
Supporting Information

**Facile and selective hydrogenolysis of β -O-4
linkages in lignin catalyzed by Pd-Ni
bimetallic nanoparticles supported on ZrO_2**

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1. General Considerations

All chemical reagents are obtained from commercial suppliers and used without further purification. ^1H NMR and ^{13}C NMR spectra are recorded on an AVANCE III 500 Bruker spectrometer operating at 500 MHz and 125 MHz in CDCl_3 , respectively, and chemical shifts were reported in ppm relative to the center of the singlet at 7.27 ppm for CDCl_3 . GC-MS was performed on an ISQ Trace 1300 in the electron ionization (EI) mode. GC analyses are performed on an Agilent 7890A instrument (Column: Agilent 19091J-413: 30 m \times 320 μm \times 0.25 μm , carrier gas: H_2 , FID detection. Transmission electron microscopy (TEM) images were taken using a PHILIPS Tecnai 12 microscope operating at 120kv. Scanning electron microscope (SEM) was performed on field-emission scanning electron microscopy (FESEM, HITACHI S-4800) and an energy-dispersive X-ray spectroscopy (EDS, Oxford instruments X-Max). X-ray photoelectron spectroscopy (XPS) were performed on a ESCALAB 250Xi spectrometer, using a Al $\text{K}\alpha$ X-ray source (1350 eV of photons) and calibrated by setting the C 1s peak to 284.80 eV. Inductively coupled plasma mass spectrometry (ICP-MS) was analyzed on Optima 7300 DV.

2. Catalysts Preparation

Nanoparticles immobilized on ZrO_2 were prepared by impregnation-reduction method, and the composition of the bimetallic nanoparticles was controlled by adjusting the ratio of the metal precursors. ZrO_2 was chosen as the support because it appears to favor activation of O-compounds on surface.^{1,2} In a typical procedure, Pd_1Ni_8 BMNPs supported on ZrO_2 were prepared as following: 2.0 g ZrO_2 was dispersed into 50 mL aqueous solution of metal precursors (0.15 mmol PdCl_2 and 1.2 mmol $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$) under ultrasonic. Lysine aqueous solution (0.53 M) was then added into the mixture with vigorous stirring for 30 min. To this suspension, NaBH_4 aqueous solution (0.05 M) was added dropwise, the color of the mixture would turn to black immediately indicating the formation of metal particles, the mixture was further stirred for 30 min and then aged for 24 h. Finally, the solid was separated, washed (water and ethanol) and dried at room temperature under *vacuum*.

3. Hydrogenolysis Reactions of Lignin Model Compounds

Model compounds were synthesized following a modified literature procedure.³ In a typical hydrogenolysis procedure, the catalyst was added into a reactor (10 mL) equipped with a magnetic

stirrer, then the hydrogen donor was induced (NaBH_4 was added directly and H_2 was provided using a hydrogen balloon). Subsequently, the substrate (0.5 mmol) dissolved in 1 mL solvent was injected into the reactor by a syringe, then, the reactor was placed into a preheated oil bath and stirred for adequate time. After the reaction, the reactor was quenched to ambient temperature using cooling water, the catalyst was collected by filtration and washed with water (2×5 mL) and ethanol (2×5 mL), and filtrate was extracted using ethyl acetate (3×5 mL). The organic fractions were combined and dried over anhydrous sodium sulfate, solvent was removed under *vacuum* at room temperature, and the obtained residue was analyzed by GC/MS.

4. Catalyst recycle

For the hydrogenolysis of model compounds, the catalyst can be easily separated by simple filtration and directly reused in the next cycle after washed with water and ethanol. For the hydrogenolysis of lignin, the catalyst can be reused after separated by filtration and washed with EtOAc and MeCN.

5. Catalyst Characterization

Inductively coupled plasma mass spectrometry (ICP-MS) was employed to analyze the metal loading of the catalyst. The Pd-Ni BMNPs were directly observed by transmission electron microscopy (TEM) and scanning electron microscope (SEM). Area-selected EDS elemental mapping was performed to determine the Ni, Pd, O element distribution in the Pd-Ni BMNPs catalyst. X-ray photoelectron spectroscopy (XPS) was applied to analyze the valence state of metal element and the surface chemical composition of the Pd-Ni/ ZrO_2 catalyst.

6. ICP-MS results of $\text{Pd}_1\text{Ni}_8/\text{ZrO}_2$ catalyst

Table S1. ICP-MS analysis of the as prepared $\text{Pd}_1\text{Ni}_8/\text{ZrO}_2$ catalyst.

Nanoparticles	Metal Content(mg/L)		%	
	Pd	Ni	Pd	Ni
Pd_1Ni_8	2.011	8.793	11.2	88.8

7. XPS spectrum of Pd₁Ni₈/ZrO₂ catalyst in O1S region

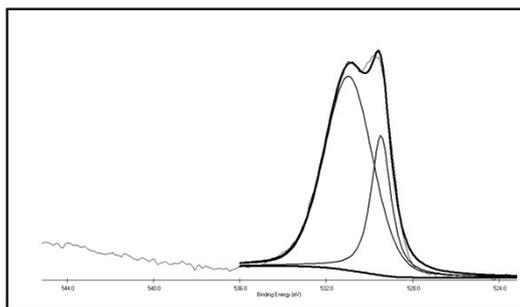
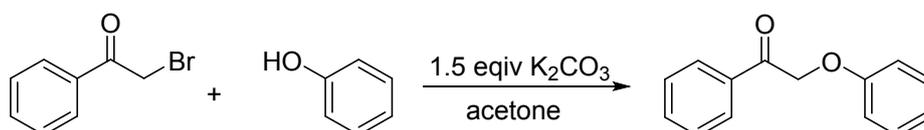


Fig. S1 XPS spectrum of as-prepared Pd₁Ni₈/ZrO₂ catalyst in O1S region

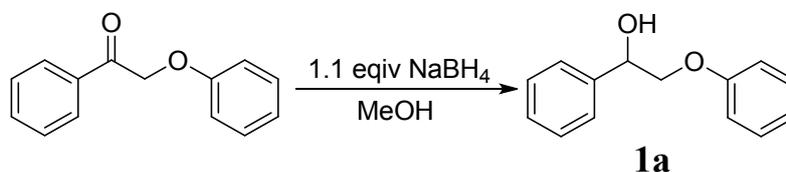
8. Synthesis of substrates

All substrates have been prepared according to literature procedures with minor modifications.^{4,5}

Synthesis of 2-phenoxy-1-phenylethanol (**1a**)



A 250 mL round bottom flask equipped with a reflux condenser and a dropping funnel was charged with phenol (0.520 g, 5.5 mmol) and K₂CO₃ (1.040 g, 7.6 mmol) in acetone (50 mL) and stirred at RT. To this solution, 2-bromoacetophenone (1.000 g, 5.0 mmol) in acetone (50 mL) was added dropwise over 30 min at RT. The resulting suspension was stirred at reflux for 4 h, after the suspension was filtered and concentrated in *vacuo*. The crude product was purified by recrystallization from petroleum ether to give 2-phenoxy-1-phenylethanone as a white solid (1.050 g, 4.9 mmol) in 98% yield.

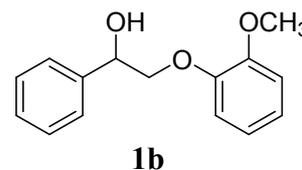


A round bottom flask was charged with **1b** (1.7 g, 8.0 mmol) and methanol (60 mL). Sodium borohydride (0.33 g, 8.8 mmol) was added to the solution in small portions at 25 °C. After stirring for 4 h, the suspension was quenched with saturated aqueous NH₄Cl (150 mL), followed by the addition of ethyl acetate (150 mL). After separation, the organic phase was washed with H₂O (2 x 100 mL), dried over MgSO₄, filtered and the solvent was evaporated under vacuum. The crude product was purified by recrystallization from hexane to give a white crystalline in 96% yield. ¹H NMR (500 MHz, CDCl₃) δ

7.50 – 7.45 (m, 2H), 7.41 (dd, $J = 8.1, 6.7$ Hz, 2H), 7.38 – 7.33 (m, 1H), 7.33 – 7.27 (m, 2H), 6.99 (t, $J = 7.4$ Hz, 1H), 6.96 – 6.91 (m, 2H), 5.14 (dt, $J = 8.8, 2.5$ Hz, 1H), 4.14 – 4.00 (m, 2H), 2.89 (d, $J = 2.4$ Hz, 1H). ^{13}C NMR (126 MHz, CDCl_3) δ 158.54 (s), 139.82 (s), 129.73 (s), 128.73 (s), 128.35 (s), 126.45 (s), 121.47 (s), 114.80 (s), 77.33 (d, $J = 31.9$ Hz), 76.95 (s), 76.87 – 76.44 (m), 73.45 (s), 72.74 (s).

2-(2-methoxyphenoxy)-1-phenylethanol (1b) was prepared in 93% yield.

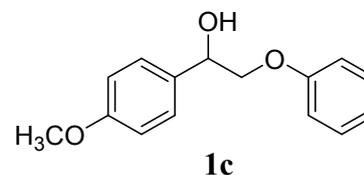
^1H NMR (500 MHz, CDCl_3) δ 7.50 – 7.44 (m, 2H), 7.39 (dd, $J = 8.1, 6.7$ Hz, 2H), 7.34 (dt, $J = 9.4, 4.3$ Hz, 1H), 7.01 (ddd, $J = 8.0, 7.0, 1.9$ Hz, 1H), 6.98 – 6.87 (m, 3H), 5.14 (d, $J = 9.3$ Hz, 1H), 4.20 (dd, $J = 10.0, 2.9$ Hz, 1H), 4.02 (t, $J = 9.7$ Hz, 1H), 3.88 (s, 3H), 3.72 (d, $J = 2.3$ Hz, 1H).



^{13}C NMR (126 MHz, CDCl_3) δ 150.23 (s), 148.15 (s), 139.78 (s), 128.63 (s), 128.15 (s), 126.46 (s), 122.63 (s), 121.24 (s), 116.00 (s), 112.16 (s), 77.48 (s), 77.10 (d, $J = 31.9$ Hz), 76.36 (s), 72.48 (s), 55.99 (s).

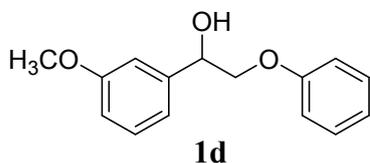
1-(4-methoxyphenyl)-2-phenoxyethanol (1c) was prepared in 95% yield.

^1H NMR (500 MHz, CDCl_3) δ 7.40 (d, $J = 8.6$ Hz, 2H), 7.31 (dd, $J = 8.6, 7.4$ Hz, 2H), 7.00 (t, $J = 7.4$ Hz, 1H), 6.97 – 6.89 (m, 4H), 5.11 – 5.06 (m, 1H), 4.09 (dd, $J = 9.6, 3.3$ Hz, 1H), 4.02 (t, $J = 9.2$ Hz, 1H), 3.84 (s, 3H), 2.90 (d, $J = 2.3$ Hz, 1H). ^{13}C NMR (126



MHz, CDCl_3) δ 159.67 (s), 158.58 (s), 132.00 (s), 129.71 (s), 127.73 (s), 121.41 (s), 114.80 (s), 114.13 (s), 77.47 (s), 77.09 (d, $J = 31.9$ Hz), 73.43 (s), 72.32 (s), 55.46 (s).

1-(3-methoxyphenyl)-2-phenoxyethanol (1d) was prepared in 94% yield.

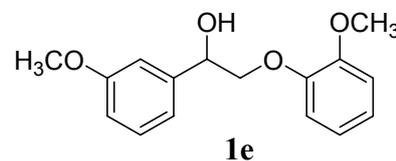


^1H NMR (500 MHz, CDCl_3) δ 7.40 (d, $J = 8.6$ Hz, 2H), 7.31 (dd, $J = 8.6, 7.4$ Hz, 2H), 7.00 (t, $J = 7.4$ Hz, 1H), 6.97 – 6.89 (m, 4H), 5.11 – 5.06 (m, 1H), 4.09 (dd, $J = 9.6, 3.3$ Hz, 1H), 4.02 (t, $J = 9.2$ Hz, 1H), 3.84 (s, 3H), 2.90 (d, $J = 2.3$ Hz, 1H). ^{13}C NMR (126

MHz, CDCl_3) δ 159.67 (s), 158.58 (s), 132.00 (s), 129.71 (s), 127.73 (s), 121.41 (s), 114.80 (s), 114.13 (s), 77.47 (s), 77.09 (d, $J = 31.9$ Hz), 73.43 (s), 72.32 (s), 55.46 (s).

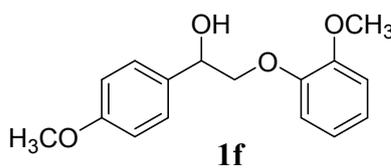
2-(2-methoxyphenoxy)-1-(3-methoxyphenyl) ethanol (1e) was prepared in 92% yield. ^1H NMR

(500 MHz, CDCl_3) δ 7.26 (t, $J = 7.9$ Hz, 1H), 6.98 (ddd, $J = 10.0, 8.6, 1.9$ Hz, 3H), 6.92 – 6.81 (m, 4H), 5.09 (dd, $J = 9.2, 2.5$ Hz,



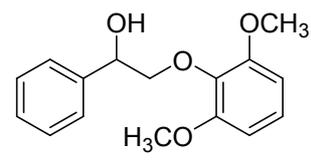
1H), 4.14 (dd, $J = 10.0, 2.9$ Hz, 1H), 3.97 (t, $J = 9.6$ Hz, 1H), 3.84 (s, 3H), 3.79 (s, 3H), 3.64 (d, $J = 4.9$ Hz, 1H). ^{13}C NMR (126 MHz, CDCl_3) δ 159.92 (s), 149.97 (s), 148.12 (s), 141.54 (s), 129.63 (s), 122.44 (s), 121.29 (s), 118.75 (s), 115.52 (s), 113.78 (s), 111.96 (d, $J = 36.6$ Hz), 78.04 – 76.98 (m), 76.98 – 76.74 (m), 76.11 (s), 72.36 (s), 55.99 (s), 55.39 (s).

2-(2-methoxyphenoxy)-1-(4-methoxyphenyl) ethanol (1f) was prepared in 91% yield. ^1H NMR

 (500 MHz, CDCl_3) δ 7.35 (t, $J = 5.7$ Hz, 2H), 6.99 – 6.94 (m, 1H), 6.93 – 6.87 (m, 5H), 5.07 (dt, $J = 9.2, 2.5$ Hz, 1H), 4.13 – 4.10 (m, 1H), 3.98 (t, $J = 9.6$ Hz, 1H), 3.83 (s, 3H), 3.79 (s, 3H), 2.04 (s, 1H). ^{13}C NMR (126 MHz, CDCl_3) δ 159.51 (s), 150.11

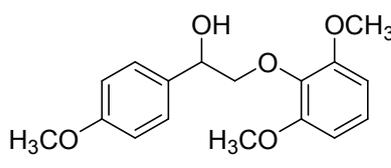
(s), 148.21 (s), 132.05 (s), 127.73 (s), 127.00 (s), 122.42 (s), 121.22 (s), 115.67 (s), 114.02 (s), 112.14 (s), 78.11 – 76.92 (m), 77.04 (s), 77.04 (s), 76.14 (s), 72.05 (s), 55.95 (s), 55.41 (s), 21.17 (s), 14.35 (s).

2-(2,6-dimethoxyphenoxy)-1-phenylethan-1-ol (1g) was prepared in 93% yield. ^1H NMR (500

 MHz, CDCl_3) δ 7.40 (d, $J = 7.6$ Hz, 2H), 7.34 (t, $J = 7.5$ Hz, 2H), 7.30 – 7.25 (m, 1H), 7.04 (t, $J = 8.4$ Hz, 1H), 6.60 (dd, $J = 15.5, 8.4$ Hz, 2H), 4.97 (d, $J = 9.7$ Hz, 1H), 4.55 (s, 1H), 4.42 (dd, $J = 11.0, 2.6$ Hz, 1H), 3.88 (s, 6H), 3.74 (t, $J = 10.5$ Hz, 1H). ^{13}C NMR (126 MHz, CDCl_3) δ

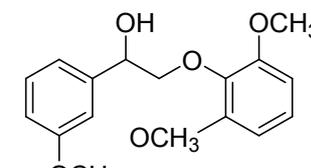
153.39 (s), 139.58 (s), 136.91 (s), 128.45 (s), 127.82 (s), 126.44 (s), 124.24 (s), 105.21 (d, $J = 27.6$ Hz), 80.16 (s), 77.45 (s), 77.07 (d, $J = 31.9$ Hz), 72.56 (s), 56.23 (s).

2-(2,6-dimethoxyphenoxy)-1-(4-methoxyphenyl)ethan-1-ol (1h) was prepared in 91% yield. ^1H

 NMR (500 MHz, CDCl_3) δ 7.33 (d, $J = 8.6$ Hz, 2H), 7.04 (t, $J = 8.4$ Hz, 1H), 6.88 (d, $J = 8.6$ Hz, 2H), 6.62 (d, $J = 8.4$ Hz, 2H), 4.93 (d, $J = 9.8$ Hz, 1H), 4.54 (s, 1H), 4.40 (dd, $J = 10.9, 2.5$ Hz, 1H), 3.88 (s, 6H), 3.79 (s, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ

159.33 (s), 153.38 (s), 136.84 (s), 131.68 (s), 127.70 (s), 124.24 (s), 113.89 (s), 105.26 (s), 80.17 (s), 77.57 (s), 77.32 (s), 77.06 (s), 72.14 (s), 56.29 (d, $J = 19.6$ Hz), 55.38 (s).

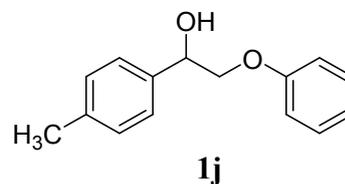
2-(2,6-dimethoxyphenoxy)-1-(3-methoxyphenyl)ethan-1-ol (1i) was prepared in 95% yield.

 ^1H NMR (500 MHz, CDCl_3) δ 7.26 (t, $J = 7.8$ Hz, 1H), 7.05 (t, $J = 8.4$ Hz, 1H), 7.02 – 6.95 (m, 2H), 6.82 (dt, $J = 12.3, 6.1$ Hz, 1H), 6.62 (t, $J = 8.3$ Hz, 2H), 4.96 (dd, $J = 9.9, 2.1$ Hz, 1H), 4.57 (s, 1H), 4.44 (dd, $J = 11.0, 2.5$ Hz, 1H), 3.89 (s, 6H), 3.81 (s, 3H). ^{13}C NMR (126 MHz,

CDCl₃) δ 159.83 (s), 153.36 (s), 141.22 (s), 136.83 (s), 129.45 (s), 124.26 (s), 118.76 (s), 113.57 (s), 111.68 (s), 105.25 (s), 80.18 (s), 77.48 (s), 77.09 (d, *J* = 31.9 Hz), 72.46 (s), 56.23 (s), 55.37 (s).

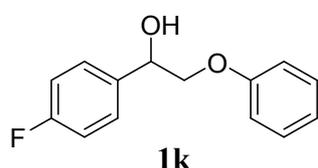
2-phenoxy-1-(p-tolyl)ethan-1-ol (1j) was prepared in 97% yield.

¹H NMR (500 MHz, CDCl₃) δ 7.42 (d, *J* = 8.0 Hz, 2H), 7.36 (dt, *J* = 18.0, 9.0 Hz, 2H), 7.28 (d, *J* = 7.9 Hz, 2H), 7.05 (t, *J* = 7.4 Hz, 1H), 7.00 (d, *J* = 7.9 Hz, 2H), 5.17 (dd, *J* = 8.8, 2.7 Hz, 1H), 4.16 (dd, *J* = 9.6, 3.2 Hz, 1H), 4.08 (t, *J* = 9.2 Hz, 1H), 2.86 (s, 1H), 2.44



(s, 3H). ¹³C NMR (126 MHz, CDCl₃) δ 158.55 (s), 138.09 (s), 136.82 (s), 129.70 (s), 129.40 (s), 126.37 (s), 121.40 (s), 114.77 (s), 77.43 (s), 77.05 (d, *J* = 31.9 Hz), 73.45 (s), 72.58 (s), 21.33 (s).

1-(4-fluorophenyl)-2-phenoxyethan-1-ol (1k) was prepared in 98% yield.

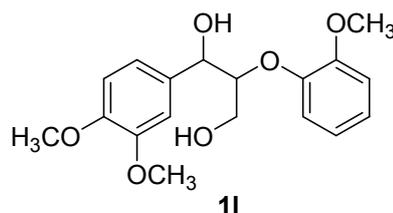


¹H NMR (500 MHz, CDCl₃) δ 7.43 (dd, *J* = 8.4, 5.5 Hz, 2H), 7.30 (t, *J* = 7.9 Hz, 2H), 7.08 (t, *J* = 8.7 Hz, 2H), 6.99 (t, *J* = 7.3 Hz, 1H), 6.93 (d, *J* = 8.3 Hz, 2H), 5.11 (dd, *J* = 8.7, 3.1 Hz, 1H), 4.08 (dd, *J* = 9.6, 3.2 Hz, 1H), 3.99 (t, *J* = 9.2 Hz, 1H), 2.90 (s, 1H). ¹³C NMR

(126 MHz, CDCl₃) δ 158.42 (s), 135.57 (s), 129.74 (s), 128.15 (d, *J* = 7.2 Hz), 121.57 (s), 115.68 (s), 115.59 (d, *J* = 21.5 Hz), 115.01 (d, *J* = 57.5 Hz), 77.73 (s), 77.42 (s), 77.73 – 76.40 (m), 73.34 (s), 72.11 (s).

1-(3,4-dimethoxyphenyl)-2-(2-methoxyphenoxy)propane-1,3-diol was prepared in 76% yield.⁶

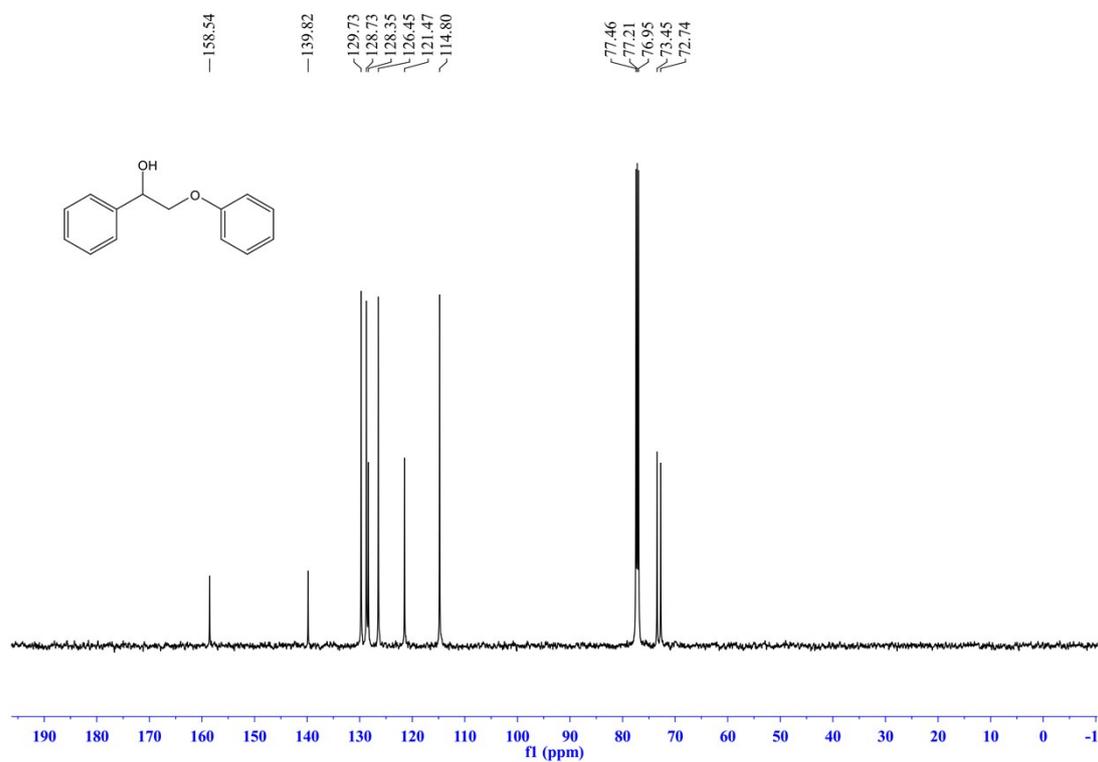
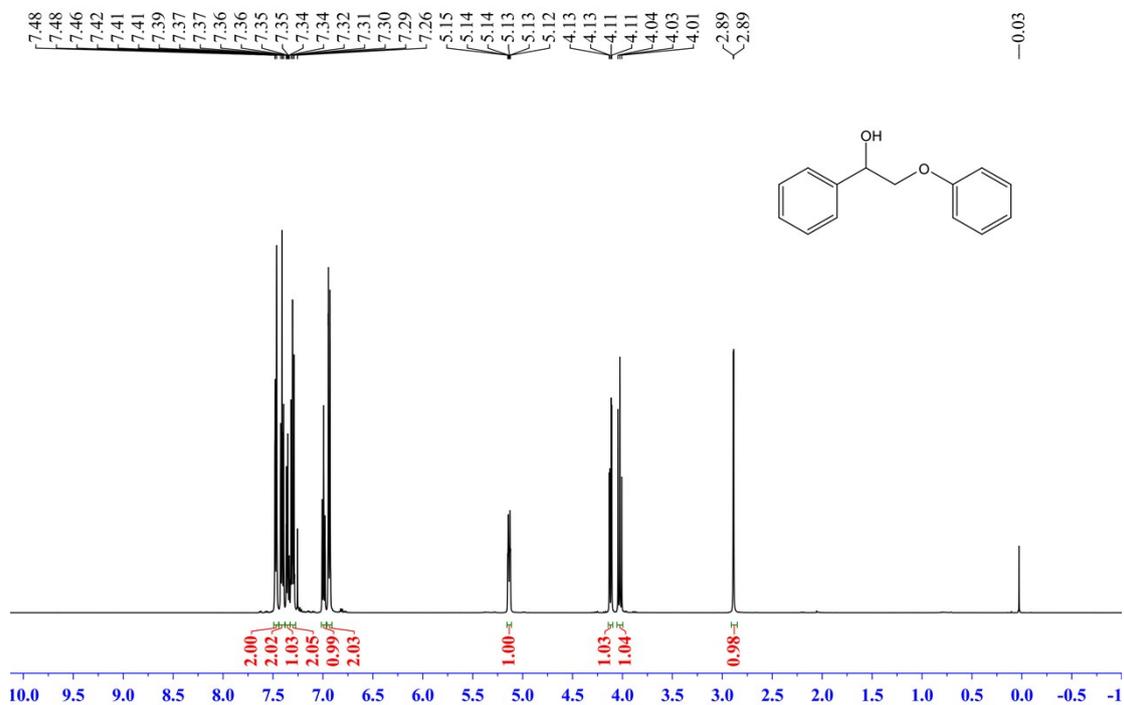
¹H NMR (500 MHz, DMSO) δ 6.97 (dd, *J* = 7.7, 1.5 Hz, 1H), 6.88 (dd, *J* = 7.8, 1.6 Hz, 1H), 6.80 (ddd, *J* = 9.5, 7.4, 3.8 Hz, 2H), 6.57 (d, *J* = 2.2 Hz, 2H), 6.31 (t, *J* = 2.2 Hz, 1H), 5.45 (d, *J* = 4.8 Hz, 1H), 4.74 (t, *J* = 4.9 Hz, 1H), 4.60 (t, *J* = 5.6 Hz, 1H), 4.35 – 4.24 (m, 1H), 3.67 (s, 9H), 3.36 (s, 2H).

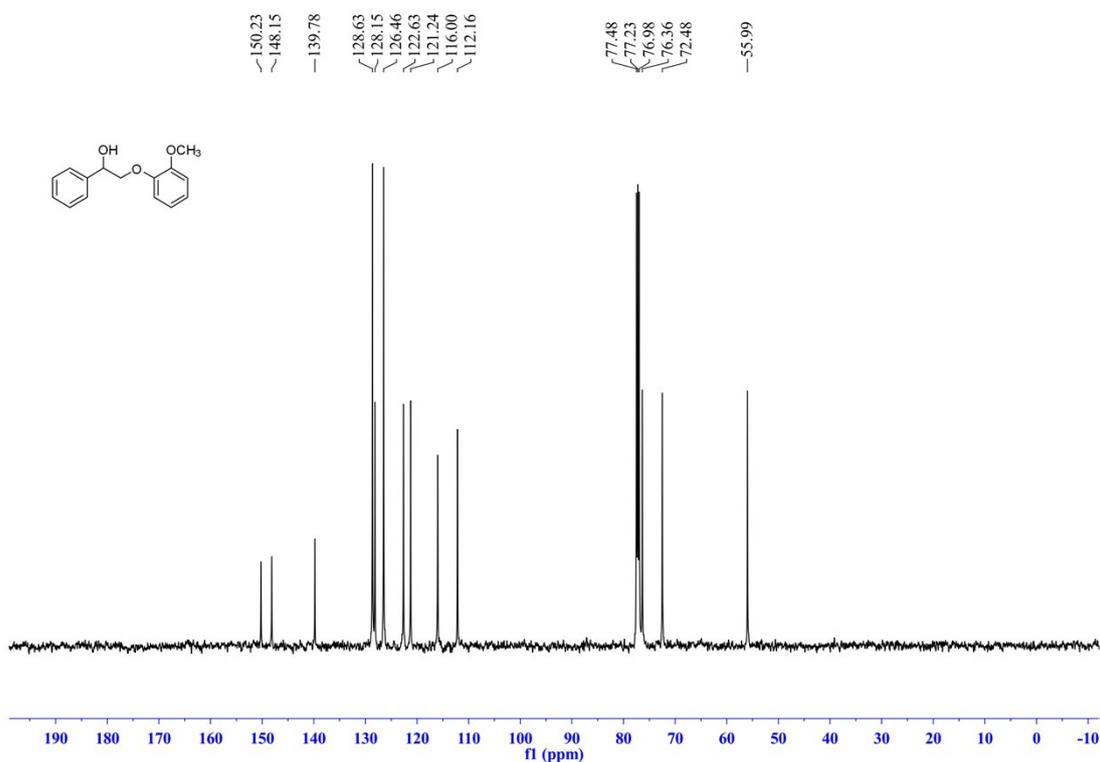
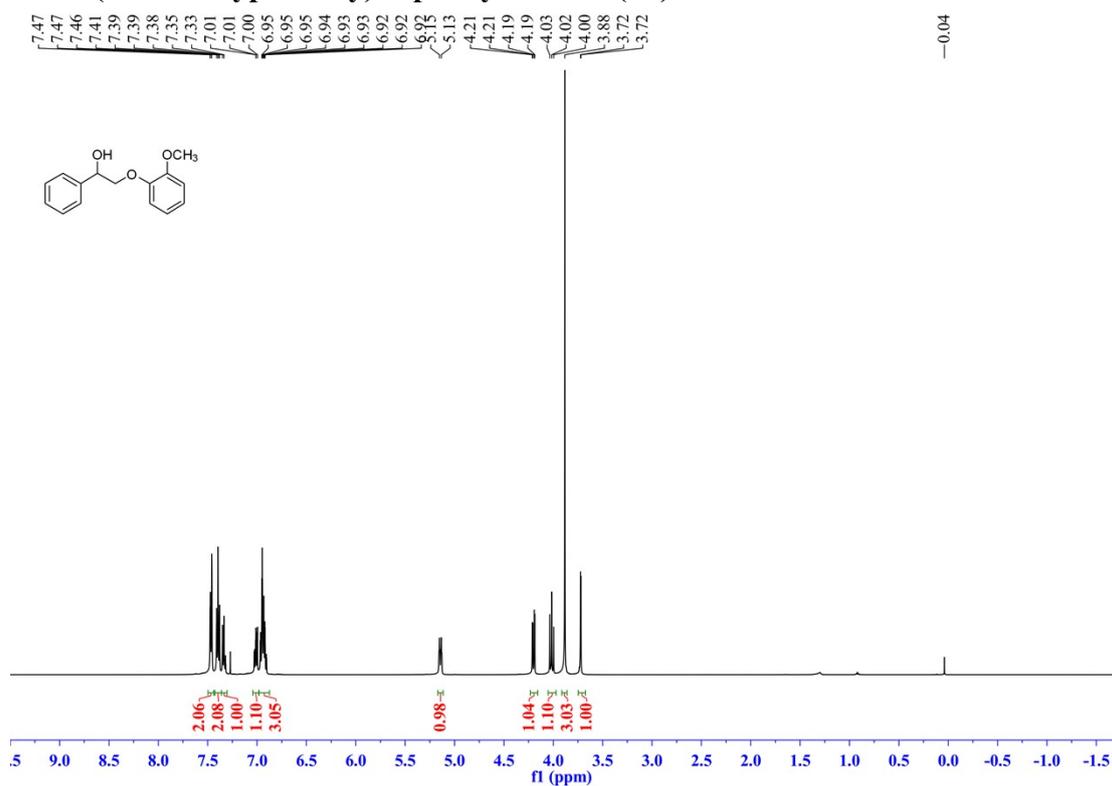


¹³C NMR (126 MHz, DMSO) δ 160.47 (s), 150.40 (s), 148.50 (s), 145.48 (s), 121.48 (d, *J* = 60.3 Hz), 116.66 (s), 113.22 (s), 105.41 (d, *J* = 53.8 Hz), 99.43 (s), 84.16 (s), 72.30 (s), 60.50 (s), 56.12 (s), 55.56 (s), 41.85 – 40.11 (m), 40.28 (dd, *J* = 41.7, 20.9 Hz), 42.26 – 38.36 (m), 41.85 – 37.82 (m), 40.27 – 38.36 (m).

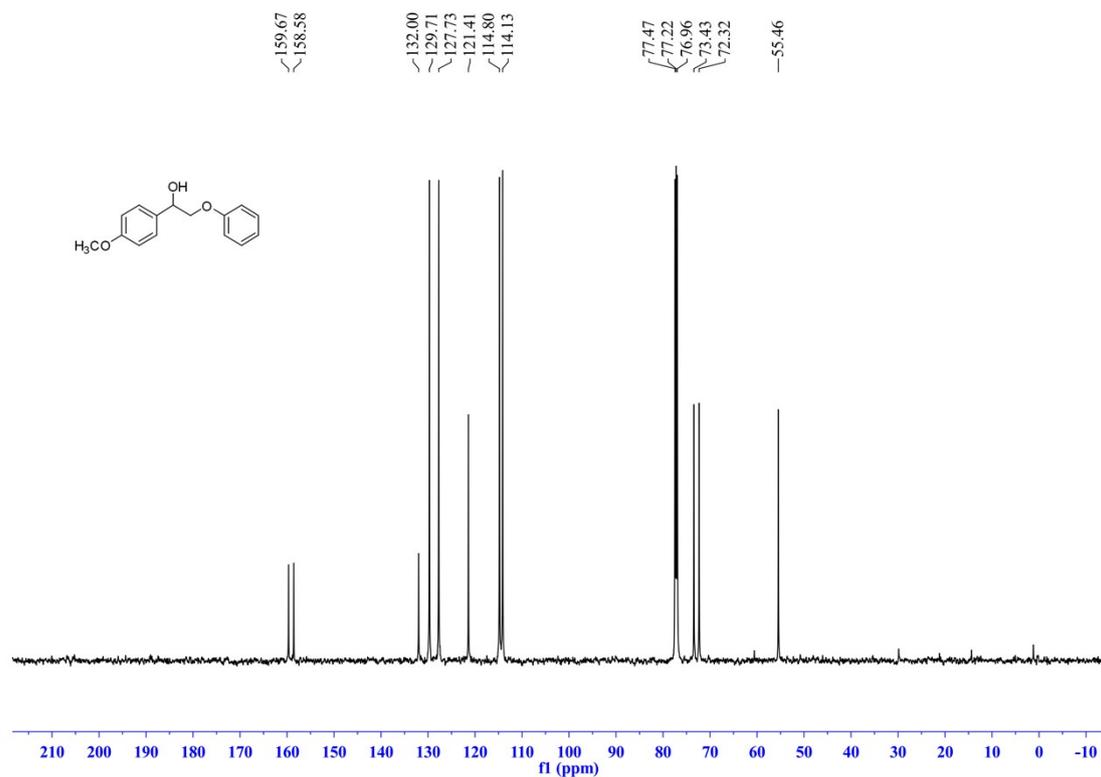
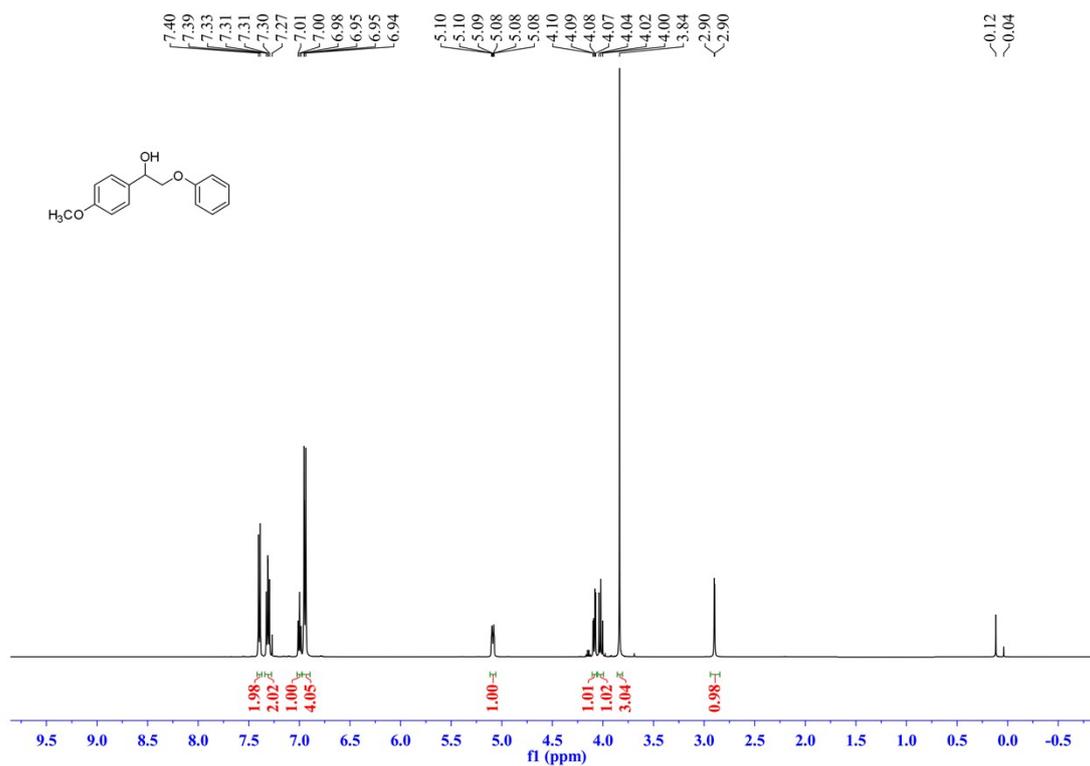
9. NMR Spectra

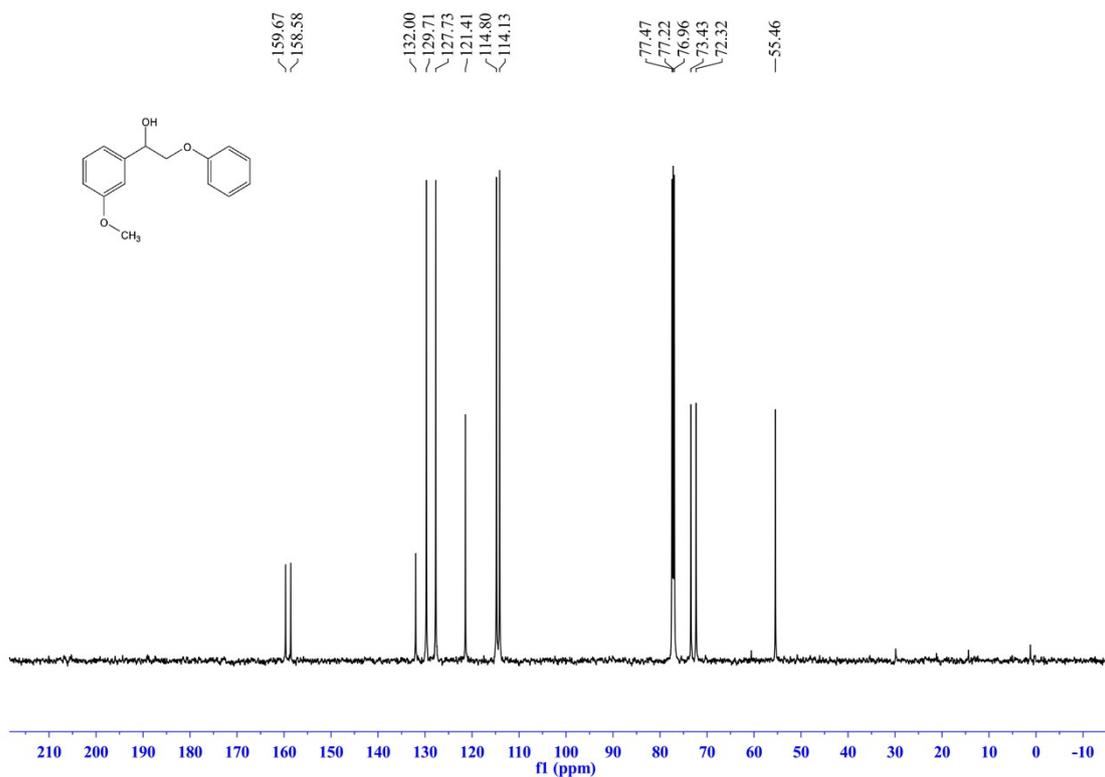
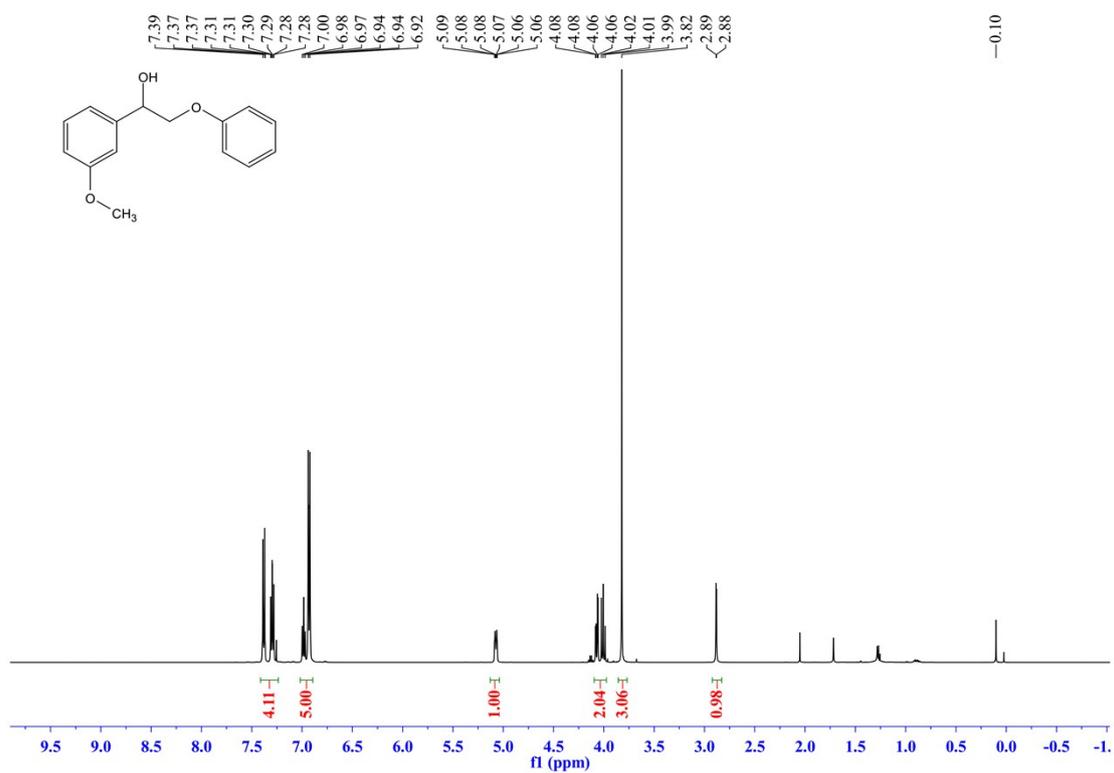
1. 2-phenoxy-1-phenylethanol (1a)



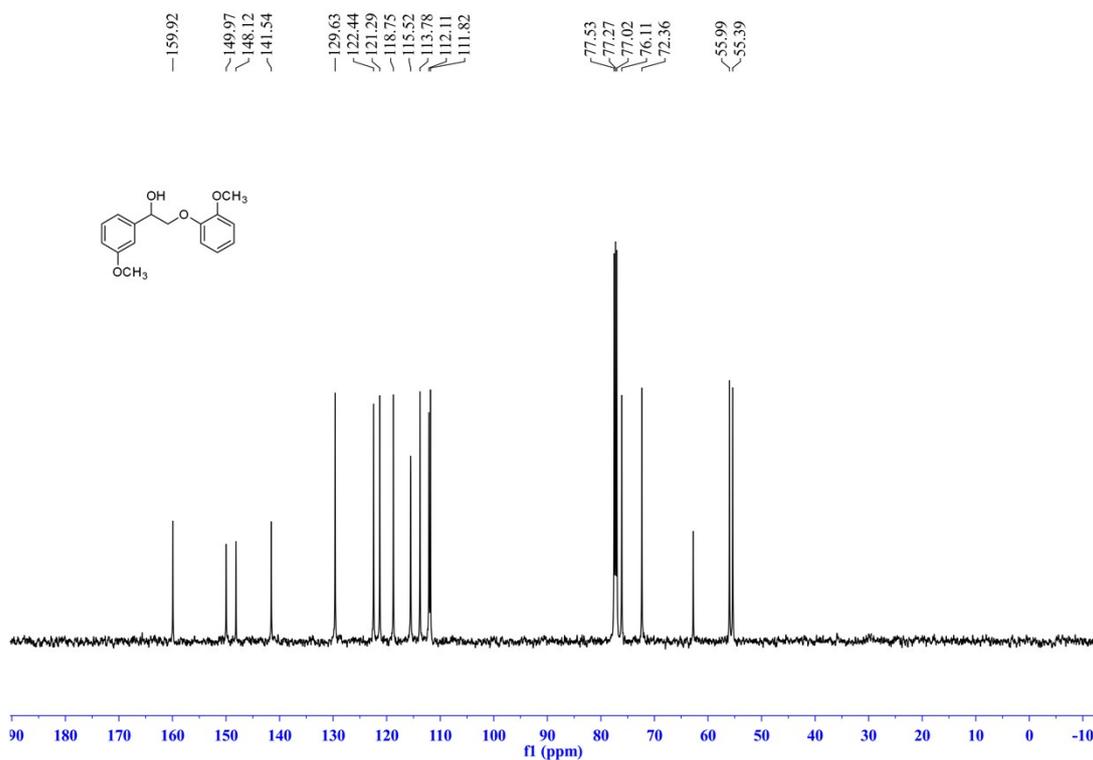
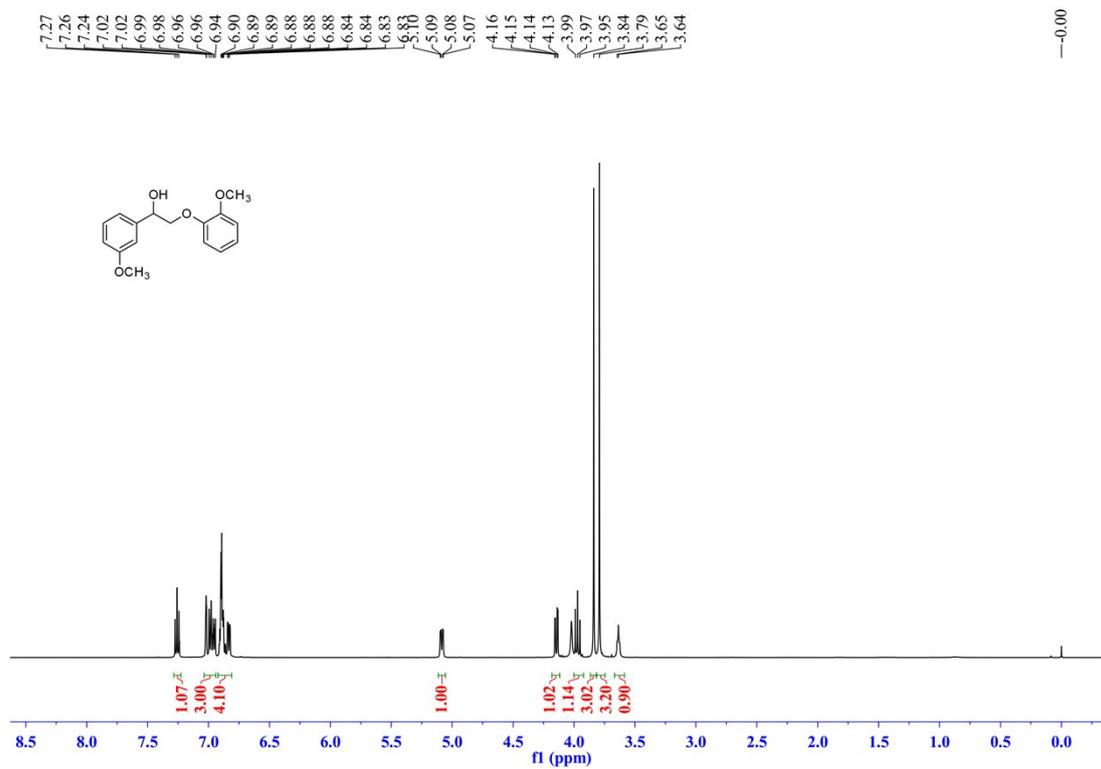
2. 2-(2-methoxyphenoxy)-1-phenylethanol (1b)

3. 1-(4-methoxyphenyl)-2-phenoxyethanol (1c)

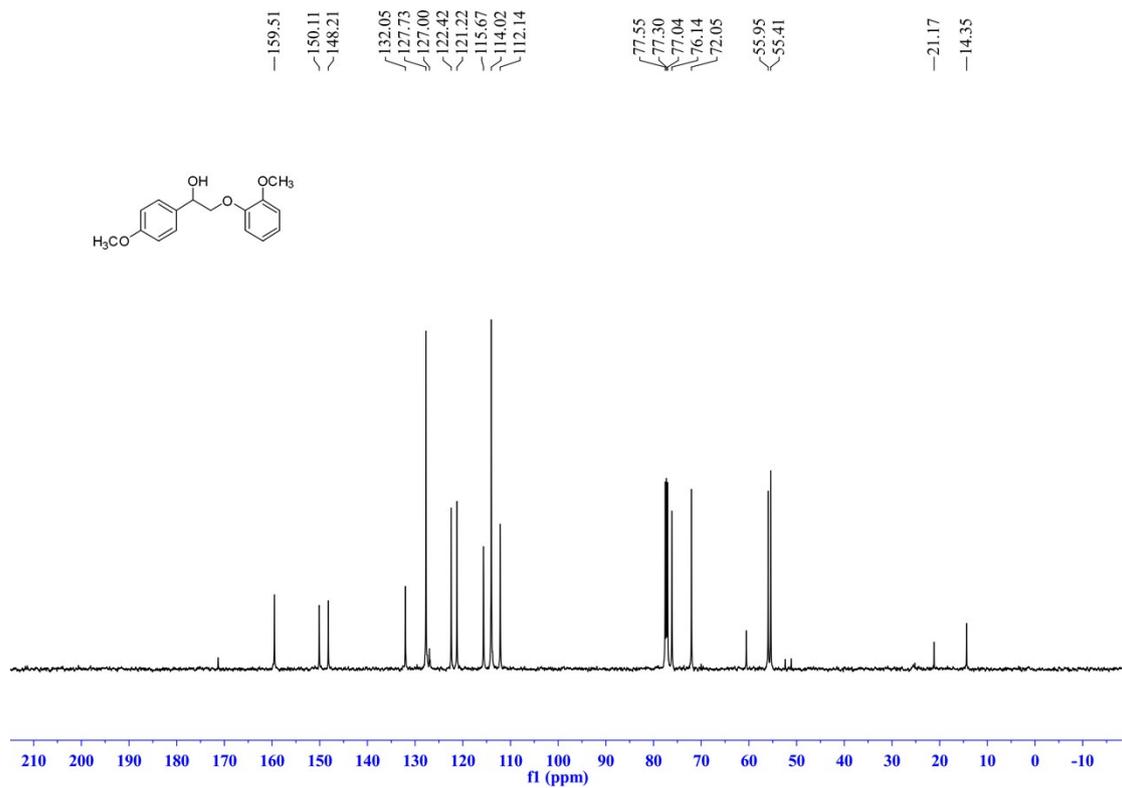
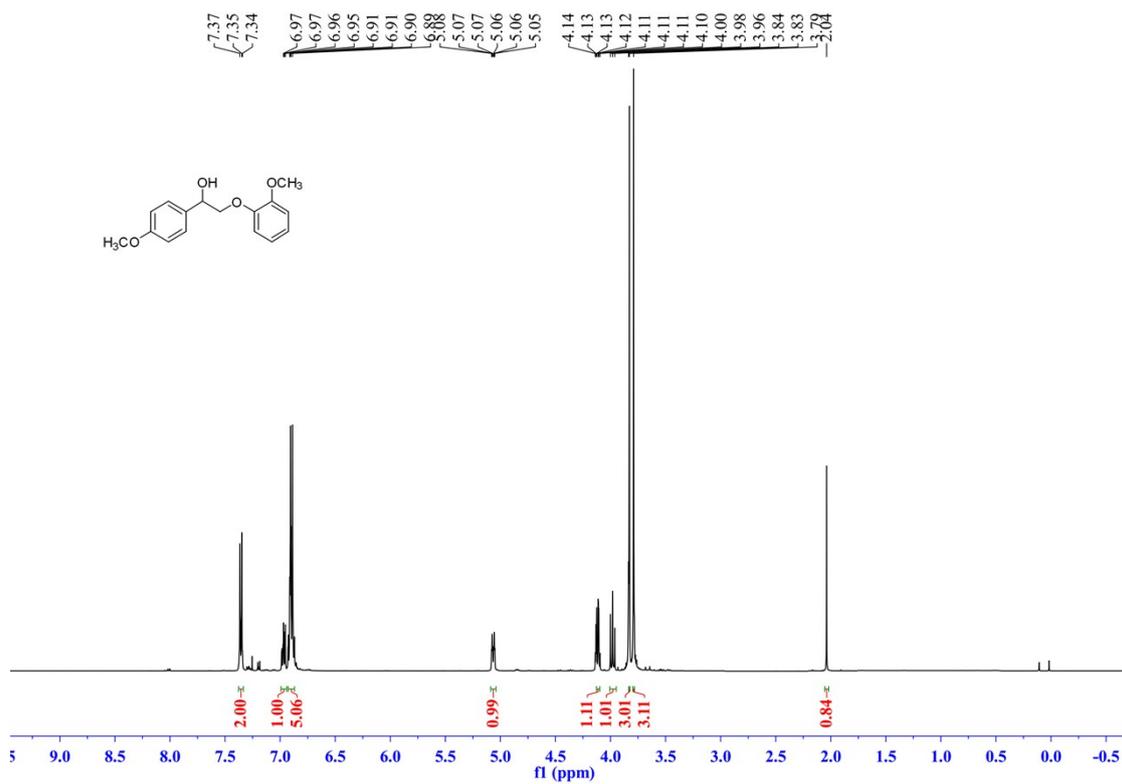


4. 1-(3-methoxyphenyl)-2-phenoxyethanol (1d)

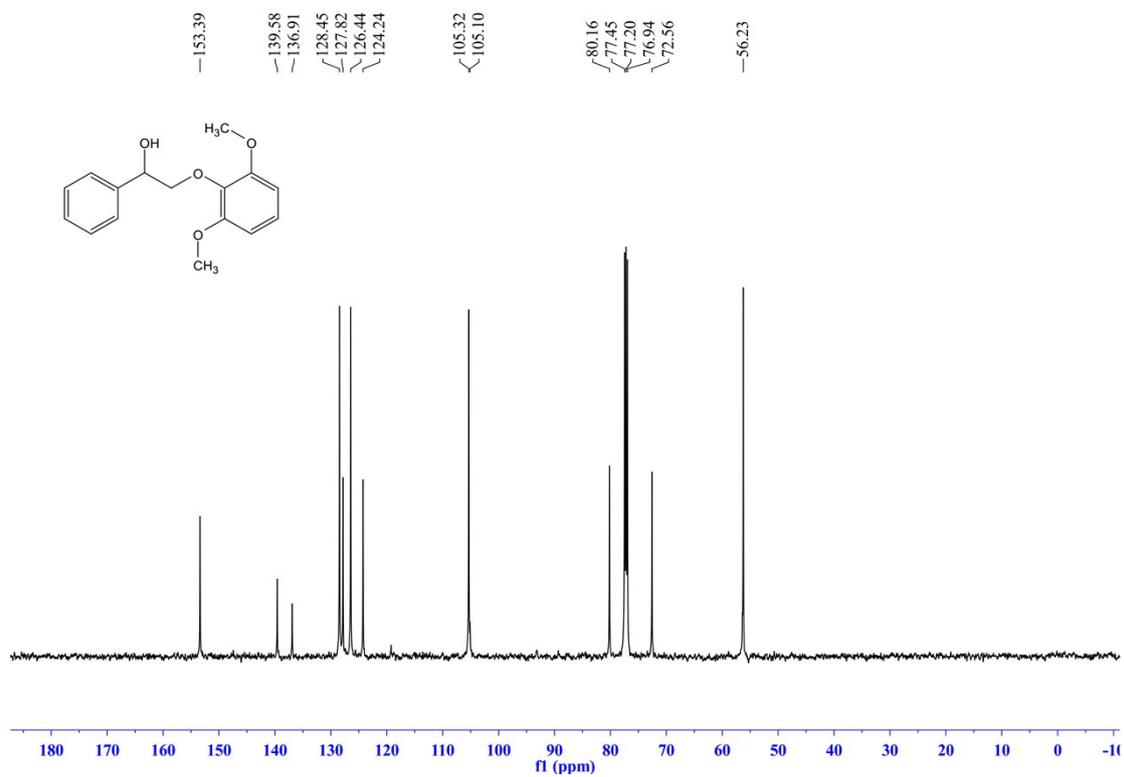
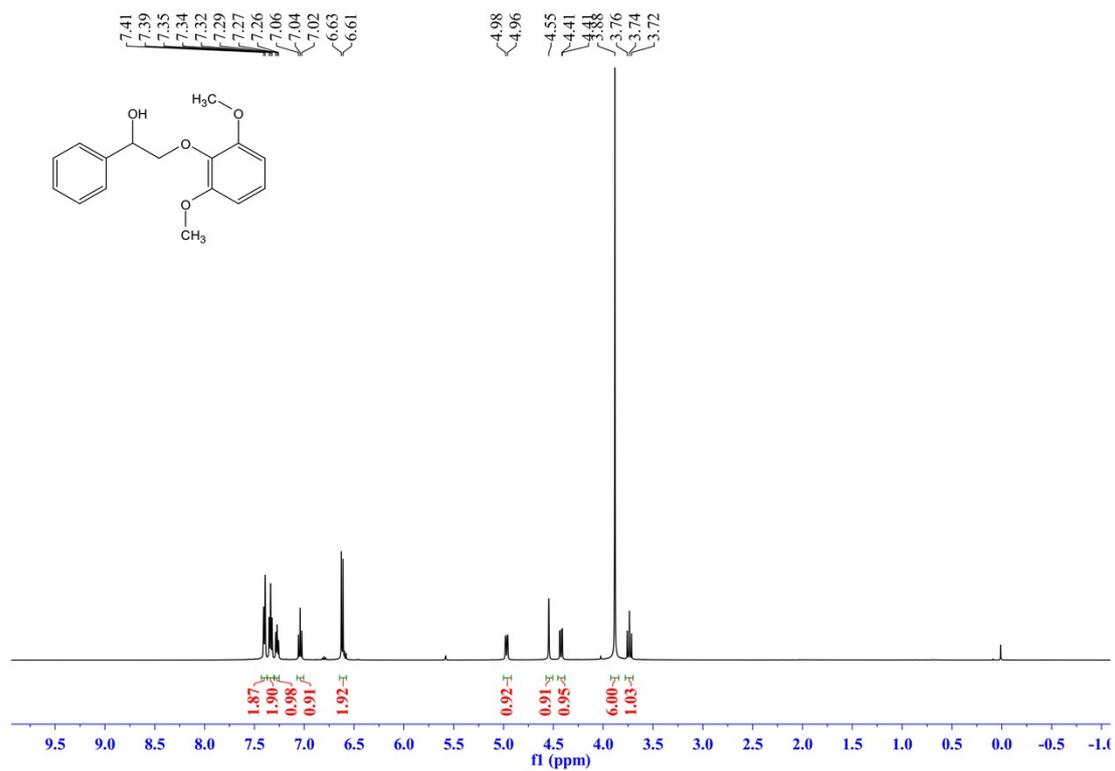
5. 2-(2-methoxyphenoxy)-1-(3-methoxyphenyl) ethanol (1e)



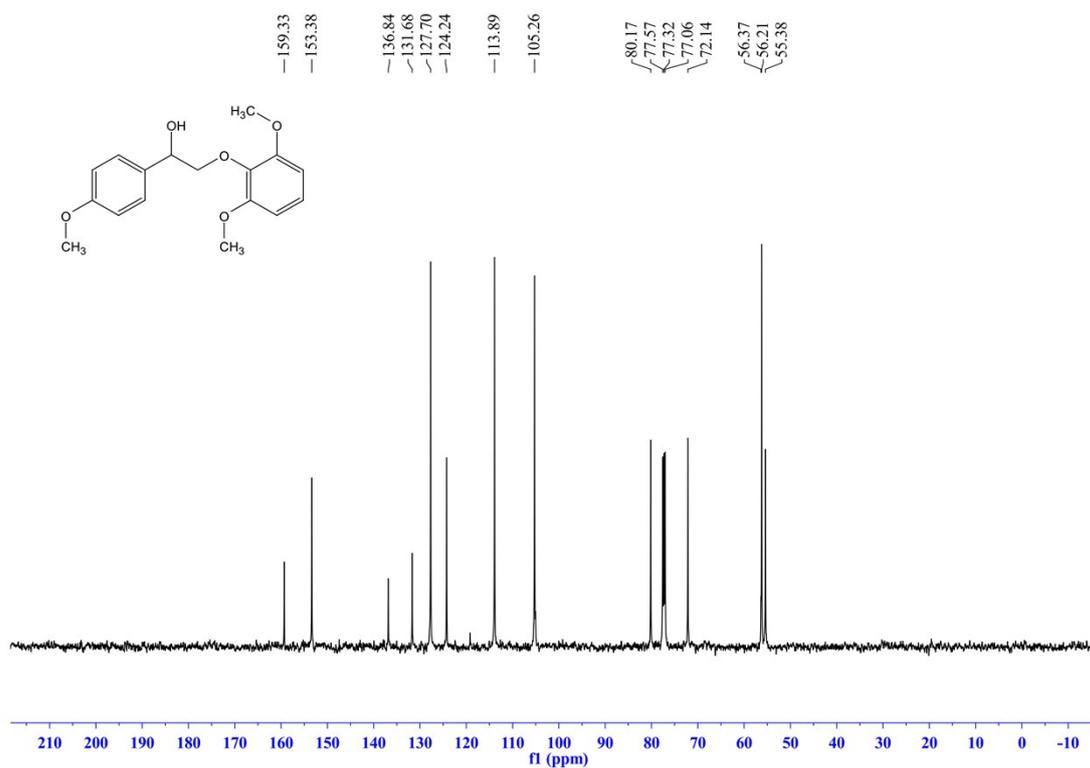
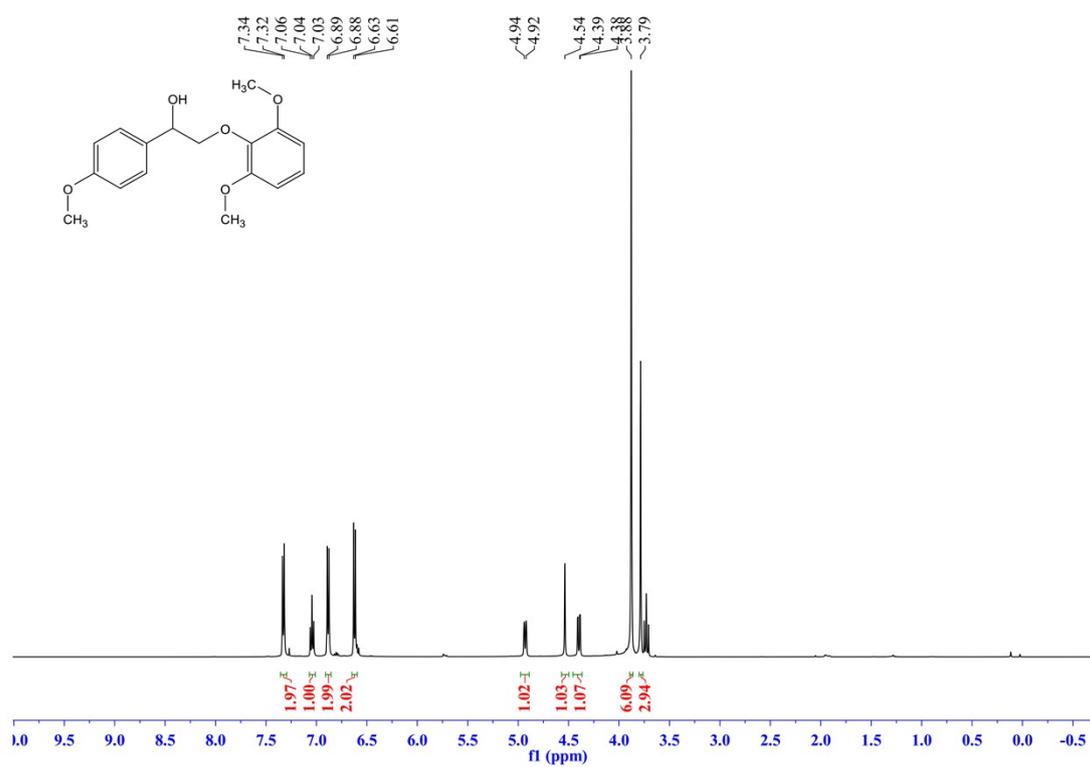
6. 2-(2-methoxyphenoxy)-1-(4-methoxyphenyl) ethanol (1f)



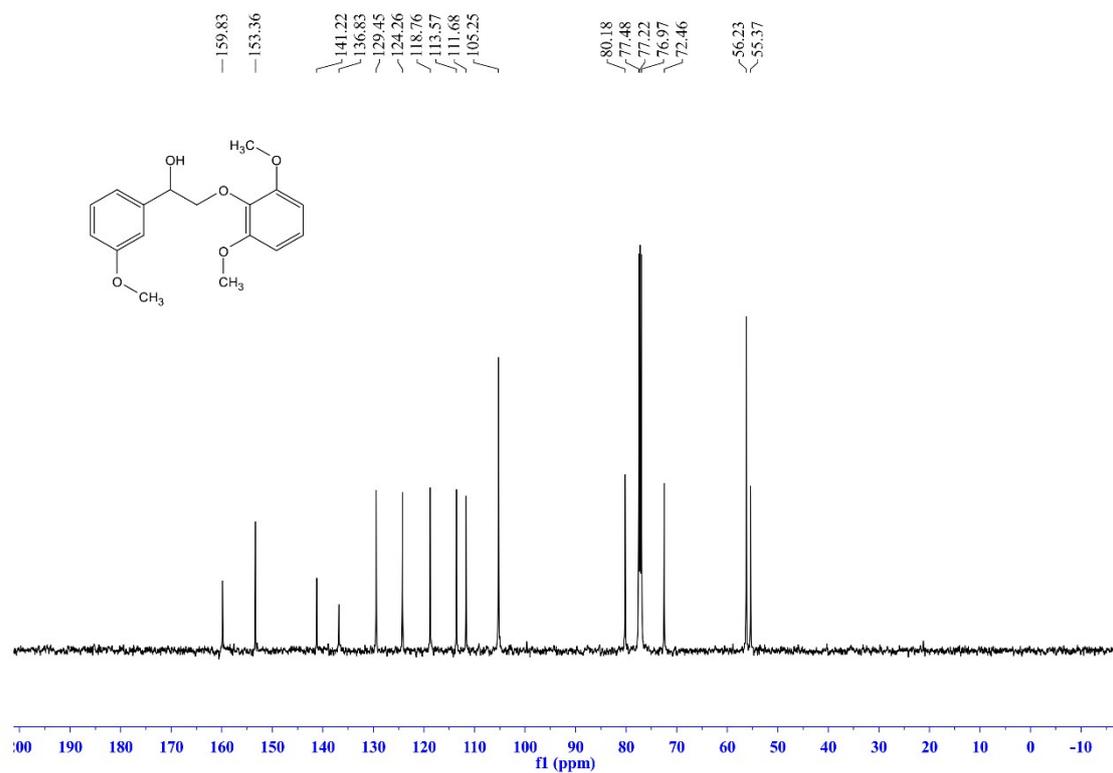
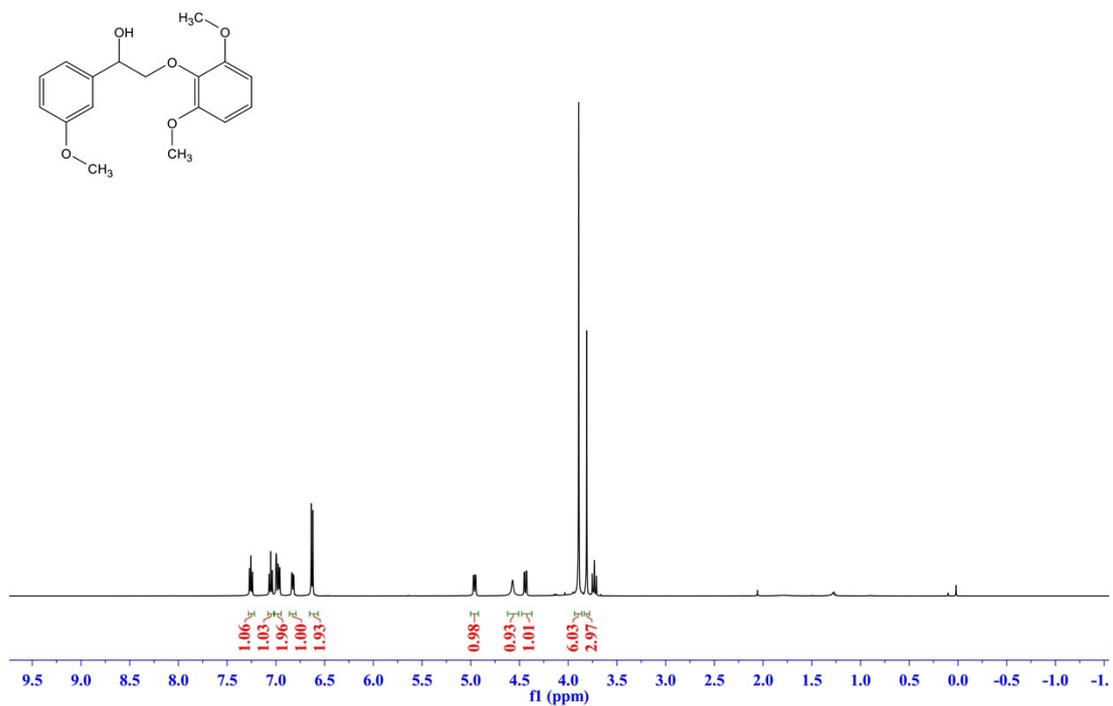
7. 2-(2,6-dimethoxyphenoxy)-1-phenylethan-1-ol (1g)



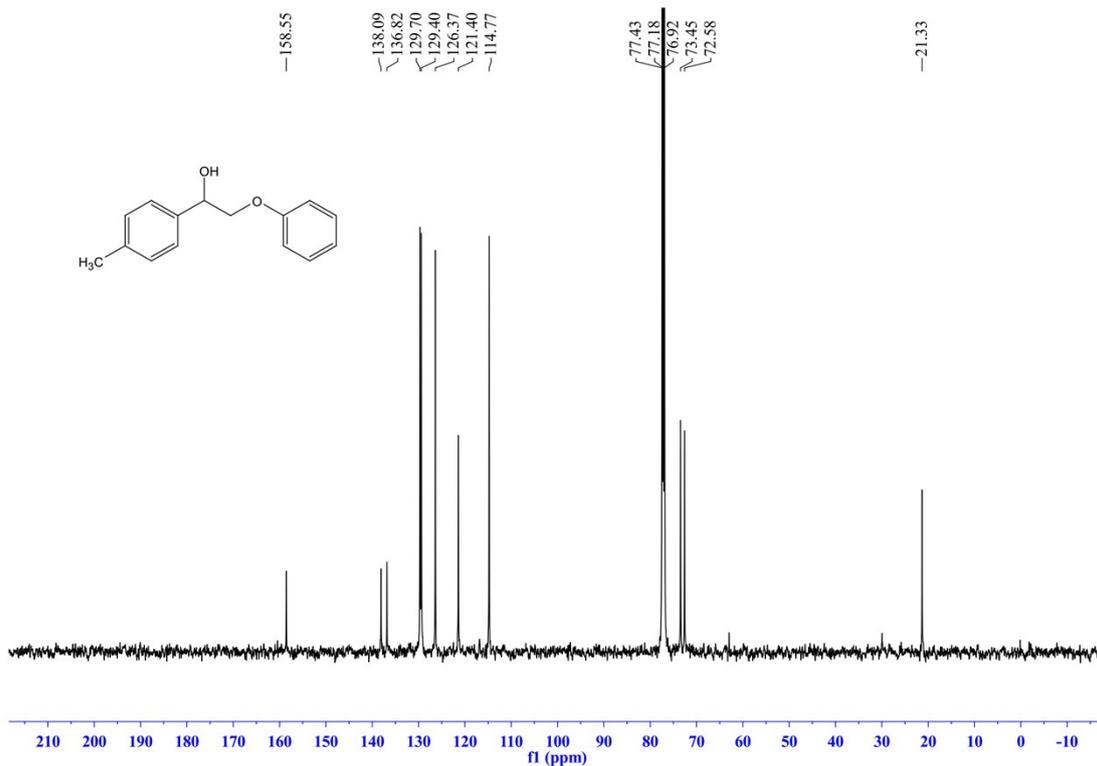
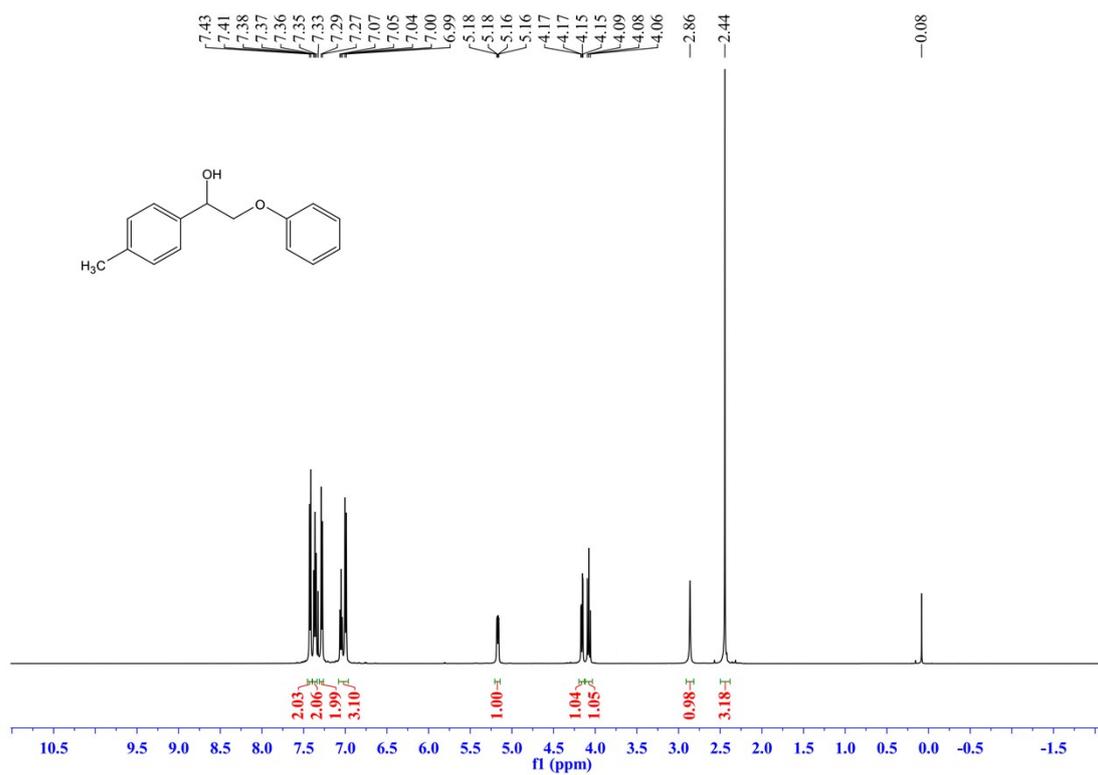
8. 2-(2,6-dimethoxyphenoxy)-1-(4-methoxyphenyl)ethan-1-ol (1h)



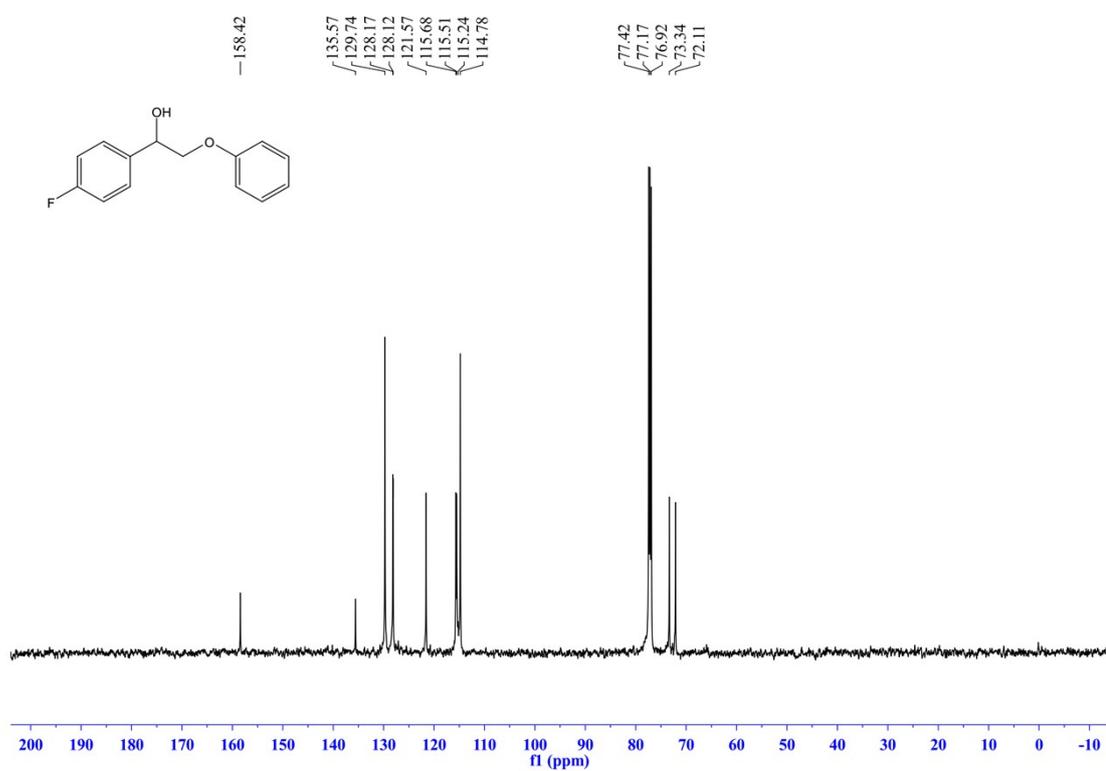
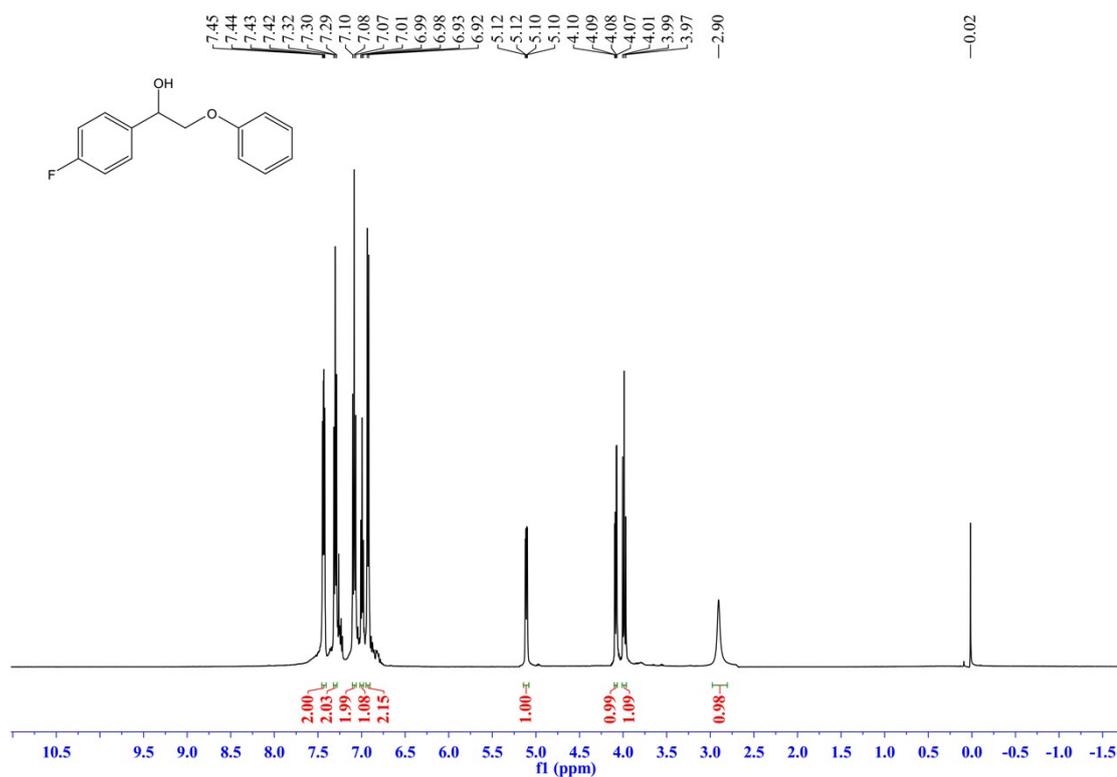
9. 2-(2,6-dimethoxyphenoxy)-1-(3-methoxyphenyl)ethan-1-ol (1i)



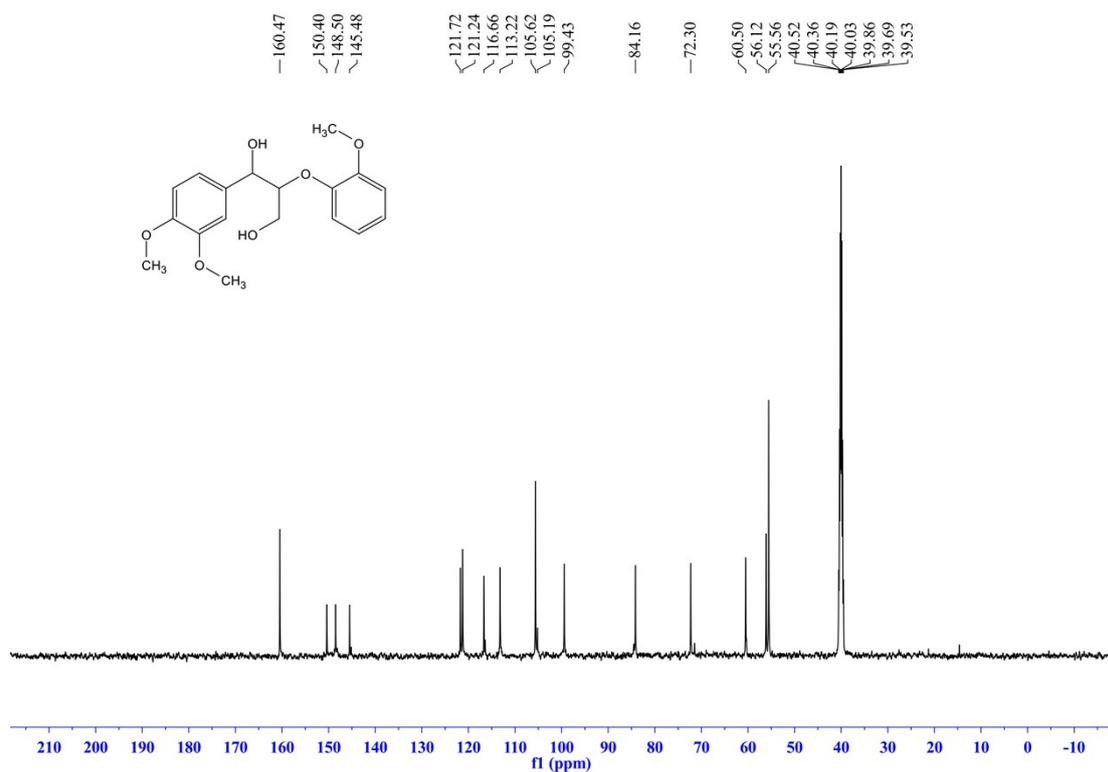
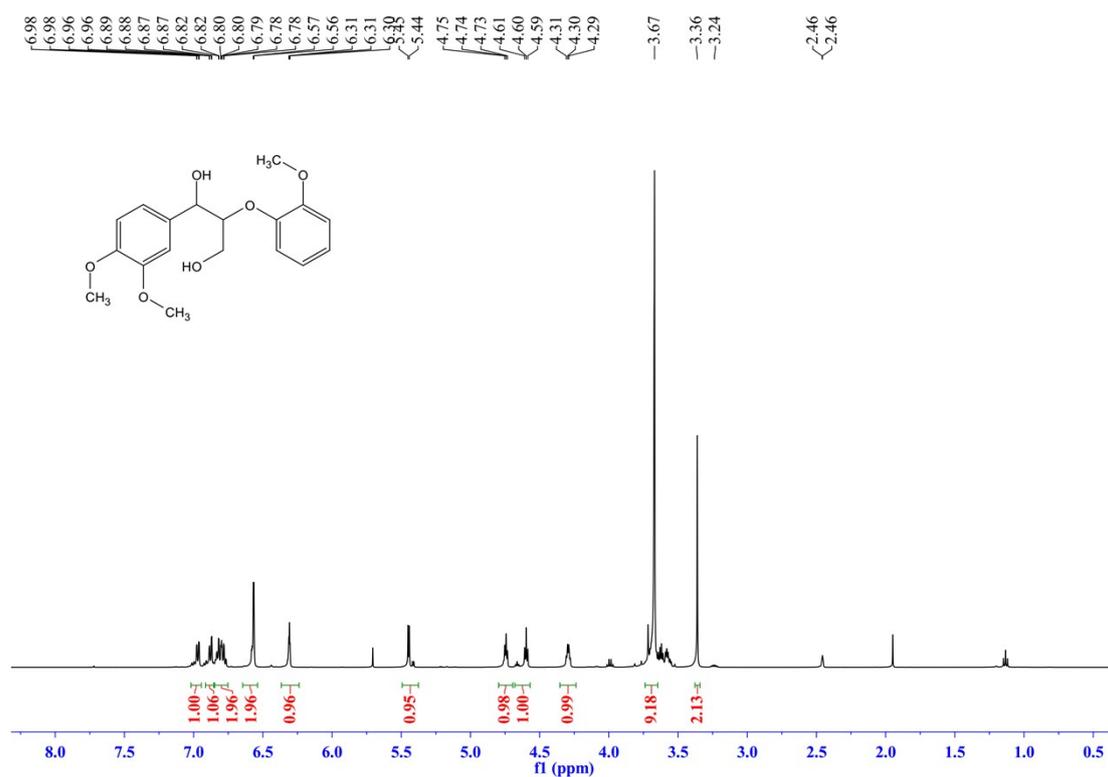
10. 2-phenoxy-1-(p-tolyl)ethan-1-ol (1j)



11. 1-(4-fluorophenyl)-2-phenoxyethan-1-ol (1k)



12. 1-(3,4-dimethoxyphenyl)-2-(2-methoxyphenoxy)propane-1,3-diol (11)



10. Extraction of Lignin²

To birch sawdust (10 g) was added 1,4-dioxane (100 mL) followed by 2 mL HCl (12M) and the mixture was heated to a gentle reflux under a N₂ atmosphere for 1 hour. The mixture was then allowed

to cool and the lignin containing liquor was collected by filtration. The collected liquor was partially concentrated *in vacuo* to give a gummy residue which was taken up in acetone/water (9:1, ~30 mL) and precipitated by addition to rapidly stirring water (150 mL). The crude lignin was collect by filtration and dried under vacuum. The dried crude lignin was taken up in acetone/methanol (9:1) and precipitated by dropwise addition to rapidly stirring Et₂O (100 mL). The precipitated lignin was collected by filtration and dried under vacuum to give a purified birch lignin (0.8 g). This lignin was used in subsequent experiments without further processing.

11. GS-MS spectrum of the lignin depolymerization product

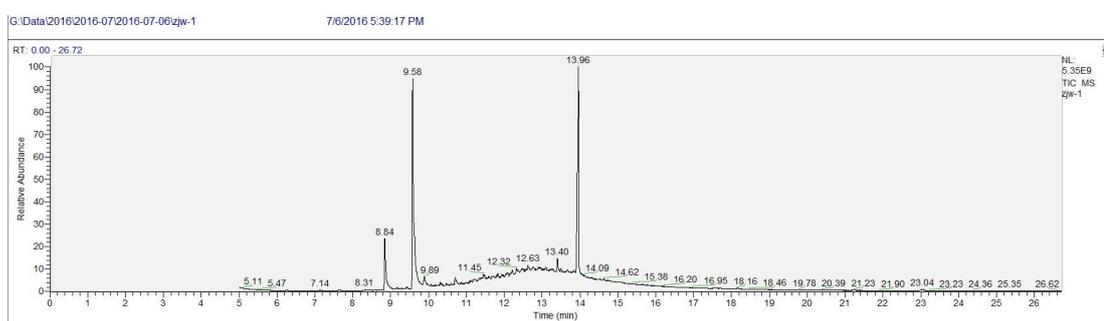


Fig. S2 GC-MS spectrum of a soluble crude product mixture from epolymerization of organosolv lignin

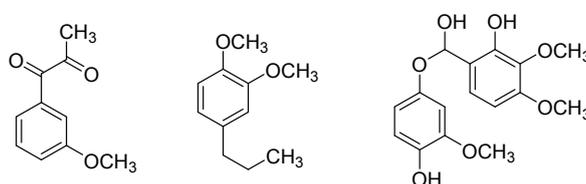
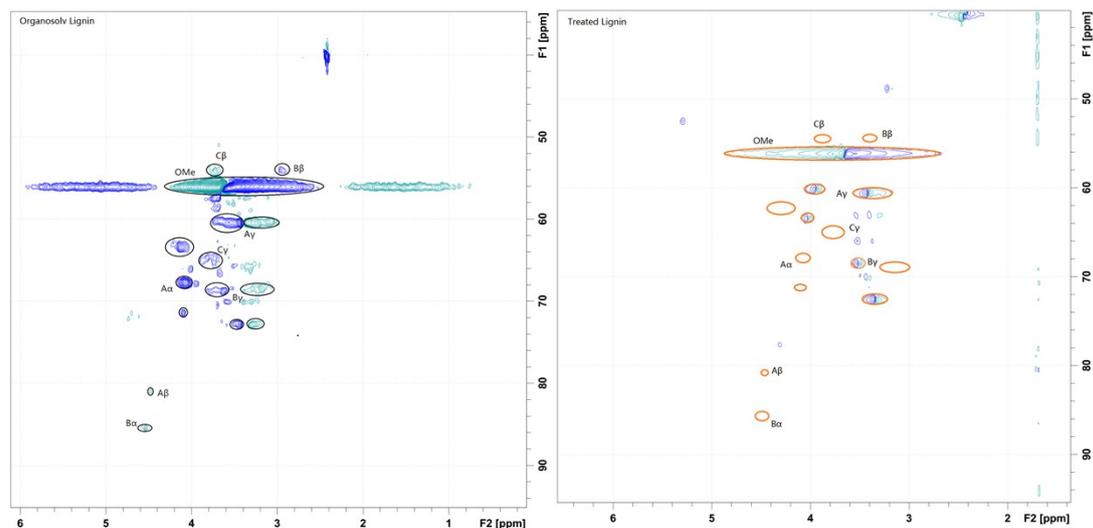


Fig. S3 Proposed structure of the three major components from depolymerization of organosolv lignin



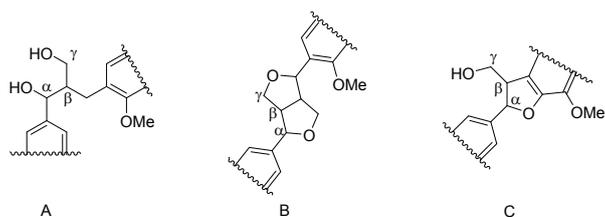
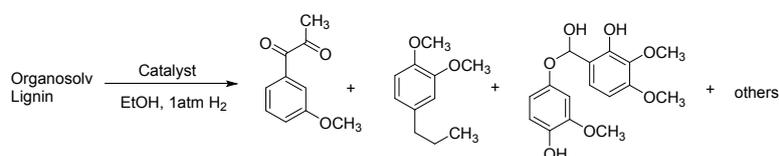


Fig. S4. 2D HSQC NMR spectra of the organosolv lignin before and after reaction

Table S2. Products distribution in hydrogenolysis of birch lignin catalyzed by Pd₁Ni₈NPs@ZrO₂.



Catalyst	Recycle	Ratio			Residual Lignin (wt%)
		45	12	43	
Pd ₁ Ni ₈ /ZrO ₂	1	45	12	43	44
Pd ₁ Ni ₈ /ZrO ₂	2	46.7	11.7	41.6	50
Pd ₁ Ni ₈ /ZrO ₂	3	46.3	10.5	43.2	43
Pd ₁ Ni ₈ /ZrO ₂	4	48.2	9.1	42.7	49
Pd ₁ Ni ₈ /ZrO ₂	5	42.1	9.0	48.9	51

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