Supporting Information

One-Pot Anodic Conversion of Symmetrical Bisamides of Ethylene Diamine to Unsymmetrical gem-Bisamides of Methylene Diamine

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1. General information

Equipment, spectral measurements, and reagents

Equipment used in this study for measurements of $^1$H NMR (400 MHz, 500 MHz), $^{13}$C NMR (100 MHz, 125 MHz), MS, HRMS, and FT-IR, as well as carrying out TLC, cyclic voltammetry, and outlining purity of reagents, electrolytes and solvents, have been described previously [1].

2. Preparation of ethylene bisamides substrates

Symmetrical bisamides 1-11 were prepared according to a previously described procedure [1] by reacting ethylenediamine with the corresponding benzoyl chloride derivative. For convenience, a typical procedure is given here in case the chloride of the acid is not available. Thionyl chloride (30 mmol) is added to a 100-ml round-bottom flask containing 22 mmol of the respective benzoic acid derivative. After 2h of reflux, the excess of thionyl chloride is evaporated and the remaining reaction mixture was dissolved in 100 ml of dichloromethane followed by addition of 10 mmol of ethylenediamine. The reaction mixture is allowed to stand overnight with stirring, followed by filtering by vacuum suction. The solid residue was recrystallized from ethyl acetate-water (9:1) and the resulting white (or yellow) precipitate was dried, weighted and its structure was verified by $^1$H NMR. Most prepared bisamides are known and fully characterized: N,N’-(ethane-1,2-diyl)dibenzamide (1) [3,4,7], N,N’-(ethane-1,2-diyl)bis(4-methylbenzamide) (2) [3], N,N’-(ethane-1,2-diyl)bis(4-methoxybenzamide) (3) [4], N,N’-(ethane-1,2-diyl)bis(4-chlorobenzamide) (4) [2,3], N,N’-(ethane-1,2-diyl)bis(4-bromobenzamide) (5) [5], N,N’-(ethane-1,2-diyl)bis(4-nitrobenzamide) (6) [2], N,N’-(ethane-1,2-diyl)bis(3-fluorobenzamide) (9) [commercially available from Aurora Fine Chemicals, LLC, San Diego, CA, USA], N,N’-(ethane-1,2-diyl)bis(2-phenylacetamide) (10) [8].

Symmetrical bisamides N,N’-(ethane-1,2-diyl)bis(4-cyanobenzamide) (7), N,N’-(ethane-1,2-diyl)bis(3-cyanobenzamide) (8), and N,N’-(ethane-1,2-diyl)bis(4-cyclobutylamide) (11) are new (vide infra).
Following the general procedure, the desired compound was obtained as a white solid, 2.06 g, 76% yield. $^1$H NMR (DMSO-$d_6$, 400 MHz) $\delta$ 3.40 – 3.52 (m, 4H), 7.42 – 7.57 (m, 6H), 7.82 – 7.91 (m, 4H), 8.62 (t, $J = 5.4$ Hz, 2H).
N,N'-(ethane-1,2-diyl)bis(4-methylbenzamide) (2) [3]

Yield: 2.05 g, 69% (white solid). $^1$H NMR (400 MHz, DMSO-$d_6$) $\delta$ 2.35 (s, 6H), 3.42 – 3.45 (m, 4H), 7.26 (d, $J$ = 7.9 Hz, 4H), 7.71 – 7.79 (m, 4H), 8.52 (t, $J$ = 5.4 Hz, 2H).
N,N'-(ethane-1,2-diyl)bis(4-methoxybenzamide) (3) [4]

Yield: 2.07g, 63% (white solid). $^1$H NMR (400 MHz, DMSO-$d_6$) $\delta$ 3.39 – 3.44 (m, 4H), 3.81 (s, 6H), 6.97 – 7.01 (m, 4H), 7.82 – 7.86 (m, 4H), 8.50 (t, $J = 4.4$ Hz, 2H).
Yield: 2.89 g, 86% (white solid). $^1$H NMR (400 MHz, DMSO-$_d$6) $\delta$ 3.40 – 3.48 (m, 4H), 7.50 – 7.59 (m, 4H), 7.82 – 7.91 (m, 4H), 8.69 (t, $J = 5.5$ Hz, 2H).
Yield: 3.29 g, 78% (white solid). $^1$H NMR (400 MHz, DMSO-$d_6$) $\delta$ 3.40 – 3.48 (m, 4H), 7.50 – 7.57 (m, 4H), 7.84 – 7.90 (m, 4H), 8.70 (t, $J = 5.4$ Hz, 2H).
N,N'- (ethane-1,2-diyl) bis(4-nitrobenzamide) (6) [2]

Yield: 1.85 g, 52% (yellow solid). $^1$H NMR (400 MHz, DMSO-$d_6$) $\delta$ 3.48 – 3.51 (m, 4H), 8.06 – 8.10 (m, 4H), 8.30 – 8.35 (m, 4H), 8.97 (t, $J = 5.5$ Hz, 2H).
N,N'-((ethane-1,2-diyl)bis(3-fluorobenzamide) (9)

Yield: 2.44 g, 80% (white solid). $^1$H NMR (400 MHz, DMSO-$d_6$) $\delta$ 3.39 – 3.53 (m, 4H), 7.38 (tdd, $J = 8.5, 2.7, 1.0$ Hz, 2H), 7.53 (td, $J = 8.0, 5.8$ Hz, 2H), 7.64 (ddd, $J = 10.1, 2.7, 1.5$ Hz, 2H), 7.70 (dt, $J = 7.8, 1.2$ Hz, 2H), 8.70 (t, $J = 5.4$ Hz, 2H).
N,N'-ethane-1,2-diylbis(2-phenylacetamide) (10) [8]

Yield: 2.23 g, 72% (white solid). $^1$H NMR (400 MHz, DMSO-$d_6$) δ 3.08 – 3.14 (m, 4H), 3.39 (s, 4H), 7.19 – 7.32 (m, 10H), 8.09 (t, $J = 5.2$ Hz, 2H).
4. Characterization of unknown new substrates

\[ N,N'-(ethane-1,2-diyl)bis(4-cyanobenzamide) \] (7)
Yield: 2.51 g, 79% (white solid). $^1$H NMR (DMSO-$d_6$) $\delta$ 3.40 – 3.58 (m, 4H), 7.96 (d, $J = 8.5$ Hz, 4H), 8.00 (d, $J = 8.4$ Hz, 4H), 8.90 (t, $J = 5.4$ Hz, 2H); $^{13}$C NMR $\delta$ 114.0, 118.8, 128.5, 132.8, 130.0, 165.6; HRMS (ESI): calculated for C$_{18}$H$_{15}$N$_4$O$_2$+H: 319.11950; found: 319.11974. m.p. 249-252$^\circ$C. IR (NaCl plate): IR (NaCl plate): 1651, 2268, 2863, 2905, 3457.
N,N'-{(ethane-1,2-diyl)bis(3-cyanobenzamide)} (8)
Yield: 2.93 g, 92% (white solid). $^1$H NMR (DMSO-$d_6$) δ 3.43 – 3.50 (m, 4H), 7.70 (t, $J = 7.8$ Hz, 2H), 8.01 (dt, $J = 7.7$, 1.4 Hz, 2H), 8.15 (dt, $J = 8.0$, 1.5 Hz, 2H), 8.26 (t, $J = 1.7$ Hz, 2H), 8.84 (t, $J = 5.5$ Hz, 2H); $^{13}$C NMR (DMSO-$d_6$) δ 111.9, 118.8, 130.2, 131.4, 132.5, 135.0 (d, $J = 1.6$ Hz), 136.0 (d, $J = 2.2$ Hz), 165.2; HRMS (ESI): calculated for C$_{18}$H$_{15}$N$_4$O$_2$+H: 319.11950; found: 319.11974; m.p. 252-255°C. IR (NaCl plate): 1478, 1657, 2263, 2860, 2931, 3412.
$N,N'-(\text{ethane-1,2-diyl})\text{dicyclobutanecarboxamide}$ (11)
Yield: 1.26 g, 56% (white-beige solid). $^1$H NMR (CDCl$_3$) $\delta$ 1.78 – 2.06 (m, 4H), 2.08 – 2.34 (m, 8H), 3.02 (pd, $J = 8.6$, 1.0 Hz, 2H), 3.39 – 3.42 (m, 4H), 6.20 (s, 2H); $^{13}$C NMR (CDCl$_3$) $\delta$ 18.1, 25.4, 39.9, 40.2, 176.4; HRMS (ESI): calculated for C$_{12}$H$_{21}$N$_2$O$_2$+H: 225.16030; found: 225.16073; m.p. 218-220°C. IR (NaCl plate): 1261, 1670, 2865, 2923, 3368.


5. Electrolysis procedure and isolation of products

Preparative anodic oxidation was performed at controlled potential electrolysis (CPE) using a PAR Potentiostat/Galvanostat Model 273A, and H-type two-compartment cell equipped with a medium glass frit as a membrane. The anode compartment contained a platinum foil, or C felt or C rod (immersed area of ~5 cm²) as working electrodes. In addition, a polished silver wire 0.1 M LiClO₄.quasi-reference electrode (commonly used in organic electrochemistry; approx. +0.19 V vs. Ag/AgCl (+0.15 V vs. SCE), was immersed in the anolyte solution in a glass cylinder equipped with a fine glass frit at its end. A stainless-steel counter electrode was used in the catholyte. Both compartments contained acetonitrile (AR grade) and CPE was conducted by controlling the potential at the Ep value for each substrate (Table 1). Initial currents were in the range of 60-120 mA, depending on the substrate and reached values of 6-12 mA, respectively, upon termination. Yields of products were determined by ¹H NMR integration using benzoquinone as internal standard. Since some of the products undergo facile hydrolysis/decomposition it is suggested that the analysis will be done immediately after terminating the electrolysis. Notably, this procedure of analyzing a mixture of products simultaneously and successfully has been based on prior separation and characterization of the individual products.

In a typical electrolysis, using the above described electrochemical setup, 1 mmol (268 mg) of substrate 1 was added to the anode compartment that contained a total volume of 35 mL of acetonitrile-0.1 M LiClO₄. The anode was a carbon rod (6 mm in dia.) and immersed about 2.5 cm of its length. The cathode was a stainless steel spatula. The cell temperature was maintained around 40 °C by water bath to allow solubility of substrates. The applied potential was 1.95 V (vs. Ag wire) and reaction mixture was stirred during electrolysis (~5 h) and terminated after passing 4 F/mol (386 Coul.). Pulsing (to 0 V for 1 s, every 25 s) was required during electrolysis to avoid passivation of a working electrode surface, probably due to the formation of the insulating polymer. The reaction mixture was evaporated to reach ~2-3 ml volume (careful, not to dryness!). Separation of products for spectral analysis was carried out by silica
gel column chromatography, using different eluents of ethyl acetate (10-100%)-hexane followed by a more polar mixture of ethyl acetate (50-90%)-acetone. The yield of the major isolated product 1A is 50% (95 mg). Yields of other minor products were determined by $^1$H NMR integration using benzoquinone as internal standard and are described in Table 2 (last line).

6. Characterization of products

Products from N,N'-{(ethane-1,2-diyl)dibenzamide, PhCONH(CH$_2$)$_2$NHCOPh (I)

(Unknown products are marked in yellow; only integrated peaks are relevant to products)

N-(acetamidomethyl)benzamide, Ph-CO-NH-CH$_2$-NH-CO-CH$_3$ (1A) [9]

Yield: 88.0 mg, 46% (white solid). Spectral data: $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 2.02 (1s, 3H), 4.83 (t, $J = 6.3$ Hz, 2H), 7.11 (s, 1H), 7.43 – 7.47 (m, 2H), 7.52 – 7.56 (m, 1H), 7.65 (s, 1H), 7.81 – 7.84 (m, 2H).
N,N'-methylene dibenzamide, Ph-CO-NH-CH₂-NH-CO-Ph (1B) [3, 10,11]

Yield: 30.5 mg, 12% (white solid). Spectral data: $^1$H NMR (400 MHz, DMSO-$d_6$) $\delta$ 4.87 (t, $J = 5.6$ Hz, 2H), 7.46 (dd, $J = 8.2$, 6.6 Hz, 4H), 7.51 – 7.56 (m, 2H), 7.91 (dt, $J = 7.0$, 1.4 Hz, 4H), 9.06 (t, $J = 5.5$ Hz, 2H).
Yield: 5.2-58.5 mg, 4-45% (Table 3) (yellowish oil). Spectral data: $^1$H NMR (400 MHz, DMSO-$d_6$) $\delta$ 1.79 (s, 6H), 4.33 (t, $J = 6.0$ Hz, 2H), 8.43 (t, $J = 6.5$ Hz, 2H).
N-formyl benzamide, PhCONHCHO (1D) is known [13].

Yield: 3.0-28.1 mg, 2-19% (Table 2) (white solid). Spectral data: $^1$H NMR (400 MHz, DMSO-$d_6$) $\delta$ 7.53 – 7.58 (m, 2H), 7.65 – 7.70 (m, 1H), 8.02 – 8.05 (m, 2H), 9.27 (d, $J$ = 8.8 Hz, 1H), 11.74 (d, $J$ = 8.6 Hz, 1H).
Products from \( N,N'-(\text{ethane-1,2-diyl})\text{bis(4-methylbenzamide)} \) (2)

\( N\)-(acetamidomethyl)-4-methylbenzamide, \( p\)-Me-C\(_6\)H\(_4\)-CO-NH-CH\(_2\)-NH-CO-CH\(_3\) (2A) [New] \[\text{New}\]
Yield: 123.6 mg, 60% (white solid). Spectral data: $^1$H NMR (400 MHz, DMSO-$d_6$) δ 1.82 (1s, 3H), 2.35 (1s, 3H), 4.58 (t, $J = 5.9$ Hz, 2H), 7.26 (d, $J = 7.9$ Hz, 2H), 7.78 – 7.81 (m, 2H), 8.46 (t, $J = 5.9$ Hz, 1H), 8.98 (t, $J = 5.8$ Hz, 1H); $^{13}$C NMR (100 MHz, DMSO-$d_6$) δ 21.42, 22.94, 44.52, 127.83, 129.27, 131.51, 141.78, 166.93, 170.00. HRMS (ESI): calculated for C$_{11}$H$_{15}$N$_2$O$_2$+H: 207.11335; found: 207.11316; R$_f$ (ethyl acetate:hexane = 9:1) 0.25; m.p. 165-167°C (white solid). IR (NaCl plate): 1115, 1343, 1440, 1563, 1662, 2865, 2922, 3467, 3525.
N,N'-methylenebis(4-methylbenzamide), p-Me-C₆H₄-CO-NH-CH₂-NHCO-C₆H₄-p-Me (2B) [3,11,12,14]

Yield: 8.5 mg, 3% (white solid). Spectral data: $^1$H NMR (400 MHz, DMSO-$d_6$) δ 2.35 (s, 6H), 4.85 (t, $J = 5.6$ Hz, 2H), 7.26 (d, $J = 8.0$ Hz, 4H), 7.81 – 7.83 (m, 4H), 8.95 (t, $J = 5.6$ Hz, 2H).
Products from $N,N'$-(ethane-1,2-diyl)bis(4-chlorobenzamide) (4)

$N$-(acetamidomethyl)-4-chlorobenzamide, p-$\text{Cl-C}_6\text{H}_4\text{-CO-NH-CH}_2\text{-NH-CO-CH}_3$ (4A) [New]
Yield: 115.5 mg, 51% (white solid). Spectral data: $^1$H NMR (400 MHz, DMSO-$d_6$) $\delta$ 1.82 (1s, 3H), 4.59 (t, $J = 5.8$ Hz, 2H), 7.52 – 7.56 (m, 2H), 7.89 – 7.92 (m, 2H), 8.50 (t, $J = 5.9$ Hz, 1H), 9.16 (t, $J = 5.8$ Hz, 1H); $^{13}$C NMR (100 MHz, DMSO-$d_6$) $\delta$ 22.94, 44.59, 128.84, 129.76, 133.09, 136.72, 166.01, 170.03. HRMS (ESI): calculated for C$_{10}$H$_{11}$N$_2$O$_2$Cl$+H$: 227.05818; found: 227.05858; R$_f$ (ethyl acetate:hexane = 9:1) 0.2; m.p. 174-178°C (white solid). IR (NaCl plate): 1112, 1442, 1545, 1658, 2882, 2930, 3425, 3546.
N,N'-methylenebis(4-chlorobenzamide), p-Cl-C₆H₄-CO-NH-CH₂-NH-CO-C₆H₄-p-Cl (4B) [3,12,14]

Yield: 32.3 mg, 10% (white solid). Spectral data: ¹H NMR (400 MHz, DMSO-d₆) δ 4.85 (t, J = 5.6 Hz, 2H), 7.53 – 7.56 (m, 4H), 7.91 – 7.94 (m, 4H), 9.17 (t, J = 5.6 Hz, 2H).
Products from N,N'-(ethane-1,2-diyl)bis(4-bromobenzamide) (5)

N-(acetamidomethyl)-4-bromobenzamide, p-Br-C₆H₄-CO-NH-CH₂-NH-CO-CH₃ (5A) [New]
Yield: 124.7 mg, 46% (white solid). Spectral data: $^1$H NMR (400 MHz, DMSO-$d_6$) δ 1.82 (s, 3H), 4.58 (t, $J = 5.9$ Hz, 2H), 7.66 – 7.70 (m, 2H), 7.81 – 7.85 (m, 2H), 8.50 (t, $J = 5.9$ Hz, 1H), 9.17 (t, $J = 5.8$ Hz, 1H). $^{13}$C NMR (100 MHz, DMSO-$d_6$) δ 22.92, 44.61, 125.69, 129.95, 131.79, 133.43, 166.20, 170.19. HRMS (ESI): calculated for C$_{10}$H$_{11}$BrN$_2$O$_2$+H: 270.00039; found: 270.00043. R$_f$ (ethyl acetate:hexane = 9:1) 0.2; m.p. 184-187°C (white solid). IR (NaCl plate): 1105, 1317, 1545, 1560, 1636, 2849, 2917, 3415, 3582.
Products from N,N’-(ethane-1,2-diyl)bis(4-nitrobenzamide) (6)

N-(acetamidomethyl)-4-nitrobenzamide, p-NO₂-C₆H₄-CO-NH-CH₂-NH-CO-CH₃ (6A) [New]
Yield: 94.8 mg, 40% (yellowish solid). Spectral data: $^1$H NMR (400 MHz, DMSO-$d_6$) δ 1.84 (s, 3H), 4.62 (t, $J = 5.8$ Hz, 2H), 8.09 – 8.12 (m, 2H), 8.30 – 8.32 (m, 2H), 8.56 (t, $J = 5.9$ Hz, 1H), 9.41 (t, $J = 5.7$ Hz, 1H). $^{13}$C NMR (100 MHz, DMSO-$d_6$) δ 22.92, 44.68, 123.95, 129.35, 139.96, 149.60, 165.46, 170.16. HRMS (ESI): calculated for C$_{10}$H$_{11}$N$_3$O$_4$+H: 238.08223; found: 238.08287. R$_f$ (ethyl acetate:hexane = 9:1) 0.21; m.p. 171-173°C (yellowish solid). IR (NaCl plate): 1115, 1343, 1440, 1563, 1662, 2865, 2922, 3467, 3525.
N,N'-methylenebis(4-nitrobenzamide), p-NO₂-C₆H₄-CO-NH-CH₂-NH-CO-C₆H₄-p-NO₂ (6B) [3,7,10,13,14]

Yield: 68.8 mg, 20% (yellowish solid). Spectral data: $^1$H NMR (400 MHz, DMSO-$d_6$) δ 4.90 (t, $J = 5.6$ Hz, 1H), 8.12 – 8.15 (m, 2H), 8.31 – 8.33 (m, 2H), 9.49 (t, $J = 5.6$ Hz, 1H).
Yield: 16.5 mg, 5% (yellowish solid). Spectral data: $^1$H (400 MHz, DMSO-$d_6$) $\delta$ 7.73 (s, 2H), 8.08 – 8.11 (m, 2H), 8.29 – 8.32 (m, 2H).
Products from $N,N'$-(ethane-1,2-diyl)bis(4-cyanobenzamide) (7)

N-(acetamidomethyl)-4-cyanobenzamide, p-CN-C₆H₄-CO-NH-CH₂-NH-CO-CH₃ (7A) [New]
Yield: 67.3 mg, 31% (white solid). Spectral data: $^1$H NMR (DMSO-$d_6$) $\delta$ 1.83 (s, 3H), 4.61 (t, $J = 5.8$ Hz, 2H), 7.94 – 7.97 (m, 2H), 8.02 – 8.05 (m, 2H), 8.54 (t, $J = 6.0$ Hz, 1H), 9.34 (t, $J = 5.7$ Hz, 1H). $^{13}$C NMR (100 MHz, DMSO-$d_6$) $\delta$ 22.92, 114.27, 118.76, 128.67, 132.88, 138.27, 165.68, 170.11. HRMS (ESI): calculated for C$_{11}$H$_{11}$N$_3$O$_2$+H: 218.09240; found: 218.09335. Rf (ethyl acetate:hexane = 9:1) = 0.23. m.p. 161-164°C (white solid). IR (NaCl plate): 1657, 2260, 2850, 2925, 3363, 3401.
N,N'-methylenebis(4-cyanobenzamide), p-CN-C₆H₄-CO-NH-CH₂-NH-CO-C₆H₄-p-CN (7B) [New]
Yield: 15.2 mg, 5% (white solid). Spectral data: $^1$H NMR (400 MHz, DMSO-$d_6$) $\delta$ 4.87 – 4.91 (m, 2H), 7.96 (dd, $J = 8.5$, 2.4 Hz, 4H), 8.04 – 8.06 (m, 4H), 9.39 (t, $J = 5.6$ Hz, 2H). $^{13}$C NMR (100 MHz, DMSO-$d_6$) $\delta$ 45.76, 114.29, 118.75, 128.75, 132.86, 138.31, 165.67. HRMS (ESI): calculated for C$_{17}$H$_{12}$N$_4$O$_2$+H: 305.10385; found: 305.10377. R$_f$ (ethyl acetate:hexane = 9:1) 0.63. m.p. 161-164°C (white solid). IR (NaCl plate): 1653, 2257, 2852, 2923, 3370.
Products from \(N,N'\)-(ethane-1,2-diyl)bis(3-cyanobenzamide) (8)

N-(acetimidomethyl)-3-cyanobenzamide, m-CN-C₆H₄-CO-NH-CH₂-NH-CO-CH₃ (8A) [New]
Yield: 65.1 mg, 30% (white solid). Spectral data: $^1$H NMR (400 MHz, DMSO-$d_6$) $\delta$ 1.83 (s, 3H), 4.61 (t, $J = 5.9$ Hz, 2H), 7.70 (t, $J = 7.8$ Hz, 1H), 8.02 (dt, $J = 7.7$, 1.4 Hz, 1H), 8.19 (dt, $J = 7.9$, 1.5 Hz, 1H), 8.31 (t, $J = 1.8$ Hz, 1H), 8.55 (t, $J = 5.9$ Hz, 1H), 9.30 (t, $J = 5.7$ Hz, 1H). $^{13}$C NMR (100 MHz, DMSO-$d_6$) $\delta$ 22.93, 44.61, 111.96, 118.74, 130.25, 131.50, 132.62, 135.31, 135.34, 165.19, 170.13. HRMS (ESI): calculated for C$_{11}$H$_{11}$N$_3$O$_2$+H: 218.09240; found: 218.09335. $R_f$ (ethyl acetate:hexane = 9:1) 0.25. m.p. 173-176°C (white solid). IR (NaCl plate): 1464, 1653, 2271, 2871, 2920, 3520.
N,N'-methylenebis(3-cyanobenzamide), m-CN-C₆H₄-CO-NH-CH₂-NH-CO-C₆H₄-m-CN (8B) [New]
Yield: 15.2 mg, 8% (white solid). Spectral data: $^1$H NMR (400 MHz, DMSO-$d_6$) $\delta$ 4.90 (t, $J = 5.6$ Hz, 2H), 7.70 (t, $J = 7.8$ Hz, 2H), 8.00 (tt, $J = 5.5$, 1.4 Hz, 2H), 8.21 (dt, $J = 8.0$, 1.5 Hz, 2H), 8.33 (t, $J = 1.8$ Hz, 2H), 9.35 (t, $J = 5.6$ Hz, 2H). $^{13}$C NMR (100 MHz, DMSO-$d_6$) $\delta$ 45.65, 111.96, 118.73, 130.21, 131.56, 132.68, 135.31, 135.34, 165.20. HRMS (ESI): calculated for C$_{17}$H$_{13}$N$_4$O$_2$+H: 305.10385; found: 305.10363. $R_f$ (ethyl acetate:hexane = 9:1) = 0.54; m.p. 156-160°C (white solid). IR (NaCl plate): 1653, 2257, 2852, 2923, 3370.
Products from \(N,N'-(\text{ethylene-1,2-diyl})\text{bis(3-fluorobenzamide})\) \((9)\)

(Commercially available from Aurora Fine Chemicals, LLC, San Diego, CA, USA)

\(N\)-(acetamidomethyl)-3-fluorobenzamide, \(m\)-F-C\(_6\)H\(_4\)-CO-NH-CH\(_2\)-NH-CO-CH\(_3\) \((9A)\) [New]
Yield: 77.7 mg, 37% (white solid). Spectral data: $^1$H NMR (400 MHz, DMSO-$d_6$) $\delta$ 1.83 (s, 3H), 4.60 (t, $J = 5.9$ Hz, 2H), 7.39 (tdd, $J = 8.5, 2.6, 1.0$ Hz, 1H), 7.52 (td, $J = 8.0, 5.9$ Hz, 1H), 7.69 (ddd, $J = 10.1, 2.7, 1.5$ Hz, 1H), 7.74 – 7.76 (m, 1H), 8.52 (t, $J = 6.0$ Hz, 1H), 9.18 (t, $J = 5.7$ Hz, 1H). $^{13}$C NMR (100 MHz, DMSO-$d_6$) $\delta$ 22.86, 44.72, 114.54 (d, $J = 22.7$ Hz), 118.87 (d, $J = 20.9$ Hz), 123.94, 131.02 (d, $J = 8.3$ Hz), 136.57, 162.39 (d, $J = 244.3$ Hz), 165.87, 170.54. HRMS (ESI): calculated for C$_{10}$H$_{11}$N$_2$O$_2$F$+H$: 211.08828; found: 211.08821; R$_f$ (ethyl acetate:hexane = 9:1) 0.22. m.p. 181-184°C (white solid). IR (NaCl plate): 1235, 1506, 1602, 1654, 2855, 2920, 3423, 3530.
Yield: 17.4 mg, 6% (white solid). Spectral data: $^1$H NMR (400 MHz, DMSO-$d_6$) $\delta$ 4.86 (t, $J = 5.4$ Hz, 2H), 7.43 – 7.36 (m, 2H), 7.53 (td, $J = 8.0$, 5.9 Hz, 2H), 7.69 – 7.73 (m, 4H), 7.77 (dd, $J = 7.6$, 1.4 Hz, 2H), 9.20 (t, $J = 5.8$ Hz, 2H).
7. References


