H), 6.90 (d, *J*=9.1 Hz, 2H), 6.76 (d, *J*=1.8 Hz, 2H), 6.28 (t, *J*=1.8 Hz, 1H), 4.60 (br s, 4H), 3.80 (s, 3H); ^{13}C NMR (75 MHz, acetone-d_6) δ 167.2, 158.6, 150.7, 146.1,132.2, 123.9, 115.6, 106.6, 106.3, 56.3; MS (APCI) m/z (relative intensity) 259 (M^++1, 100).

Diimide(19a). Following General Procedure A with the modification that 20 equiv of triethylamine was substituted for the zinc acetate, diamine (**18a**) in THF was converted into diimide (**19a**) as a yellow powder following chromatographic purification with 7:3 hexane/EtOAc as eluent (44%). mp 160-162 °C. IR (KBr) 1713, 1672 cm^{-1}; ^{1}H NMR (360 MHz, acetone-d_6) δ 8.71 (d, *J*=7.6 Hz, 2H), 8.69 (d, *J*=7.6 Hz,

2H), 8.28 (d, *J*=2.0 Hz, 2H), 8.05 (d, *J*=7.6 Hz, 2H), 7.94 (d, *J*=7.6 Hz, 2H), 7.87 (t, *J*=2.2 Hz, 1H), 7.85 (d, *J*=7.9 Hz, 4H), 7.75 (d, *J*=16.2 Hz, 2H), 7.54 (d, *J*=7.9 Hz, 4H), 6.66 (d, *J*=15.8 Hz, 2H), 5.28 (m, 1H), 4.32~4.29 (m, 4H), 3.60~3.55 (m, 4H), 1.38 (d, *J*=6.5 Hz, 6H), 1.12~1.07 (m, 4H), 0.91~0.86 (m, 4H), 0.10 (s, 18H), -0.03 (s, 18H); ^{13}C NMR (90 MHz, acetone-d_6) δ 168.5, 166.8, 165.1, 163.9, 163.7, 146.5, 144.04, 144.02,138.8, 137.4, 135.6, 135.4, 133.3, 131.7, 131.4, 131.06, 130.96 130.93, 130.5, 129.8, 129.2, 127.9, 126.4, 123.7, 120.3, 69.4, 64.5, 62.9, 22.2, 18.2, 17.6, -1.3, -1.4; MS (MALDI) m/z (relative intensity) 1357 (M^++Na, 100).

Diimide(19b). Following General Procedure A with the modification that 20 equiv of triethylamine was substituted for the zinc acetate, diamine (**18b**) in xylene was converted into diimide (**19b**) as a yellow powder following chromatographic purification with 4:1 hexane/EtOAc as eluent (24%). mp 162-164 °C. IR (KBr) 1713, 1672 cm^{-1}; ^{1}H NMR (360 MHz, acetone-d_6) δ 8.69 (d, *J*=7.7 Hz, 2H), 8.68 (d, *J*=7.5 Hz, 2H), 8.30 (d, *J*=2.0 Hz, 2H), 8.03 (d, *J*=7.5 Hz, 2H), 7.93 (d, *J*=7.5 Hz, 2H), 7.86 (t, *J*=1.8 Hz, 1H), 7.84 (d, *J*=8.2 Hz, 4H), 7.75 (d, *J*=15.9 Hz, 2H), 7.53 (d, *J*=8.4 Hz, 4H), 6.64 (d, *J*=15.9 Hz, 2H), 5.05 (m, 1H), 4.31 (m, 4H), 3.56 (m, 4H), 1.96 (m, 2H), 1.77 (m, 2H), 1.58 (m, 4H), 1.42 (m, 2H), 1.09 (m, 4H), 0.87 (m, 4H), 0.10 (s, 18H), -0.04 (s, 18H); ^{13}C NMR (75 MHz, acetone-d_6) δ 168.6, 166.9, 165.1, 164.2, 164.1, 146.5, 144.12, 144.10,143.8, 138.8, 137.8, 135.4, 133.4, 131.8, 131.6, 131.3, 131.1, 130.9, 130.5, 129.8, 129.3, 127.8, 126.3, 123.7, 120.3, 74.3, 64.7, 62.9, 32.3, 26.1, 24.3, 19.3, 18.8, -1.3, -1.4; MS (MALDI) m/z (relative intensity) 1397 (M^++Na, 100).

Diimide(19c). Following General Procedure A with the modification that 20 equiv of triethylamine was substituted for the zinc acetate, diamine (**18c**) in xylene was converted into diimide (**19c**) as a yellow powder following chromatographic purification with 4:1 hexane/EtOAc as eluent (34%). mp 122-124 °C. IR (KBr) 1713, 1678 cm^{-1}; ^{1}H NMR (360 MHz, acetone-d_6) δ 8.69 (d, *J*=7.7 Hz, 2H), 8.67 (d, *J*=7.5 Hz, 2H), 8.29 (d, *J*=2.0 Hz, 2H), 8.02 (d, *J*=7.5 Hz, 2H), 7.91 (d, *J*=7.5 Hz, 2H), 7.86 (t, *J*=2.0 Hz, 1H), 7.83 (d, *J*=8.2 Hz, 4H), 7.74 (d, *J*=16.0 Hz, 2H), 7.52 (d, *J*=8.2 Hz, 4H), 7.25~7.05 (m, 5H), 6.64 (d, *J*=16.2 Hz, 2H), 4.32 (m, 4H), 3.57 (m, 4H), 2.67 (t, *J*=7.1 Hz, 2H), 1.80 (m, 4H), 1.10 (m, 4H), 0.88 (m, 4H), 0.10 (s, 18H), -0.02 (s, 18H); ^{13}C NMR (90 MHz, acetone-d_6) δ 168.5, 166.9, 165.7, 164.1, 163.9, 146.4, 144.0, 143.8, 143.0,138.7, 137.6, 135.6, 135.3, 132.8, 132.3, 131.7, 131.6, 131.2, 131.1, 130.8, 130.5, 129.8, 129.2, 129.0, 127.7, 126.5, 126.2, 123.5, 120.2, 65.9, 64.6, 62.9, 35.9, 29.0, 28.5, 17.9, 17.4; MS (MALDI) m/z (relative intensity) 1447 (M^++Na, 85).

Diimide(19d). Following General Procedure A with the modification that 20 equiv of triethylamine was substituted for the zinc acetate, diamine (**18d**) in THF was converted into diimide (**19d**) as a yellow

powder following chromatographic purification with 7:3 hexane/EtOAc as eluent (48%). mp 118-120 °C. IR (KBr) 1713, 1672 cm^{-1}; ^{1}H NMR (360 MHz, acetone-d_6) δ 8.72 (d, *J*=7.5 Hz, 2H), 8.70 (d, *J*=7.5 Hz, 2H), 8.28 (d, *J*=2.2 Hz, 2H), 8.05 (d, *J*=7.5 Hz, 2H), 7.95 (d, *J*=7.5 Hz, 2H), 7.88 (t, *J*=2.2 Hz, 1H), 7.85 (d, *J*=8.6 Hz, 4H), 7.76 (d, *J*=15.8 Hz, 2H), 7.55 (d, *J*=7.9 Hz, 4H), 7.40~7.10 (m, 5H), 6.66 (d, *J*=16.2 Hz, 2H), 4.58 (t, *J*=7.2 Hz, 2H), 4.39~4.29 (m, 4H), 3.60~3.55 (m, 4H), 3.11 (t, *J*=7.2 Hz, 2H), 1.12~1.09 (m, 4H), 0.91~0.86 (m, 4H), 0.11 (s, 18H), -0.02 (s, 18H); ^{13}C NMR (90 MHz, acetone-d_6) δ 168.5, 166.9, 165.5, 164.1, 163.9, 146.4, 144.0, 143.7, 138.8, 138.7, 137.5, 135.4,135.3, 132.6, 131.7, 131.6, 131.2, 131.1, 130.7, 130.5,129.8, 129.7, 129.3, 129.2, 127.6, 127.2, 126.1, 123.4, 120.2, 66.6, 64.6, 62.9, 35.7, 17.9, 17.4, -1.4, -1.5; MS (MALDI) m/z (relative intensity) 1419 (M^{+}+Na).

Diimide(19e). Following General Procedure A with the modification that 20 equiv of triethylamine was substituted for the zinc acetate, diamine (**18e**) in xylene was converted into diimide (**19e**) as a yellow powder following chromatographic purification with 1:1 hexane/EtOAc as eluent (25%). mp 119-122 °C. IR(KBr), 1713, 1672 cm^{-1}; ^{1}H NMR (360 MHz, acetone-d_6) δ 8.70 (d, *J*=7.7 Hz, 2H), 8.69 (d, *J*=7.5 Hz, 2H), 8.30 (d, *J*=1.8 Hz, 2H), 8.04 (d, *J*=7.5 Hz, 2H), 7.93 (d, *J*=7.7 Hz, 2H), 7.87 (t, *J*=1.8 Hz, 1H), 7.84 (d, *J*=8.2 Hz, 4H), 7.74 (d, *J*=16.0 Hz, 2H), 7.53 (d, *J*=8.4 Hz, 4H), 6.64 (d, *J*=16.2 Hz, 2H), 4.38 (t, *J*=6.6 Hz, 2H), 4.31 (m, 4H), 3.57 (m, 4H), 3.49 (t, *J*=5.7 Hz, 2H), 1.79 (m, 2H), 1.53~1.41 (m, 6H), 1.09 (m, 4H), 0.87 (m, 4H), 0.08 (s, 18H), -0.05 (s, 18H); ^{13}C NMR (90 MHz, acetone-d_6) δ 168.5, 166.9, 165.7, 164.1, 164.0, 146.4, 144.1, 143.7, 138.7, 137.6, 135.6,135.3, 132.9, 131.7, 131.6, 131.2, 131.1, 130.7, 130.5, 129.8, 129.2, 127.6, 126.1, 123.4, 120.2, 66.1, 64.7, 62.9, 62.3, 33.6, 29.4, 26.6, 26.3, 17.9, 17.4, -1.3, -1.4; MS (MALDI) m/z (relative intensity) 1415 (M^{+}+Na, 90).

Diimide(19f). Following General Procedure A with the modification that 20 equiv of triethylamine was substituted for the zinc acetate, diamine (**18f**) in xylene was converted into diimide (**19f**) as a yellow powder following chromatographic purification with 1:4 hexane/EtOAc as eluent (30%). mp 188-190 °C; IR (KBr) 3450, 1707, 1672 cm^{-1}; ^{1}H NMR (360 MHz, acetone-d_6) δ 8.71 (d, *J*=7.7 Hz, 2H), 8.69 (d, *J*=7.5 Hz, 2H), 8.31 (d, *J*=1.8 Hz, 2H), 8.05 (d, *J*=7.5 Hz, 2H), 7.94 (d, *J*=7.5 Hz, 2H), 7.88 (t, *J*=1.8 Hz, 1H), 7.85 (d, *J*=8.2 Hz, 4H), 7.76 (d, *J*=16.0 Hz, 2H), 7.54 (d, *J*=8.2 Hz, 4H), 6.65 (d, *J*=16.0 Hz, 2H), 4.49 (d, *J*=6.2 Hz, 2H), 4.31 (m, 4H), 3.73 (d, *J*=5.7 Hz, 4H), 3.57 (m, 4H), 2.15(m, 1H), 1.09 (m, 4H), 0.88 (m, 4H), 0.09 (s, 18H), -0.03 (s, 18H); ^{13}C NMR (90 MHz, acetone-d_6) δ 168.5, 166.9, 165.8, 164.1, 164.0, 146.4, 144.1, 143.7, 138.7, 137.6, 135.6, 135.3, 132.9, 131.8, 131.6, 131.24,131.16,130.8, 130.5, 129.8, 129.2, 127.7, 126.1, 123.5, 120.2, 64.7, 64.4, 62.9, 61.1, 44.3, 18.0, 17.4, -1.3, -1.4; MS (MALDI) m/z (relative intensity) 1403 (M^{+}+Na, 90).

Diimide(19g). Following General Procedure A with the modification that 20 equiv of triethylamine was substituted for the zinc acetate, diamine (**18g**) in THF was converted into diimide (**19g**) as a yellow powder following chromatographic purification with 7.5:2.5 hexane/EtOAc as eluent (51%). mp 108-111 ^{o}C. IR (KBr) 1707, 1672 cm^{-1}; ^{1}H NMR (360 MHz, acetone-d_6) δ 8.71 (d, *J*=7.7 Hz, 2H), 8.69 (d, *J*=7.5 Hz, 2H), 8.43 (d, *J*=1.8 Hz, 2H), 8.04 (d, *J*=7.5 Hz, 2H), 7.95 (t, *J*=2.1 Hz, 1H), 7.93 (d, *J*=7.7 Hz, 2H), 7.83 (d, *J*=8.4 Hz, 4H), 7.75 (d, *J*=16.2 Hz, 2H), 7.53 (d, *J*=8.4 Hz, 4H), 7.30~7.20 (m, 4H), 6.64(d, *J*=16.2 Hz, 2H), 4.31 (m, 4H), 3.57 (m, 4H), 2.35 (s, 3H), 1.09 (m, 4H), 0.87 (m, 4H), 0.10 (s, 18H), -0.03 (s, 18H); ^{13}C NMR (90 MHz, acetone-d_6) δ 168.5, 166.8, 164.4, 163.9, 163.8, 150.1, 146.6, 144.06, 144.02, 138.9, 137.6, 136.1,135.6, 132.5, 132.3, 131.9, 131.7, 131.5, 131.0, 130.59, 130.57, 129.9, 129.2, 127.9, 126.4, 123.7, 122.4, 120.3, 108.4, 64.5, 62.9, 29.9, 18.2, 17.6, -1.4, -1.5; MS (MALDI) m/z (relative intensity) 1405 (M^{+}+Na, 100).

Diimide(19h). Following General Procedure A with the modification that 20 equiv of triethylamine was substituted for the zinc acetate, diamine (**18h)** in xylene was converted into diimide (**19h)** as a yellow powder following chromatographic purification with 7:3 hexane/EtOAc as eluent (37%). mp 102-104 ^{o}C. IR (KBr), 1708, 1672 cm^{-1}; ^{1}H NMR (300 MHz, acetone-d_6) δ 8.73 (d, *J*=7.5 Hz, 2H), 8.72 (d, *J*=7.6 Hz, 2H), 8.44 (d, *J*=1.8 Hz, 2H), 8.06 (d, *J*=7.5 Hz, 2H), 7.96 (t, *J*=1.8 Hz, 1H), 7.95 (d, *J*=7.7 Hz, 2H), 7.85 (d, *J*=8.5 Hz, 4H), 7.76 (d, *J*=15.6 Hz, 2H), 7.54 (d, *J*=8.1 Hz, 4H), 7.29 (d, *J*=9.2 Hz, 2H), 7.00 (d, *J*=9.2 Hz, 2H), 6.65 (d, *J*=16.0 Hz, 2H), 4.30 (m, 4H), 3.82 (s, 3H), 3.57 (m, 4H), 1.08 (m, 4H), 0.87 (m, 4H), 0.10 (s, 18H), -0.02 (s, 18H); ^{13}C NMR (75 MHz, acetone-d_6) δ 168.5, 166.9, 164.6, 164.0, 163.9, 158.3, 146.3, 145.3, 144.0, 143.6, 138.5, 137.5, 136.1, 135.2, 132.1, 131.74, 131.69, 131.67, 131.2, 130.6, 130.4, 129.7, 129.1, 127.5, 125.9, 123.4, 123.2, 120.1, 115.1, 64.6, 62.9, 55.8, 17.9, 17.4, -1.3, -1.5; MS (MALDI) m/z (relative intensity) 1405 (M^{+}+Na, 45).

Tetraacid (20a). Following General procedure B, the tetra(silylethyl)ester (**19a)** was converted to the tetraacid (**20a)** (88%, light yellow needles). mp > 350 ^{o}C (decomp). IR (KBr) 3424, 2931, 1707, 1672 cm^{-1}; ^{1}H NMR (360 MHz, THF-d_8): δ 8.69 (d, *J*=7.2 Hz, 2H), 8.67 (d, *J*=7.2 Hz, 2H), 8.14 (d, *J*=1.8 Hz, 2H), 8.03 (d, *J*=7.2 Hz, 2H), 7.85 (d, *J*=7.2 Hz, 2H), 7.73~7.68 (m, 7H), 7.51 (d, *J*=7.2 Hz, 2H), 6.58 (d, *J*=16.2 Hz, 2H), 5.27 (hept, *J*=6.1 Hz, 1H), 1.37 (d, *J*=6.1 Hz, 6H); ^{13}C NMR (90 MHz, THF-d_8 + DMSO-d_6): δ 170.2, 168.6, 164.4, 164.3, 164.2, 147.2, 144.27, 144.26, 137.8, 136.2,135.5, 134.1,133.3, 131.2, 131.0, 130.5, 129.9,129.6, 129.2, 128.3, 125.6, 123.4, 123.3, 120.7, 114.4, 70.0, 22.4; MS (MALDI) m/z (relative intensity) 957 (M^{+}+Na, 100).

Tetraacid (20b). Following General procedure B, the tetra(silylethyl)ester (**19b**) was converted to the tetraacid (**20b**) (52%, yellow needles). mp 340 °C (decomp). IR (KBr) 3436, 1707, 1672 cm^{-1}; ^1H NMR (360 MHz, THF-d_8) δ 8.69 (d, *J*=7.7 Hz, 2H), 8.67 (d, *J*=7.7 Hz, 2H), 8.16 (d, *J*=1.8 Hz, 2H), 8.03 (d, *J*=7.7 Hz, 2H), 7.85 (d, *J*=7.5 Hz, 2H), 7.72 (d, *J*=8.2 Hz, 4H), 7.70 (d, *J*=16.2 Hz, 2H) 7.68 (t, *J*=1.8 Hz, 1H), 7.51 (d, *J*=8.4 Hz, 4H), 6.58 (d, *J*=15.8 Hz, 2H), 5.05 (m, 1H), 2.0~1.0 (m, 10H); ^{13}C NMR (75 MHz, THF-d_8+ DMSO-d_6) δ 170.3, 168.7, 165.2, 164.4, 164.3, 147.3, 144.2, 144.1, 137.8, 135.5, 133.1, 131.6, 131.43, 131.40, 131.36, 131.0, 130.50, 130.48, 129.5, 129.2, 127.7, 125.5, 123.4, 121.17, 121.16, 74.2, 32.4, 30.4, 24.5; MS (MALDI) m/z (relative intensity) 997 (M^++Na, 45).

Tetraacid (20c). Following General procedure B, the tetra(silylethyl)ester (**19c**) was converted to the tetraacid (**20c)** (100%, yellow powder). mp 240 °C (decomp). IR (KBr) 3424, 2954, 1707, 1672 cm^{-1}; ^1H NMR (360 MHz, THF-d_8) δ 8.68 (d, *J*=7.7 Hz, 2H), 8.66 (d, *J*=8.0 Hz, 2H), 8.16 (d, *J*=2.0 Hz, 2H), 8.02 (d, *J*=7.5 Hz, 2H), 7.84 (d, *J*=7.5 Hz, 2H), 7.71 (d, *J*=15.7 Hz, 2H), 7.70 (d, *J*=7.5 Hz, 4H), 7.69 (t, *J*=2.0 Hz, 1H), 7.50 (d, *J*=8.1 Hz, 4H), 7.20~7.00 (m, 5H), 6.57 (d, *J*=15.9 Hz, 2H), 4.37 (t, *J*=6.2 Hz, 2H), 2.67 (t, *J*=6.7 Hz, 2H), 1.79 (m, 4H); ^{13}C NMR (75 MHz, THF-d_8) δ169.9, 168.6, 165.8, 164.3, 164.1, 147.0, 144.2, 143.0, 140.1, 137.7, 135.9, 135.6, 132.7, 131.6, 131.4, 131.20, 131.18, 131.0, 130.5, 129.7, 129.3, 129.23, 129.21, 129.1, 127.8, 126.5, 125.9, 123.5, 121.1, 65.9, 36.1, 29.2, 28.6; MS (MALDI) m/z (relative intensity) 1047 (M^++Na, 100).

Tetraacid (20d). Following General procedure B, the tetra(silylethyl)ester (**19d**) was converted to the tetraacid (**20d**) (28%, yellow needles). mp 320 °C (decomp). IR (KBr) 3433, 1711, 1678 cm^{-1}; ^1H NMR (360 MHz, THF-d_8) δ 8.72 (d, *J*=7.2 Hz, 2H), 8.70 (d, *J*=7.2 Hz, 2H), 8.18 (d, *J*=3.6 Hz, 2H), 8.06 (d, *J*=7.2 Hz, 2H), 7.87 (d, *J*=7.2 Hz, 2H), 7.75~7.71 (m, 7H), 7.53 (d, *J*=7.2 Hz, 4H), 7.33~7.17 (m, 5H), 6.60 (d, *J*=14.4 Hz, 2H), 4.55 (t, *J*=7.2 Hz, 2H), 3.10 (t, *J*=7.2 Hz, 2H); ^{13}C NMR (90 MHz, DMSO-d_6) δ 169.8, 168.9, 164.4, 164.2, 164.0, 153.7, 149.2, 144.7, 143.6, 139.6, 139.1, 138.7, 135.1, 131.9, 131.6, 131.59, 131.55, 130.4, 130.3, 129.7, 129.3, 129.2, 129.18, 127.3, 127.2, 125.0, 122.4, 121.4, 119.4, 64.0, 35.4; MS (MALDI) m/z (relative intensity) 1019 (M^++Na, 90).

Tetraacid (20e). Following General procedure B, the tetra(silylethyl)ester (**19e**) was converted to the tetraacid (**20e**) (57%, yellow powder). mp 340 °C (decomp). IR (KBr) 3436, 1709, 1672 cm^{-1}; ^1H NMR (360 MHz, THF-d_8) δ 8.69 (d, *J*=7.7 Hz, 2H), 8.66 (d, *J*=7.9 Hz, 2H), 8.16 (d, *J*=2.0 Hz, 2H), 8.02 (d, *J*=7.5 Hz, 2H), 7.84 (d, *J*=7.7 Hz, 2H), 7.71 (d, *J*=15.7 Hz, 2H), 7.70 (d, *J*=8.2 Hz, 4H), 7.69 (t, *J*=2.0 Hz, 1H), 7.50 (d, *J*=8.2 Hz, 4H), 6.57 (d, *J*=15.9 Hz, 2H), 4.35 (t, *J*=6.5 Hz, 2H), 3.46 (t, *J*=6.1 Hz, 2H),

1.77 (m, 2H), 1.50~1.40 (m, 6H); ^{13}C NMR (90 MHz, THF-d_8) δ 169.3, 167.7, 165.7, 163.9, 163.8, 146.9, 144.4, 144.3, 139.5, 137.4, 135.6, 132.8, 132.4, 131.5, 131.3, 131.1, 131.0, 130.9, 130.6, 129.6, 128.9, 127.9, 126.2, 123.6, 120.3, 66.0, 62.4, 33.9, 30.6, 26.9, 26.6; MS (MALDI) m/z (relative intensity) 1015 (M^++Na, 100).

Tetraacid (20f). Following General procedure B, the tetra(silylethyl)ester (**19f**) was converted to the tetraacid (**20f**) (100%, yellow powder). mp 270 °C (decomp). IR (KBr) 3424, 3072, 2954, 1713 cm^{-1}; ^{1}H NMR (360 MHz, THF-d_8) δ 8.69 (d, *J*=7.7 Hz, 2H), 8.67 (d, *J*=8.0 Hz, 2H), 8.17 (d, *J*=2.0 Hz, 2H), 8.03 (d, *J*=7.5 Hz, 2H), 7.85 (d, *J*=7.5 Hz, 2H), 7.71 (d, *J*=8.4 Hz, 4H), 7.70 (t, *J*=2.0 Hz, 1H), 7.69 (d, *J*=16.2 Hz, 2H), 7.50 (d, *J*=8.2 Hz, 4H), 6.57 (d, *J*=16.0 Hz, 2H), 4.43 (d, *J*=6.1 Hz, 2H), 3.66 (d, *J*=5.7 Hz, 4H), 2.06 (m, 1H); ^{13}C NMR (90 MHz, THF-d_8) δ 167.9, 166.5, 164.5, 162.6, 162.5, 145.6, 143.02, 142.97, 138.2, 136.0, 134.2, 130.1, 130.0, 129.73, 129.70, 129.3, 129.2, 128.2, 127.58, 127.56 126.6, 124.7, 122.2, 119.9, 118.9, 66.2, 59.8, 29.0; MS (MALDI) m/z (relative intensity) 1003 (M^++Na, 45).

Tetraacid (20g). Following General procedure B, the tetra(silylethyl)ester (**19g**) was converted to the tetraacid (**20g**) (61%, yellow powder). mp 200 °C (decomp). IR (KBr) 3424, 3060, 1707, 1672 cm^{-1}; ^{1}H NMR (360 MHz, THF-d_8) δ 8.70 (d, *J*=7.7 Hz, 2H), 8.68 (d, *J*=7.7 Hz, 2H), 8.32 (d, *J*=1.8 Hz, 2H), 8.03 (d, *J*=7.5 Hz, 2H), 7.85 (d, *J*=7.7 Hz, 2H), 7.78 (d, *J*=1.8 Hz, 1H), 7.72 (d, *J*=8.4 Hz, 4H), 7.70 (d, *J*=15.9 Hz, 2H), 7.50 (d, *J*=8.2 Hz, 4H), 7.18 (d, *J*=6.1 Hz, 4H), 6.58 (d, *J*=16.1 Hz, 2H), 2.34 (s, 3H); ^{13}C NMR (90 MHz, THF-d_8) δ 169.3, 167.8, 164.0, 163.9, 163.8, 147.0, 146.9, 144.45, 144.42, 144.39, 139.55, 139.47, 137.6, 136.0, 135.6, 135.59, 131.4, 131.0, 130.59, 130.54, 130.49, 129.7, 129.0, 128.9, 128.0, 126.2, 123.6, 122.4, 120.3, 21.0; MS (MALDI) m/z (relative intensity) 1005 (M^++Na, 40).

Tetraacid (20h). Following General procedure B, the tetra(silylethyl)ester (**19h**) was converted to the tetraacid (**20h**) (61%, yellow clusters). mp 350 °C (decomp). IR (KBr) 3436, 1707, 1672 cm^{-1}; ^{1}H NMR (360 MHz, THF-d_8) δ 8.70 (d, *J*=7.5 Hz, 2H), 8.68 (d, *J*=7.7 Hz, 2H), 8.31 (d, *J*=1.8 Hz, 2H), 8.04 (d, *J*=7.5 Hz, 2H), 7.85 (d, *J*=7.5 Hz, 2H), 7.77 (d, *J*=1.8 Hz, 1H), 7.71 (d, *J*=8.2 Hz, 4H), 7.70 (d, *J*=15.9 Hz, 2H), 7.51 (d, *J*=8.4 Hz, 4H), 7.20 (d, *J*=9.1 Hz, 2H), 6.93 (d, *J*=9.1 Hz, 2H), 6.58 (d, *J*=16.1 Hz, 2H), 3.78 (s, 3H); ^{13}C NMR (90 MHz, THF-d_8 + DMSO-d_6) δ 170.1, 168.7, 165.0, 164.5, 164.3, 158.5, 147.2, 145.4, 144.3, 144.1, 140.7, 138.1, 135.5, 131.9, 131.7, 131.5, 131.4, 131.0, 130.53, 130.51, 129.7, 129.35, 129.29, 127.7, 125.7, 123.6, 123.4, 121.1, 115.5, 56.2; MS (MALDI) m/z (relative intensity) 1021 (M^++Na, 45).

Triimide (23). Following General Procedure A, anhydride (**5**) and 1,3,5-triaminobenzene (**22**)[5] were combined to furnish triimide (**23**) as a yellow solid following silica gel flash chromatography with 40% EtOAc/hexane as eluent. mp 182-188 ºC. IR ($CHCl_3$) 1714, 1681, 1638 cm^{-1}; ^1H NMR (360 MHz, $CDCl_3$) δ 8.77 (overlapping doublets, *J* = 7.8, 8.5 Hz, 6 H), 7.96 (d, *J* = 7.5 Hz, 3 H), 7.79 (d, *J* = 7.6 Hz, 3 H), 7.72 (d, *J* = 16.0 Hz, 3 H), 7.66 - 7.63 (m, 9H), 7.49 (d, *J* = 8.1 Hz, 6 H), 6.50 (d, *J* = 16.0 Hz, 3 H), 4.33 (t, *J* = 8.4 Hz, 6 H), 3.56 (t, *J* = 8.5 Hz, 6 H), 1.09 (t, *J* = 8.4 Hz, 6 H), 0.83 (t, *J* = 9.1 Hz, 6 H), 0.10 (s, 27 H), 0.09 (s, 27H); ^{13}C NMR (90 MHz, $CDCl_3$) δ 168.2, 166.8, 163.1, 162.9, 145.7, 143.2, 142.8, 137.9, 135.2, 134.5, 131.6, 131.1, 130.9, 129.9, 129.8, 129.5, 129.1, 128.2, 127.0, 124.9, 122.2, 119.5, 64.4, 62.9, 17.4, 17.1, -1.4, -1.6; MS *m/z* (relative intensity) 1835 (MH^+, 80); HRMS calcd for 1834.6557, found 1834.6638.

Hexaacid (24). Following General Procedure B, triimide (**23**) was deprotected to yield hexaacid (**24**) as a yellow solid. mp ≥ 360ºC (decomp). IR (KBr) 3448, 1711, 1676 cm^{-1}; ^1H NMR (200 MHz, THF-d_8) δ 8.71 - 8.65 (overlapping doublets, 6 H), 8.01 (d, *J* = 7.4 Hz, 3 H), 7.82 (d, *J* = 7.5 Hz, 3 H), 7.73 - 7.62 (m, 12 H), 7.49 (d, *J* = 8.0 Hz, 6 H), 6.56 (d, *J* = 16.1 Hz, 3 H); ^{13}C NMR (90 MHz, THF-d_8) δ 169.2, 167.6, 163.6, 163.5, 146.6, 144.5, 144.4, 139.2, 138.1, 136.5, 135.5, 131.4, 131.3, 130.9, 130.8, 129.6, 128.8, 127.9, 126.3, 125.9, 123.7, 120.2; MS *m/z* (relative intensity) 1234 (M^+, 100); HRMS calcd for $C_{72}H_{40}N_3O_{18}$ 1234.2307, found 1234.2312.

Tetraimide (29a). Following General Procedure A, tetraamine (**28a**) and anhydride (**5**) were combined in mesitylene to afford **29a** as an orange solid following silica gel flash chromatography with CH_2Cl_2 as eluent (34%). mp 186-188°C; IR ($CDCl_3$) 1700, 1658, 1639, 1591 cm^{-1}; ^1H NMR ($CDCl_3$) δ 8.75 - 8.69 (m, 8H,), 7.94 (d, *J* = 7.6 Hz, 4H), 7.77 (m, 6H), 7.69 - 7.63 (m, 10H), 7.49 (d, *J* = 8.1 Hz, 8H), 6.51 (d, *J* = 16.0 Hz, 4H), 4.75 (m, 8H), 4.33 (t, *J* = 8.4 Hz, 8H), 3.55 (t, *J* = 8.9 Hz, 8H), 2.18 (m, 8H), 1.09 (t, *J* = 8.4 Hz, 8H), 0.83 (t, *J* = 8.9 Hz, 8H), 0.09 (s, 36 H), -0.03 (s, 36H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.5, 166.9, 163.8, 163.6, 145.3, 143.3, 143.0, 137.5, 134.4, 131.2, 130.8, 130.6, 129.8, 129.5, 129.0, 128.2, 126.9, 125.0, 122.3, 119.4, 64.4, 62.9, 36.9, 35.0, 29.7, 17.4, 17.1, -1.4, -1.5; MS *m/z* (relative intensity) 2469 (M^+, 100); HRMS Calcd for $C_{137}H_{160}N_4O_{24}Si_8$ 2469.9655, found 2469.9549.

Tetraimide (29b). Following General Procedure A, tetraamine (**28b**) and anhydride (**5**) were combined in mesitylene to afford **29b** as an orange solid following silica gel flash chromatography with CH_2Cl_2 to 20% EtOAc/hexane as eluent (27%). mp 131-133°C. IR ($CDCl_3$) 1702, 1659 cm^{-1}; ^1H NMR (200 MHz, $CDCl_3$) δ 8.57 (d, *J* = 7.6 Hz, 8H), 7.90 (d, *J* = 7.5 Hz, 4H), 7.76 - 7.67 (m, 16H), 7.43 (d, *J* = 8.1 Hz, 8H),

6.51 (d, *J* = 16.0 Hz, 4H), 4.35 (t, *J* = 8.5 Hz, 8H), 4.15 (m, 8H), 3.54 (t, *J* = 8.9 Hz, 8H), 1.72 (m, 8H), 1.42 (s, 8H), 1.10 (t, *J* = 8.5 Hz, 8H), 0.81 (t, *J* = 8.9 Hz, 8H), 0.10 (s, 36H), -0.03 (s, 36H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.3, 166.7, 163.5, 163.3, 145.0, 143.2, 142.9, 137.2, 134.3, 130.9, 130.6, 130.3, 129.6, 129.4, 128.8, 128.1, 126.7, 124.9, 122.2, 119.4, 64.2, 62.8, 41.1, 37.3, 33.4, 21.9, 17.3, 17.0, -1.4, -1.6; HRMS Calcd for $C_{141}H_{168}N_4O_{24}Si_8$ 2525.0262, found 2525.0203.

Octaacid (30a). Following General Procedure B, 83 mg (0.03 mmol) of the tetraimide (**29a**) was deprotected to furnish octaacid (**30a**). This crude solid was dissolved in 1:1 THF:EtOAc (20 mL) and then washed with 0.5 N HCl. The organic layer was then washed with 0.5 N NaOH. The aqueous layer was extracted with 1:1 THF:EtOAc (3 x 10 mL) to remove any organic impurities and then it was acidified to pH 2 with 0.5 N HCl, at which point a precipitate deposited. The precipitate was extracted from the aqueous layer with 1:1 THF:EtOAc (3 x 15 mL). The combined organic layers were washed with H_2O (3 x 10 mL), then dried over Na_2SO_4, filtered, and concentrated *in vacuo* to afford 44 mg (78%) of octaacid (**30a**) as a bright yellow solid. mp >310°C; IR (KBr) 3448, 2958, 1701, 1655, 1637, 1591 cm^{-1}; ^{1}H NMR (360 MHz, DMSO-d_6) δ 12.6 (br s, 8H), 8.49-8.44 (m, 8H), 7.97 (d, *J* = 7.3 Hz, 4H), 7.78-7.74 (m, 12H), 7.66 (d, *J* = 16.0 Hz, 4H), 7.40 (d, *J* = 8.0 Hz, 8H), 6.61 (d, *J* = 15.3 Hz, 4H), 4.47-4.41 (m, 8H), 2.03-2.00 (m, 8H); ^{13}C NMR (90 MHz, DMSO-d_6) δ 168.9, 167.6, 163.1, 162.9, 151.5, 145.1, 143.4, 142.7, 139.2, 138.3, 134.0, 130.7, 129.3, 128.7, 128.0, 126.1, 124.9, 124.3, 121.8, 119.7, 34.4, 30.4, 21.1; MS *m/z* (relative intensity) 1669 (MH^+, 100); HRMS Calcd for $C_{97}H_{65}N_4O_{24}$ 1669.3989, found 1669.3976.

Octaacid (30b). Following General Procedure B, 126 mg (0.05 mmol) of the tetraimide (**29b**) was deprotected to furnish the octaacid (**30b**). The crude yellow-green solid was dissolved in 1:1 THF:EtOAc (30 mL) and then washed with 0.5 N HCl. The separated organic layer was then washed with 0.5 N NaOH. The aqueous layer was extracted with 1:1 THF:EtOAc (3 x 15 mL) then acidified to pH 2 with 0.5 N HCl, at which point a precipitate deposited. The precipitate was removed from the aqueous layer with 1:1 THF:EtOAc (3 x 20 mL). The combined organic layers were washed with H_2O (3 x 10 mL), dried over Na_2SO_4, filtered, and concentrated *in vacuo* to afford 69 mg (80%) of yellow solid. mp >310°C. IR (KBr) 3448, 1702, 1655, 1631, 1591 cm^{-1}; ^{1}H NMR (300 MHz, DMSO-d_6) δ 12.7 (bs, 8H), 8.36 (dd, *J* = 7.3 Hz, 4H), 8.30 (dd, *J* = 7.3 Hz, 4H), 7.89 (d, *J* = 7.8 Hz, 4H), 7.70 - 7.60 (m, 16H), 7.25 (d, *J* = 8.2 Hz, 8H), 6.60 (d, *J* = 16.4 Hz, 4H), 3.92 (m, 8H), 1.48 (m, 8H,), 1.21 (m, 8H); ^{13}C NMR (90 MHz, DMSO-d_6) δ 168.8, 167.6, 162.9, 162.8, 151.5, 145.0, 143.4, 142.6, 139.2, 138.2, 134.0, 130.6,

128.9, 128.3, 128.0, 125.9, 124.9, 123.9, 121.5, 119.7, 34.4, 30.4, 21.2, 21.0; HRMS Calcd for $C_{101}H_{73}N_4O_{24}$ 1725.4615, found 1725.4741.

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