Supplementary Information (SI) for Environmental Science: Advances.
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Supporting Information

for the article

Green Valorisation of Bio-wastes – Electrochemical "One-pot" Reductive Amination of Furfural on Graphite Electrode in Water

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1. NMR spectra of the reaction mixtures

1.1 Working potential dependence

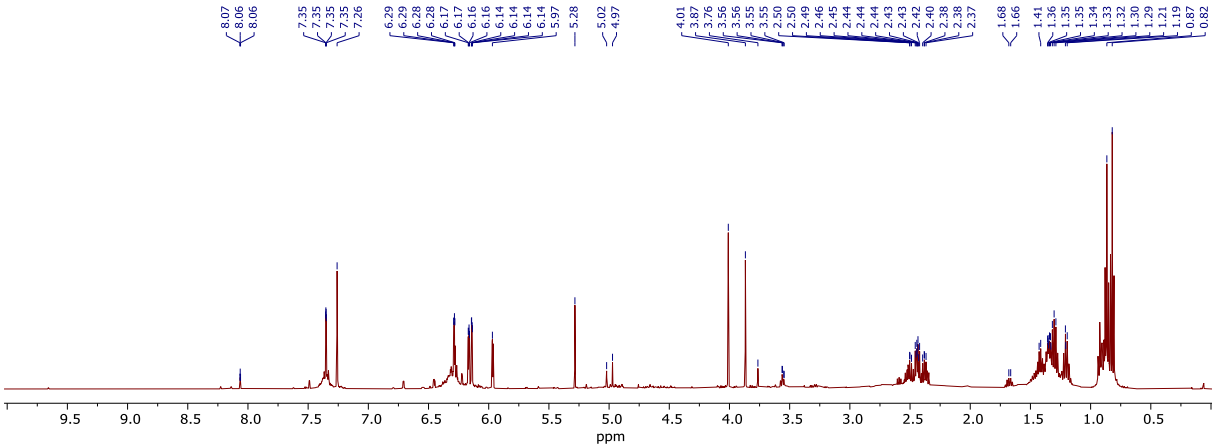

Fig S1. ^{1}H NMR spectrum of the isolated reaction mixture from the electroreductive amination of FF with *n*-BuNH$_2$ at pH 11, potential –1.7 V.

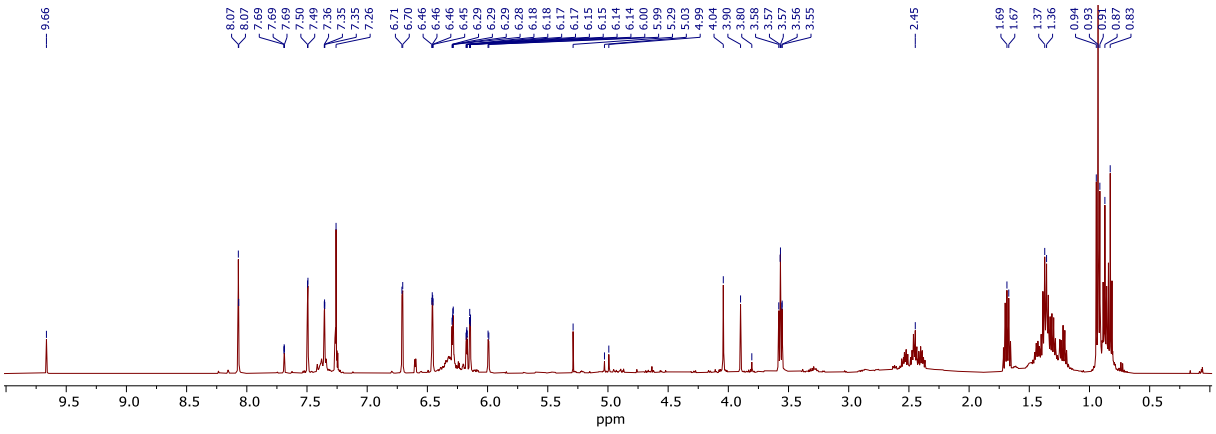

Fig S2. ^{1}H NMR spectrum of the isolated reaction mixture from the electroreductive amination of FF with *n*-BuNH$_2$ at pH 11, potential –1.6 V.

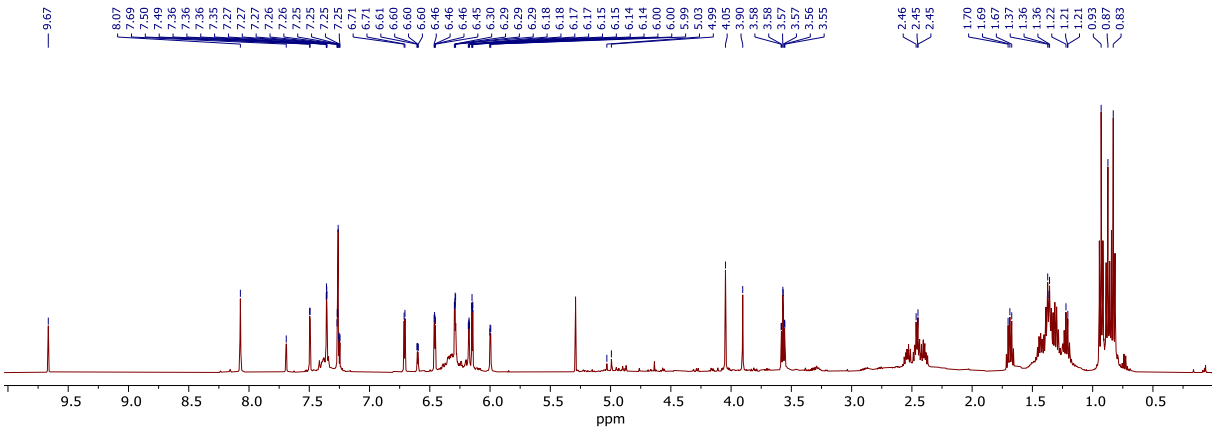

Fig S3. ^{1}H NMR spectrum of the isolated reaction mixture from the electroreductive amination of FF with *n*-BuNH$_2$ at pH 11, potential –1.5 V.

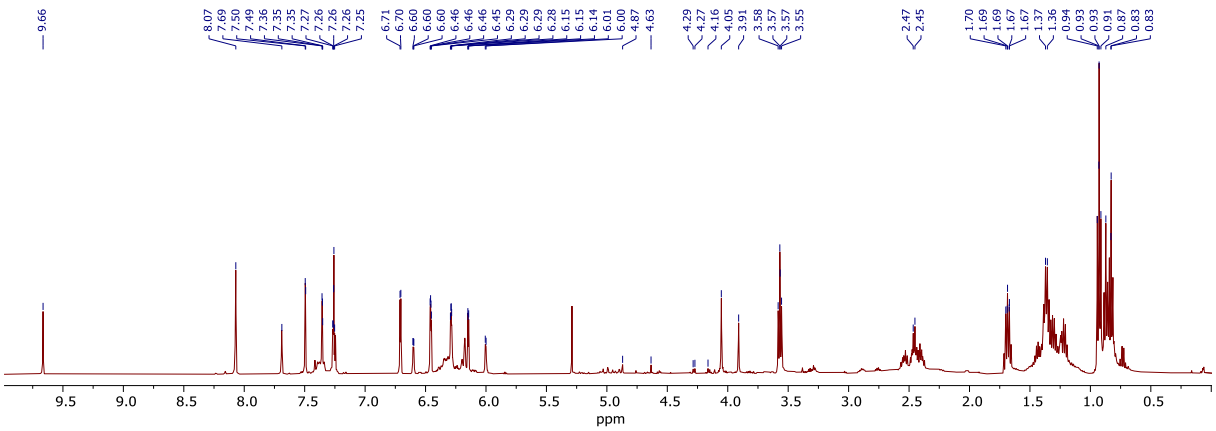

Fig S4. ^{1}H NMR spectrum of the isolated reaction mixture from the electroreductive amination of FF with *n*-BuNH$_2$ at pH 11, potential –1.4 V.

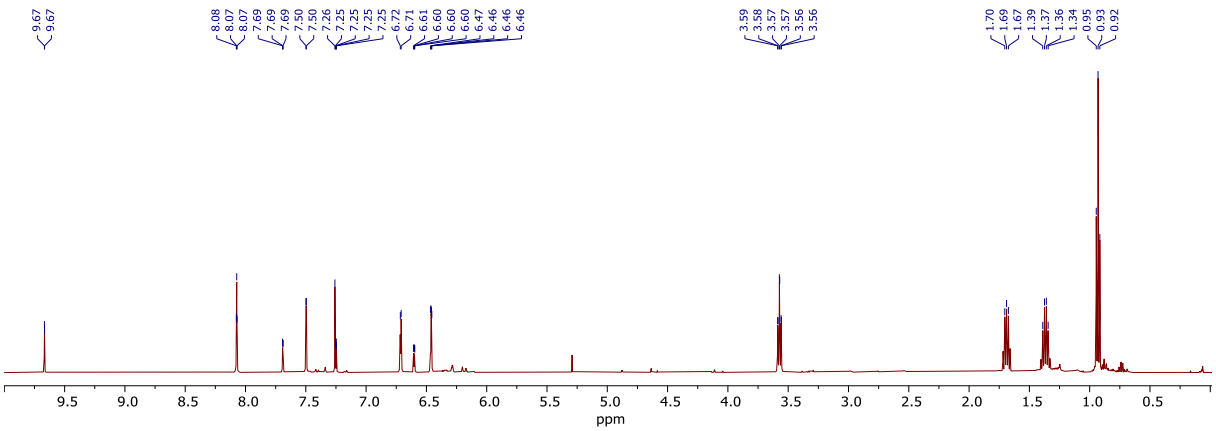

Fig S5. ^{1}H NMR spectrum of the isolated reaction mixture from the electroreductive amination of FF with *n*-BuNH$_2$ at pH 11, potential –1.3 V.

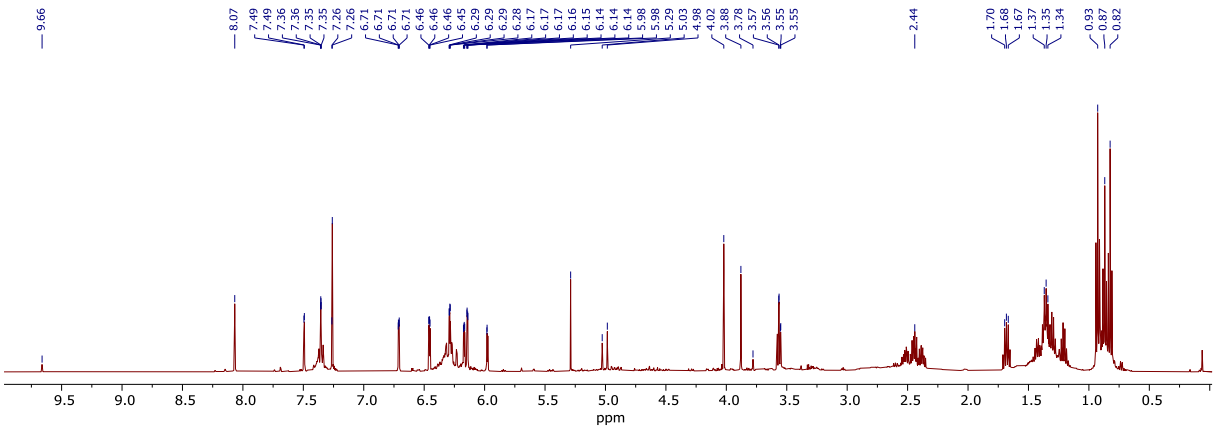

Fig S6. ^{1}H NMR spectrum of the isolated reaction mixture from the electroreductive amination of FF with *n*-BuNH$_2$ at pH 7.5, potential –1.7 V.

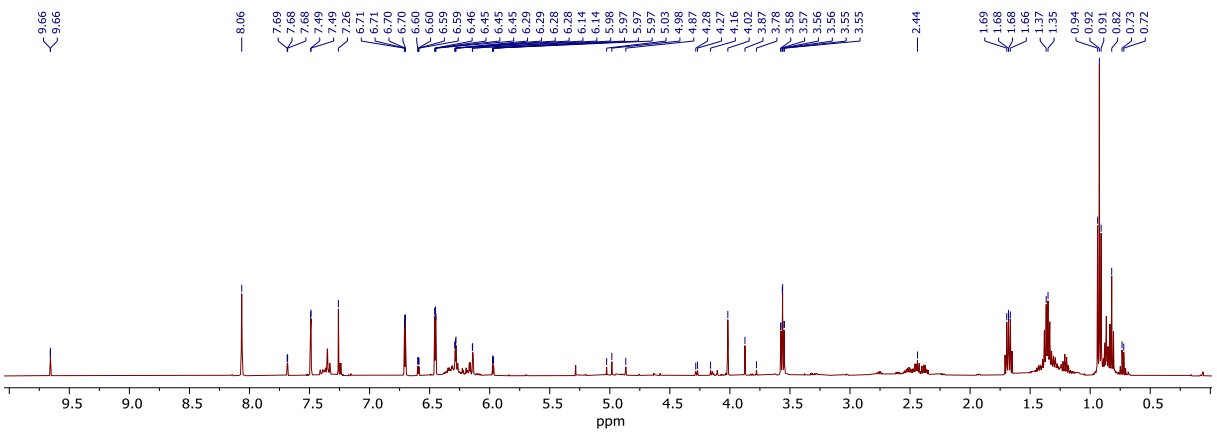

Fig S7. ^{1}H NMR spectrum of the isolated reaction mixture from the electroreductive amination of FF with *n*-BuNH$_2$ at pH 7.5, potential –1.6 V.

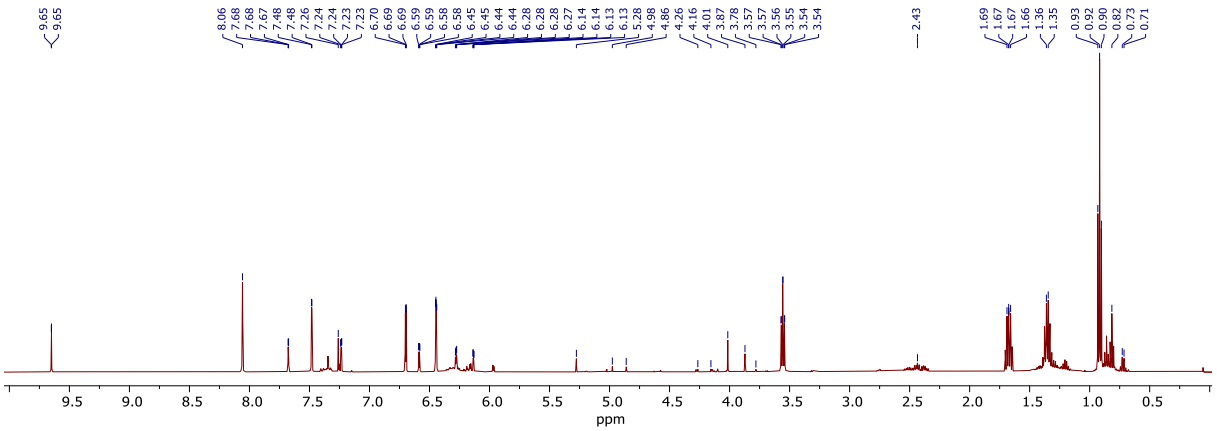

Fig S8. ^{1}H NMR spectrum of the isolated reaction mixture from the electroreductive amination of FF with *n*-BuNH$_2$ at pH 7.5, potential –1.5 V.

1.2 pH dependence (at potential –1.7 V)

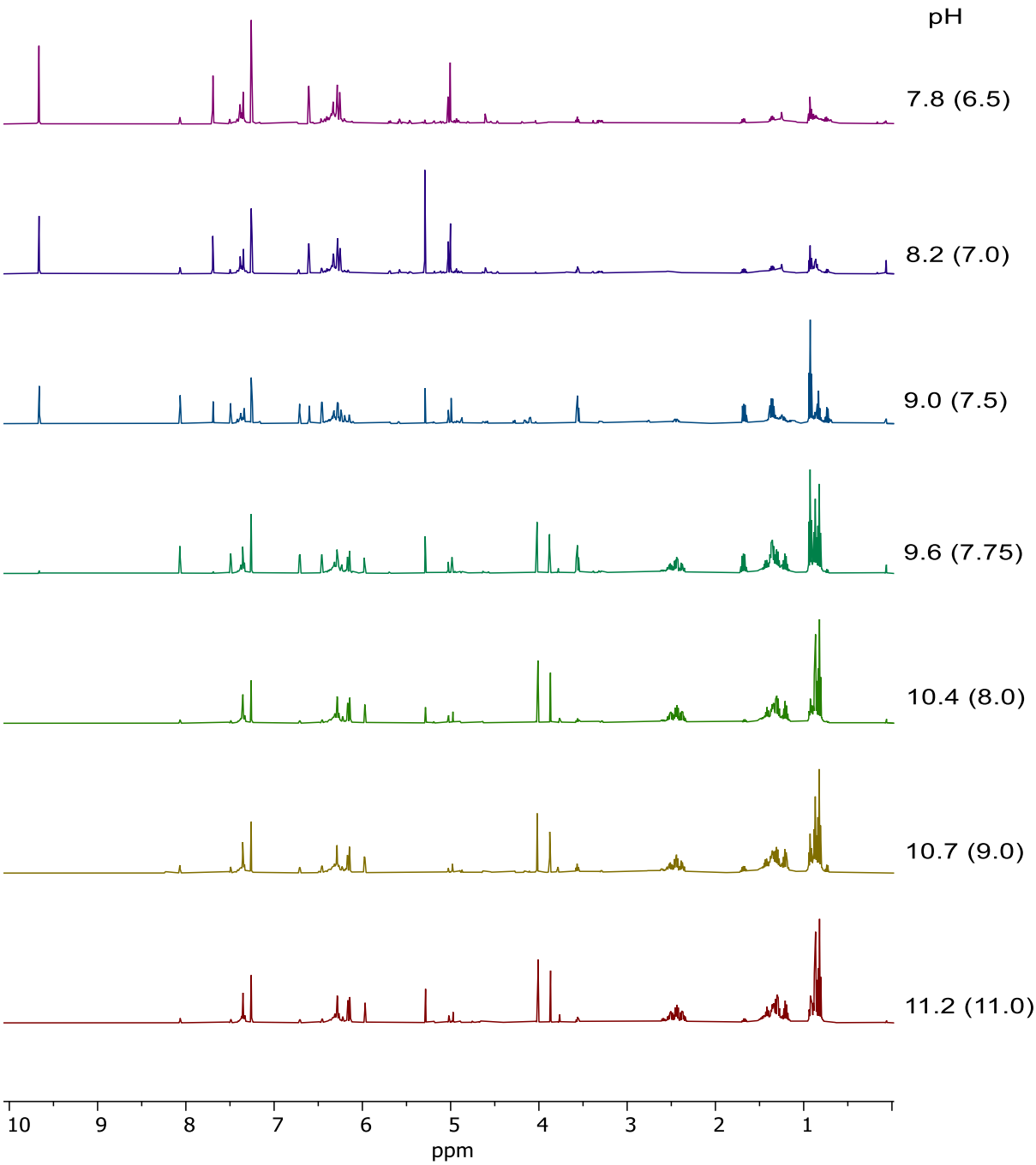

Fig S9. ^{1}H NMR spectra of the isolated reaction mixtures from the electroreductive amination of FF with *n*-BuNH$_2$ at various pH (measured pH and pH of the initial phosphate electrolyte in parenthesis).

1.3 Electroreduction using various amines (at potential –1.7 V, pH 11)

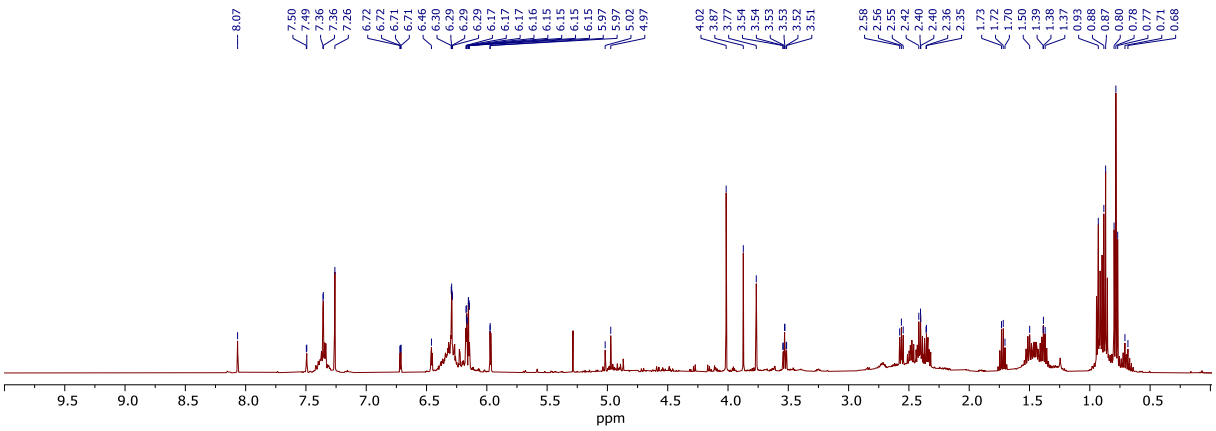

Fig S10. ^{1}H NMR spectrum of the isolated reaction mixture from the electroreductive amination of FF with *n*-PrNH$_2$.

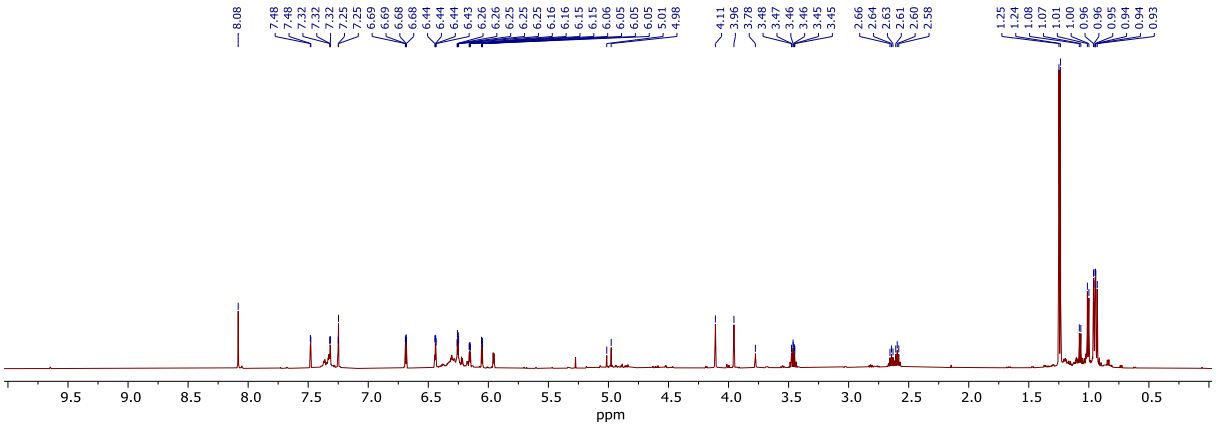

Fig S11. ^{1}H NMR spectrum of the isolated reaction mixture from the electroreductive amination of FF with *i*-PrNH$_2$.

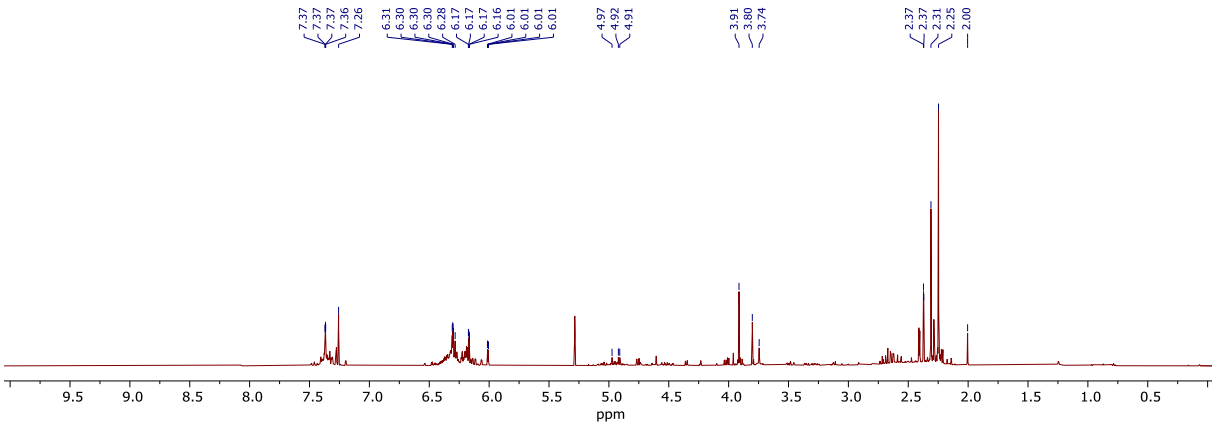

Fig S12. ^{1}H NMR spectrum of the isolated reaction mixture from the electroreductive amination of FF with MeNH$_2$.

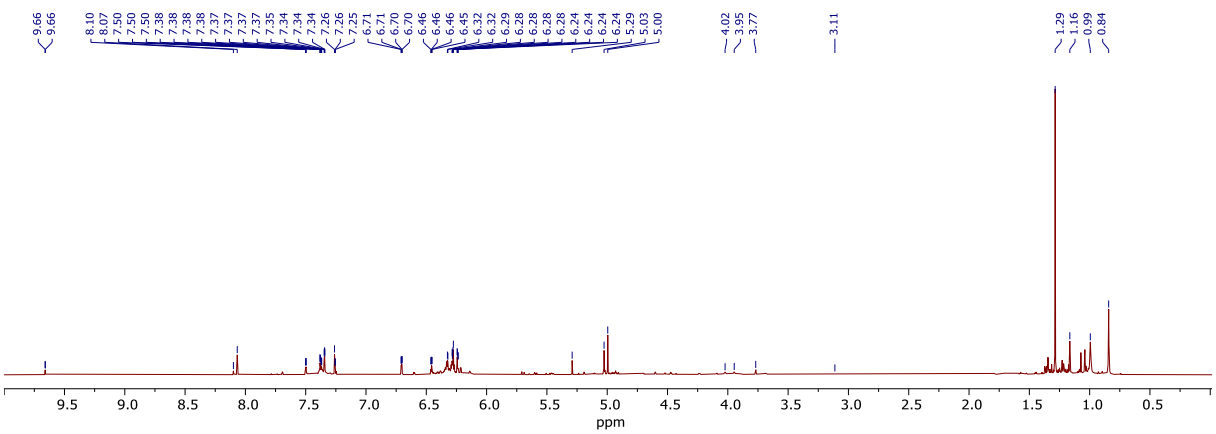

Fig S13. ^{1}H NMR spectrum of the isolated reaction mixture from the electroreductive amination of FF with *t*-BuNH_2.

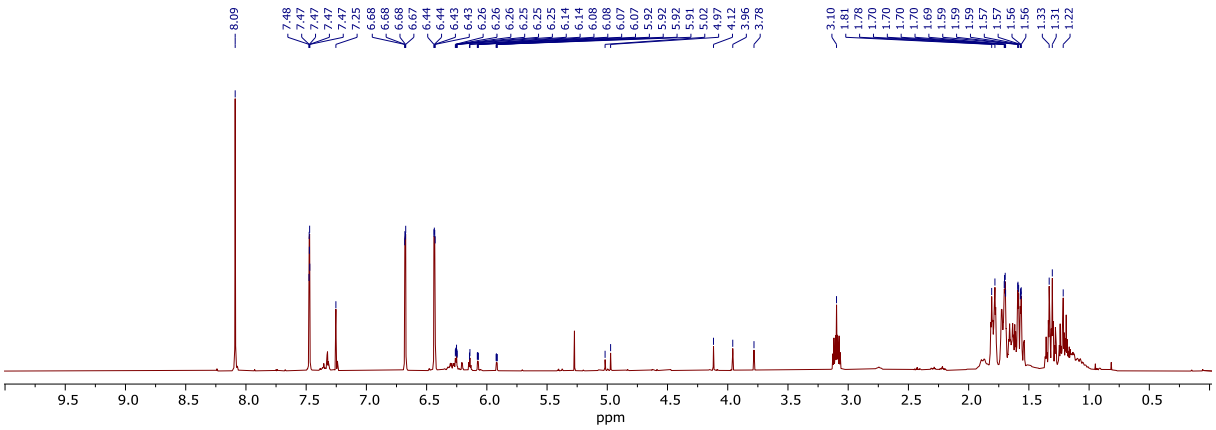

Fig S14. ^{1}H NMR spectrum of the isolated reaction mixture from the electroreductive amination of FF with *cy*-HxNH_2.

2. Isolation of the diamine product 4a

Crude products of electrolysis of FF with *n*BuNH_2 were purified by column chromatography on silicagel using *n*-Hx/AcOEt/NEt_3 5:1:0.1 as an eluent. A red oil was obtained: 34 mg (56 % yield based on FF) as a mixture of *rac*- and *meso*-**4**a in ratio ca. 1.1:1.

Data for **4a**: NMR ($CDCl_3$, 500 MHz) ^{1}H: δ 0.83 (t, ^{3}J = 7.2 Hz, 6H, 2×*Me* of *rac*-), 0.87 (t, ^{3}J = 7.2 Hz, 6H, 2×*Me* of *meso*-), 1.18–1.47 (m, 16H, 4×CH_2 of both isomers), 2.46 (m, 8H, 2×CH_2 of both isomers), 3.90 (s, 1H, C*H*-N of *meso*-), 4.05 (s, 1H, C*H*-N of *rac*-), 6.00 (d, ^{3}J = 3.2 Hz, 1H, C^{2}H of *meso*-), 6.15 (dd, ^{3}J = 3.2 Hz, ^{4}J = 0.8 Hz, 1H, C^{2}H of *rac*-), 6.17 (dd, ^{3}J = 3.2 Hz, ^{3}J = 1.8 Hz, 1H, C^{3}H of *meso*-), 6.29 (dd, ^{3}J = 3.2 Hz, ^{3}J = 1.8 Hz, 1H, C^{3}H of *rac*-), 7.27 (dd, ^{3}J = 1.8 Hz, ^{4}J = 0.8 Hz, 1H, C^{4}H of *meso*-), 7.36 (dd, ^{3}J = 1.8 Hz, ^{4}J = 0.8 Hz, 1H, C^{4}H of *rac*-) ppm; ^{13}C{^{1}H}: δ 14.05, 14.10 (2×*Me*), 20.37, 20.54, 32.10, 32.30 (4× *C*H_2), 47.33, 47.52 (2× *C*H_2-N), 59.94, 60.46 (2× *C*H-N), 107.45, 108.28, 109.86, 110.06 (4× C^{2}, C^{3}), 141.53, 142.02 (2× C^{4}), 154.5 (C^{1}) ppm. Anal. calcd. for $C_{18}H_{28}O_2N_2$ (M_r = 304.43): C 71.02, H 9.27, N 9.20 %. Found: C 70.91, H 9.30, N 9.15 %.

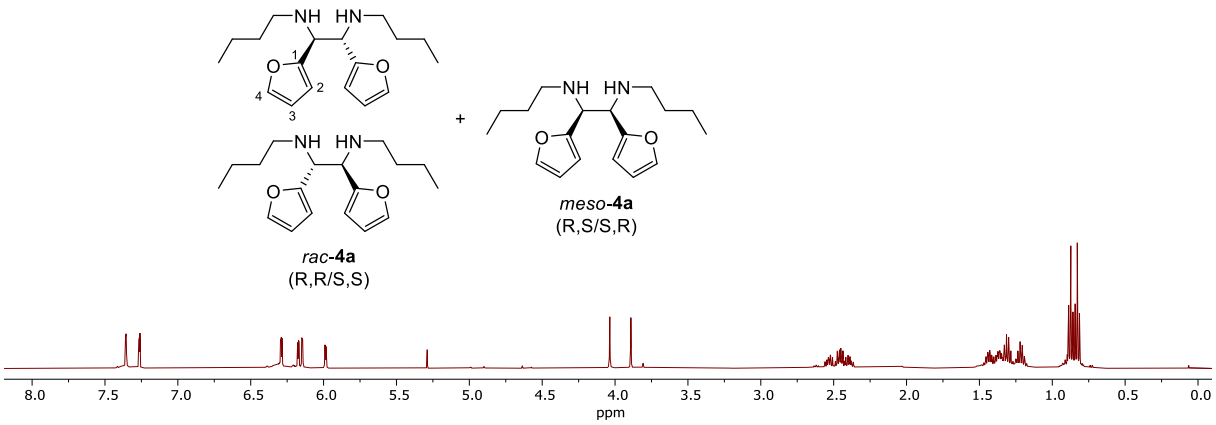

Fig S15. ^{1}H NMR in $CDCl_3$ of **4a** purified by column chromatography on SiO_2 (*n*-Hx/AcOEt/NEt_3). Assignment of the isomers.