Supporting Information:

One-pot hydrodeoxygenation of biomass furan derivatives into decane under mild conditions over Pd/C combined with phosphotungstic acid

Song Li, [abcd] Long Yan, *[abc] Qiying Liu, [abc] Jianguo Liu, [abc] Qiyu Liu,[e] Wei Fan,[e] Xuelai Zhao,[f] Xinghua Zhang, [abc] Chenguang Wang, [abc] Longlong Ma, [abc] Qi Zhang*[abc]

[a]Guangzhou Institute of Energy Conversion, Chinese Academy of Sciences, Guangzhou 510640, PR China. E-mail: zhangqi@ms.giec.ac.cn; yanlong@ms.giec.ac.cn; Fax: +86 20 87057789; Tel: +86 20 87057789.

[b]CAS Key Laboratory of Renewable Energy, Guangzhou 510640, PR China.

[c]Guangdong Provincial Key Laboratory of New and Renewable Energy Research and Development, Guangzhou 510640, PR China.

[d]University of Chinese Academy of Sciences, Beijing 100049, PR China.

[e]Department of Chemical Engineering, University of Massachusetts-Amherst, 159 Goessman Lab, 686 N Pleasant Street, Amherst, 01003, USA.

[f]Department of Thermal Science and Energy Engineering, University of Science and Technology of China, Hefei, 233022, China.

Contents page

Preparation details

Preparation of FDLA[1]: 18.6 g LA was neutralized with 24.0 g Na_2CO_3 at room temperature in 100 ml deionized water. The solution was heated to refluxed under stirring and then added with a mixture of 7.7 g furfural in 16 ml ethanol dropwise. It was cooled in ice water after no furfural was detected. The cooled solution was slowly poured into excessive diluted hydrochloric acid to collect the solid condensation products. FDLA was obtained by recrystallization of the solid products from hot water.

Preparation of HPW-300: HPW-300 was prepared by calcination of HPW at 300 ^oC for 4 h in air.

Preparation of Pd/$C_{Reduced}$: 50 mg Pd/C was reduced in 10 ml cyclohexane under 3 MPa H_2 at 170 ^oC for 4 h. Pd/$C_{Reduced}$ was obtained after evaporation of solvent.

Preparation of Pd/$C_{Reduced}$-HPW(50:50): 50 mg Pd/C was reduced in 10 ml cyclohexane under 3 MPa H_2 at 170 ^oC for 4 h. 50 mg HPW was added after cooling to room temperature. The sealed reactor was purged with H_2 four times and charged with 3 MPa H_2. Then, the reactor was heated to 170 ^oC and kept for 4 h. Pd/$C_{Reduced}$-HPW(50:50) was obtained after evaporation of solvent.

HDO of lactones at different conditions

Table S1 HDO of lactones at different conditions[a]

Entry	Model compounds	Pd/C (mg)	HPW (mg)	Temperature (°C)	Conversion (%)	Yield (%)							
						Nonane	Decane	Decanoic alcohol	Decanoic acid	γ-Decalactone	Decanoic ether	Decanoic ester	Sum
1	DC[b]	/	50	170	65.83	0	0	0	0	0	0	0	0
2	γ-Decalactone	/	50	170	27.54	0	0	0	1.05	-	0	0	1.05
3	DC	50	/	170	53.14	0	0	0	0	0.71	0	0	0.71
4	γ-Decalactone	50	/	170	24.96	0	0	0	1.66	-	0	0	1.66
5	DC	50	50	170	100	3.08	94.58	0	0	0	0	0	97.66
6	γ-Decalactone	50	50	170	100	3.15	95.09	0	0	-	0	0	98.24
7	γ-Decalactone	50	50	160	100	1.58	33.84	1.27	11.98	-	19.14	27.32	95.13
8	γ-Decalactone	50	50	150	100	0.89	12.66	1.07	23.17	-	21.82	36.96	96.57
9	γ-Decalactone	50	50	140	100	0.47	8.28	0.77	37.11	-	13.99	35.82	96.44
10	γ-Decalactone	50	50	130	100	0	3.54	0	58.41	-	0	5.57	67.51
11	γ-Decalactone	25	50	170	100	2.11	17.47	0.84	24.05	-	11.16	27.43	83.06
12	γ-Decalactone	50	25	170	100	2.86	15.71	0.80	23.65	-	14.69	31.49	89.20

[a]Reaction conditions: lactones (0.515 mmol), cyclohexane (10 mL), 3 MPa H_2 at room temperature, stirred at 400 rpm, reacted at desired temperature for 4 h; [b]DC: 5-(2-(tetrahydrofuran-2-yl)ethyl)-dihydrofuran-2(3H)-one.

TEM/EDS images of catalysts

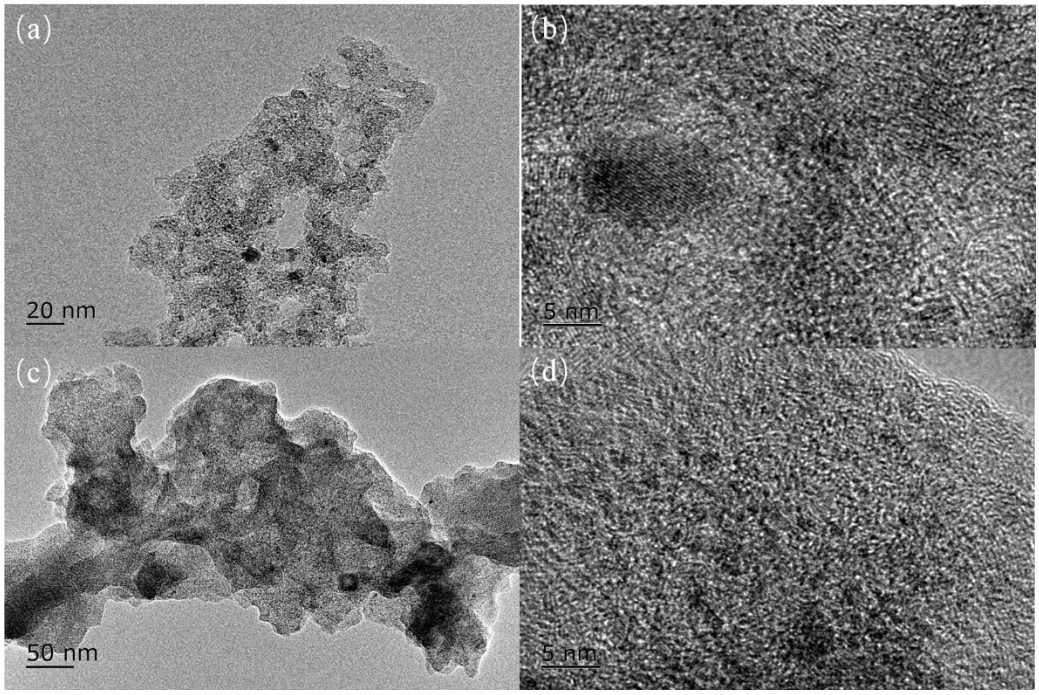


Figure S1 TEM images of Ru/C (a, b) and Ru/C-HPW (50:50) (c, d) catalysts

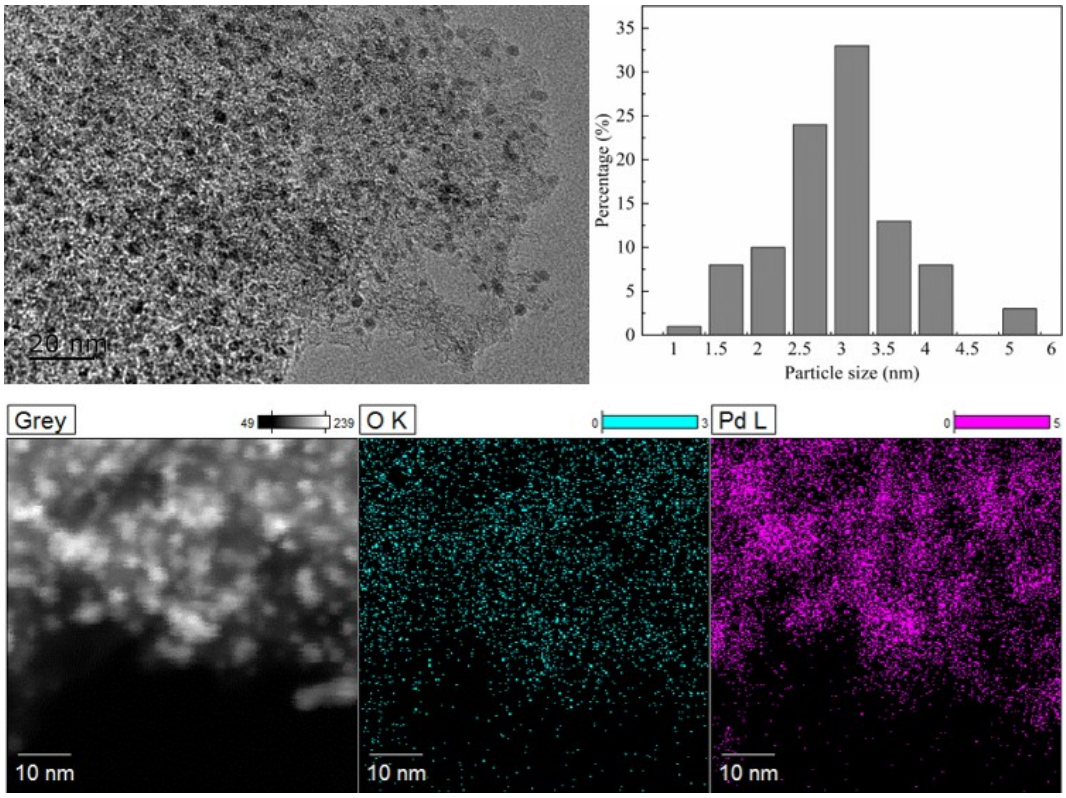


Figure S2 TEM/EDS images of Pd/C catalyst

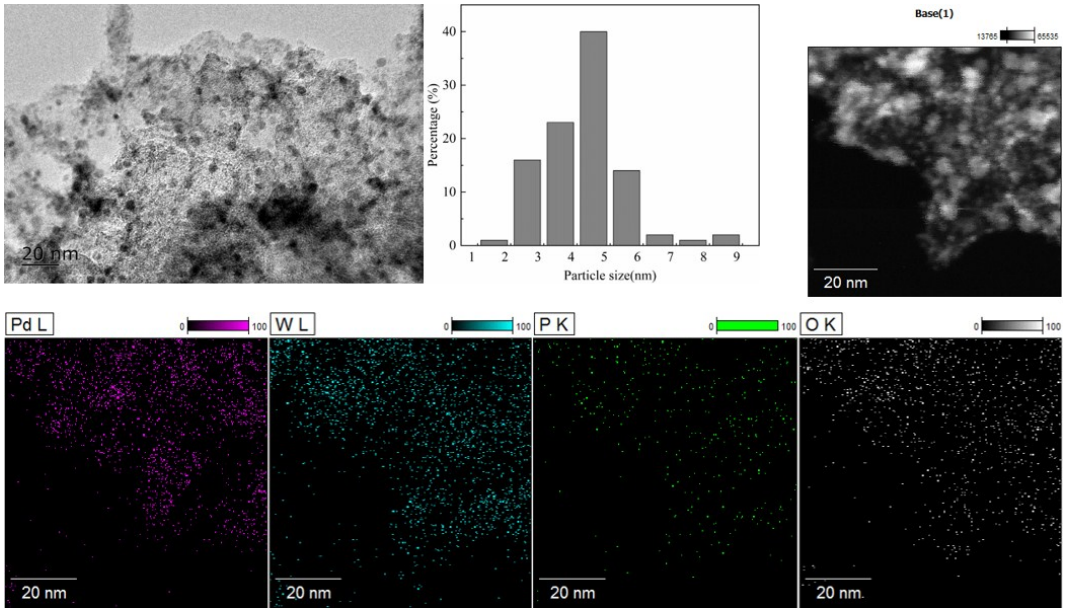


Figure S3 TEM/EDS images of Pd/C-HPW (50:50) catalyst

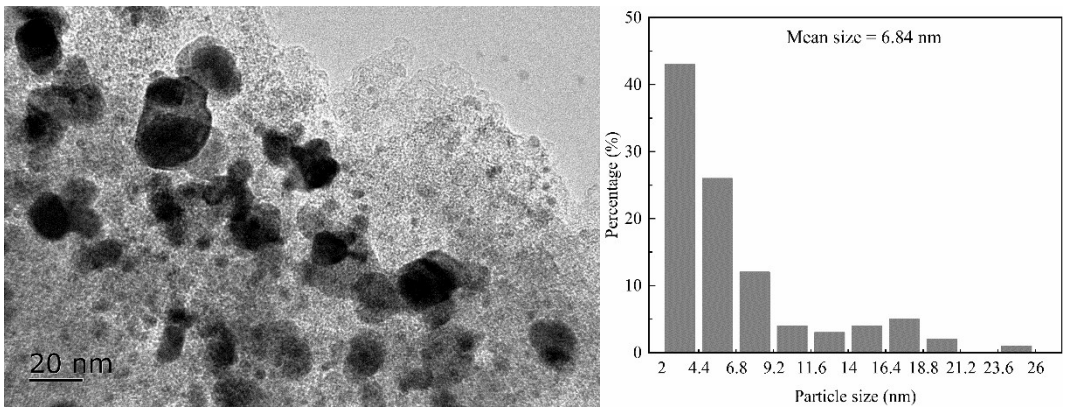


Figure S4 TEM image of Pd/$C_{\mathbf{Reduced}}$ catalyst

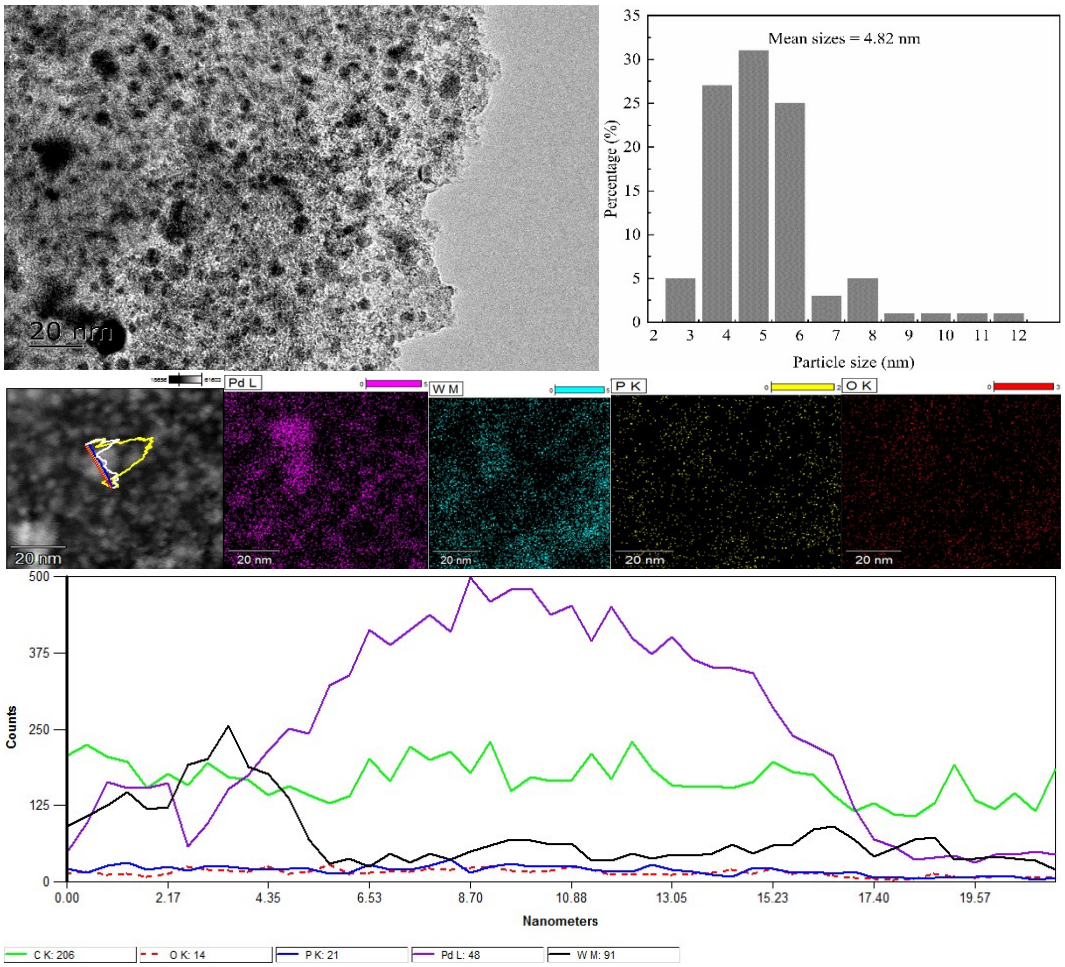


Figure S5 TEM/EDS images of Pd/$C_{\mathbf{Reduced}}$-HPW (50:50) catalyst

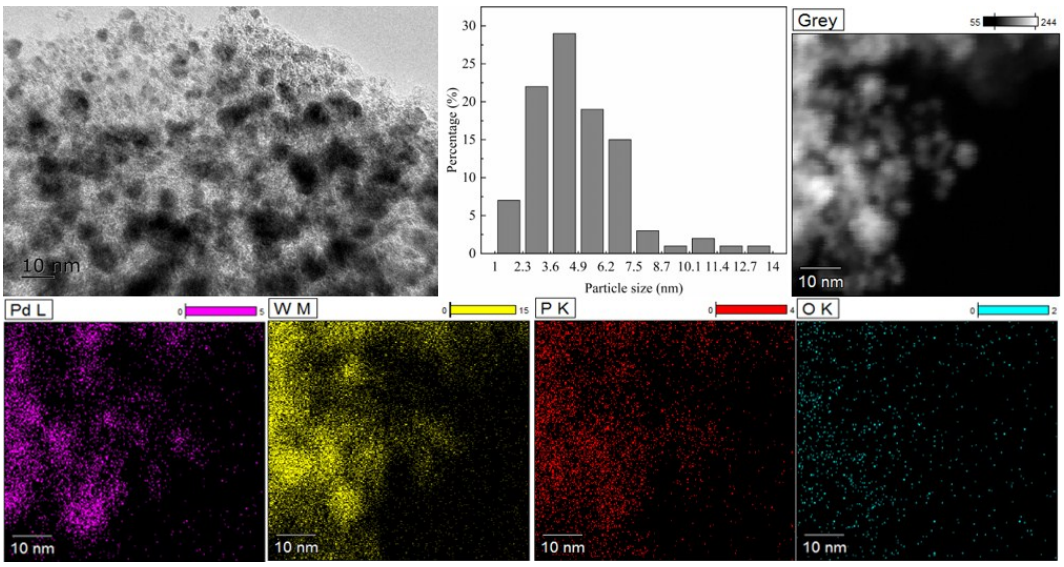


Figure S6 TEM/EDS images of recycled Pd/C-HPW (50:50) catalyst

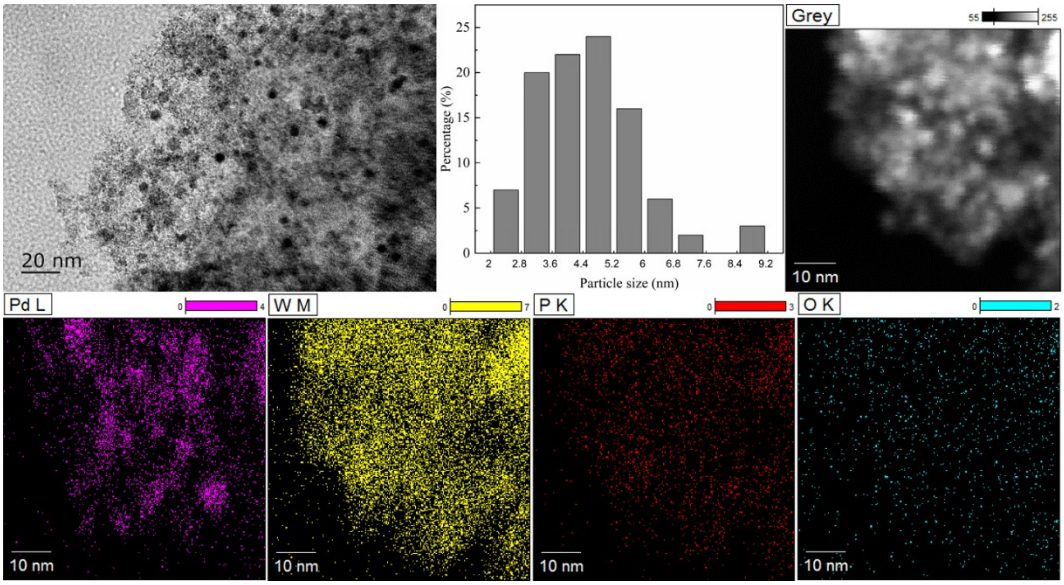
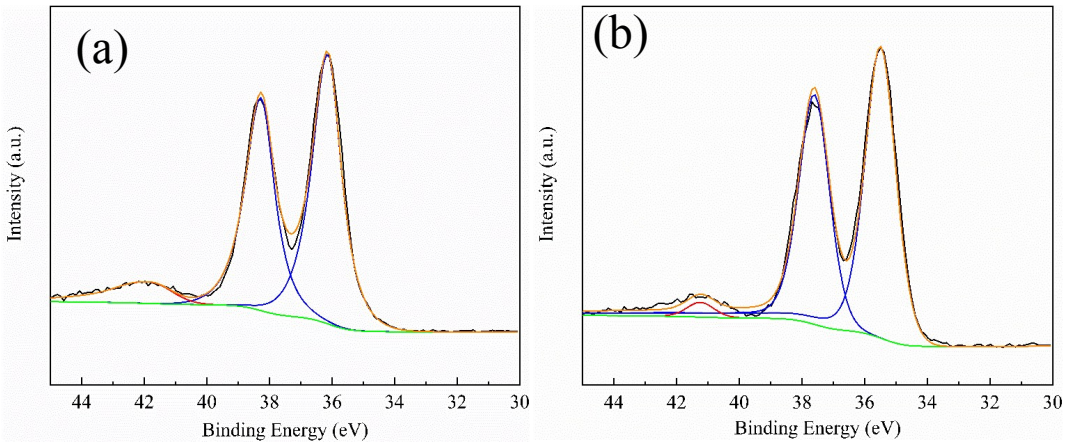


Figure S7 TEM/EDS images of recycled Pd/C-HPW (50:50) catalyst washed with water and ethanol



W 4f XPS spectra of HPW and Pd/C-HPW (50:50) catalysts

Figure S8 W 4f XPS spectra of HPW (a) and Pd/C-HPW (50:50) (b)catalysts

XRD spectra of fresh/used Pd/C and Pd/C-HPW (50:50) catalysts

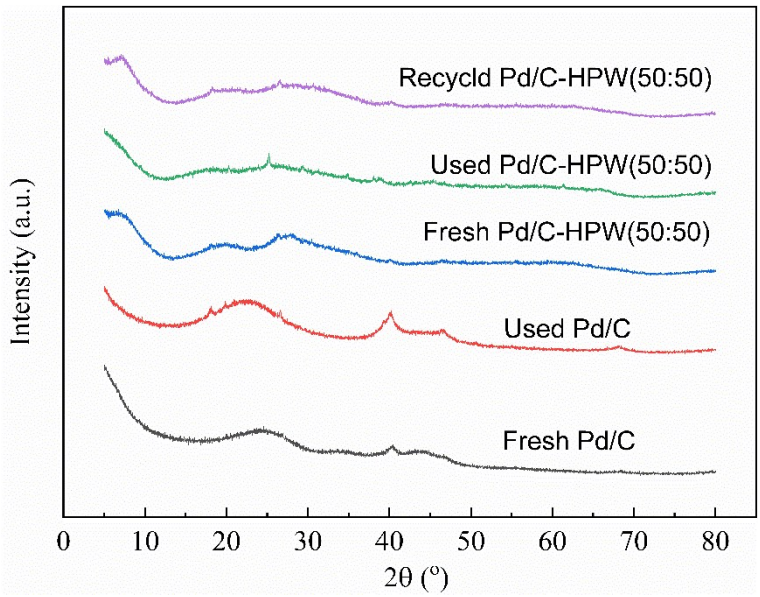


Figure S9 XRD spectra of fresh/used Pd/C and Pd/C-HPW (50:50) catalysts

NMR spectra of FDLA/DC/γ-decalactone

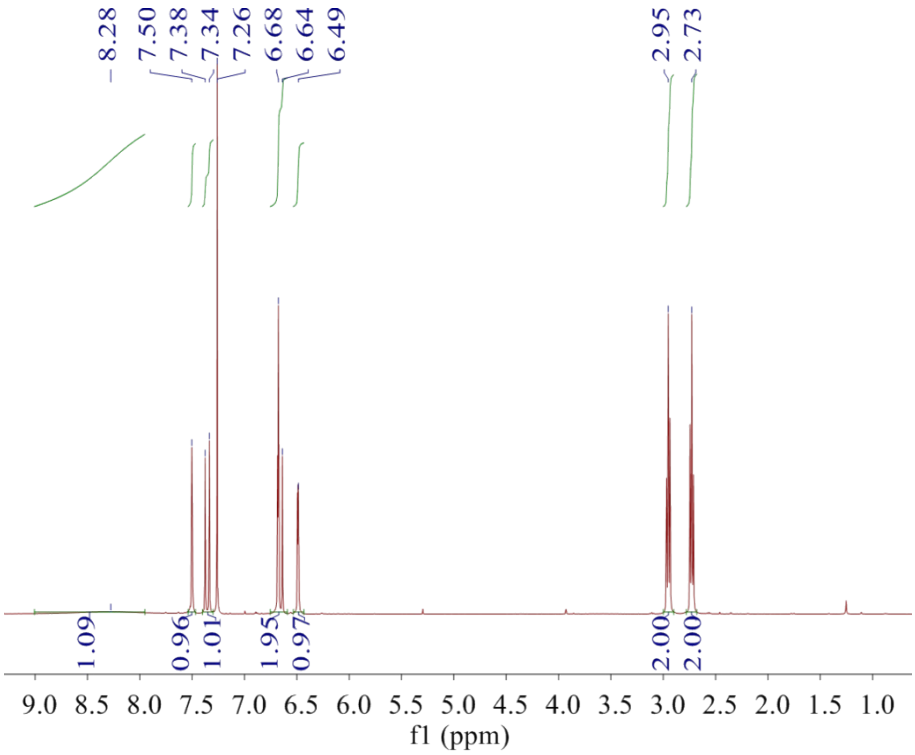


Figure S10 ^{1}H-NMR spectra of FDLA

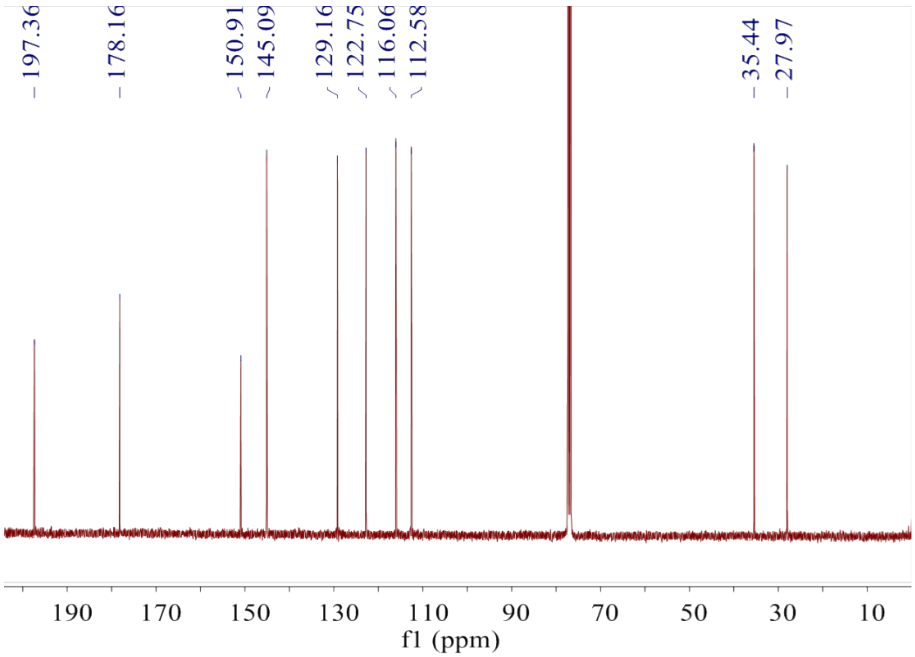


Figure S11 ^{13}C-NMR spectra of FDLA

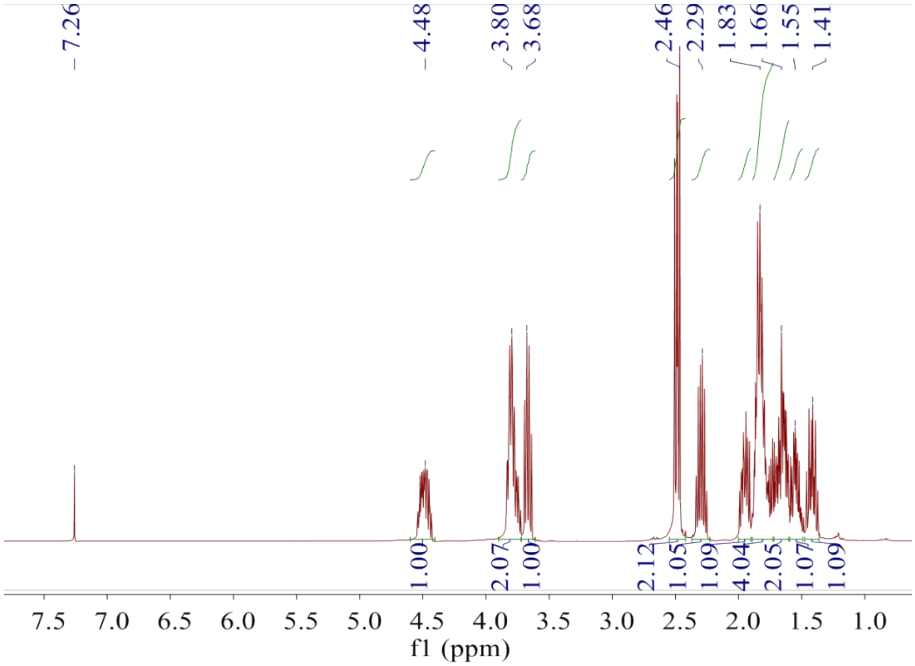


Figure S12 ^{1}H-NMR spectra of DC

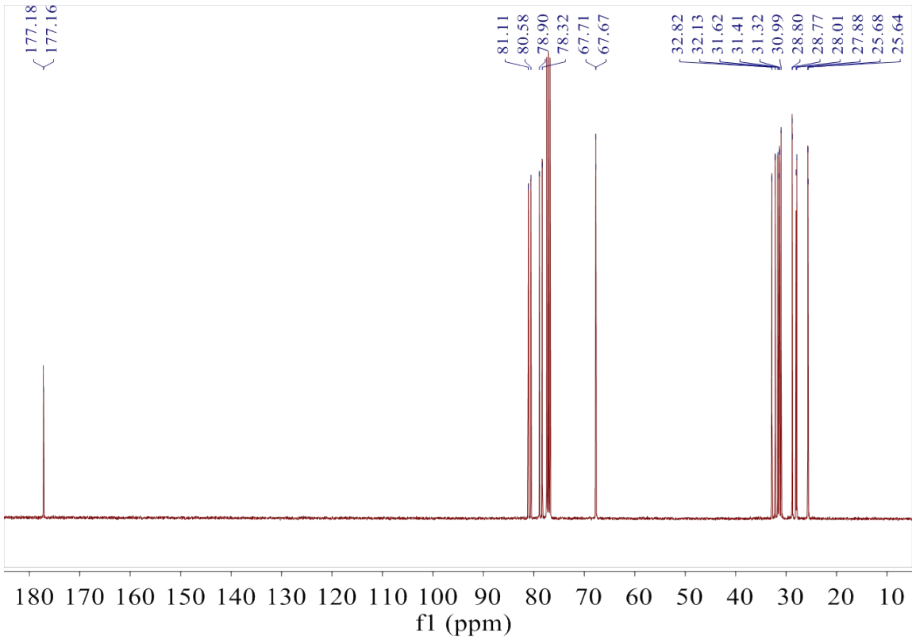


Figure S13 ^{13}C-NMR spectra of DC

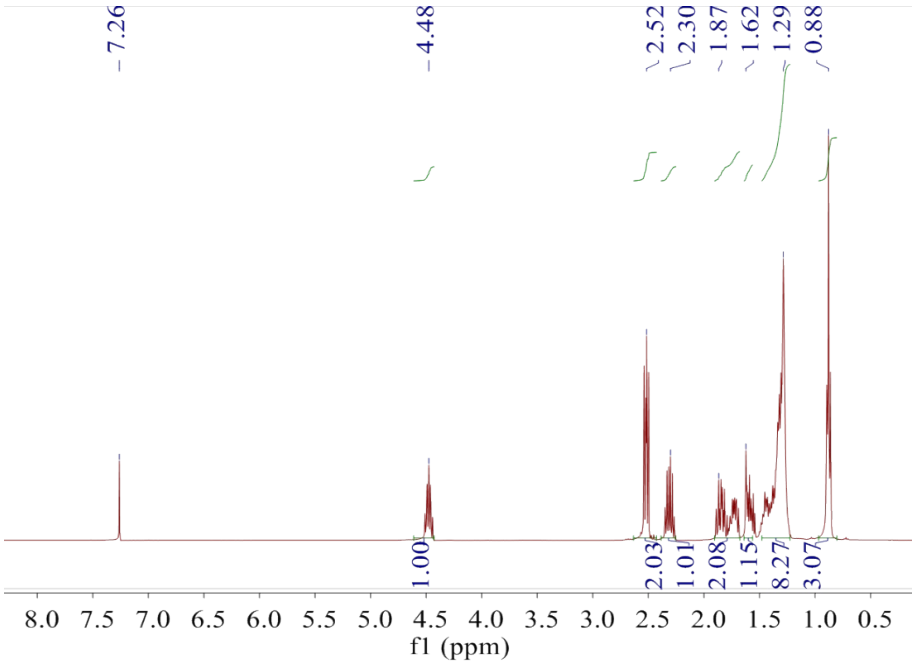


Figure S14 ^{1}H-NMR spectra of γ-decalactone

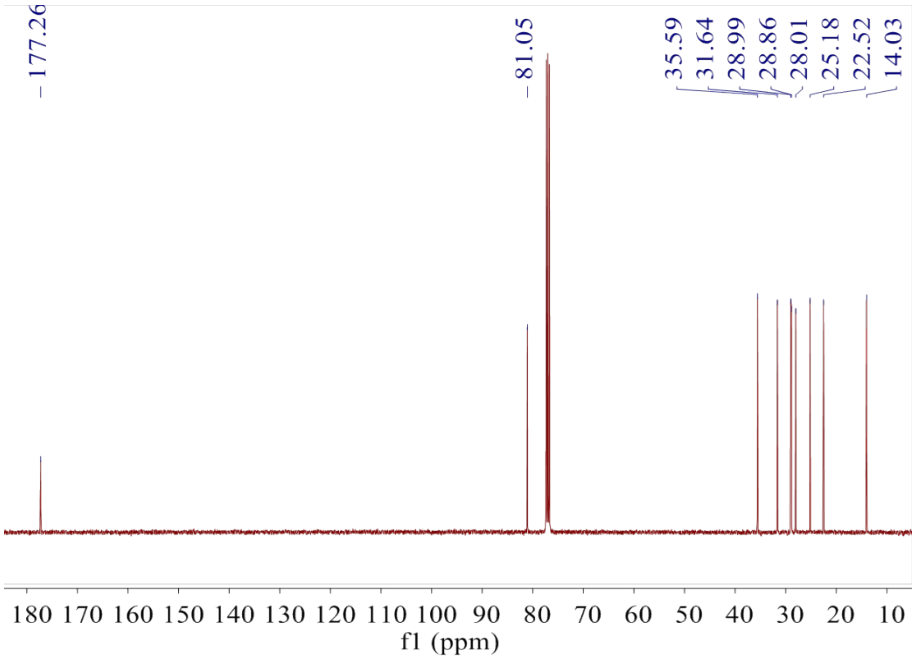


Figure S15 ^{13}C-NMR spectra of γ-decalactone

References

Reference：
1. Y. Hachihama and I. Hayashi, 1954, **13**, 201-209.