

Electronic Supplementary Material (ESI) for New Journal of Chemistry

**Electrochemical coupling halobenzene into biphenyl on reusable
Pd nanoparticles-coated carbon-paper electrode at ambient
conditions**

Chao-Nan Wang[†], Yong-Heng Lu[†], Yue Liu, Jun Liu, Yao-Yue Yang*, Zhi-Gang Zhao*

Key Laboratory of Basic Chemistry of State Ethnic Commission, School of Chemistry and Environment, Southwest Minzu University, Chengdu 610041, Sichuan, China.

*Corresponding author: YYY, *E-mail: yaoyueyoung@swun.edu.cn*; ORCID ID: 0000-0002-4573-9437; ZZG, *E-mail: zzg63129@163.com*

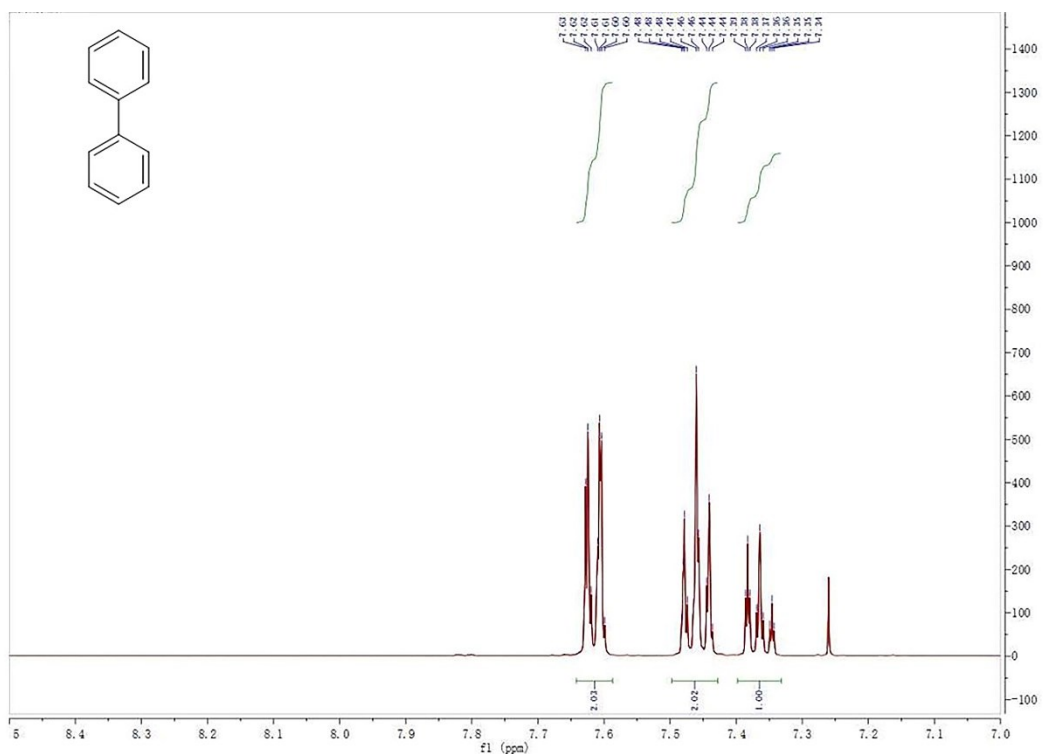


Figure S1. The typical ^1H NMR spectra of the product obtained from the reaction conditions described in entry 1-2, table 2 in the manuscript.

Biphenyl: ^1H NMR (400 MHz, CDCl_3), δ (ppm) 7.599-7.628 (m, 2H), 7.436-7.483 (m, 2H), 7.343-7.386 (m, 1H).

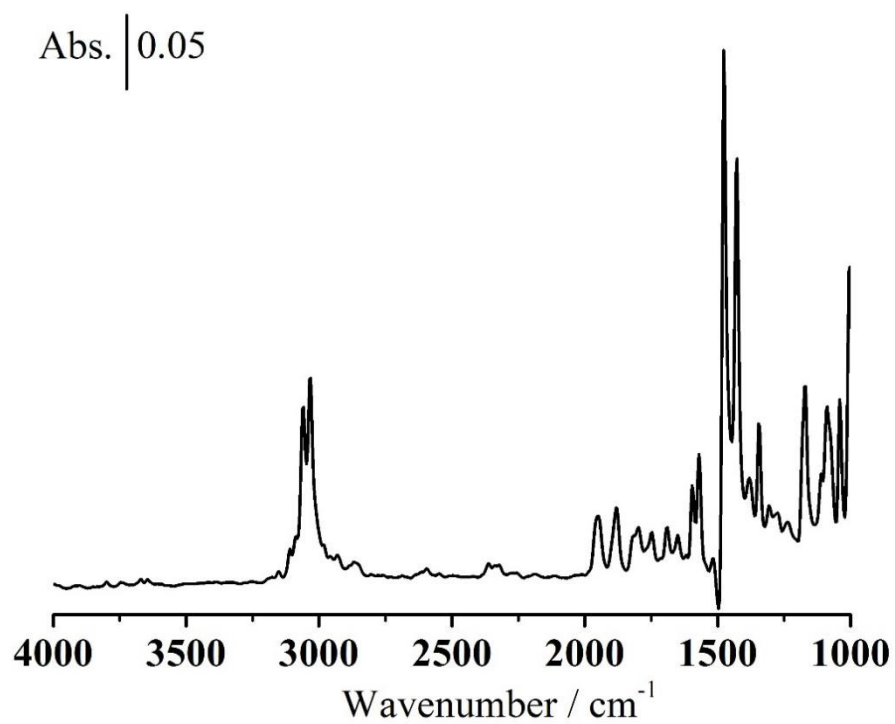


Figure S2. Infrared spectrum of the product obtained from the reaction conditions described in entry 1-2, table 2 in the manuscript.

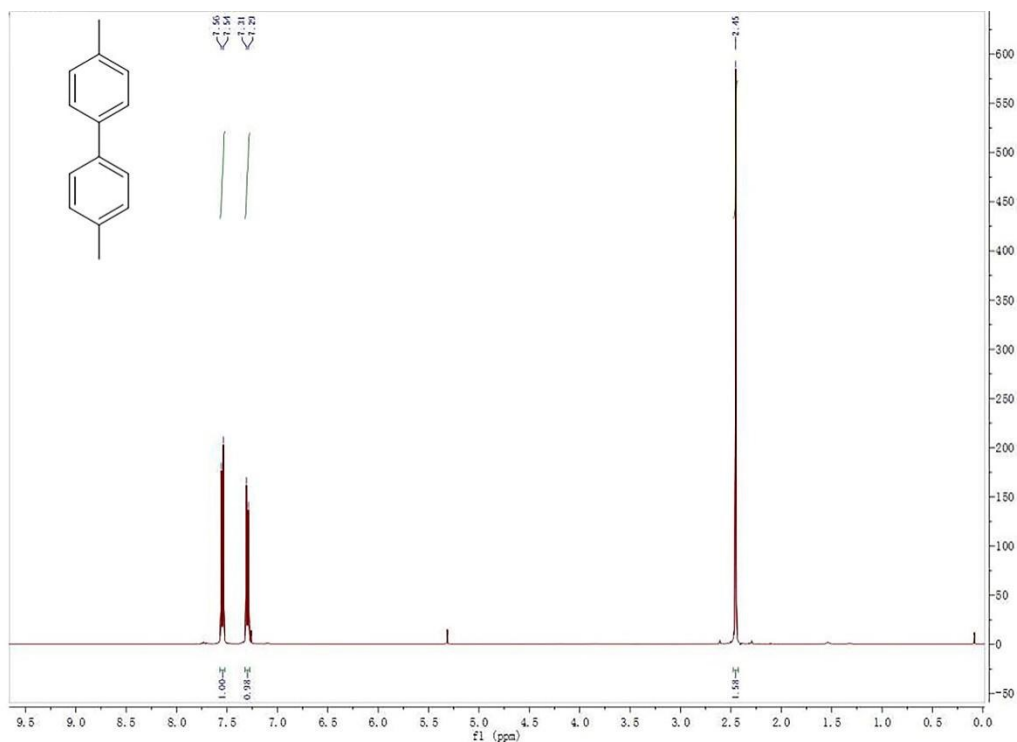


Figure S3. The typical ¹H NMR spectra of the product obtained from the reaction conditions described in entry 4-5, table 2 in the manuscript.

4,4'-Dimethylbiphenyl: ¹H NMR(400 MHz, CDCl₃) δ (ppm) 7.55 (d, *J*=8 Hz, 1H), 7.30 (d, *J*=8 Hz, 1H), 2.453 (s, 1.5H).

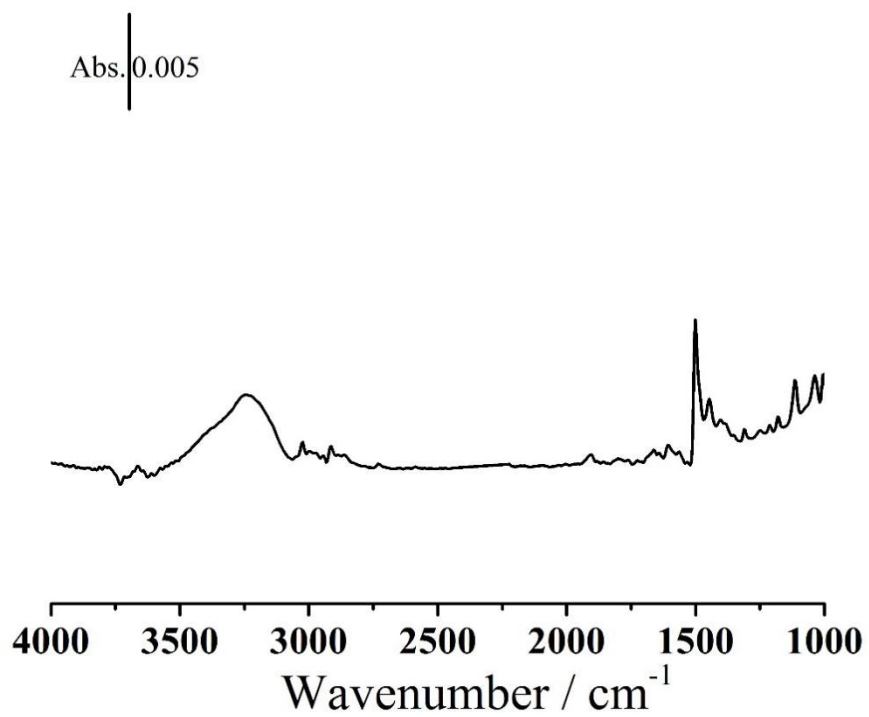


Figure S4. Infrared spectrum of the product obtained from the reaction conditions described in entry 4-5, table 2 in the manuscript.

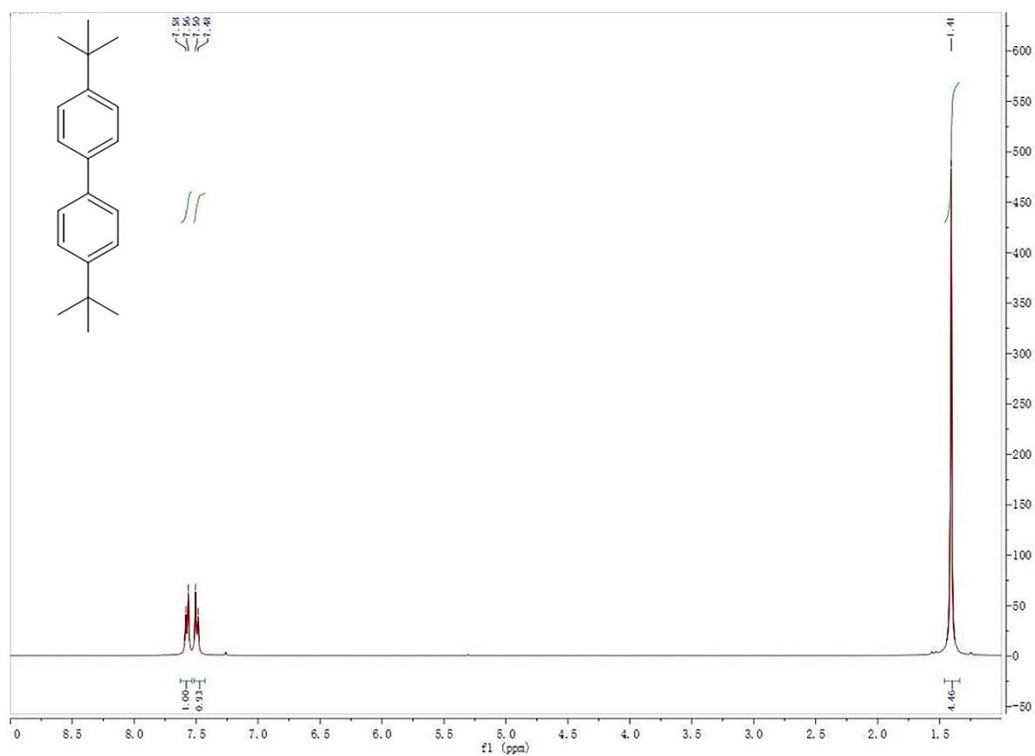


Figure S5. The typical ¹H NMR spectra of the product obtained from the reaction conditions described in entry 6-7, table 2 in the manuscript.

4,4'-Di-tert-butyl-1,1'-biphenyl: ¹H NMR(400 MHz, CDCl₃) δ (ppm) 7.57 (d, *J*=8.4 Hz, 1H), 7.49 (d, *J*=8.4 Hz, 1H), 1.405 (s, 4.5H)

Abs. | 0.01

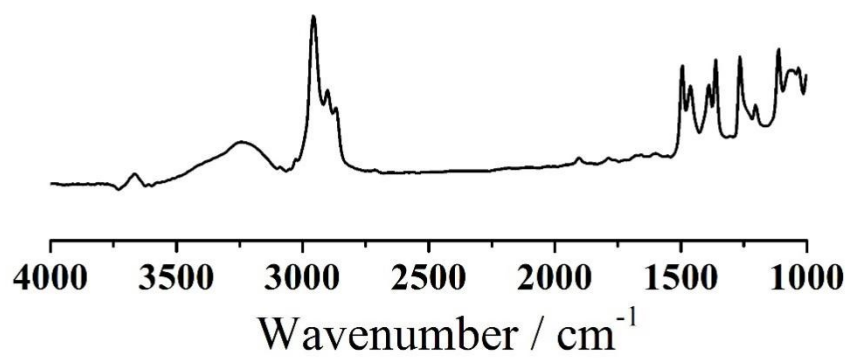


Figure S6. Infrared spectrum of the product obtained from the reaction conditions described in entry 6-7, table 2 in the manuscript.

Abs. | 0.01

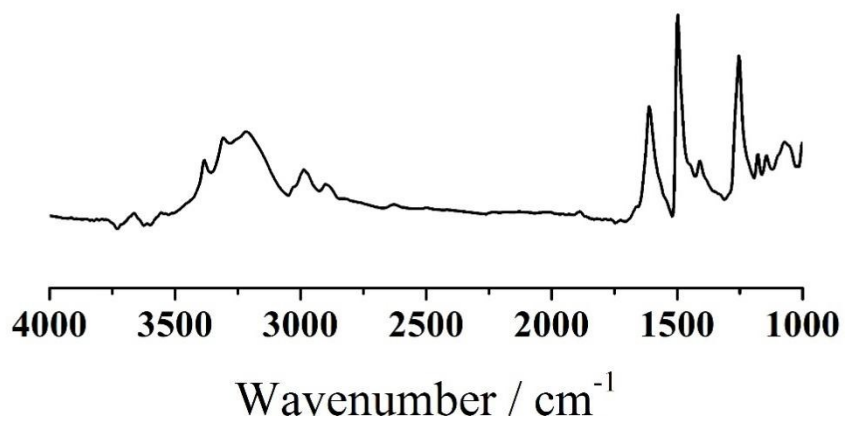


Figure S8. Infrared spectrum of the product obtained from the reaction conditions described in entry 10-11, table 2 in the manuscript.

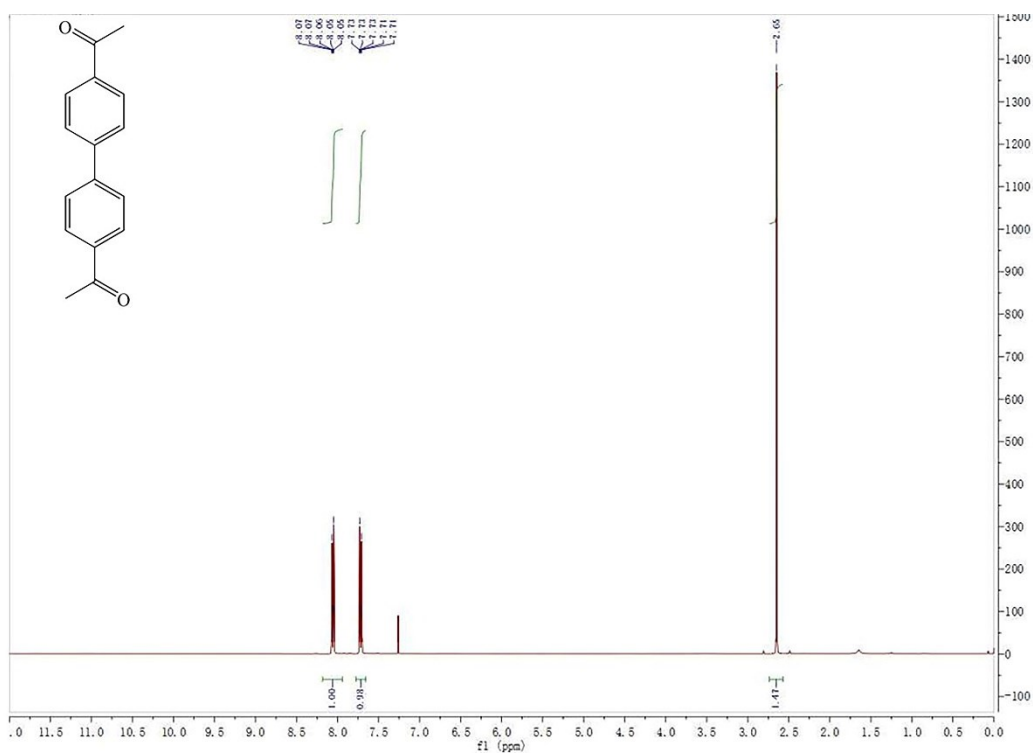


Figure S9. The ¹H NMR spectra of the product obtained from the reaction conditions described in entry 12-13, table 2 in the manuscript.

4,4'-Diacetylbiphenyl: ¹H NMR(400 MHz, CDCl₃) δ (ppm) 8.05-8.07 (m, 1H)
7.71-7.73 (m, 1H) 2.65 (s, 1.5H)

Abs. | 0.02

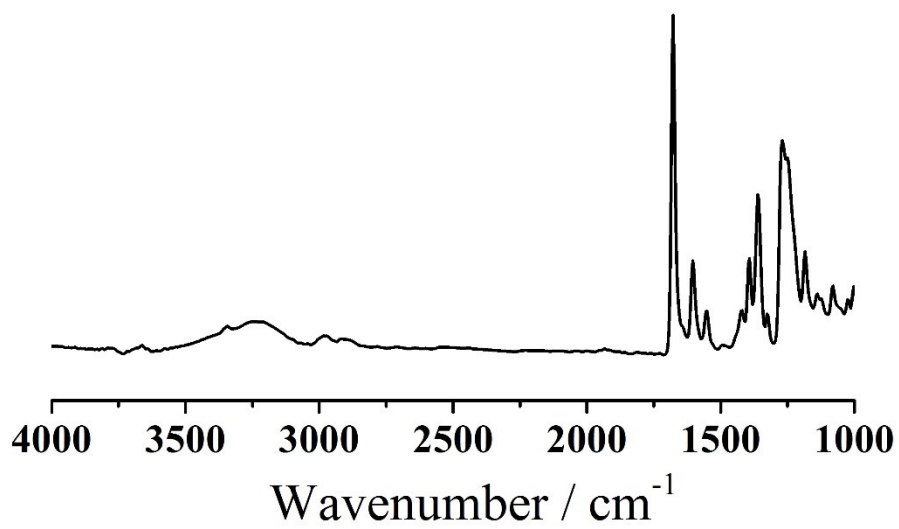
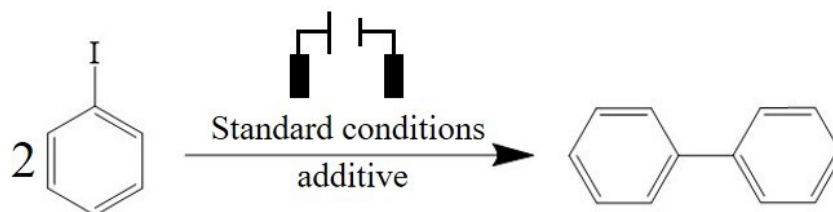


Figure S10. Infrared spectrum of the product obtained from the reaction conditions described in entry 12-13, table 2 in the manuscript.

Table S1. The result with additional dose of 2,6-di-tert-butyl-4-methylphenol (BHT, 1.0 equiv.) as the radical scavenger during the electrochemical coupling of PhI under the same reaction conditions described in entry 7, Table 1 in the manuscript.



additive	Q	yield
BHT (1.0 equiv)	3.9 F mol ⁻¹	12 %

Table S2. Comparison of catalysis performance on palladium-based catalysts on biphenyl synthesis.

Catalyst	Solvent	Base	T (°C)	Time (h)	Yield (%)	Ref.
Pd–Au ₃ /HT	IPA	KOH	40	2	97	[1]
Pd/CN-C	EtOH	K ₂ CO ₃	30 (Ar)	5	94	[2]
Pd @CAP	DMF	K ₂ CO ₃	r.t. (MW,400W)	6 mins	92	[3]
Pd/MOF-253	DMSO/EtOH	KOAc	120 (N ₂)	10	99	[4]
FLA-Pd	H ₂ O	K ₂ CO ₃	reflux	1	93	[5]
Pd@Fe ₃ O ₄ -lignin	EtOH/H ₂ O	K ₂ CO ₃	90	2.3	95	[6]
Pd/KB nanocomposite	EtOH/H ₂ O	K ₂ CO ₃	90	2.5	91	[7]
Mesh-GO/Pd	EtOH/H ₂ O	K ₂ CO ₃	90	1.5	97	[8]
Pd@Fe ₃ O ₄ /CS-AG	H ₂ O	K ₂ CO ₃	r.t. (MW,400W)	5 mins	96	[9]
Pd@Kao/Fe ₃ O ₄ /Pyr	—	K ₂ CO ₃	r.t. (MW,400W)	6 mins	95	[10]
PdB/C	MeCN	—	r.t.	6	77	This Work

References:

- [1] J. Wang, A. Xu, M. Jia, S. Bai, X. Cheng and B. Zhaorigetu, *New J. Chem.*, 2017, **41**, 1905-1908.
- [2] Q. Jia, S. Zhang and Q. Gu, *J. Energy Chem.*, 2019, **30**, 152-161.
- [3] T. Baran and M. Nasrollahzadeh, *Int. J. Biol. Macromol.*, 2020, **148**, 565-573.
- [4] L. Chen, Z. Gao and Y. Li, *Catal. Today*, 2015, **245**, 122-128.
- [5] M. Nasrollahzadeh, Z. Issaabadi and R.S. Varma, *ACS Omega*, 2019, **4**, 14234-14241.
- [6] M. Nasrollahzadeh, N.S.S. Bidgoli, Z. Issaabadi, Z. Ghavamifar, T. Baran and R. Luque, *Int. J. Biol. Macromol.*, 2020, **148**, 265-275.
- [7] B. Feizi Mohazzab, B. Jaleh, M. Nasrollahzadeh and Z. Issaabadi, *Catal. lett.*, 2018, **149**, 169-179.
- [8] B. Feizi Mohazzab, B. Jaleh, Z. Issaabadi, M. Nasrollahzadeh and R.S. Varma, *Green Chem.*, 2019, **21**, 3319-3327.
- [9] T. Baran and M. Nasrollahzadeh, *Carbohydr. Polym.*, 2019, **222**, 115029.
- [10] T. Baran and M. Nasrollahzadeh, *J. Phys. Chem. Solids*, 2020, **146**, 109566.