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Energy Harvesting: Synthetic Use of Recovered Energy in Electrochemical Late-Stage Functionalization

Tobias A. Karl, Max Seidl, and Burkhard König*[a]

An induction-based energy harvesting (EH) device was presented. It converted part of the rotational energy of magnetic stirrers back into electrical energy, making it accessible for electrochemical transformations. After rectification, the induced AC voltage was optionally provided as a constant voltage or constant current whereby the available voltage could directly be adjusted by the stirring rate of the reaction. The comparability of the results with reactions carried out with commercial power supplies has been demonstrated on six different late-stage functionalization, including methylation, carboxylation, trifluoromethylation, imidation, hydrolysis, and keto-olefin coupling. Therefore, the described EH device is a low-cost, resource-efficient alternative to a commercial electrochemical set-up and enables laboratories without specialized equipment to perform electrochemical reactions.

Introduction

Energy Harvesting (EH) refers to a process in which ambient energy is captured, to power devices.[1] In addition to recovering otherwise wasted energy, it also helps to decentralize the generation of electricity for more energy autonomy. Self-powered electrochemical and photoelectrochemical systems are used for waste stream treatment,[2,3] sensor systems,[4,5] corrosion protection[6] and water splitting.[7] To our knowledge, applications in the synthesis of organic molecules have not been described so far.[8]

We report here the application of a self-powered rotary energy harvesting (EH) device (Figure 1) to power an electrolytic cell for the synthesis of complex organic molecules. Based on the concept of induction, which is defined as the occurrence of an electromotive force across an electrical conductor due to a changing magnetic flux, parts of the rotating magnetic field of laboratory stirring plates are converted back into electric energy.[9] This method is not intended to reduce overall energy consumption, but to enable performance of electrochemical transformation without the need of additional power supplies. It may be therefore a cost-effective and resource-saving alternative for low budget chemical laboratories. The approach simplifies operations in synthetic laboratories and allows performing electrochemical reactions with limited equipment. Simple design ensures that all components can be modified and repaired as easily as possible.

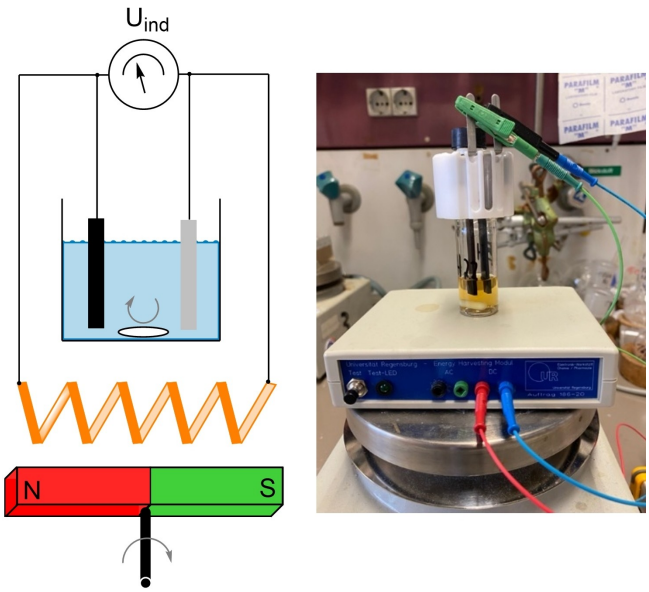

Figure 1. Energy Harvesting (EH) device. Left: schematic representation, right: Prototype on a magnetic stirring plate during the operation of an electrolysis.

Results and Discussion

The EH device essentially consists of an induction coil inside a plastic housing, AC and DC outputs, a rectifier made of four diodes, a capacitor and a test LED with switch and ballast resistor (Figure S1, S2). Providing an open-circuit voltage of $U_{ind} = 18$ V and a current of $I_{max} = 40$ mA, the requirements for most electrochemical reactions are fulfilled.[10–13] The sinusoidal voltage is smoothed to a high level, ensuring operation in DC mode (Figure 2). An external module, consisting of a tunable rotary potentiometer was conceived for constant current (c.c.) operation mode. Connected to the DC-output of the EH device, constant current is delivered to the modules output jacks. In

[a] T. A. Karl, M. Seidl, Prof. Dr. B. König
Faculty of Chemistry and Pharmacy
University of Regensburg
93040 Regensburg, Germany
E-mail: Burkhard.koenig@chemie.uni-regensburg.de

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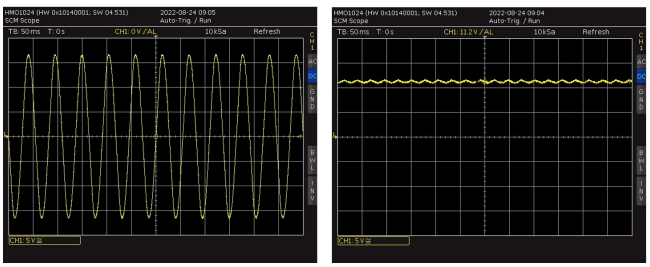
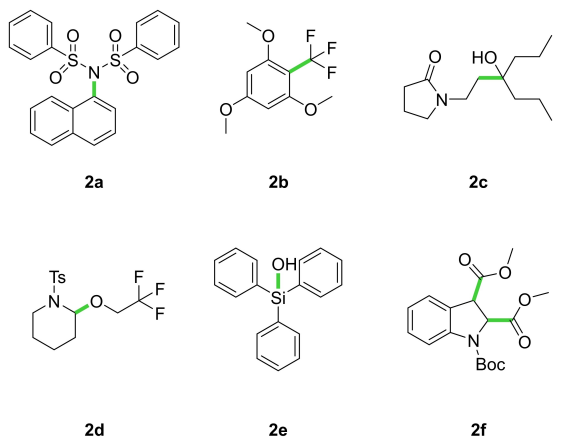


Figure 2. Oscillogram of the output voltage of the EH device. Left: open circuit without rectification, right: closed circuit under load with rectification.

order to assess the viability of this EH concept in organic electrochemistry, a comparison between conventional power supply (PS) and the newly developed EH device was carried out as follows. To determine whether and to what extent the EH device can be used as a substitute for a commercial energy source, the yield was chosen as the decisive parameter. Due to their importance in the synthesis of natural products,[14] pharmaceuticals,[15,16] agrochemicals[17] and new materials,[16] six late-stage functionalization (Scheme 1) were selected as model reactions for this purpose.

Nitrogen-containing aromatics are omnipresent and new approaches for Buchwald-Hartwig[18] coupling and Ullman amination[19,20] receive much attention.[21] We employed a method for C–N cross-coupling, developed by Lei[22] to demonstrate the direct dependency between rotational speed and induced voltage. Operating at constant voltage (c.v.) mode, a slow stirring speed of $f_{rot} = 350$ rpm provides a voltage of 3.5 V, which furnishes the imidation of naphthalene in 18% yield. The control reaction with a conventional power supply led to similar low outcome. Higher temporal change of the magnetic field generates a higher output voltage $U_{ind} = 4.0$ V, furnishing 50% respectively 47% yield at 550 rpm. An extreme stirring speed of 1050 rpm delivers $U_{ind} = 8.0$ V, causing an overoxidation which in turn diminishes the yield (12%). Overall, the yields varied only slightly between the two power sources for all rotation speeds. Therefore, poor mixing at extremely high or low stirring speeds hardly has any effect, while the induced voltage does.



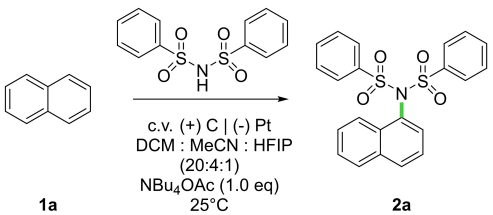
Scheme 1. Selected late stage functionalization.

Operation in c.c. mode enhanced the reaction outcome, as described by the authors.[22] The reaction outcome is also almost identical for both power sources in this operating mode (Table 1). To confirm the initial findings, we applied our EH approach to other reactions like the trifluoromethylation of 1,3,5-trimethoxybenzene. The introduction of trifluoromethyl groups into organic compounds often improves properties related to bioavailability, lipophilicity and metabolic stability which makes them appealing for all kinds of applications.[23–25] There are numerous strategies for the installation of a trifluoromethyl functionality, including photoredox[26,27] and electrochemical[28,29] approaches. Processes that do not require an external oxidant are of particular interest. That is why we chose a work of Lei[30] for another comparison between the different power sources in c.v. and c.c. mode. The applied voltage of 3.3 V corresponds to the initial voltage at 10 mA.

Once again, no significant difference between the power sources has been observed, neither in c.v. nor in c.c. mode (Table 2). Both modes furnished the product formation in >60% yield. The deviation in yield in c.v. mode is slightly higher than in c.c. mode, which was also observed during the previous imidation.

A keto olefin coupling of 1-vinylpyrrolidin-2-one with 4-heptanone was chosen for the last example in c.v. mode

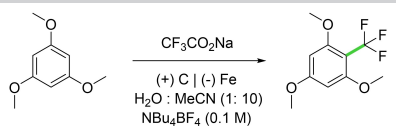
Table 1. Influence of stirring speed on electrochemical reactions using the imidation of naphthalene as an example.



1a 2a

Entry	Source	f_{rot} [rpm]	Mode	U [V]	I [mA]	Yield [%]
1	PS	350	c.v.	3.5		12
2	EH	350	c.v.	3.5		18
3	PS	550	c.v.	4.6		47
4	EH	550	c.v.	4.6		50
5	PS	1050	c.v.	8.0		11
6	EH	1050	c.v.	8.0		12
	PS	550	c.c.		10	59
6	EH	550	c.c.		10	56

Table 2. Comparison of c.v and c.c. operation using the example of the trifluoromethylation of 1,3,5-trimethoxybenzene.

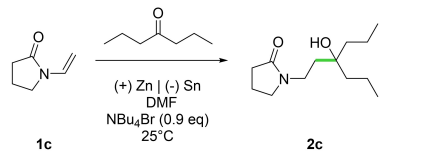


1b 2b

Entry	Source	Mode	U [V]	I [mA]	Yield [%]
1	PS	c.v.	3.3		64
2	EH	c.v.	3.3		60
3	PS	c.c.		10	64
4	EH	c.c.		10	64

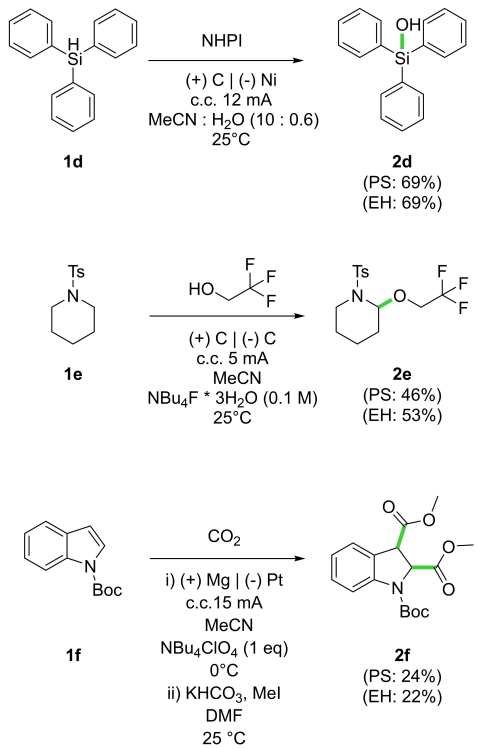
(Table 3). As carbonyl and olefin compounds are well available,[31,32] such couplings have found use in complex molecule synthesis.[33] Following a electro reductive procedure reported by Baran,[32] we obtained the coupling product in fair yields. The yield in c.v. mode was in average by 14% lower than in c.c. mode and the fluctuation between PS and EH was double (4%) compared those at c.c. mode (2%). In conclusion, the c.v. mode is more reliable in terms of reaction outcome than the c.v. mode. Fluctuations seem to be smaller and yields tend to be higher. Photoelectrochemical reactions are an exception here.[34] As a consequence, the last three examples (Scheme 2) were focusing on the comparisons of both power sources in c.c. mode.

Table 3. Comparison of c.v. and c.c. operation using the example of a keto-olefin coupling.



1c 2c

Entry	Source	Mode	*U* [V]	*I* [mA]	Yield [%]
1	PS	c.v.	1.9		54
2	EH	c.v.	1.9		50
3	PS	c.c.		10	68
4	EH	c.c.		10	66



1d 2d (PS: 69%) (EH: 69%)

1e 2e (PS: 46%) (EH: 53%)

1f 2f (PS: 24%) (EH: 22%)

Scheme 2. Comparison of yields from EH and classical electrolysis, based on the hydrolysis of triphenylsilylane (top), the synthesis of an N–O-acetal as a methylation precursor (middle) and the dual carboxylation of Boc-indole.

Silanols are valuable compounds as they are employed in several organic transformations, including metal-catalyzed cross-couplings,[35] C–H functionalization,[36,37] and organocatalysis.[38] They are furthermore known from polymeric materials[39] and as isosteres in bioactive molecules.[40,41] One elegant way to access silanols is the hydrolysis of hydrosilane.

Using NHPI (*N*-hydroxyphthalimide) as HAT mediator, we synthesized triphenylsilanol in satisfactory yield, without any detectable difference based on the power source used.[42] For the provision of desirable intermediates in late stage functionalization a Shono-oxidation of *N*-*P*-tosylpyridine gives access to the corresponding *N*,*O*-acetal, which acts as a methylation precursor. Methylations are an important modification to increase biological activity. Activity changes of more than two orders of magnitude have been observed for bioactive compounds[43] and methyl groups are a structural elements in top-selling drugs.[44] We applied a procedure by Lin,[45] which uses trifluoroethanol (TFE) to capture an iminium ion intermediate.

TFE is more stable towards anodic oxidation then MeOH, while retaining its reactivity to undergo methylation when treated with organozinc reagents.[45] Under low current electrolysis (5 mA) both PS and EH forged the protected *N*,*O*-acetal of piperidine in fair yield. After the applicability at low currents was demonstrated, the upper limit of the device was examined. Carbon dioxide is considered to be the most widespread source of carbon on the planet.[46] CO_2 is omnipresent, but its extensive emission from fossil fuels is the main cause of global warming, leading to increasing attention to CO_2-capturing syntheses.[47] In this regard, Mita[48] has developed an electrochemically carboxylation procedure for the esterification of indole derivatives. Even though the described method exceeds the available current, provided by the EH device, we could demonstrate that under slightly modified conditions Boc-Indole was successfully converted. After a presumably quantitative methylation with MeI, we managed to isolate the two-fold carboxylated product in 22% (EH) respectively 24% (PS) yield, manifesting that there are no significant drawbacks in terms of yield when a commercial PS is replaced by EH. For the application in small lab scale organic synthesis, the EH performs equally well than a potentiostat.

Conclusion

Six different late-stage functionalization reactions including C–CF_3, C–Me, C–CO_2Me, sp^2C–sp^3C, Si–O and C–N bond formation demonstrate that the EH of rotational energy of a conventional laboratory magnetic stirrer could be used to perform electrochemical reactions. We observed no significant deviation in yield within the individual operational modes, between conventional power sources and the EH device. The built-in rectifier was sufficient to smooth the induced AC voltage to the extent required for different organic transformations. The simple to operate and cost-effective set-up may allow more chemical laboratories including laboratories in developing countries to use electrochemical reactions in

education and research. The overall cost for all parts of the EH device was estimated to be below 90 €.

Experimental Section

Power sources and electrodes

All electrochemical transformations, powered by EH, were performed by placing the EH device between the magnetic stirrer and the reaction vessel. If necessary, the c.c. module was added. Voltage or current was set to the specific value *via* the speed of the stirrer and monitored by a connected multimeter. Reactions performed with a commercial device were powered by a PeakTech® 6080 A digital DC power supply. If indicated in literature, an IKA ElectraSyn® 2.0 was employed instead. The used electrode materials were: reticulated vitreous carbon (RVC) foam electrodes, thickness: 6.35 mm, porosity: 96.5% (Goodfellow, product code: 613-422-20); carbon from Faber-Castell 2.0 mm HB pencil lead, Pt 99.9 from Sigma Aldrich; Fe from DC01 CR1 (C 0.12, P 0.045, S 0.045, Mn 0.60, Ti 0.0) by Thyssen Krupp; Mg 99.9% from Sigma Aldrich, Zinc from MARAWE (part01-74-00000) (200×170×0.6 mm). Sn 99.9% (Aldrich).

Energy harvesting

N-(Naphthalen-1-yl)-N-(phenylsulfonyl) benzene sulfonamide (2a):[22] In a 25 mL Schlenk tube, naphthalene (51.3 mg, 0.4 mmol, 1.0 equiv.), dibenzenesulfonimide (178 mg, 0.6 mmol, 1.5 equiv.) and $^{n}Bu_4NOAc$ (121 mg, 0.4 mmol, 1.0 equiv.) were added. The tube was sealed with a rubber septum, equipped with an RVC (15 mm× 5.0 mm×5.0 mm) anode and a Pt-foil (10 mm×5.0 mm×0.1 mm) cathode. Dry, degassed DCM (2.5 mL), MeCN (0.5 mL) and HFIP (0.125 mL) were added through the septum. Then, the mixture was stirred and electrolyzed at a constant current of 10 mA for 4 hours. The crude was purified by flash column chromatography on silica (hexane/EtOAc = 90/10 to 0/100) to receive a white solid: EHD: 94 mg, 56% yield; PS: 100 mg, 59% yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H [ppm] = 7.96 (dd, J = 8.5, 1.2 Hz, 5H), 7.85 (d, J = 8.2 Hz, 1H), 7.67 (t, J = 7.5 Hz, 2H), 7.59–7.49 (m, 5H), 7.48–7.38 (m, 2H), 7.29 (ddd, J = 8.3, 6.9, 1.1 Hz, 1H), 7.12 (dd, J = 7.4, 1.0 Hz, 1H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ_C [ppm] = 139.25, 134.78, 134.21, 132.92, 131.28, 131.13, 130.67, 129.18, 129.06, 128.23, 127.11, 126.66, 125.10, 124.06; **HRMS** (ESI) m/z calcd. for $C_{22}H_{18}NO_4S_2+$ $[M+H]+$ = 424.0672 found 424.0674. **MP** 197–198 °C

1,3,5-Trimethoxy-2-(trifluoromethyl) benzene (2b):[30] In a 25 mL Schlenk tube 1,3,5-trimethoxybenzene (84.0 mg, 0.5 mmol, 1.0 equiv.), CF_3SO_2Na (156 mg, 1 mmol, 2.0 equiv.), $^{n}Bu_4NBF_4$ (82.3 mg, 0.25 mmol, 5.0 equiv.) were dissolved in a mixture of MeCN (10 mL) and water (1 mL) under nitrogen atmosphere. The tube was sealed with a rubber septum, equipped with a carbon (RVC: 20 mm×5.0 mm×5.0 mm) anode and a steel (20 mm× 5.0 mm×0.2 mm) cathode. The mixture was degassed by purging with nitrogen for 5 min and electrolyzed at a constant current of 10 mA at room temperature for 5 h. Then water (10 mL) and ethyl acetate were added and the organic layer was separated. The aqueous phase was extracted in ethyl acetate (3x 20 mL) and the combined organic phase was washed with brine and dried over anhydrous Na_2SO_4. The solvent was removed under reduced pressure and the crude was purified by flash column chromatography (hexane/EtOAc = 95/5) to receive a white solid: EH: 76 mg, 64%; PS: 76 mg, 64% yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H [ppm] = 6.12 (s, 2H), 3.83 (s, 9H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ_C [ppm] = 163.67, 160.53, 160.52, 124.51 (q, $J^1_{c\text{-}F}$ = 182.81 Hz), 100,385 (q, $J^2_{c\text{-}F}$ = 20.20 Hz), 91.33, 56.31, 55.45; **^{19}F NMR** (377 MHz, $CDCl_3$) δ_F [ppm] = −54.68; **HRMS** (EI+) m/z calcd. for $C_{10}H_{12}O_3F_3+$ $[M+H]+$ = 236.06548, found 236.06502; **MP** 60–63 °C.

1-(3-Hydroxy-3-propylhexyl)pyrrolidin-2-one (2c):[32] In a 2 mL Eppendorf® tube, 4-heptanon (98 μL, 0.7 mmol, 2.0 equiv.), 1-vinyl-2-pyrrolidon (37 μL, 0.35 mmol, 1.0 equiv.) and $^{n}Bu_4NBr$ (96.7 mg, 0.3 mmol) were dissolved in DMF (1 mL). A zinc anode (10 mm× 5.0 mm×0.3 mm) and a Sn-cathode (10 mm×5.0 mm×0.2 mm) were immersed. Then the mixture was electrolyzed at air with a constant current of 10 mA at room temperature for 4 h. The reaction mixture was filtered through a plug of silica, rinsed with EtOAc (30 mL) and dried over anhydrous Na_2SO_4. The solvent was removed under reduced pressure and the crude was purified by flash column chromatography on silica (petroleum ether/EtOAc = 90/10) to receive a colorless oil: EH: 54 mg, 68% yield; PS: 54 mg, 68% yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H [ppm] = 3.40–3.29 (m, 4H), 2.63 (brs, 1H), 2.33 (t, J = 8.1 Hz, 2H), 2.05–1.90 (m, 2H), 1.62–1.56 (m, 2H), 1.43–1.36 (m, 4H), 1.33–1.20 (m, 4H), 0.88 (t, J = 7.1 Hz, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ_C [ppm] = 175.22, 73.36, 47.51, 41.62, 38.29, 36.16, 31.17, 17.95, 16.93, 14.74. **HRMS** (EI+) m/z calcd. for $C_{13}H_{26}NO_2+$ $[M+H]+$ = 227.1885, found 227.1885

Triphenylsilanol (2d):[42] In a 10 mL ElectraSyn® undivided cell, triphenylsilane (157 mg, 0.6 mmol, 1.0 equiv.), NHPI (98 mg, 0.6 mmol, 1.0 equiv.), and $^{n}Bu_4NPF_6$ (325 mg, 0.84 mmol, 1.4 equiv.) were dissolved in a mixture of MeCN (7.0 mL) and water (0.4 mL). The vial was sealed with a cap, equipped with an RVC (20 mm× 5.0 m×3.0 mm) anode and a nickel foam (4.0 cm×0.8 cm×0.2 cm) cathode and the mixture was purged with nitrogen for 15 min. Then, the mixture was stirred and electrolyzed at a constant current of 12 mA for 8 hours at room temperature. After completion, the solvent was removed under reduced pressure and the crude was purified by flash column chromatography on silica (petroleum ether/ EtOAc = 95/5) to receive a white solid: EH: 140 mg, 69% yield; PS: 140 mg, 69% yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H [ppm] = 7.66 (d, J = 6.5 Hz, 6H), 7.47 (t, J = 7.3 Hz, 3H), 7.40 (t, J = 7.2 Hz, 6H), 2.74 (brs, 1H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ_C [ppm] = 135.13, 130.24, 128.05; **HRMS** (EI+) m/z calcd. for $C_{18}H_{17}OSi+$ $[M+H]^+$ = 276.09649, found 276.09676; **MP**: 151–152 °C.

Piperidine,1-[(4-methylphenyl)sulfonyl]-2-(2,2,2-trifluoroethoxy) (2e):[45] In a 5 mL ElectraSyn vial, equipped with graphite (20 mm× 6.0 mm×3.0 mm) anode and graphite (20 mm×6.0 mm×3.0 mm) cathode, 1-tosylpiperidine (**1f**) (239 mg, 1.0 mmol, 1.0 equiv.), trifluoroethanol (4 mL, 55 mmol, 55 equiv.) and TBAF trihydride (126 mg, 0.4 mmol) were added. The mixture was electrolyzed at a constant current of 5 mA at room temperature for 16 hours (2.9 F/ mol). Then, the crude was purified by flash column chromatography (petroleum ether /DCM = 40/60) to receive a colorless oil: EH: 147 mg, 53% yield; PS:127 mg, 46% yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H [ppm] = 7.70 (d, J = 8.3 Hz, 2H), 7.30 (d, J = 8.0 Hz, 2H), 5.33 (s, 1H), 3.98–3.83 (m, 2H), 3.59 (d, J = 13.5 Hz, 1H), 3.12 (td, J = 13.3, 2.7 Hz, 1H), 2.43 (s, 3H), 1.92–1.89 (m, 1H), 1.77–1.63 (m, 1H), 1.53–1.41 (m, 2H), 1.37–1.34 (m, 1H), 1.42–1.21 (m, 1H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ_C [ppm] = 143.73, 137.70, 129.92, 128.19, 124,05 (q, $J^1_{c\text{-}F}$ = 279.77 Hz), 83.52, 64.65 (q, $J^2_{c\text{-}F}$ = 34.34 Hz) 40.85, 29.61, 23.92, 21.65, 17.66; **^{19}F NMR** (377 MHz, $CDCl_3$) δ_F [ppm] = -74.66 (s); **HRMS** (ESI+) m/z calcd. for $C_{14}H_{19}F_3NO_3S+$ $[M+H]+$ = 338.1032 found 338.1033.

1-(tert-Butyl) 2,3-dimethyl (2S,3S)-indoline-1,2,3-tricarboxylate (2f):[48] In a 5 mL Schlenk tube, 1-Boc indole (**1f**) (43.5 mg, 0.2 mmol, 1.0 equiv.) and $^{n}NBu_4ClO_4$ (68.4 mg, 0.2 mmol, 1.0 equiv.) were dissolved in dry MeCN (2 mL) under nitrogen atmosphere. The vial was capped with a septum, equipped with a magnesium foil (20 mm ×3.0 mm×0.15 mm) anode and a platinum foil (10 mm ×5.0 mm ×0.1 mm) cathode. Then the mixture was sparged with CO_2 for 10 min and electrolyzed under a constant current of 15 mA

and continuous CO_2 sparging at 0 °C for 3 h. The reaction mixture was acidified with 1 N HCl (2 mL), extracted with ethyl acetate (3× 15 mL) and the combined organic phase was dried over anhydrous Na_2SO_4. The solvent was removed under reduced pressure and the crude was methyl esterified as following: In a 5 mL crimp cap vial, the crude dicarboxylic acid and $KHCO_3$ (80 mg, 0.8 mmol, 2 equiv.) were dissolved in anhydrous DMF (3 mL) under nitrogen atmosphere. Then MeI (38 μL, 0.6 mmol) was slowly added and mixture was stirred at room temperature for 12 h. The reaction mixture was diluted with EtOAc (10 mL), washed with brine (10 mL) and dried over anhydrous Na_2SO_4. The solvent was removed under reduced pressure and the crude product was purified flash column chromatography on silica (EtOAc-PE, 3:17) to give a colorless oil: EH: 15 mg, 22% yield; PS: 16 mg, 24% yield. **^{1}H NMR** (600 MHz, $CDCl_3$) δ_H [ppm] = 7.33 (d, J = 7.6 Hz, 1H), 7.30–7.26 (m, 2H), 6.98 (t, J = 7.4 Hz, 1H), 5.34 (brs, 1H), 4.17 (brs, 1H), 3.79 (s, 3H), 3.76 (s, 3H), 1.51 (brs, 9H); **^{13}C NMR** (151 MHz, $CDCl_3$) δ_C [ppm] = 171.42, 170.54, 151.37, 142.39, 129.65, 125.57, 125.18, 122.86, 115.14, 81.97, 62.80, 53.20, 52.73, 49.98, 28.38; **HRMS** (ESI+) m/z calcd. for $C_{17}H_{22}NO_6^+$ $[M+H]^+$ = 358.1261; found 358.1269.

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Conflict of Interest

The authors declare no conflict of interest.

Data Availability Statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

Keywords: Electrochemistry · Energy harvesting · Late-stage functionalization · Radicals · Self-sustaining

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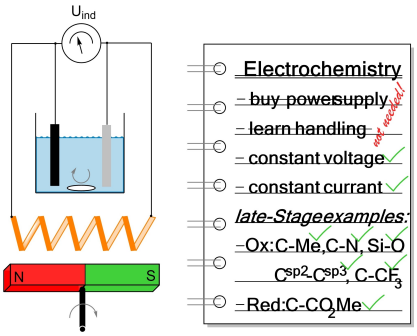
RESEARCH ARTICLE

Powering up: An induction-based energy harvesting (EH) device converted rotational energy of magnetic stirrers back to electrical energy, making it accessible for electrochemical late-stage functionalization. The device optionally provided constant voltage (AC, DC) or constant current (DC) whereby the available voltage could be directly adjusted by the stirring rate of the reaction.



*T. A. Karl, M. Seidl, Prof. Dr. B. König**

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Energy Harvesting: Synthetic Use of Recovered Energy in Electrochemical Late-Stage Functionalization