**Supporting Information for “Flow Electrosynthesis and Molecular Weight Control of Polyphenylene Deriving from 1,4-Bis(trimethylsilyl)benzene: Effect of a Silyl Substituent on the Coupling Position” Published in *Electrochemistry*, 2020, 88, 336-339.**

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**Preparation procedure for the electrochemical flow microreactor**

The reactor was constructed from Pt plate anode and cathode (3 cm width, 3 cm length). A spacer (80 μm thickness double faced adhesive tape) was used to leave a rectangular channel exposed, and the two electrodes were simply sandwiched together (area of the two electrodes: 1×3 cm2). After connecting Teflon tubing to inlets and outlet, the reactor was sealed with epoxy resin (Figure S1).



**Figure S1.** Schematic illustration of preparation procedure for the electrochemical flow microreactor.

**Computational Data**

Computation was carried out in the Gaussian 09 Program suite (7) at the B3LYP/6-31G(d) level of theory.

**Table S1** B3LYP/6-31G(d) calculated mulliken charges and spin densities with hydrogens of 1,4-bis(trimethylsilyl)benzene radical cation summed into heavy atoms

|  |  |  |  |
| --- | --- | --- | --- |
| Label | Atom | Mulliken charges | Spin densities |
| 1 | C | 0.048445 | 0.022015 |
| 2 | C | 0.048458 | 0.022065 |
| 3 | C | -0.015823 | 0.364736 |
| 4 | C | 0.048445 | 0.022015 |
| 5 | C | 0.048458 | 0.022064 |
| 6 | C | -0.015823 | 0.364736 |
| 11 | Si | 0.688605 | -0.008061 |
| 12 | Si | 0.688606 | -0.008061 |
| 13 | C | -0.062503 | 0.087811 |
| 17 | C | -0.103588 | 0.005725 |
| 21 | C | -0.103594 | 0.00571 |
| 25 | C | -0.103594 | 0.00571 |
| 29 | C | -0.062503 | 0.087811 |
| 33 | C | -0.103588 | 0.005725 |

**13C-and 1H- NMR spectra**

Each raw data is set in csv data.

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**Figure S2**. 13C-NMR spectrum of polyphenylene synthesized by electro-oxidative polymerization of 1,4-bis(trimethylsilyl)benzene and following desilylation procedure.

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**Figure S3**. 1H-NMR spectrum of polyphenylene synthesized by electro-oxidative polymerization of 1,4-bis(trimethylsilyl)benzene and following desilylation procedure.