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Cyclic voltammetry experiments were carried out at 25 °C in N₂ purged 0.1 M NaCl aqueous solutions with a Metrohm Eco Chemie Autolab PGSTAT12 potentiostat, working on GPES v.4.9 software. A three-electrode configuration was used with a 3 or 1.6 mm glassy carbon working electrode, a platinum counter electrode and RE-5B Ag/AgCl BASI reference electrode. The glassy carbon electrode was polished before each measurement using a 0.05 µm alumina-H₂O slurry on a polishing cloth. Cyclic voltammetry was performed at 1 mM concentrations of active materials using a scan rate of 20 mV s⁻¹.

Whereas the presence of dimers could not be determined directly (as a result of both high radical concentrations affecting observation by NMR and the known EPR silence of viologen p-dimers⁴⁴ affecting observation by EPR), in situ PFG NMR experiments carried out for 10, 11 and 13 at 0%, 50% and 100% SOC (Extended Data Table 1) indicated a general decrease in diffusivity (D) from 0% to 100% SOC, especially for compounds 11 and 13 that show low radical concentrations at all SOC. These results indicate an increase in size of the cations on monoradical generation, consistent with the formation of dimers.

To gain further insights into the nature of monoradical dimerization, ex situ ultraviolet-visible light (UV-vis) spectroelectrochemical studies were carried out for 10, 11 and 13. Degassed solutions of 10⁴⁺ (0.5 mM), 11⁴⁺ (0.5 mM) and 13⁴⁺ (0.5 mM) were prepared using the Schlenk technique. The solutions were transferred into quartz cuvettes (10 mm path length) under N₂ and the cuvettes were kept under a positive flow of N₂. The samples were electrochemically reduced using a carbon paper working electrode and a gold counter electrode. Spectral data were acquired using a UV-vis spectrometer (Horiba, Duetta) immediately after the solutions were transferred into the cuvettes and electrochemically reduced.

OEMS experiments were performed using a custom-made H cell, connected to gas flow system previously described using 1% and 20% O₂ in Ar at 1.2 bar(a)^{5,32}.

Flow cells were assembled as described above. Full cells were assembled from 250 mM 11 or 17 (10.0 ml for 11, 12.5 ml for 17) and 250 mM 4-hydroxy-TEMPO in 1 M NaCl (50 ml). Currents of 20 mA cm⁻² (at a flow rate of 40 rpm), 30 mA cm⁻² (at a flow rate of 60 rpm) and 40 mA cm⁻² (at a flow rate of 80 rpm) were used in both cases. Both reservoirs were purged with N₂, degassed for 1 h and then kept under active N₂ flow during cycling for the first five cycles, after which the nitrogen flow was disconnected and the reservoirs opened to air. The flow cells were galvanostatically charged and discharged at room temperature using a portable potentiostat. Electrochemical data were processed using EC-lab 11.36 (BioLogic).

The experimental dataset generated and/or analysed during the current study is available from the corresponding authors on request.

Any code used during analysis is available from the corresponding authors on request.

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These authors contributed equally: Mark E. Carrington, Kamil Sokołowski

M.E.C., K.S., C.P.G. and O.A.S. together with Cambridge Enterprise (University of Cambridge) filed a patent application to protect intellectual property embedded within this report and its relation to the use of the molecules described for RFBs and their operation in air.

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Web: <https://kary.com.pl/contact-us/>

Email: energystorage2000@gmail.com

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