Supplementary Information

Site-Selective Sequential Coupling Reactions Controlled by "Electrochemical Reaction Site Switching": a Straightforward Approach to 1,4-Bis(diaryl)buta-1,3-diynes

Koichi Mitsudo, Natsuyo Kamimoto, Hiroki Murakami, Hiroki Mandai, Atsushi Wakamiya, Yasujiro Murata, and Seiji Suga**

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1. X-ray Crystallography

Details of the crystal data and a summary of the intensity data collection parameters for bis(diaryl)butadiyne **3g** are listed in Table S1. X-ray single crystal analysis was conducted with a Rigaku VariMax with Saturn. Graphite-monochromated Mo Kα radiation (λ = 0.71075 Å) was used. The structures were solved by direct methods with (SHELXS-97)[1] and refined by full-matrix least-squares techniques against F^2 (SHELXL-97). The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were placed using AFIX instructions. In the subsequent refinement, the function $\Sigma w(F_o^2–F_c^2)^2$ was minimized, where $|F_o|$ and $|F_c|$ are the observed and calculated structure factor amplitudes, respectively. The agreement indices are defined as $R_1 = \Sigma(||F_o|–|F_c||)/\Sigma|F_o|$ and $wR_2 = [\Sigma w(F_o^2–F_c^2)^2/\Sigma(wF_o^4)]^{1/2}$. All calculations were performed by using Yadokari-XG 2009[2] and illustrations were drawn by using ORTEP-3.

Table S1. Crystallographic data and structure refinement details for **3g**.

Empirical formula	$C_{32}H_{22}O_2$	
Formula weight	438.50	
Temperature	93(2) K	
Wavelength	0.71075 Å	
Crystal system	Triclinic	
Space group	P-1 (#2)	
Unit cell dimensions	a = 9.271(7) Å	α = 90.570(11)°.
	b = 9.707(7) Å	β = 97.203(5)°.
	c = 20.928(14) Å	γ = 114.198(13)°.
Volume	1700(2) Å^3	
Z	3	
Density (calculated)	1.285 Mg/m^3	
Absorption coefficient	0.079 mm^{-1}	
F(000)	690	
Crystal size	0.68 × 0.20 × 0.05 mm^3	
Theta range for data collection	3.12 to 27.47°	
Index ranges	-12<=h<=12, -12<=k<=12, -27<=l<=27	
Reflections collected	20748	
Independent reflections	7713 [R(int) = 0.0871]	
Completeness to theta = 27.47°	99.1%	
Max. and min. transmission	0.9961 and 0.9484	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	7713 / 71 / 483	
Goodness-of-fit on F^2	0.912	
Final R indices [I > 2sigma(I)]	R_1 = 0.0581, wR_2 = 0.1153	
R indices (all data)	R_1 = 0.1239, wR_2 = 0.1405	

(1) SHELX, Program for the Solution of Crystal Structures (a) Sheldrick, G. M.; University of Göttingen: Göttingen, Germany, 1997. (b) Sheldrick, G.M. *Acta Cryst.* **2008**, *A64*, 112–122.
(2)Yadokari-XG, Software for Crystal Structure Analyses, K. Wakita (2001); Release of Software (Yadokari-XG 2009) for Crystal Structure Analyses, Kabuto, C.; Akine, S.; Nemoto, T.; Kwon, E. *J. Cryst. Soc. Jpn.* **2009**, *51*, 218–224.