Electronic Supporting Information

Alternating polarity for enhanced electrochemical synthesis

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Table of Contents

1 Non-constant Current or Potential Profiles

In addition to constant current (also called DC, direct current), three different kinds of non-constant currents are usually discussed: a) alternating current (AC), which refers to a sinusoidal change of the current over time, b) pulsed current, where the current is switched on and off, and c) reverse pulse current, where alternating the direction of the current results in a square wave change (Figure S1). The period of the current is thereby the time of a whole cycle, i.e. when the current is back to the starting point (indicated by the red arrows in the figures). The frequency is the inverse of this period in Hz.

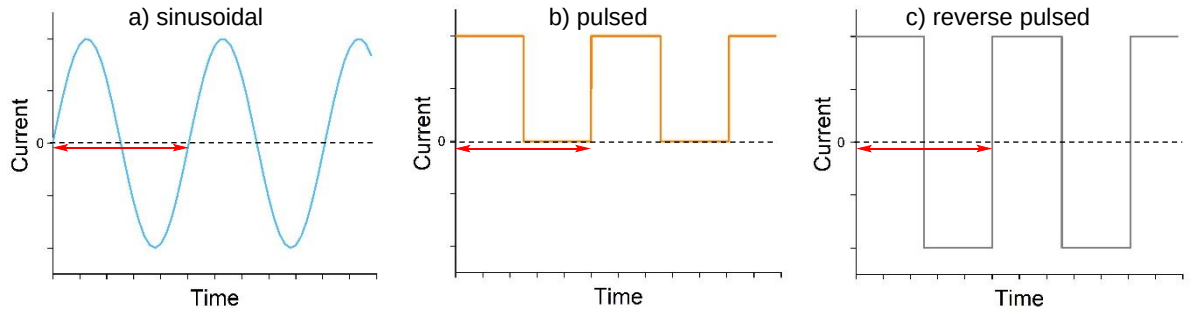


Figure S1. Descriptions of the different types of non-constant current, with the period shown with red arrow: a) sinusoidal curve, b) pulsed, c) reverse pulse

In contrast, when talking about the potential, alternating polarity usually refers to a square wave in potential, similar to the reverse pulse current (Figure S2). The use of alternating polarity means that the potential is constant within intervals maintaining the selectivity of the system in the context of a synthetic reaction. Alternating polarity is used in this research.

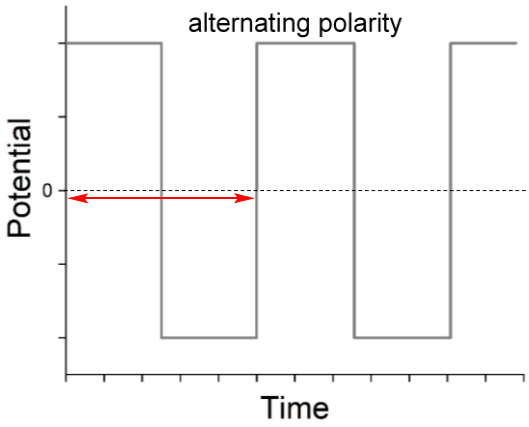
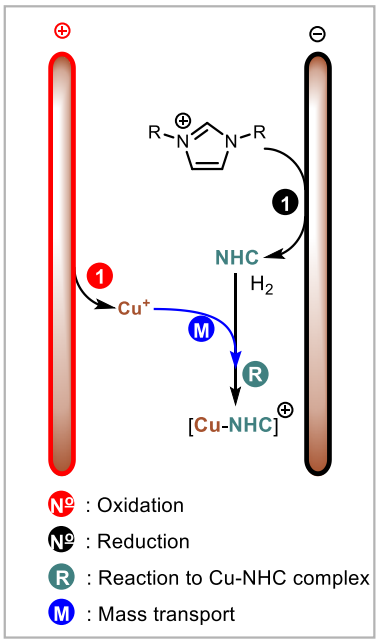


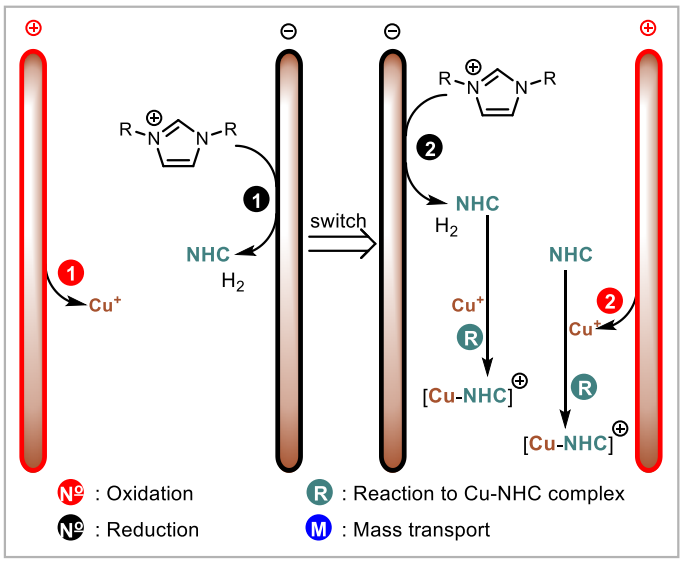
Figure S2. Alternating Polarity with the period shown with red arrow

2 Considerations Frequency Effects

Normally, NHC and the copper(I) are produced on opposite sides (red and black 1) in the reactor channel on each electrode and then have to move towards each other (blue M) to form the complex (turquois R) (Scheme S1). When alternating polarity is employed, the components are still formed on the two separate electrodes, but then another equivalent of each component is formed, but on opposite electrodes, after the polarity switch (red and black 2), avoiding the need for mass transport from one to the other electrode (Scheme S2). This is particularly important as under the continuous flow conditions employed a laminar flow regime is likely which restricts substrate movement by convection.[1] If mass transfer is the rate limiting step in the reaction, the use of alternating polarity would result in improved observed reaction rates and shorter reaction times.

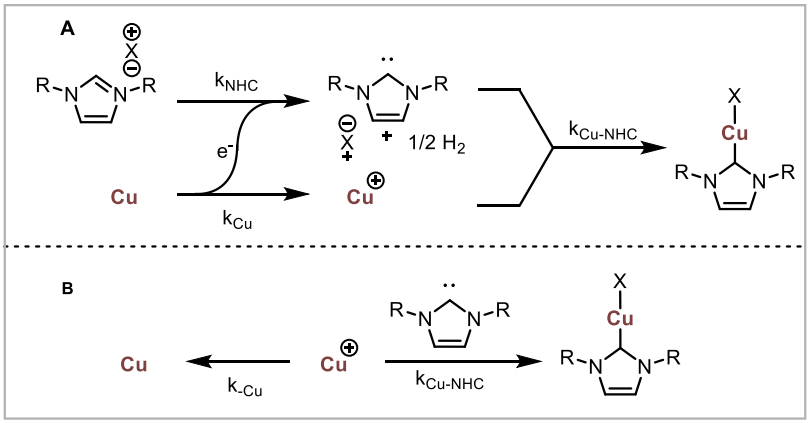


Scheme S1: Reaction at constant potential dependent on mass transfer

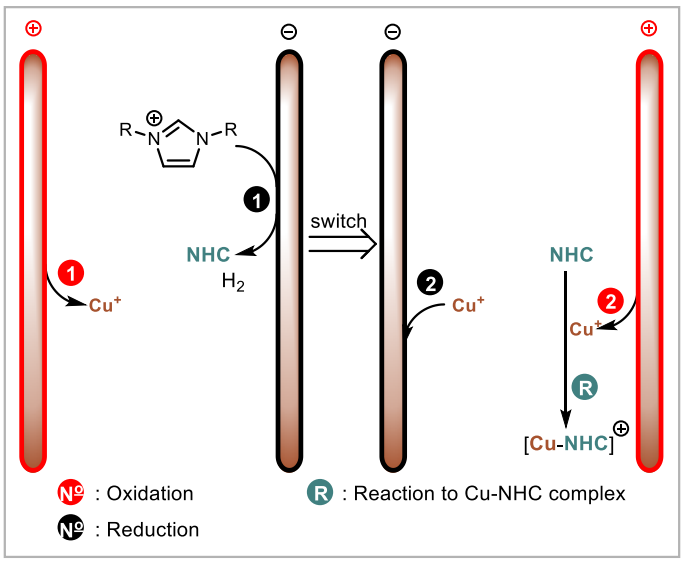


Scheme S2. Visual explanation for possible change in mass transfer rate in the system under alternating polarity

At high frequencies, the reactants might not have the time to react with another reactant, instead as they are still near the electrodes, the reverse electrochemical process can happen (Scheme S3). In the studied example the reverse reaction happening will be most likely the copper(I) being reduced back to copper(0) and being redeposited on the electrode surface (k_{-Cu}) (Scheme S4, black 2). Even though the oxidation of hydrogen and subsequent reaction of the free NHC to the imidazolium salt is possible, similar reactions usually require high system pressures.[2] If the frequency is slow, the complex formation has time to occur ($k_{Cu\text{-}NHC}$). At fast frequencies, the reverse reaction (k_{-Cu}) can occur if the copper(I) is not removed from the surface area quickly enough, in this case by forming the Cu-NHC complex.



Scheme S3. A: Complete reaction scheme, B: possible reactions for Cu(I)



Scheme S4. Visual explanation for the reverse reaction of Cu(I) ions in solution

3 General Experimental Information

Unless otherwise noted, all reagents were commercially available and used without purification. Anhydrous acetonitrile was acquired from a Dow-Grubbs solvent purification system, where it was passed over activated alumina to remove water, a copper catalyst to remove oxygen, and molecular sieves to remove any remaining water. It was then degassed by purging with argon.

NMR data were obtained on either a Bruker A500 (CH dual cryoprobe) or Bruker Ascend 400 spectrometer. ^{1}H NMR and ^{13}C{1H} chemical shifts are reported in ppm and are referenced against residual solvent peaks. Spin-spin coupling constants *J* are given in Hz and refer to apparent multiplicities rather than true coupling constants. Data is reported as: chemical shift, multiplicity and integration. Carbon shifts are reported to the nearest 0.1 ppm. Carbons in an identical environment giving one signal are not indicated further.

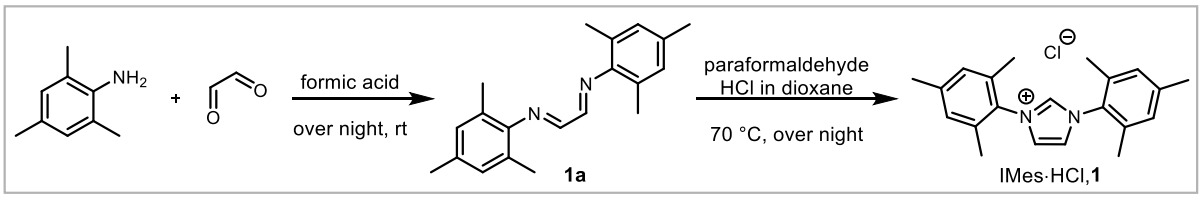
References to spectroscopic data are given for known compounds.

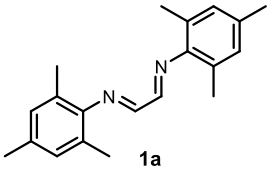
The flow setup consists of PFA tubing of an 0.8 mm ID, the electrochemical reactor (see section on electrochemical reactor), a Tenma power supply unit (72-10480) and a pump.

Reactions were either monitored by ^{1}H NMR or HPLC (Agilent Infinity 1260 with InfinityLab Poroshell 120 EC-C18 column).

4 Synthesis of Starting Materials

1,3-Dimesityl-1H-imidazolium chloride, 1 (IMes·HCl)[3]

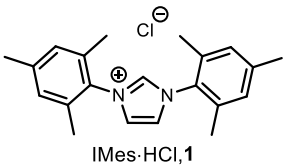




To a solution of 2,4,6-trimethylaniline (23.2 mL, 164 mmol, 2 equiv) in EtOH (150 mL) was added glyoxal (40% in water, 9.6 mL, 84 mmol, 1.02 equiv) and 20 drops of formic acid. After a few minutes a yellow precipitate formed, and the reaction solution was left to stir overnight. The solid was filtered off and washed with EtOH and MeOH to yield the product, *N*,*N*-dimesitylethane-1,2-diimine **1a**, as a bright yellow solid (78%, 18.95 g, 64 mmol). The diimine **1a** was used without further purification.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.10 (s, 2H), 6.91 (s, 4H), 2.30 (s, 6H), 2.16 (s, 12H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 163.6, 147.6, 134.4, 129.1, 126.7, 20.9, 18.4.

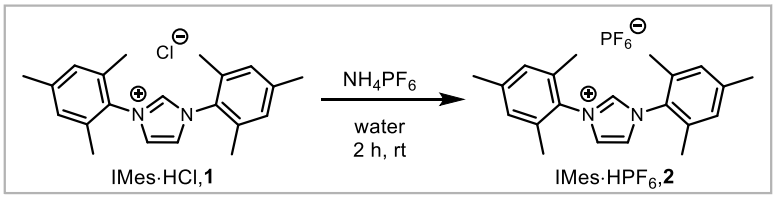


A solution of *N*,*N*-dimesitylethane-1,2-diimine **1a** (11.0 g, 37.5 mmol) and paraformaldehyde (1.2 g, 41 mmol, 1.1 equiv) in anhydrous toluene (300 mL) was prepared under N_2 and then heated up to 100 °C till mostly dissolved (about 30 min). After cooling to 40 °C HCl in dioxane (4 M, 10.3 mL, 41 mmol, 1.1 equiv) was added slowly turning the reaction mixture red. The reaction solution was then stirred at 70 °C overnight forming a precipitate in a dark brown solution. The precipitate was filtered off, washed with EtOAc until the filtrate ran clear yielding the crude product as a pale brown solid. The solid was then triturated from MeCN and Et_2O, filtered and washed with Et_2O to give the product, 1,3-dimesityl-1H-imidazolium chloride **1**, as an off-white solid (37%, 4.67 g, 13.7 mmol).

^{1}H NMR (501 MHz, $CDCl_3$) δ 11.07 (s, 1H), 7.56 (s, 2H), 7.04 (s, 4H), 2.35 (s, 6H), 2.20 (s, 12H).

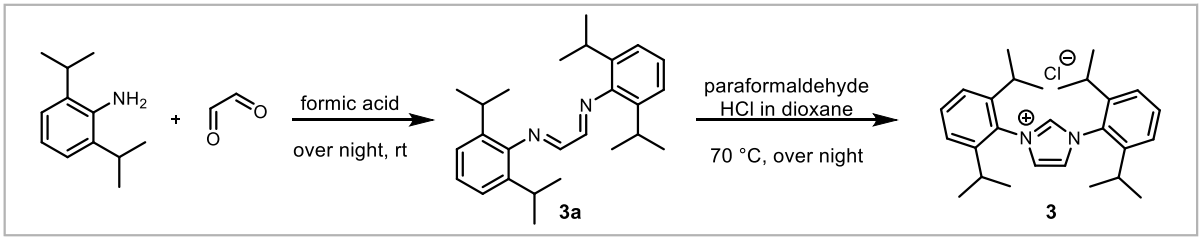
^{13}C NMR (126 MHz, $CDCl_3$) δ 141.6, 140.4, 134.2, 130.8, 130.1, 124.3, 21.3, 17.9.

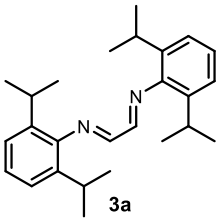
1,3-Dimesityl-1H-imidazolium hexafluorophosphate, 2 (IMes·HPF_6)



The product was prepared following a known literature procedure.[4]

1,3-Bis(2,6-diisopropylphenyl)-1H-imidazolium chloride, 3[3]

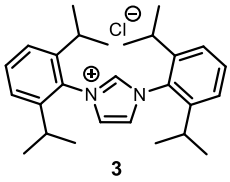




To a solution of 2,6-diisopropylaniline (10.6 mL, 10 g, 56 mmol, 2.1 equiv) in MeOH (50 mL) was added glyoxal (40% in water, 3.2 mL, 28 mmol) and 2 drops of formic acid. The reaction solution was left to stir overnight forming a yellow precipitate. The precipitate was filtered and washed with MeOH yielding the product (8.2 g, 22 mmol, 78%). The product was used without further purification (*N*,*N*-bis(2,6-diisopropylphenyl)ethane-1,2-diimine, **3a**).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.11 (s, 2H), 7.22 – 7.15 (m, 6H), 2.95 (hept, *J* = 6.9 Hz, 4H), 1.21 (d, *J* = 6.9 Hz, 24H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 163.3, 148.2, 136.9, 125.3, 123.3, 28.2, 23.5.

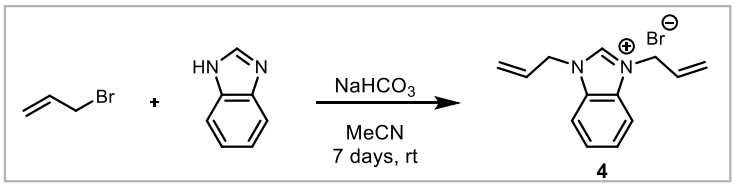


A solution of the diimine **3a** (5 g, 13 mmol) and paraformaldehyde (0.40 g, 13 mmol) in toluene (100 mL) was heated up to 100 °C till mostly dissolved (about 30 min). After cooling to 40 °C HCl in dioxane (4 M, 3.3 mL, 13 mmol) was added slowly turning the reaction mixture red. The reaction solution was then stirred at 70 °C overnight forming a precipitate. The precipitate was washed with EtOAc until the filtrate ran clear. The solid was then triturated from MeOH and Et_2O, filtered and washed with Et_2O to give the product, 1,3-bis(2,6-diisopropylphenyl)-1H-imidazol-3-ium **3**, as a beige solid (32%, 1.78 g, 4.2 mmol).

^{1}H NMR (400 MHz, $CDCl_3$) δ 10.03 (s, 1H), 8.15 (s, 1H), 7.57 (t, *J* = 7.8 Hz, 1H), 7.34 (d, *J* = 7.8 Hz, 1H), 2.44 (hept, *J* = 6.8 Hz, 1H), 1.28 (d, *J* = 6.7 Hz, 1H), 1.24 (d, *J* = 6.8 Hz, 1H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 145.2, 138.7, 132.3, 130.0, 126.9, 124.9, 29.3, 24.9, 24.0.

1,3-Diallyl-1H-benzimidazolium bromide, 4



The product was prepared according to a known literature procedure.[5]

5 Setup

5.1 Electrochemical Reactor

The reactor consists of stainless steel plates at the top and bottom , PTFE spacers and copper electrodes similar to previous reactors developed in the group (Figure S3, Table S1).[4] The reactor has a 4.8 cm diameter. The stainless-steel plates enhance structural stability and are bolted together. The electrodes and PTFE spacers are stacked in the middle. The stainless-steel plates exhibit inlet and outlet with ¼-28 threads for standard fittings (1). All elements, the stainless-steel plates, the spacers and the electrodes have three big holes for the M5 bolts (2), and two small ones (3) to align all elements while stacking. Two of the holes for the bolts are slotted on the spacers and electrodes (2 in Figure S3D). For aligning the elements drill bits (3.8 mm) are inserted into the bottom stainless steel plate, then the reactor is assembled and bolted together, and then the bits removed. PTFE spacers are 1 mm thick and have cut in channels to direct the liquid flow (4). The electrodes have a lip to connect to the power supply (5) and a hole for the reaction mixture to flow through to the next PTFE spacer (6). Depending on the number of spacers and electrodes the volume of the reactor can be changed, each spacer exhibits a flow channel with a 0.464 mL volume. If not otherwise mentioned, in this work two spacers and three copper electrodes have been used resulting in a 0.928 mL volume. The reactor can be attached to a power supply through lips on the electrodes.

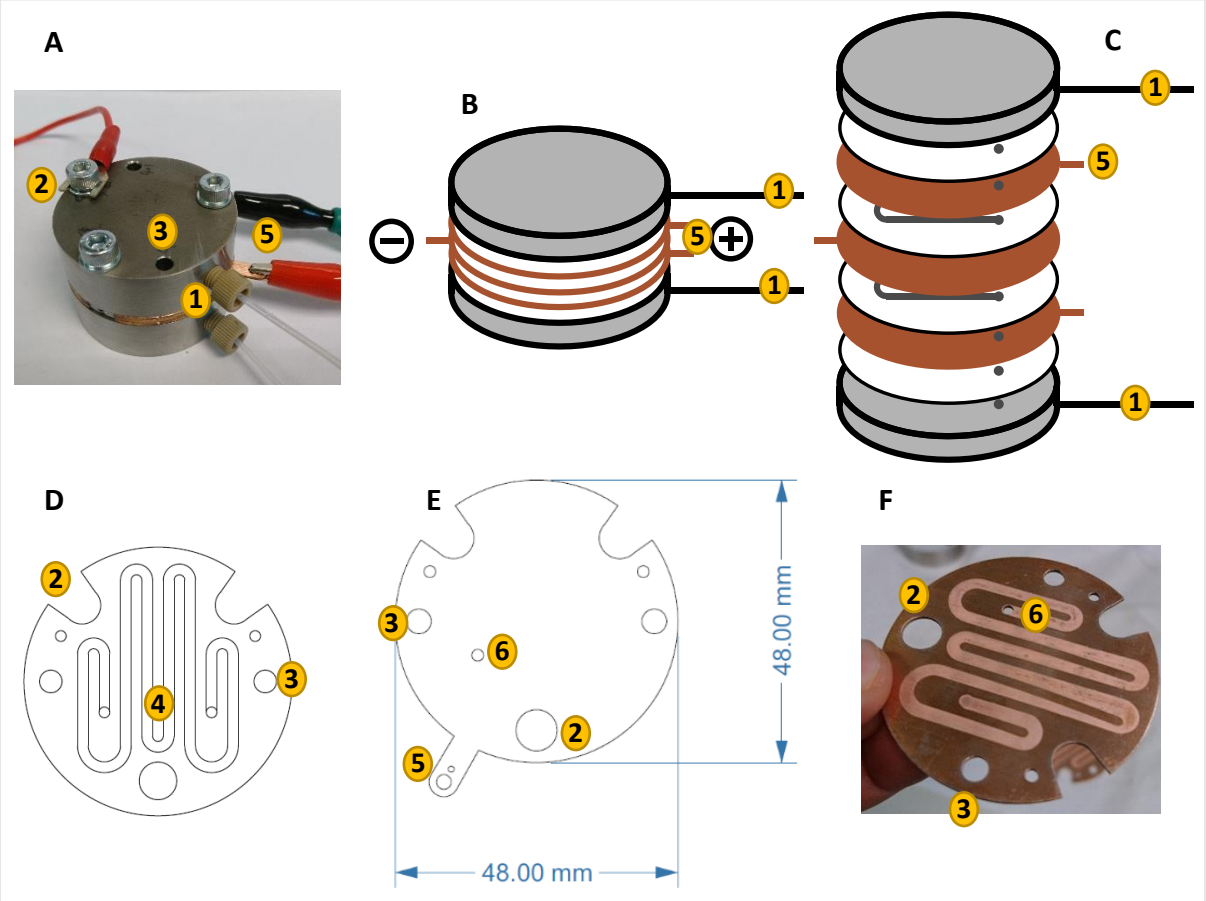


Figure S3: Continuous electrochemical reactor, A: picture of the assembled reactor, B: drawing of the assembled reactor, C: drawing of the elements of the reactor, D: spacer drawing, E: electrode drawing, F: picture of the electrode, 1: inlet and outlet, 2: holes for bolts, 3: holes for alignment, 4: flow channel, 5: connection lip, 6: hole for flow through

Table S1: Parts of the electrochemical reactor

Part	Supplier	Part number
Stainless steel plates	Machined internally	Stainless Steel 304
Copper plates	Laser cut	C160 grade, 0.9 mm thickness
PTFE spacers	Laser cut	Virgin grade PTFE, 1 mm thickness
Screws (M5)	RS	4838281
Nuts (M5)	RS	525931
Drill bits (3.8 mm)	RS	7683012
Power supply	Tenma	72-10480

5.2 Alternating Polarity Microcontroller (APM)

The alternating polarity controller (APM, Figure S4) consists of an Arduino MKR ZERO (1697584) with an Arduino MKR Relay Proto Shield (1697588) with a micro USB power supply (131-4662) all purchased from RS. The relay clicks between different positions to afford the alternating polarity (Figure S5). Coding supplied with the data.

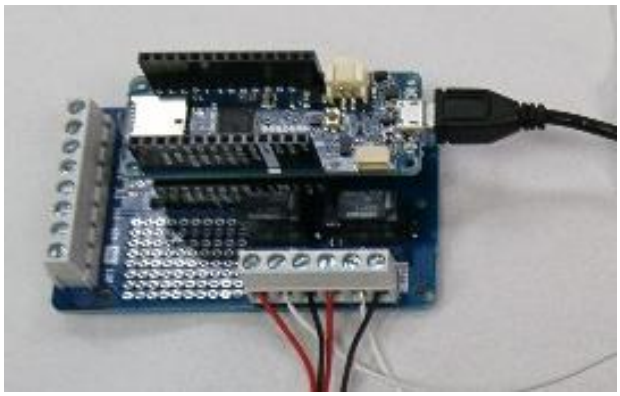


Figure S4: Alternating polarity controller (APM)

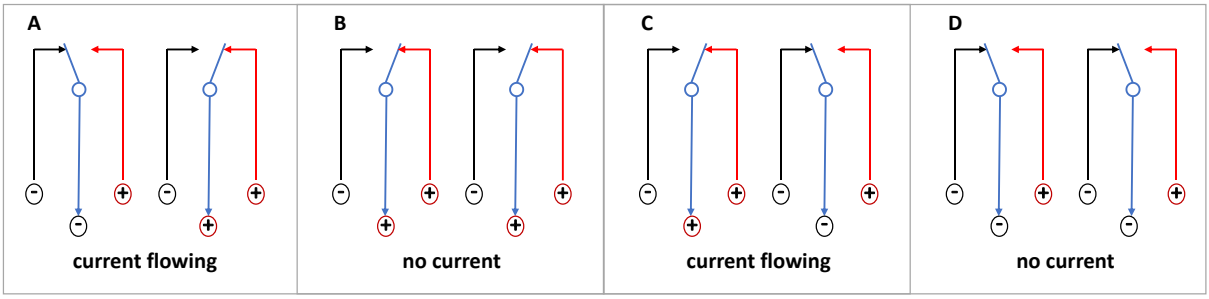
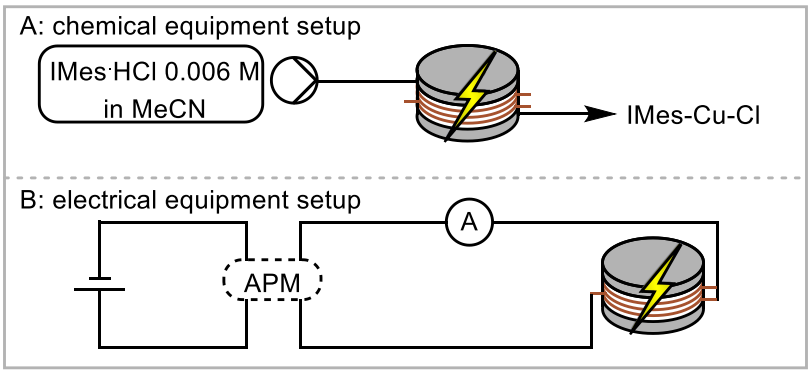


Figure S5: Relay principle, red and black from power supply, blue to reactor (white wire in Figure S4)

5.3 Experiment Setup

The setup was assembled according to Scheme S4 (see picture in Figure S7). The inlet of the electrochemical reactor was connected to a syringe pump via a short piece of tubing (Scheme S4 A). The outlet had a short piece of tubing going into a collection vessel or for online analysis. The power supply was connected to the APM, which was then connected to the reactor and optionally a multimeter measuring the current (Figure S4 B).



Scheme S5: A: chemical equipment setup, B: electrical equipment setup

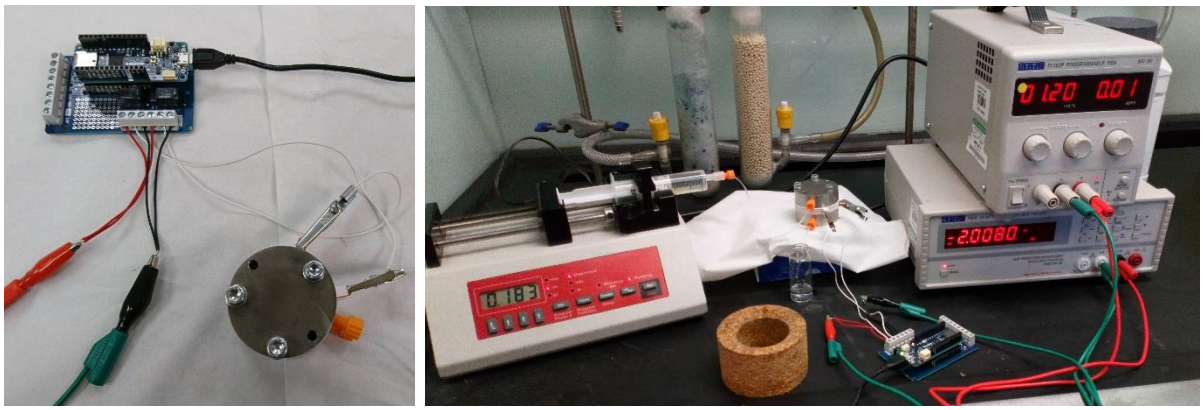


Figure S7: Picture of the APM and reactor (left) and full reaction setup (right)

5.3.1 Analysis

Reactions were typically analysed *via* ^{1}H NMR, measuring conversions, or HPLC, measuring yields. For the calculation of NMR conversions, the peaks of the starting material (IMes·HCl) and product (IMes-Cu-Cl) were compared.

SM: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.01 (s, 2H), 6.90 (s, 4H), 2.43 (s, 6H), 1.68 (s, 12H).

P: ^{1}H NMR (501 MHz, $CDCl_3$) δ 11.07 (s, 1H), 7.56 (s, 2H), 7.04 (s, 4H), 2.35 (s, 6H), 2.20 (s, 12H).

Figure S8 shows a typical NMR obtained. The starting material peak at 7.56 ppm was integrated to 2H. The peaks around 7 ppm were then integrated. Product peaks typically were observed higher in the crude NMR spectra than in the spectra of recrystallised material. The starting material peak at 7.05 ppm and the product peak at 7.01 overlapped (7.05 and 7.02 ppm in Figure S8), the peak corresponding to the product peak at 6.90 ppm was taken to calculate the conversion (7.00 ppm in Figure S8).

In this case the conversion X can be calculated as:

$$X = \frac{\frac{I_P}{H_P}}{\frac{I_P}{H_P} + \frac{I_{SM}}{H_{SM}}} \cdot 100\% = \frac{\frac{1.15}{4}}{\frac{1.15}{4} + \frac{2}{2}} \cdot 100\% = 22\%$$

I_P: integration for product peak

I_{SM}: integration for starting material peak

H_P: number of protons for product peak

H_{SM}: number of protons for starting material peak

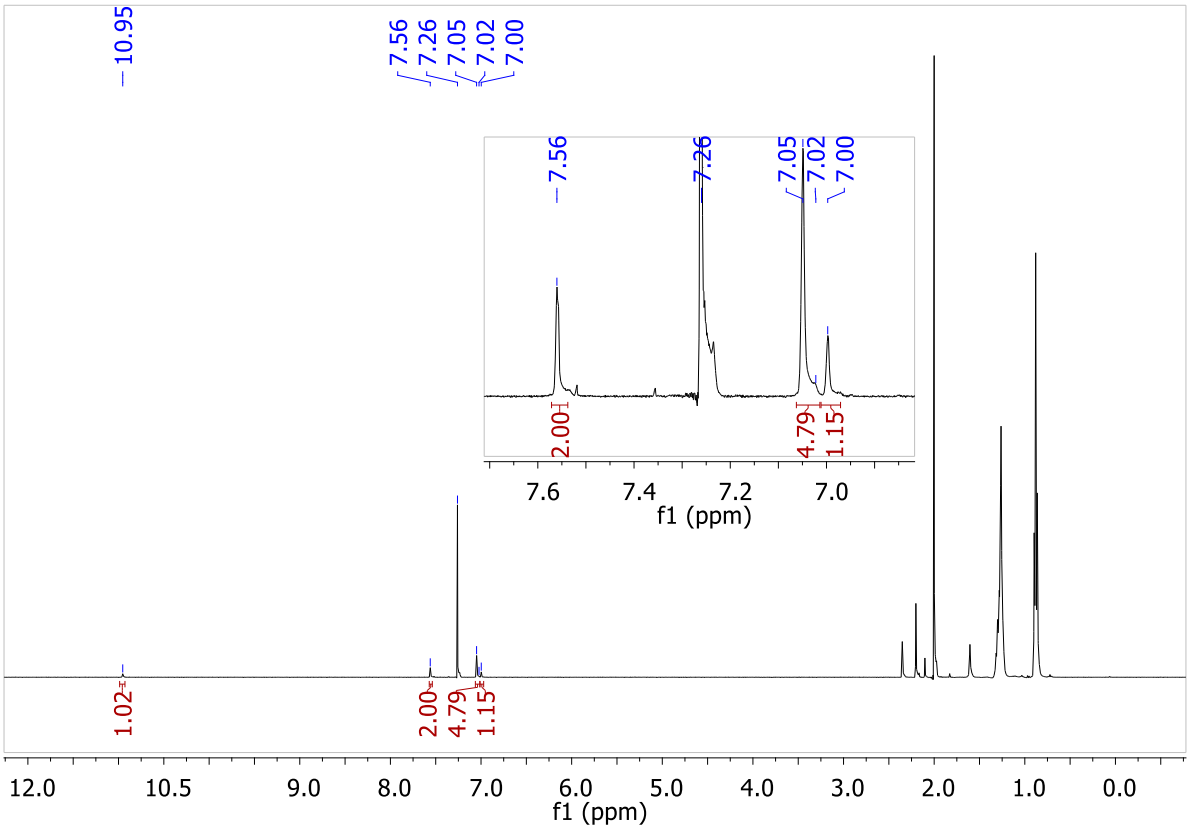
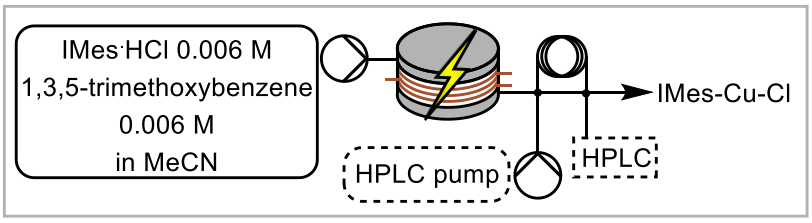


Figure S8: Typical NMR for the electrochemical reaction of IMes·HCl to IMes-Cu-Cl

The setup was also employed using online HPLC measurements. For that the setup was slightly modified, whereby the outlet passed through a 6-port-2-way valve, sampling 0.06 µL of the reaction solution for online HPLC analysis (Scheme S6). An internal standard (1,3,5-trimethoxybenzene 0.006 M) was used to determine yields. The HPLC method had been optimised (Table S2) and the HPLC calibrated for the starting material IMes·HCl and the product IMes-Cu-Cl (Figure S9, Table S3). The valve switch could be programmed to occur at a certain interval using Matlab (2.5 s delay between positions) or switched manually (with a minimum time delay of 9 min due to the length of the HPLC method), initiating the HPLC method upon reversing to the initial position.



Scheme S6: Reaction setup for HPLC monitoring

Table S2: HPLC method

Time [min]	A [%]	B [%]	Flow [mL/min]	
	95	5	0.3	no run, wait position
0	95	5	0.8	
0.2	95	5	0.8	
5	5	95	0.8	Gradient 0.2 min to 5 min
7	5	95	0.8	
7.1	95	5	0.8	Gradient back
8.1	95	5	0.8	

A (water + 0.1% formic acid), B (MeCN + 0.1% formic acid)

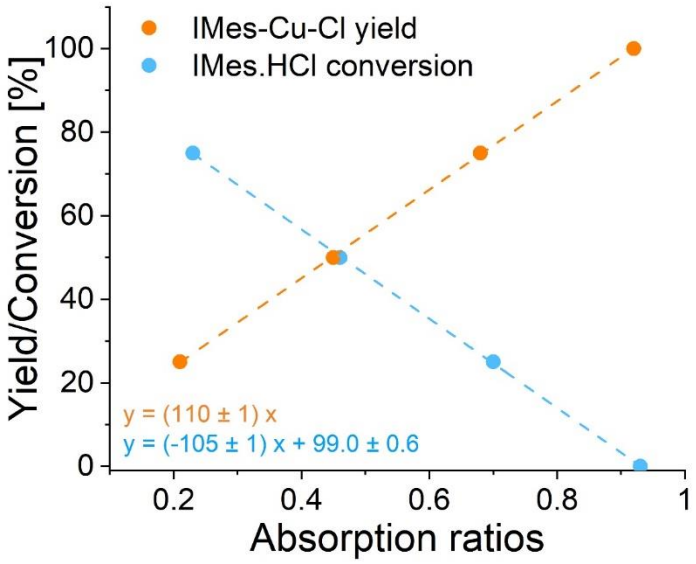


Figure S9: HPLC calibration for starting material conversion and product yield

Table S3: HPLC calibration data for Figure S9

solution IMes.HCl	conversion [%]	ratio	error
A0	0	0.93	0.002
A1	50	0.46	0.001
A2	25	0.70	0.002
A3	75	0.23	0.002

solution Cu(IMes)Cl	yield [%]	ratio	error
B0	100	0.92	0.002
B1	50	0.45	0.001
B2	75	0.68	0.001
B3	25	0.21	0.002

6 Experiments

6.1 Optimisation

A solution of 1,3-dimesityl-1H-imidazolium chloride (IMes·HCl) (0.006 M) in anhydrous MeCN was prepared under argon. The setup was prepared by programming the APM and assembling according to Scheme S5. The reactor was filled with the solution and the pump started at 0.034 mL min^{-1} (27.3 min residence time). The power supply was then switched on at the desired potential to start the reaction. After waiting for steady state (1 h), a sample was collected for 15 min into a round bottom flask under air. The solvent was removed under reduced pressure and the sample taken up in $CDCl_3$ for ^1H NMR analysis. The results are given in Figure S10 and Table S4.

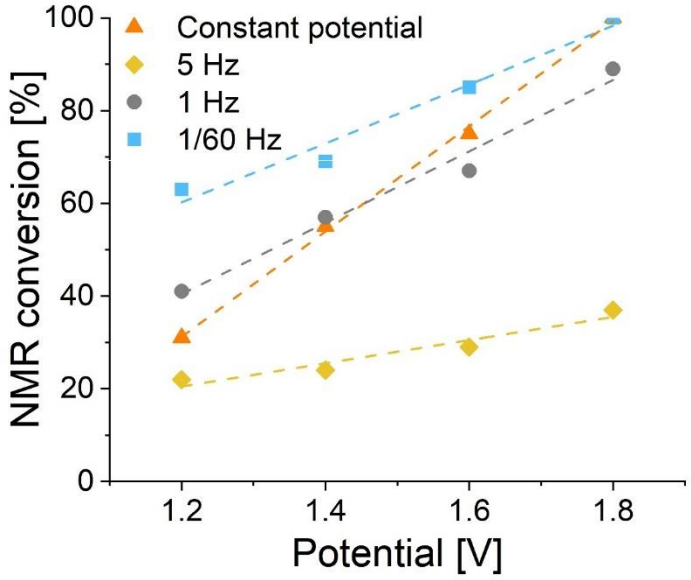


Figure S10: Single sweep voltammetry at constant potential (orange), 5 Hz (yellow), 1 Hz (grey) and 1/60 Hz (grey), ^1H NMR conversions

Table S4: Data for the single sweep voltammetry in Figure S10

entry	frequency [Hz]	potential [V]	conversion [%]
1	-	1.2	31
2	-	1.4	55
3	-	1.6	75
4	-	1.8	100
5	5	1.2	22
6	5	1.4	24
7	5	1.6	29
8	5	1.8	37
9	1	1.2	41
10	1	1.4	57
11	1	1.6	67
12	1	1.8	89
13	1/60	1.2	63
14	1/60	1.4	69
15	1/60	1.6	85
16	1/60	1.8	100

6.2 Long Term Stability

The reactions were prepared as in the optimisation studies with the APM at 1/60 Hz or at a constant potential of 1.8 V.

A solution of 1,3-dimesityl-1H-imidazolium chloride (IMes·HCl) (0.006 M) in anhydrous MeCN was prepared under argon. The setup was prepared by programming the APM and assembling according to Scheme S5. The reactor was filled with the solution and the pump started at 0.034 mL min^{-1} (27.3 min residence time). The power supply was then switched on at the desired potential to start the reaction. The reaction output was collected in aliquots into a round bottom flask under air. The solvent was removed under reduced pressure and the sample taken up in $CDCl_3$ for ^1H NMR analysis. The results are given in Figure S11, with the time points referring to the stop of the collection for that respective aliquot. The reaction was stopped after 6 h.

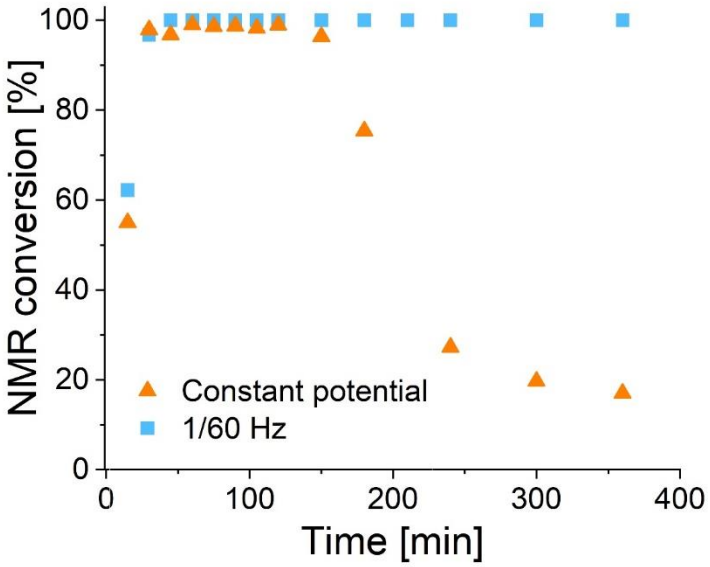


Figure S11: Long term stability with and without APM, ^{1}H NMR conversion measured over 6 h

6.3 Frequency Screen

The reactions were prepared as in the optimisation studies varying the APM frequency at 1.2 V and 1.8 V. For 1.8 V the reactor volume was halved by only using one spacer and two electrodes achieving a volume of 0.464 mL.

A solution of 1,3-dimesityl-1H-imidazolium chloride (IMes·HCl) (0.006 M) in anhydrous MeCN was prepared under argon. The setup was prepared by programming the APM and assembling according to Scheme S5. The reactor was filled with the solution and the pump started at 0.034 mL min^{-1} (residence time: 27.3 min for 1.2 V and 13.7 min for 1.8 V). The power supply was then switched on at the desired potential to start the reaction. The reaction output was collected in aliquots into a round bottom flask under air. The solvent was removed under reduced pressure and the sample taken up in $CDCl_3$ for ^{1}H NMR analysis. The results are given in Figure S12 and Table S5 for 1.2 V and Figure S13 and Table S6 for 1.8 V.

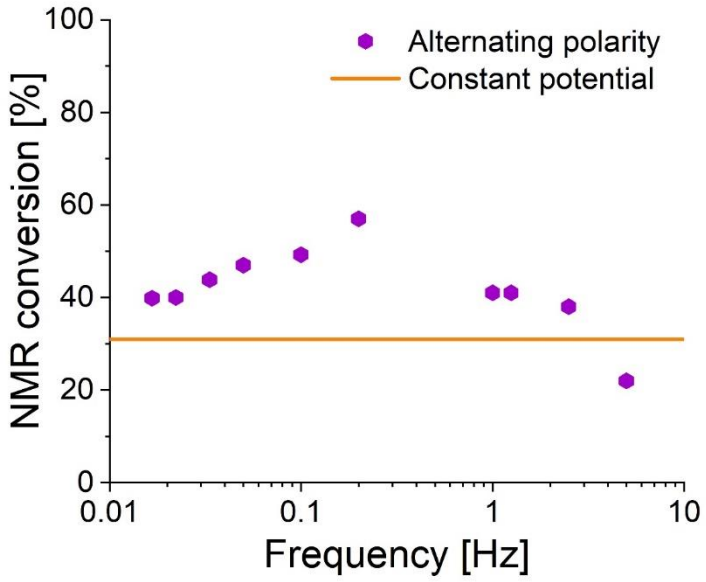


Figure S12: Frequency screen at 1.2 V

Table S5: Frequency screen data at 1.2 V for Figure S12

entry	frequency [Hz]	period [s]	conversion [%]
1	-	-	30
2	5	0.2	22
3	2.5	0.4	38
4	1.25	0.8	41
5	1	1	41
6	0.2	5	57
7	0.1	10	49
8	0.05	20	47
9	0.03	30	44
10	0.02	45	40
11	0.02	60	40
12	0.01	120	33

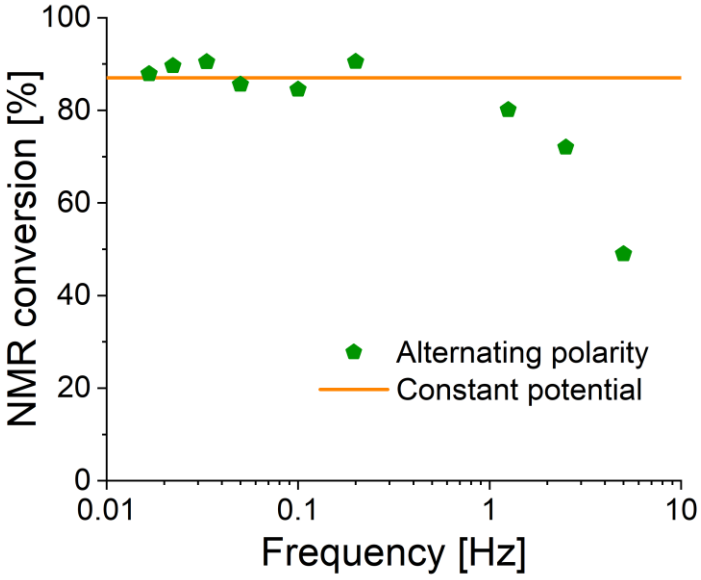


Figure S13: Frequency screen at 1.8 V

Table S6: Frequency screen data at 1.8 V for Figure S13

entry	frequency [Hz]	period [s]	X [%]
1	-	-	87
2	5	0.2	49
3	2.5	0.4	72
4	1.25	0.8	80
5	0.2	5	91
6	0.1	10	85
7	0.05	20	86
8	0.03	30	90
9	0.02	45	90
10	0.02	60	88
11	0.01	120	93

6.4 Kinetics

The reactions were prepared as in the optimisation studies varying the flowrate from fast to slow and feeding the output into the valve for HPLC analysis.

A solution of 1,3-dimesityl-1H-imidazolium chloride (IMes·HCl) (0.006 M) in anhydrous MeCN was prepared under argon. The setup was prepared by programming the APM and assembling according to Scheme S6. The reactor was filled with the solution and the pump started at the desired flow rate. The power supply was then switched on at the desired potential to start the reaction. The reaction output was sampled for HPLC after reaching steady state (2x residence time). The power supply and

pump were switched off to wait for the HPLC measurement and then restarted with the next flow rate. The yield for each experiment was calculated using the HPLC calibration. The experiment at 1/60 Hz was repeated with half the reactor volume and half the flow rate to achieve the same residence times. For each APM frequency the kinetic parameters were fitted and observed reaction rates identified using the kinetic fitting software Compunetics.[6] The model was based on the conversion of starting material to product (see Scheme S7). The results are given in Figure S14 and Table S7.

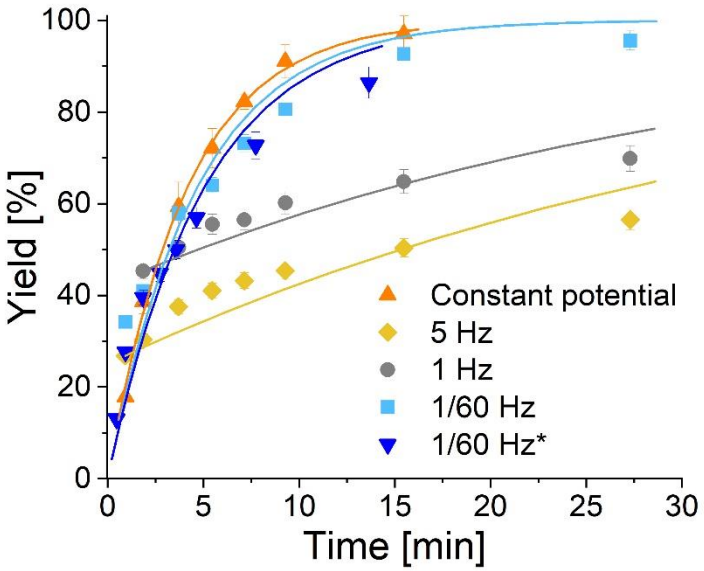
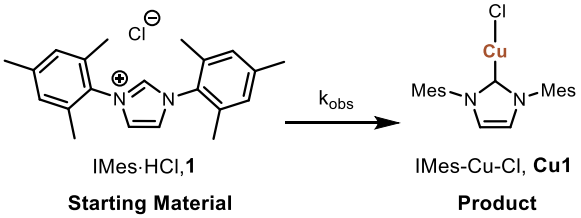


Figure S14: Kinetic experimental data with kinetic fitting, *half reactor volume



Scheme 7: Reaction scheme used for kinetic model

Table S7: Kinetic experimental data and observed reaction rates

entry	frequency [Hz]	flow rate [mL/min]	residence time [min]	yield [%]	k_{obs} [10^{-3} s^{-1}]
1	-	1.000	0.9	18	4.1 ± 0.8
2	-	0.500	1.9	39	
3	-	0.250	3.7	59	
4	-	0.170	5.5	72	
5	-	0.130	7.1	82	
6	-	0.100	9.3	91	
7	-	0.060	15.5	97	

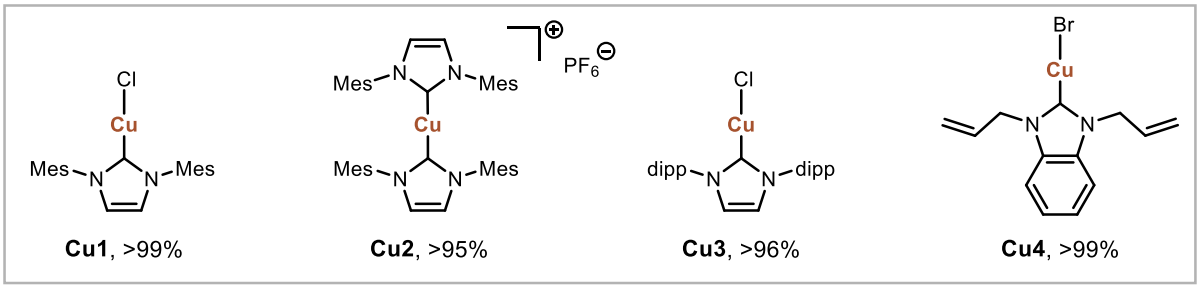
8	-	0.034	27.3		
9	5	1.000	0.9	27	0.44 ±
10	5	0.500	1.9	30	0.7
11	5	0.250	3.7	37	
12	5	0.170	5.5	41	
13	5	0.130	7.1	43	
14	5	0.100	9.3	45	
15	5	0.060	15.5	50	
16	5	0.034	27.3	56	
25	1/60	1.000	0.9	34	3.6 ± 0.3
26	1/60	0.500	1.9	41	
27	1/60	0.250	3.7	58	
28	1/60	0.170	5.5	64	
29	1/60	0.130	7.1	73	
30	1/60	0.100	9.3	81	
31	1/60	0.060	15.5	93	
32	1/60	0.034	27.3	96	
33	1/60*	1.000	0.5	13	3.3 ± 0.3
34	1/60*	0.500	0.9	28	
35	1/60*	0.250	1.9	39	
36	1/60*	0.170	2.7	45	
37	1/60*	0.130	3.6	50	
38	1/60*	0.100	4.6	57	
39	1/60*	0.060	7.7	73	
40	1/60*	0.034	13.6	86	
41	1	0.500	1.9	45	0.52 ±
42	1	0.250	3.7	50	0.1
43	1	0.170	5.5	55	
44	1	0.130	7.1	56	
45	1	0.100	9.3	60	
46	1	0.060	15	65	
47	1	0.034	27.3	70	

*half reactor volume

6.5 Substrate Scope

The reactions were prepared in a similar fashion to the optimisation studies.

A solution of the starting imidazolium salt (0.006 M) in anhydrous MeCN was prepared under argon. The setup was prepared by programming the APM to 1/60 Hz and assembling according to Scheme S5. The reactor was filled with the solution and the pump started at 0.034 mL min^{-1} (27.3 min residence time). The power supply was then switched on at 1.8 V to start the reaction. After waiting for steady state (1 h), a sample was collected for 15 min into a round bottom flask under air. The solvent was removed under reduced pressure and the sample taken up in $CDCl_3$ for ^1H NMR analysis. The conversions were calculated by comparing starting material and product peaks (Scheme S8).



Scheme S8: Substrate Scope, ^{1}H NMR conversions

Cu1 (IMes-Cu-Cl):[4]

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.01 (s, 2H), 6.90 (s, 4H), 2.43 (s, 6H), 1.68 (s, 12H).

Cu2:[4]

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.05 (s, 4H), 7.00 (s, 8H), 2.35 (s, 12H), 2.10 (s, 24H).

Cu3: [4]

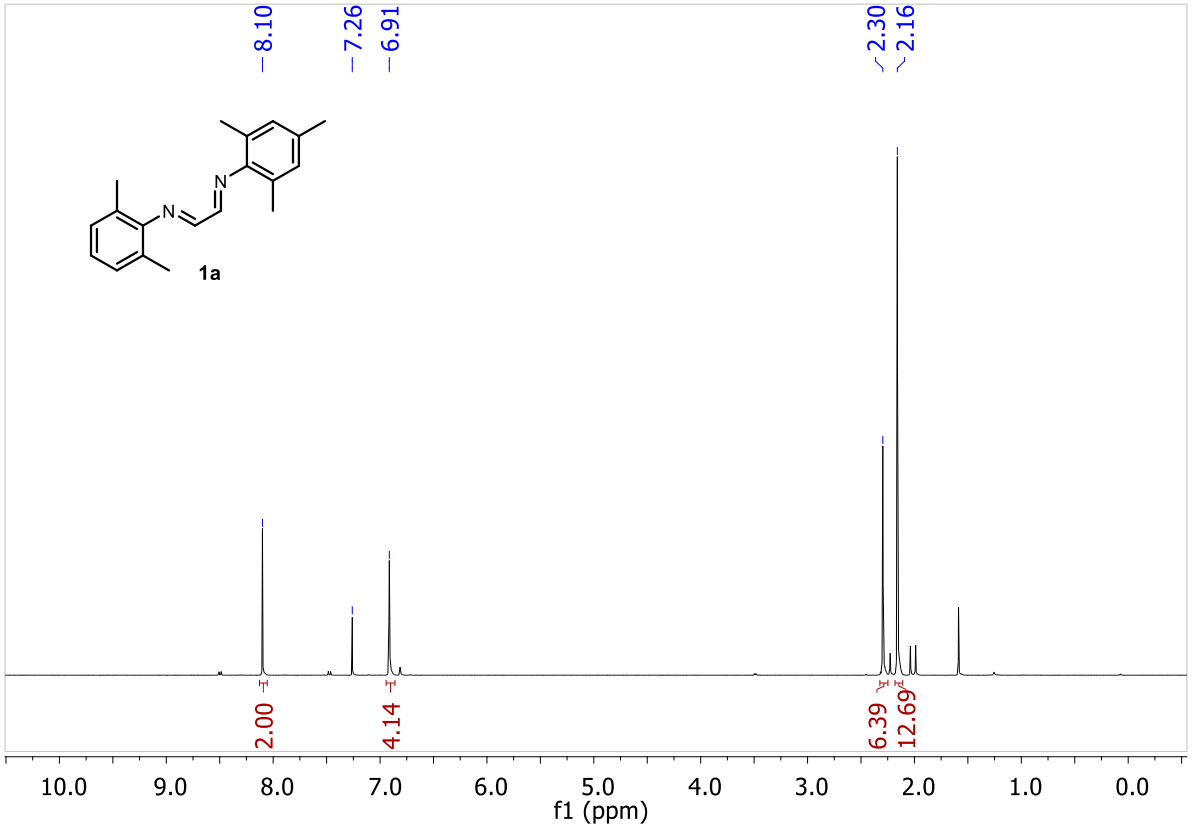
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.49 (t, *J* = 7.8 Hz, 2H), 7.30 (d, *J* = 7.8 Hz, 4H), 7.13 (s, 2H), 2.57 (hept, *J* = 6.8 Hz, 4H), 1.30 (d, *J* = 6.9 Hz, 12H), 1.23 (d, *J* = 6.9 Hz, 12H).

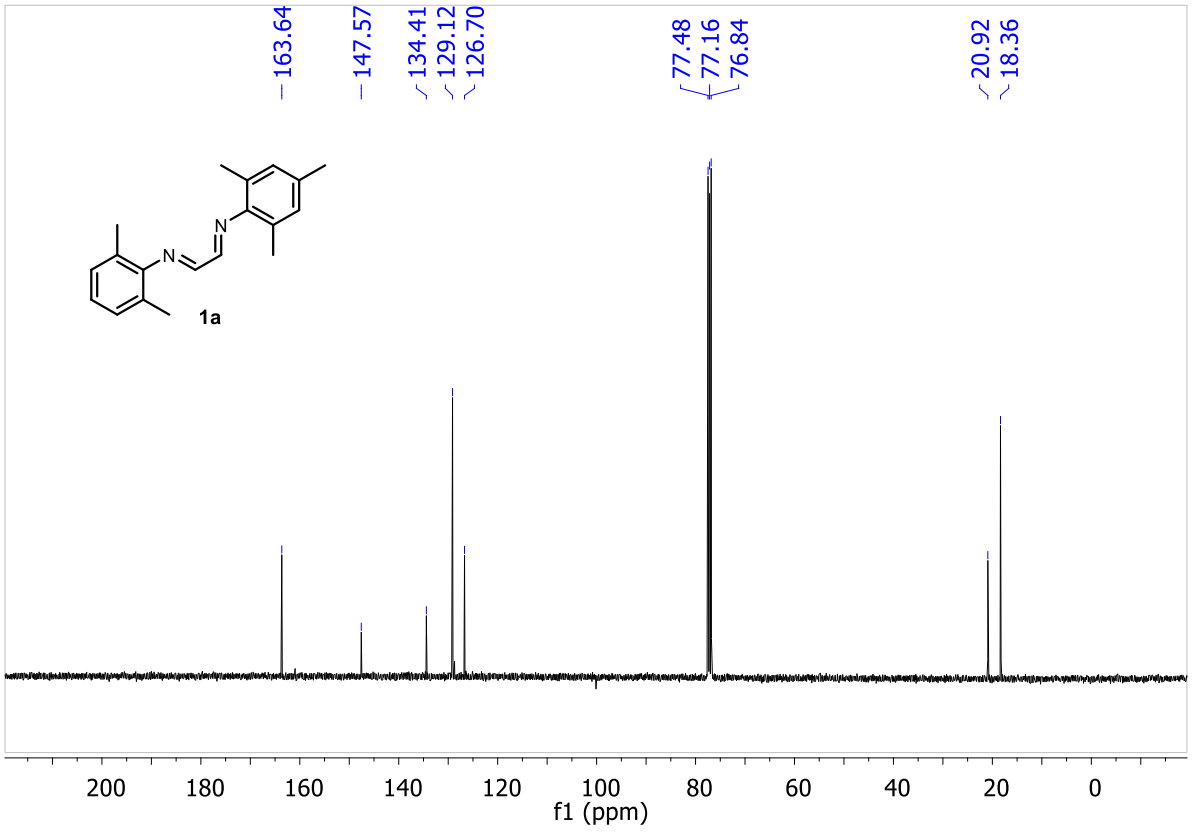
Cu4:[5]

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47 – 7.44 (m, 2H), 7.41 – 7.36 (m, 2H), 6.04 (ddd, *J* = 15.9, 11.0, 5.7 Hz, 2H), 5.39 – 5.23 (m, 4H), 5.09 – 5.01 (m, 4H).

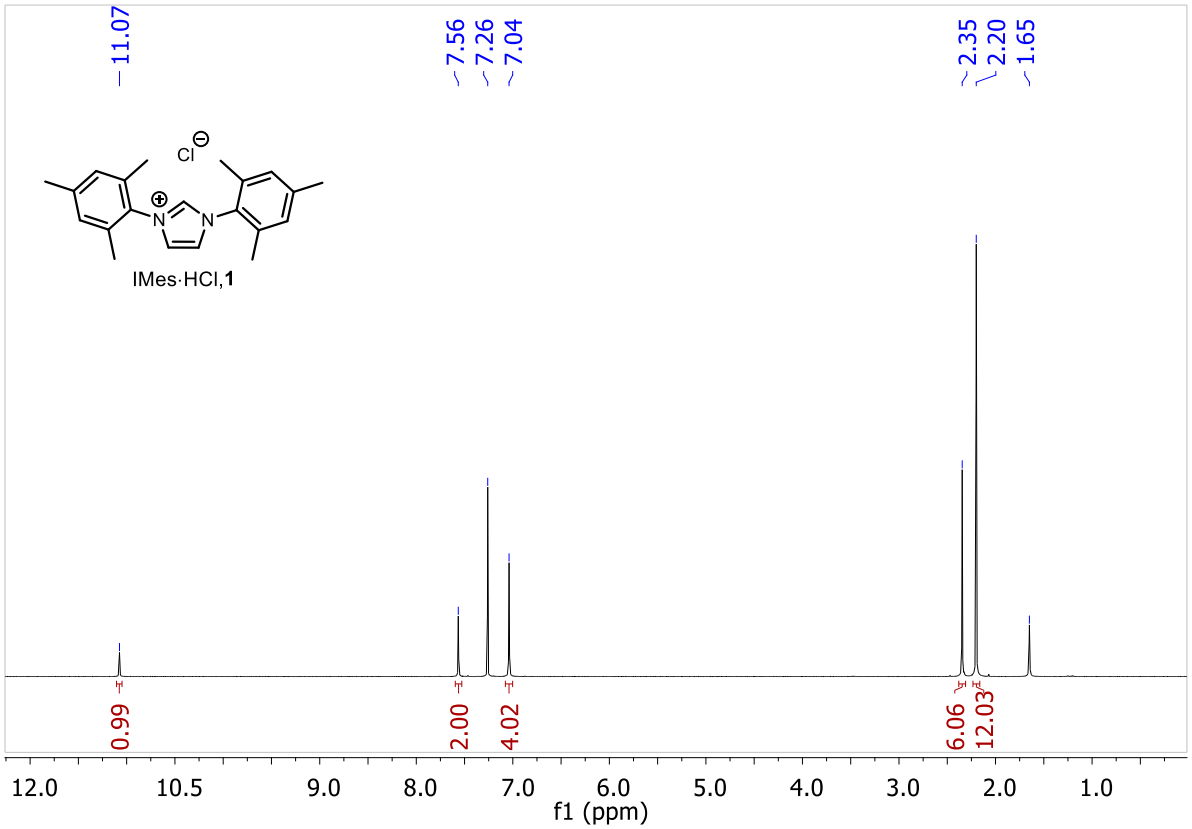
7 NMR data

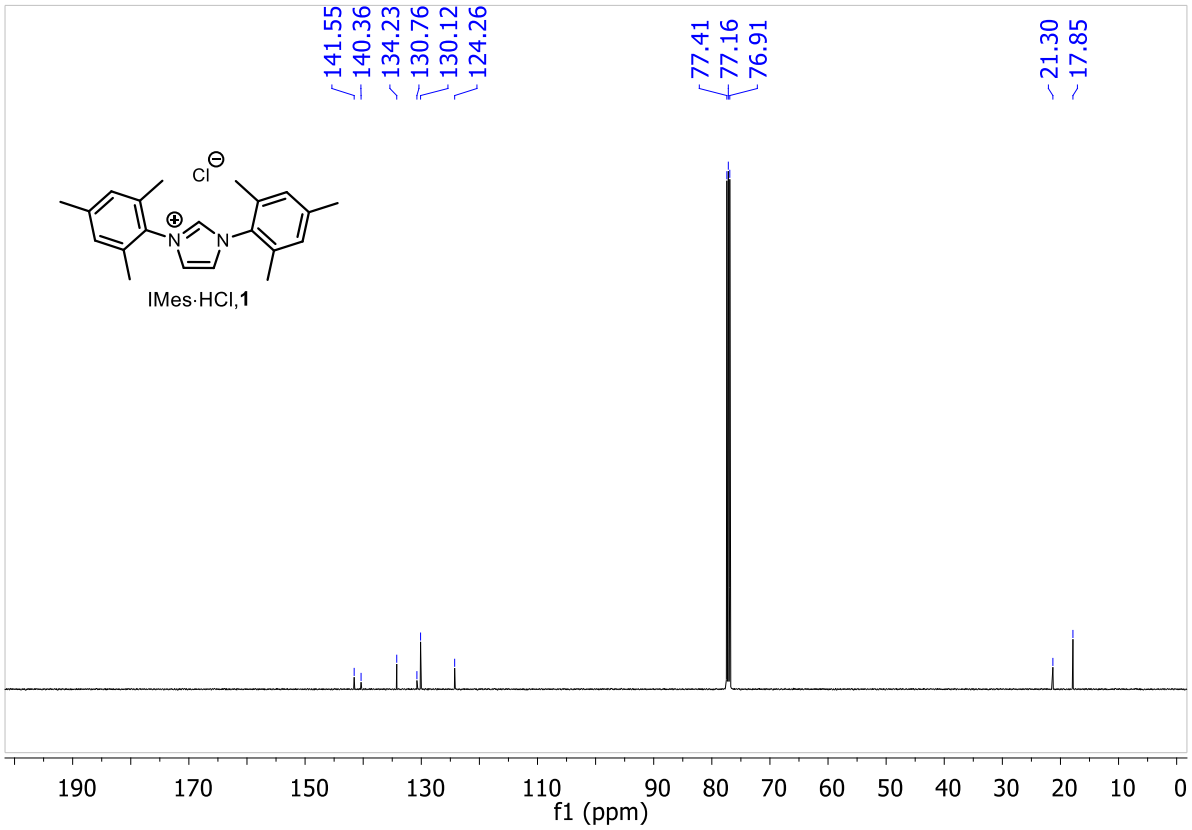
N,*N*-Dimesitylethane-1,2-diimine **1a**



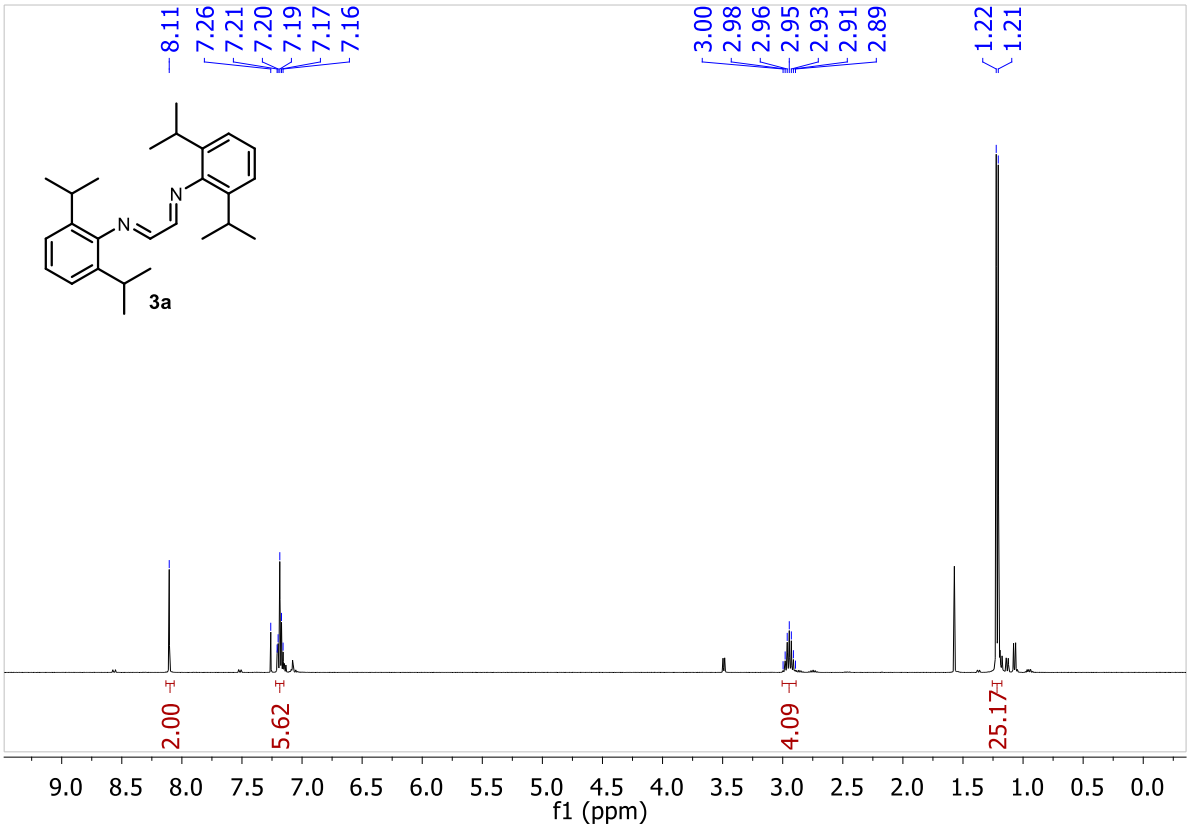


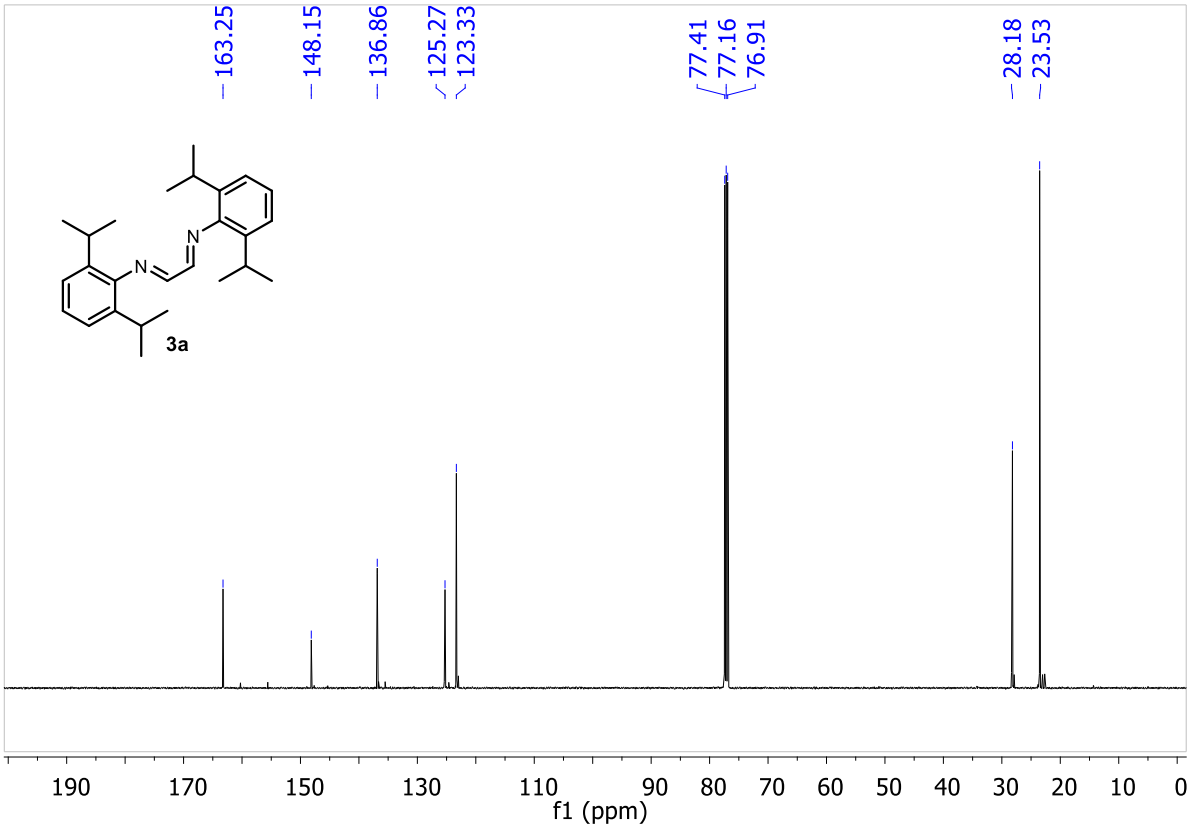
1,3-Dimesityl-1H-imidazolium chloride (IMes·HCl) **1**



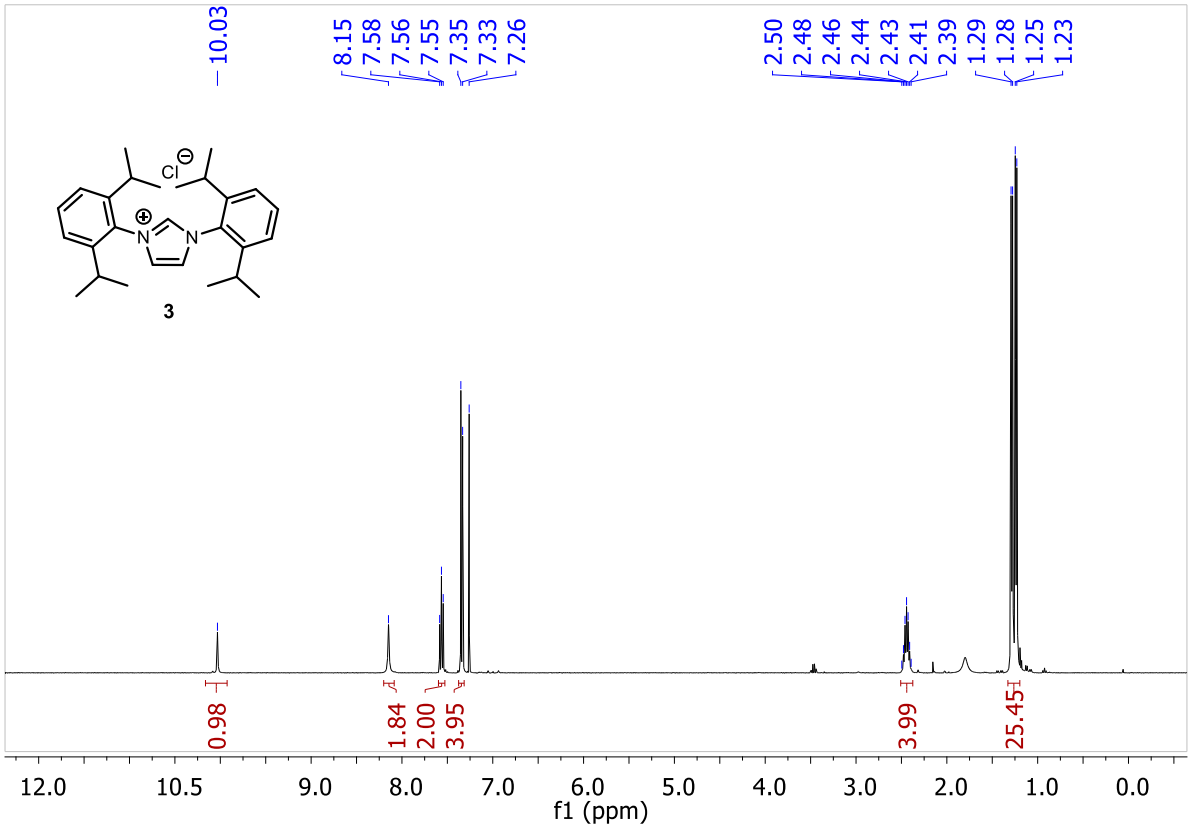


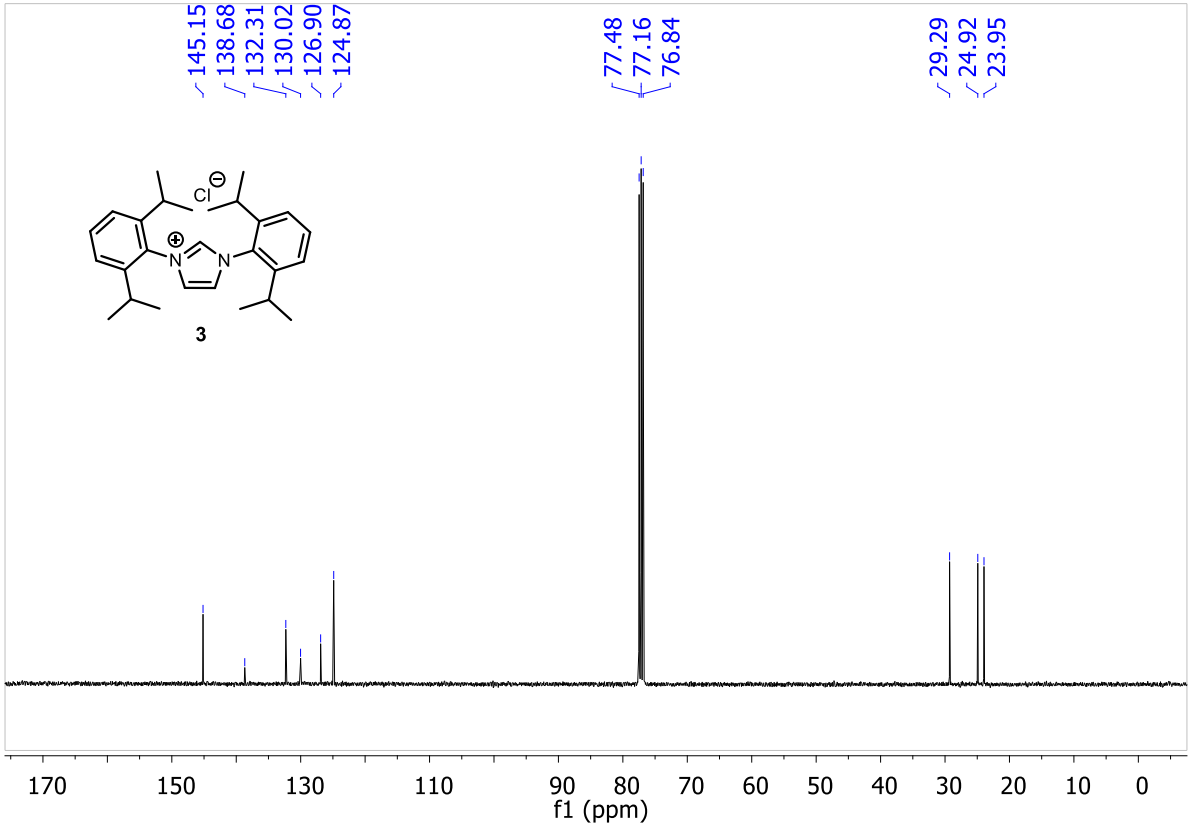
N,*N*-Bis(2,6-diisopropylphenyl)ethane-1,2-diimine **3a**

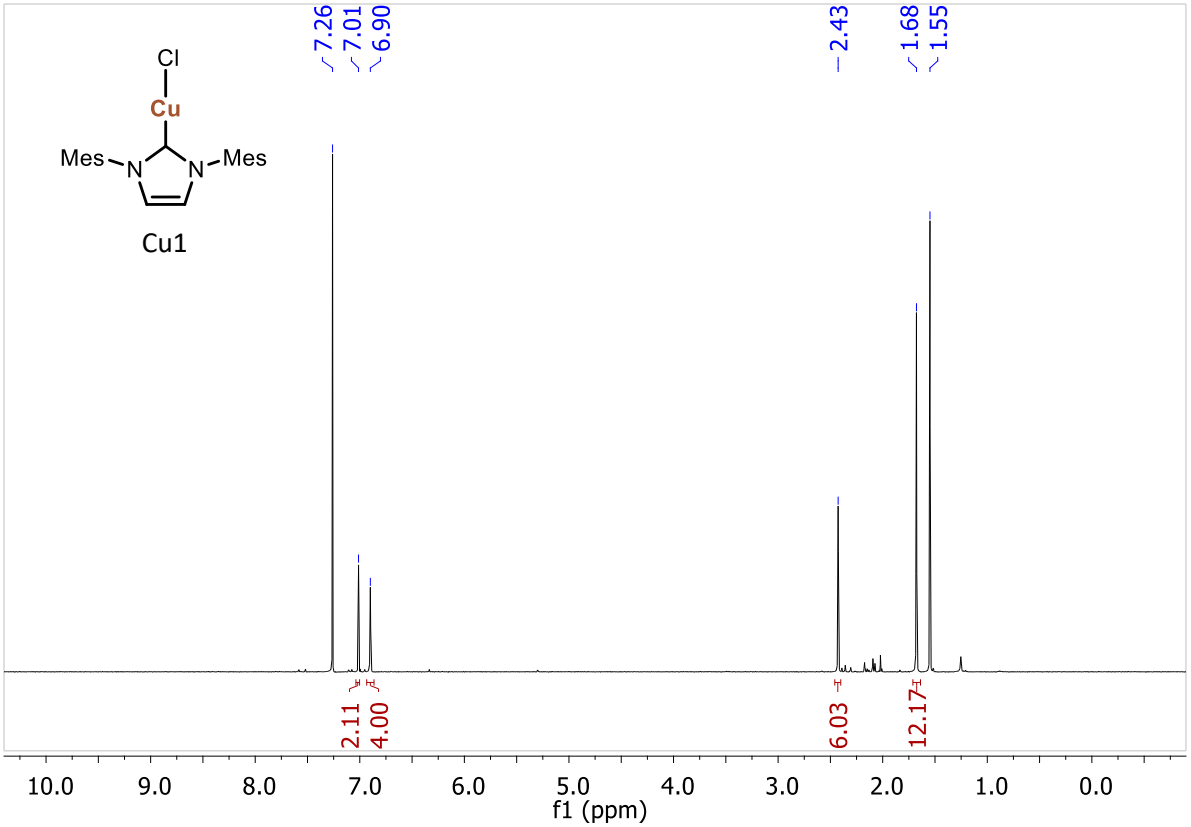




1,3-Bis(2,6-diisopropylphenyl)-1H-imidazol-3-ium **3**



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N
N
Mes
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1.68
1.55
2.11
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6.03
12.17
10.0
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7.0
6.0
5.0
4.0
3.0
2.0
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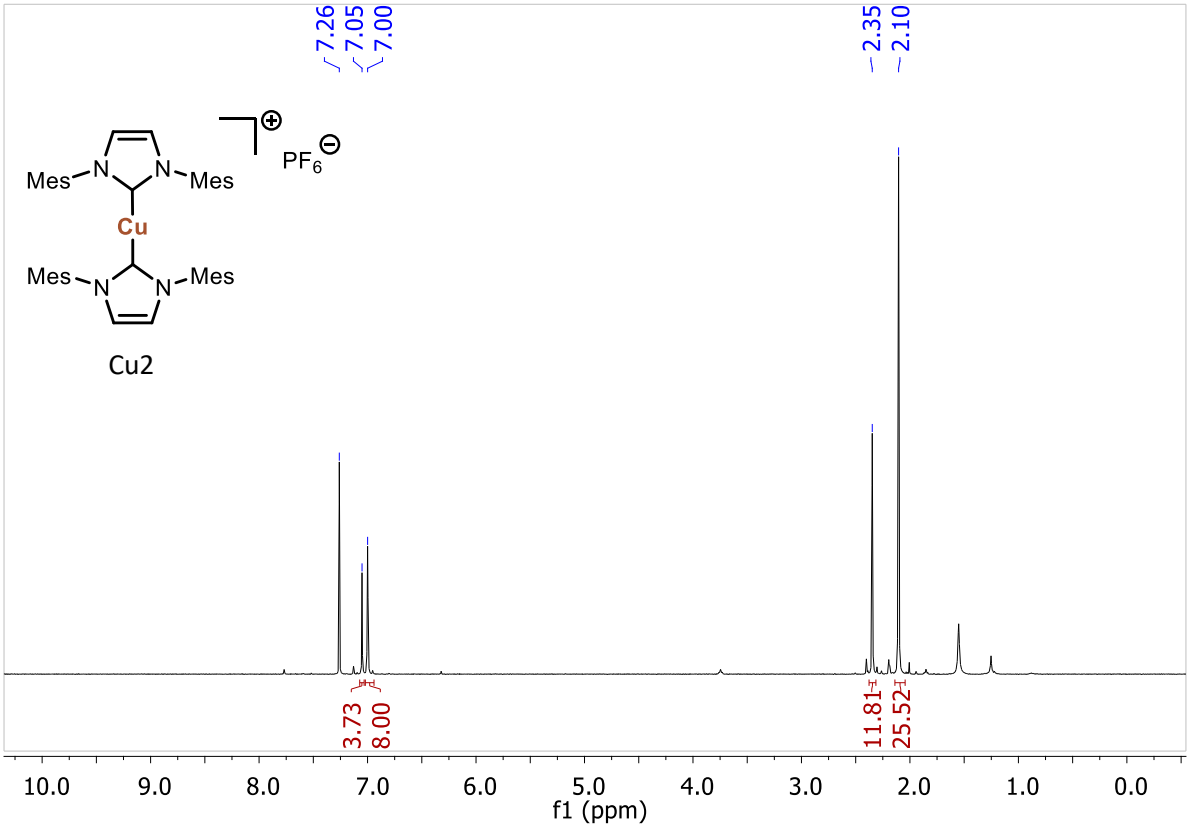
0.0
f1 (ppm)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Mes
Cu
Mes
N
N
Mes
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11.81
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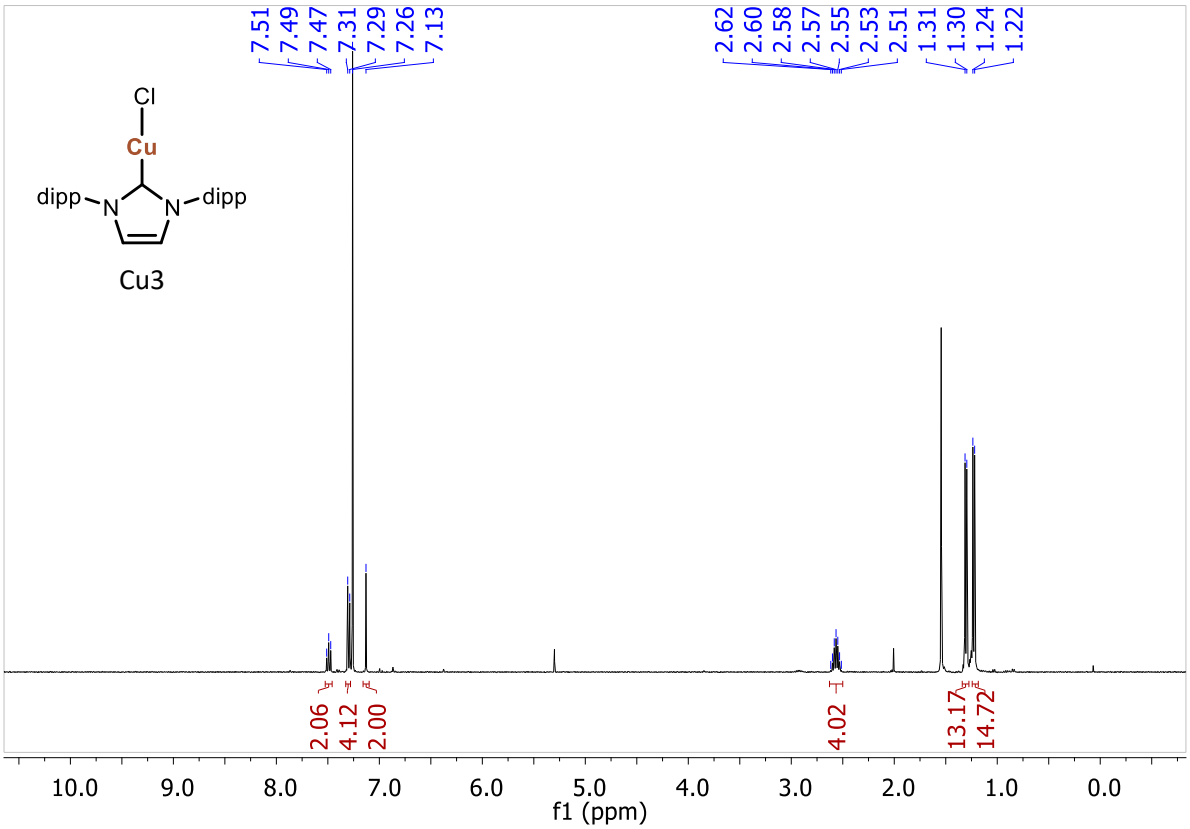
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1.30
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dipp
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dipp
Cu3
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2.00
4.02
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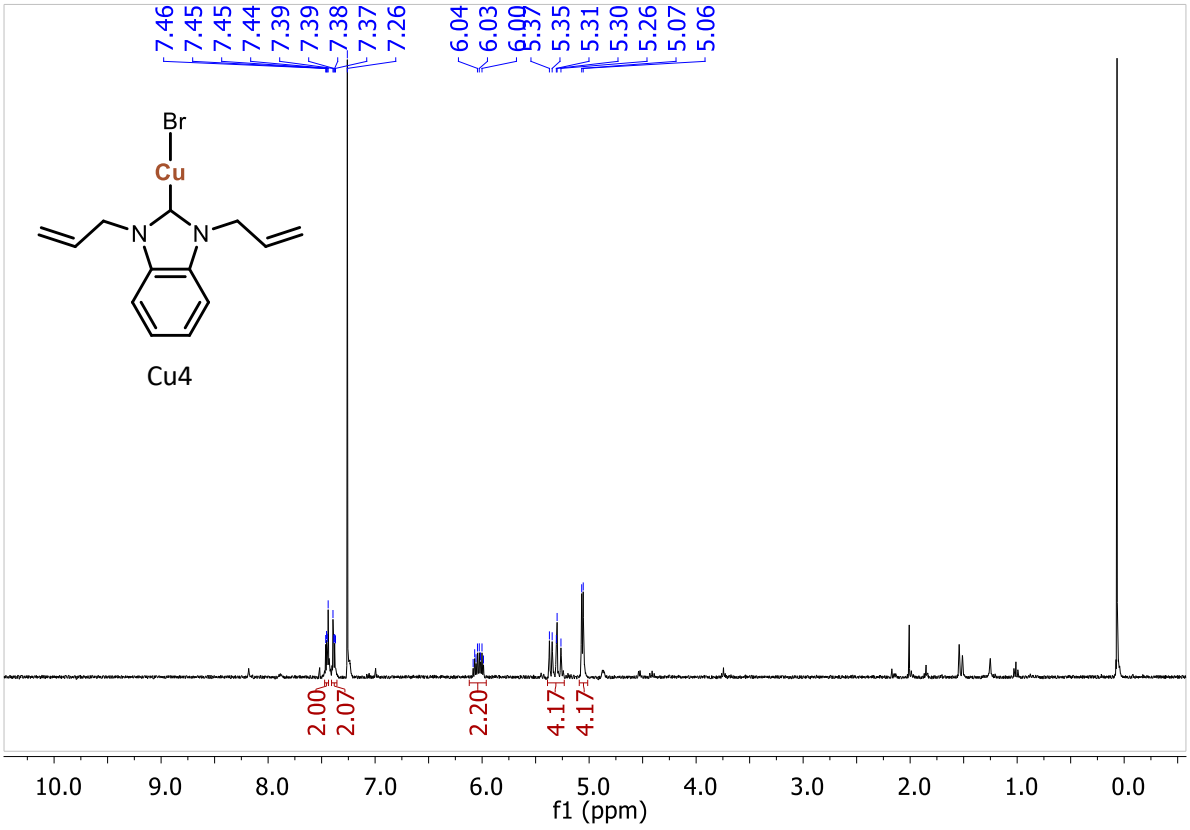
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5.0
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2.0
1.0
0.0
f1 (ppm)

8 References

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