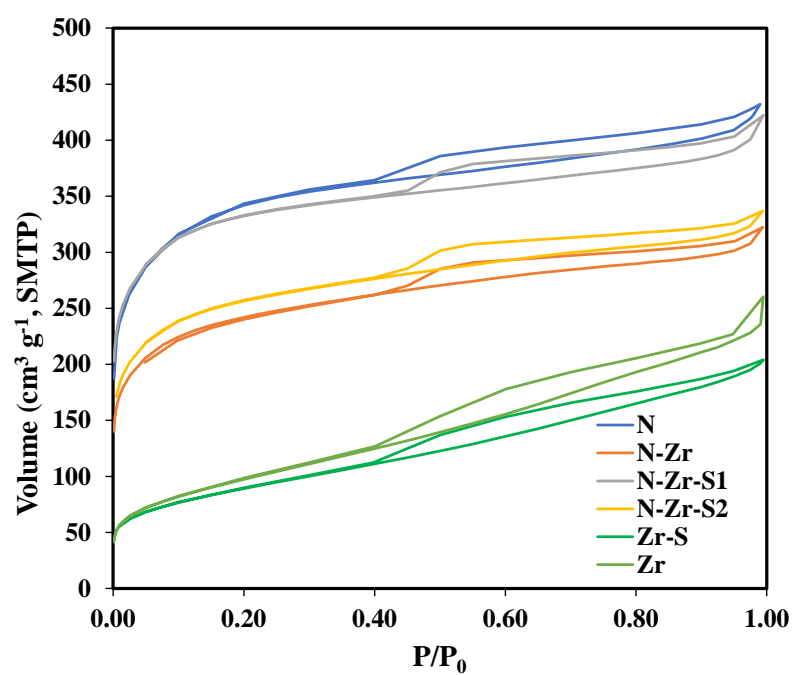


# Catalytic Screening for 1,2-Diol Protection: A Saccharose-Derived Hydrothermal Carbon Showcases Enhanced Performance

## Supplementary Materials

### Content

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**Figure S1.** N<sub>2</sub> adsorption isotherms of N, N-Zr, N-Zr-S1, N-Zr-S2, Zr-S and Zr.

**Table S1.** XPS components of C 1s peaks after deconvolution.

<b>Catalyst</b>	<b>Component 1</b>	<b>Component 2</b>	<b>Component 3</b>	<b>Component 4</b>
<b>N</b>	284.8 (57.20%)	285.7 (23.13%)	289.6 (19.66%)	
<b>Zr</b>	---	---	---	
<b>Zr-S</b>	---	---	---	
<b>N-Zr</b>	284.8 (53.66%)	285.7 (23.05%)	289.9 (23.29%)	
<b>N-Zr-S1</b>	284.8 (48.49%)	285.7 (26.86%)	289.6 (24.65%)	
<b>N-Zr-S2</b>	284.8 (44.20%)	285.7 (32.61%)	289.6 (23.19%)	
<b>N-HT</b>	284.8 (57.03%)	285.7 (24.43%)	289.6 (18.55%)	
<b>N-HT-S</b>	284.8 (51.49%)	285.9 (28.93%)	290.1 (19.60%)	
<b>M</b>	284.8 (52.75%)	285.7 (23.95%)	288.6 (23.3%)	
<b>M-S</b>	284.8 (49.34%)	285.6 (22.90%)	287.3 (27.76%)	
<b>M-N</b>	284.8 (41.22%)	285.7 (24.32%)	287.6 (18.64%)	290.7 (15.82%)
<b>HT</b>	284.8 (17.1%)		289.0 (82.9%)	
<b>HT-S</b>	284.8 (25.98%)	286.5 (74.02%)		

**Table S2.** XPS components of O 1s peaks after deconvolution.

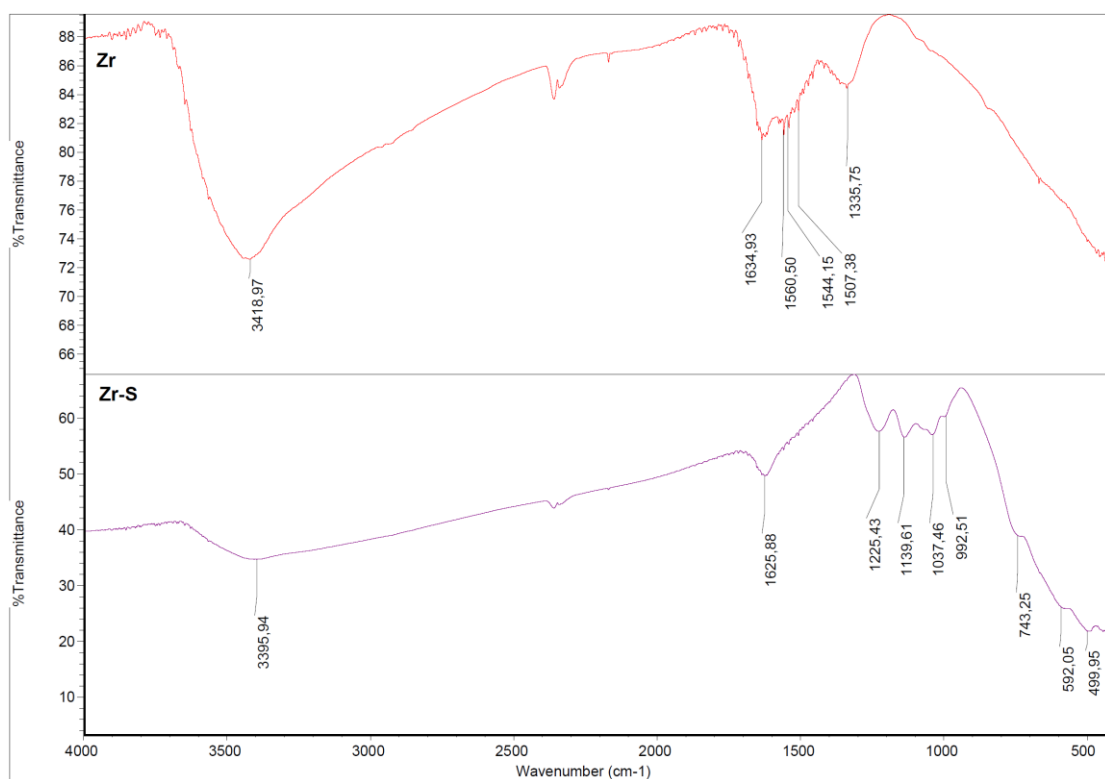
<b>Catalyst</b>	<b>Component 1</b>	<b>Component 2</b>	<b>Component 3</b>	<b>Component 4</b>
<b>N</b>	531.1 (85.26%)	534.5 (3.15%)	536.4 (11.59%)	
<b>Zr</b>	531.7 (100%)			
<b>Zr-S</b>	531.0 (100%)			
<b>N-Zr</b>	531 (19.44%)	532.5 (77.44%)	534.9 (3.12%)	
<b>N-Zr-S1</b>	530.7 (13.58%)	532.6 (56.17%)	535.9 (30.24%)	
<b>N-Zr-S2</b>	530.9 (6.54%)	532.6 (77.19%)	535.7 (16.27%)	
<b>N-HT</b>	532 (81.74%)	535.9 (14.74%)	538.8 (3.52%)	
<b>N-HT-S</b>	532.4 (85.29%)		537.4 (4.71%)	
<b>M</b>	533.0 (100%)			
<b>M-S</b>	532.8 (100%)			
<b>M-N</b>	532.9 (96.66%)	536.9 (3.34%)		
<b>HT</b>	534.5 (35.54%)		537.2 (64.46%)	
<b>HT-S</b>	533.2 (7.02%)	536.4 (16.75%)	539.2 (46.21%)	541.3 (30.02%)

**Table S3.** XPS components of N 1s peaks after deconvolution.

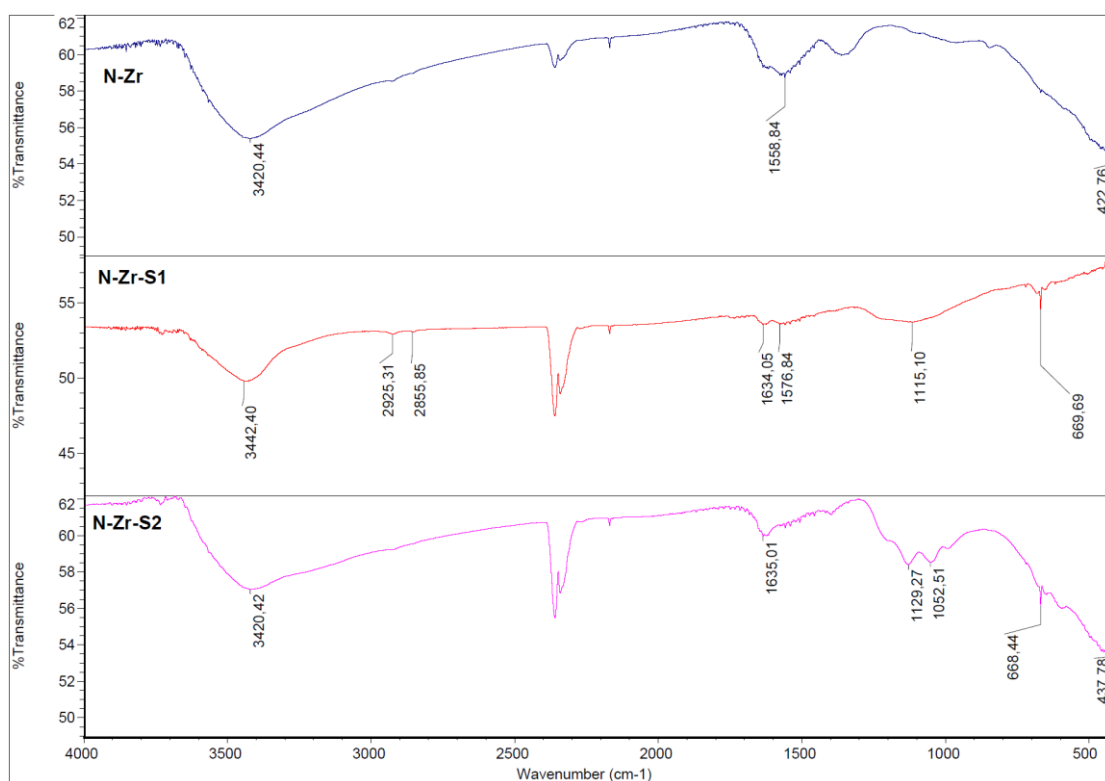
<b>Catalyst</b>	<b>Component</b>
<b>N</b>	401.0 (100%)
<b>N-Zr</b>	400.0 (100%)
<b>N-Zr-S1</b>	401.1 (100%)
<b>N-Zr-S2</b>	400.6 (100%)
<b>N-HT</b>	400.9 (100%)
<b>N-HT-S</b>	400.9 (100%)
<b>M</b>	400.7 (100%)
<b>M-S</b>	400.6 (100%)
<b>M-N</b>	400.3 (55.24%)

**Table S4.** XPS components of S 2p peaks after deconvolution.

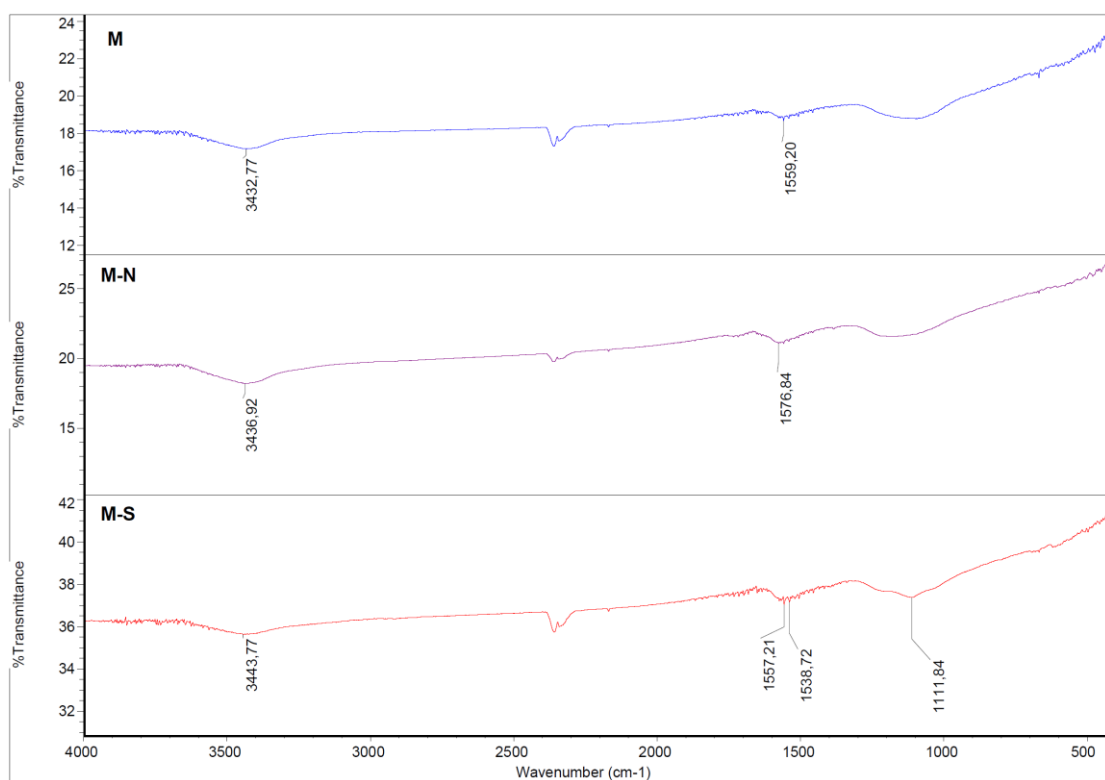
<b>Catalyst</b>	<b>Component 1</b>	<b>Component 2</b>	<b>Component 3</b>	<b>Component 4</b>
<b>N</b>	164.5 (50.2%)	168.6 (49.8%)		
<b>N-Zr</b>	164.3 (47.26%)	169.5 (52.74%)		
<b>N-Zr-S1</b>	163.9 (10.73%)	168.4 (89.27%)		
<b>N-Zr-S2</b>	170.1 (25.76%)	171.3 (74.24%)		
<b>N-HT</b>	163.8 (33.63%)	168.4 (66.37%)		
<b>N-HT-S</b>	164.5 (11.04%)	168.7 (88.96%)		
<b>HT-S</b>	163.4 (6.47%)	164.5 (5.52%)	167.4 (27.40%)	168.5 (60.61%)



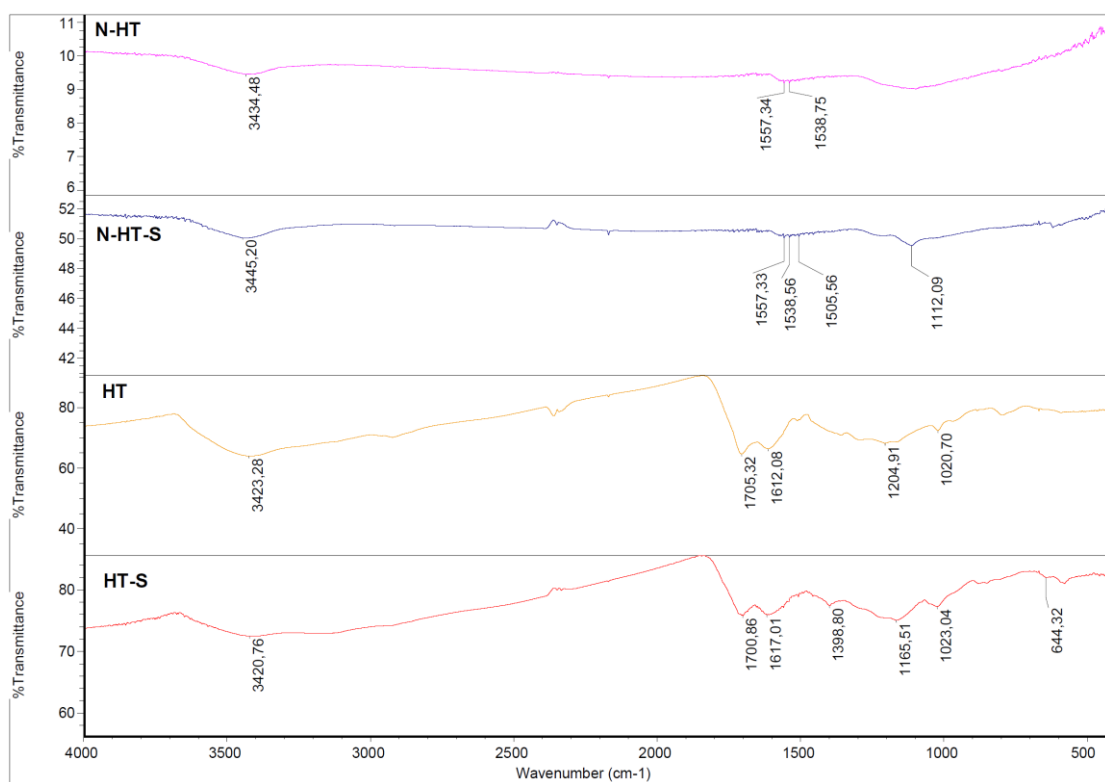
**Figure S2.** FT-IR spectra for catalysts **Zr** and **Zr-S**.



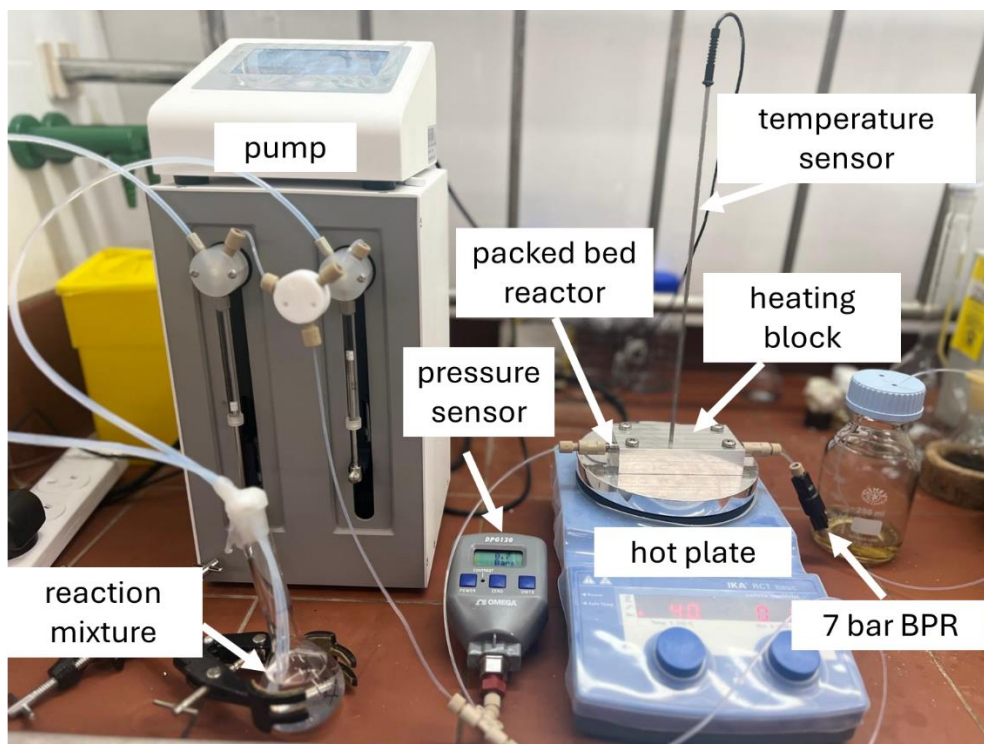
**Figure S3.** FT-IR spectra for catalysts **N-Zr**, **N-Zr-S1** and **N-Zr-S2**.



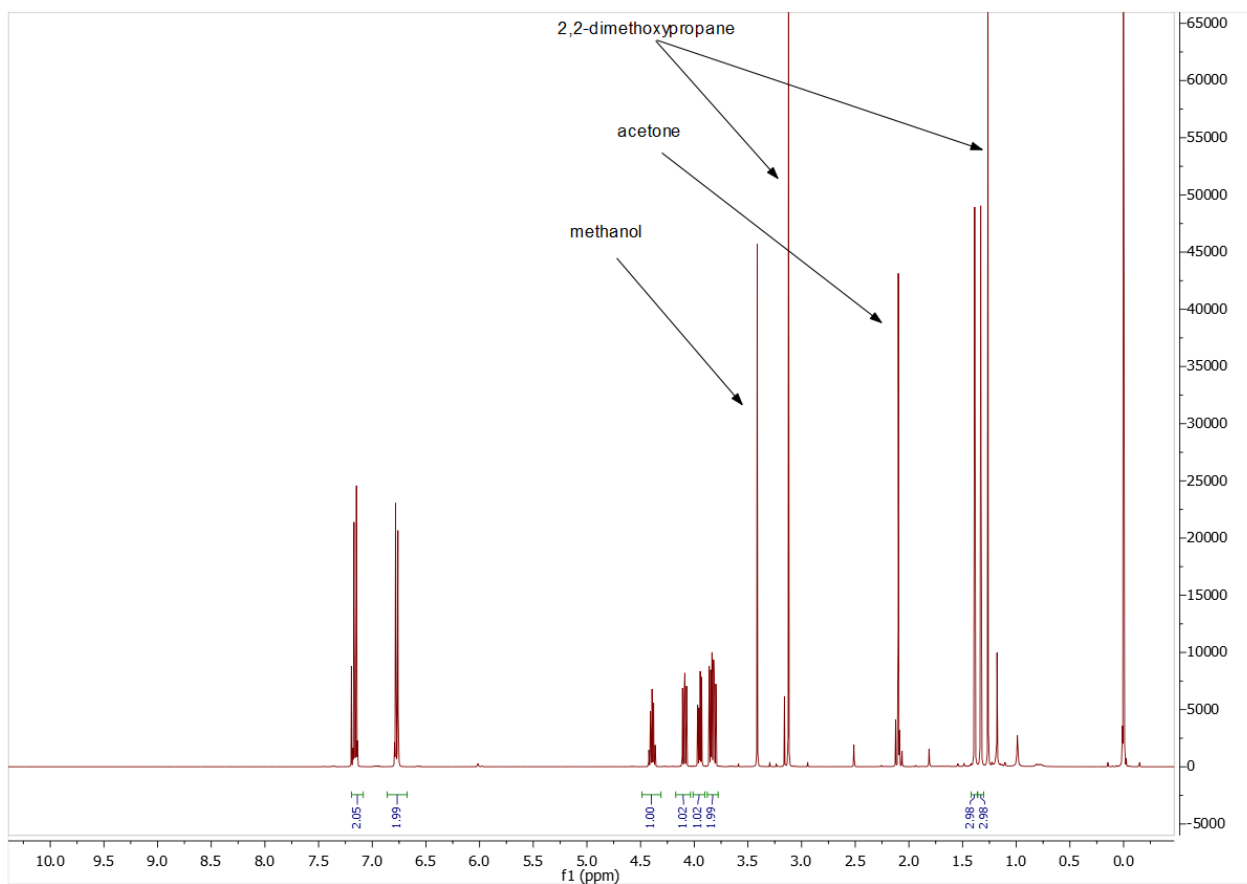
**Figure S4.** FT-IR spectra for catalysts M, M-N and M-S.



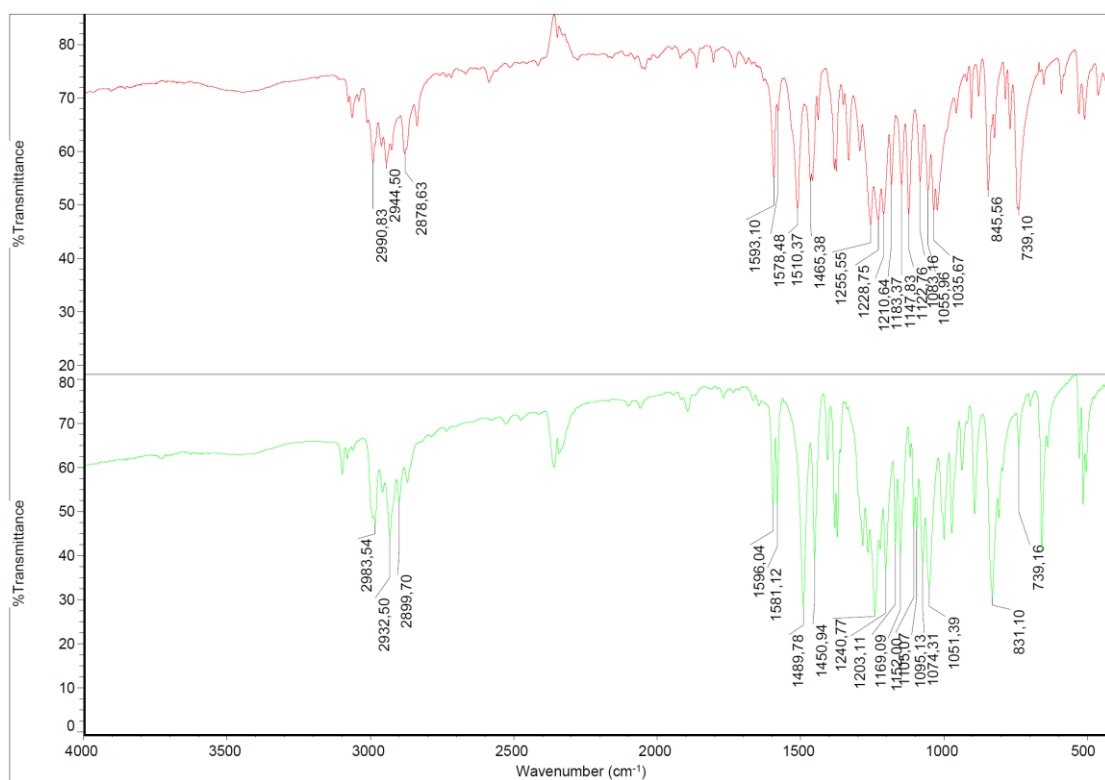
**Figure S5.** FT-IR spectra for catalysts N-HT, N-HT-S, HT and HT-S.



**Figure S6.** Set-up of the continuous flow reactor.

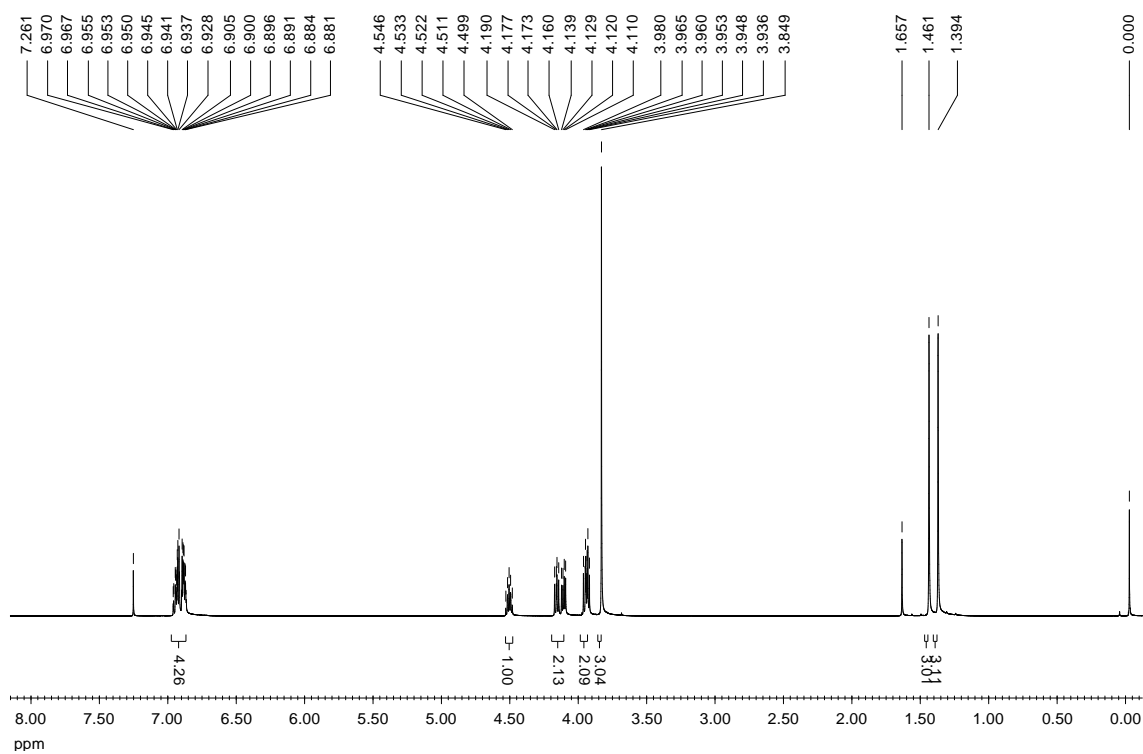


**Figure S7.** Proton NMR spectrum of crude reaction mixture obtained in continuous flow at a temperature of 60 °C and a residence time of 30 s, after partial evaporation of the solvent.

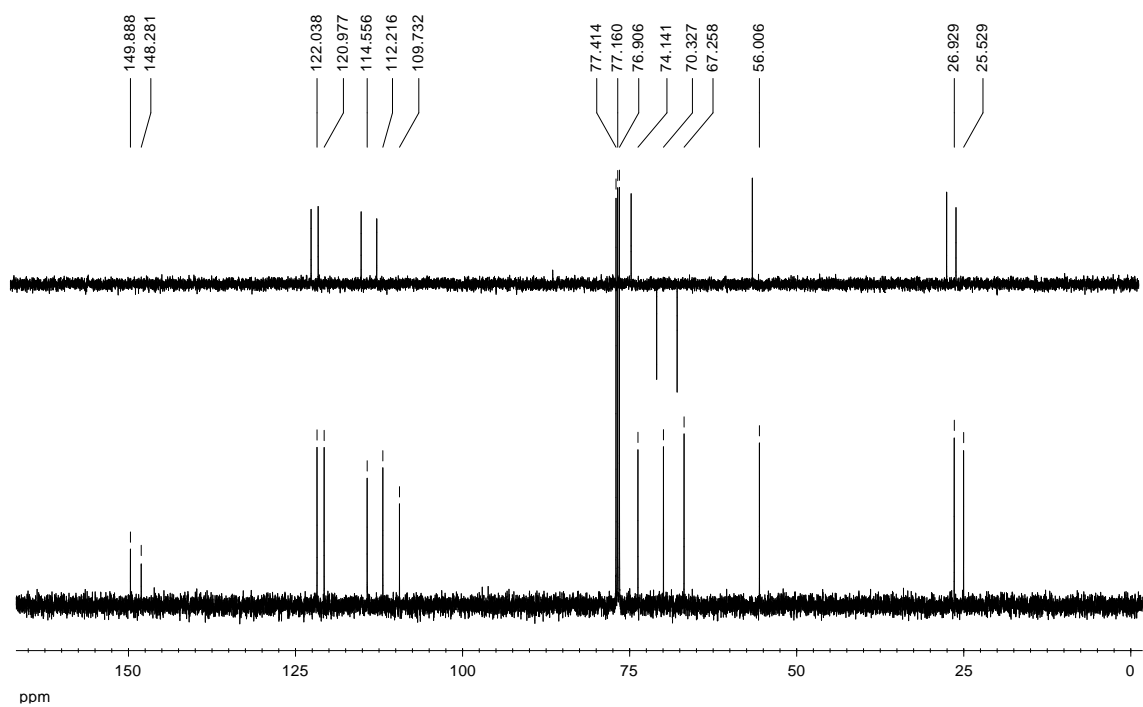


**Figure S8.** FT-IR spectra for 1,3-dioxolanes **3** (top) and **4** (bottom).

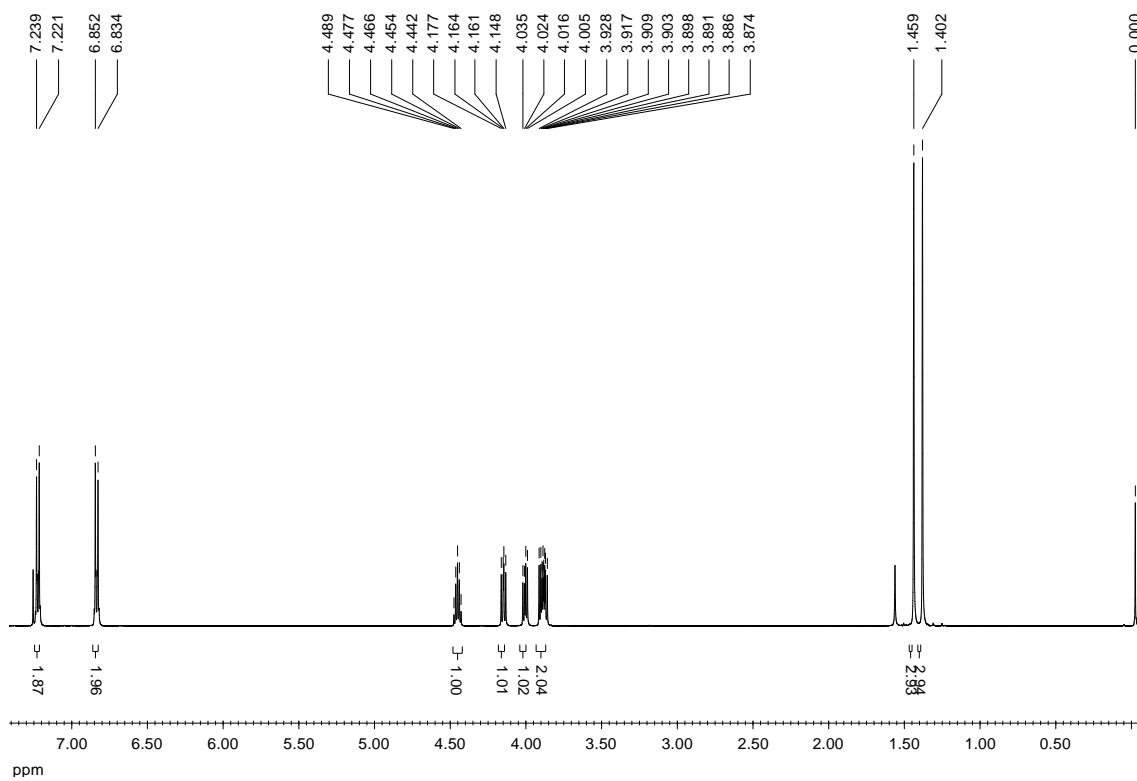




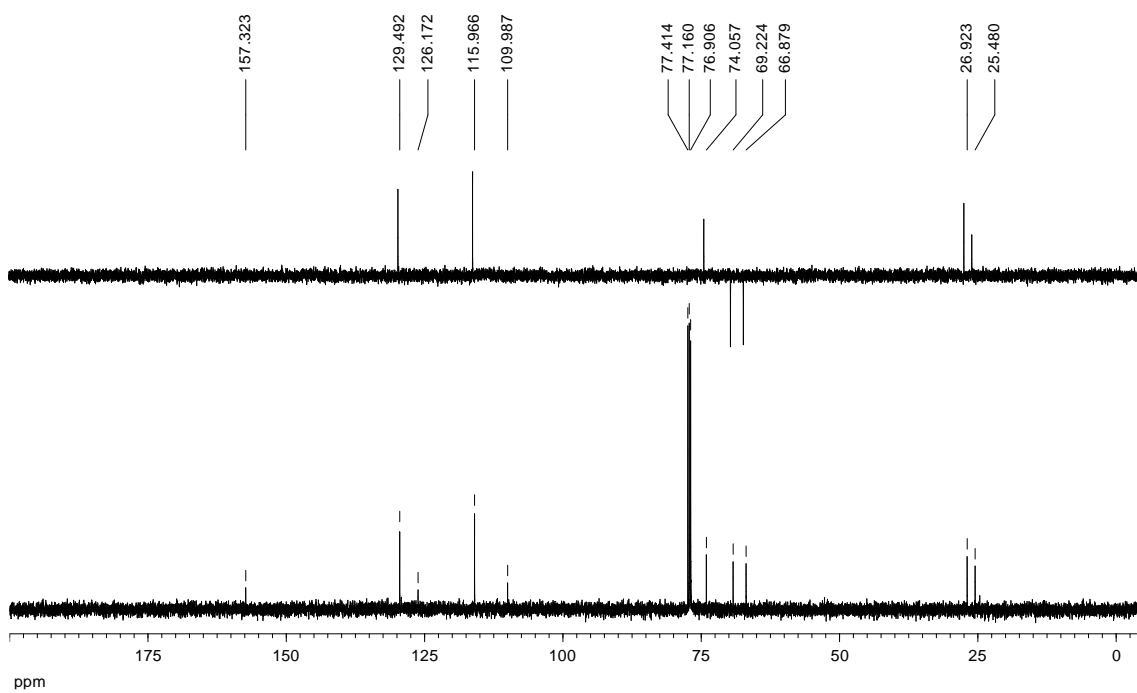
**Figure S9.** <sup>1</sup>H-NMR spectrum (CDCl<sub>3</sub>, 500 MHz) of **3**.



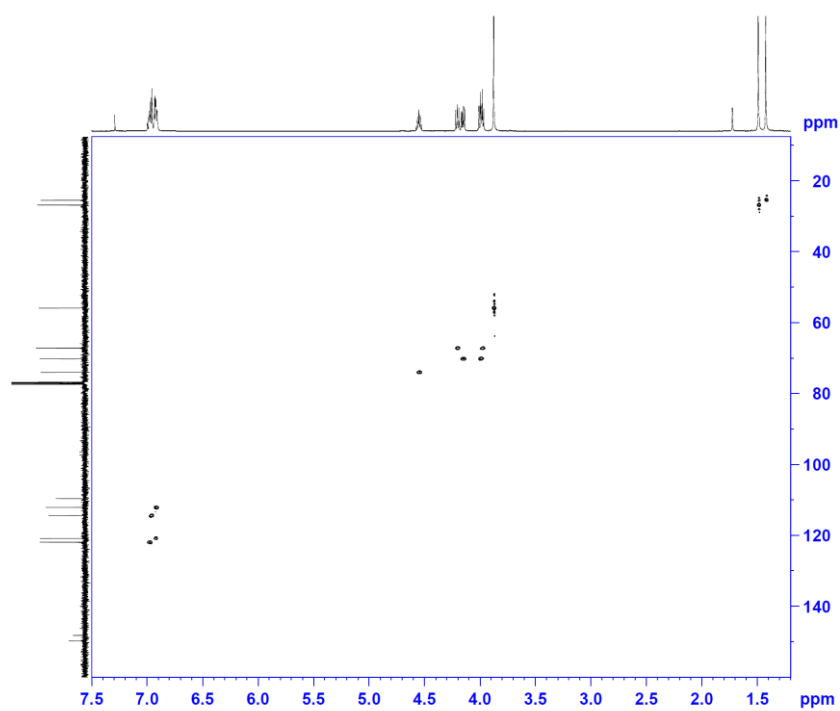
**Figure S10.** <sup>13</sup>C-NMR and DEPT spectra (CDCl<sub>3</sub>, 125 MHz) of **3**.



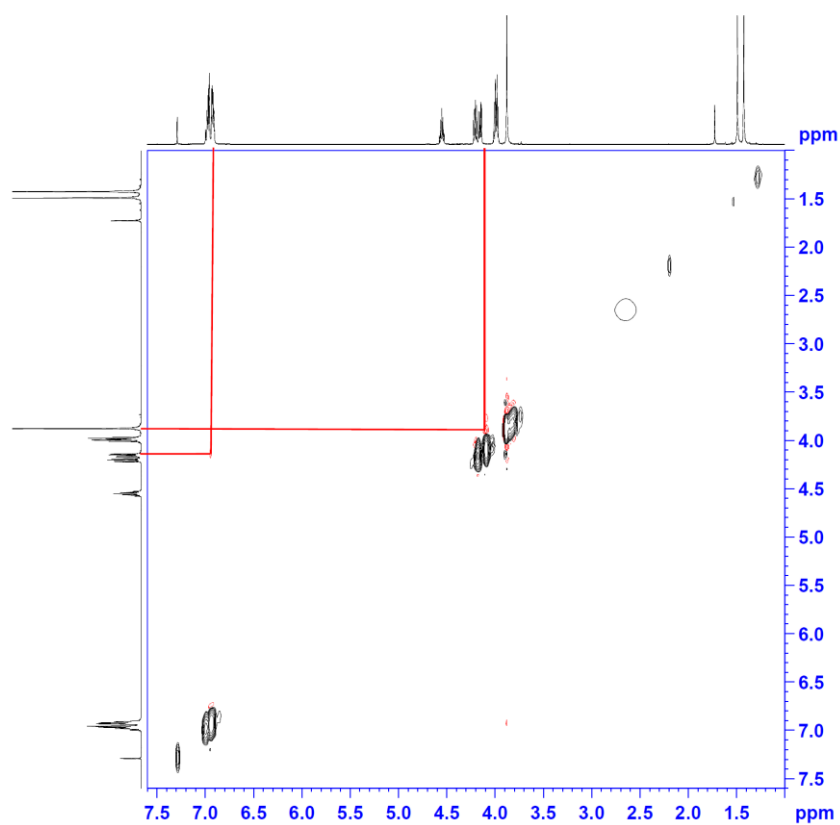
**Figure S11.** <sup>1</sup>H-NMR spectrum (CDCl<sub>3</sub>, 500 MHz) of **4**.



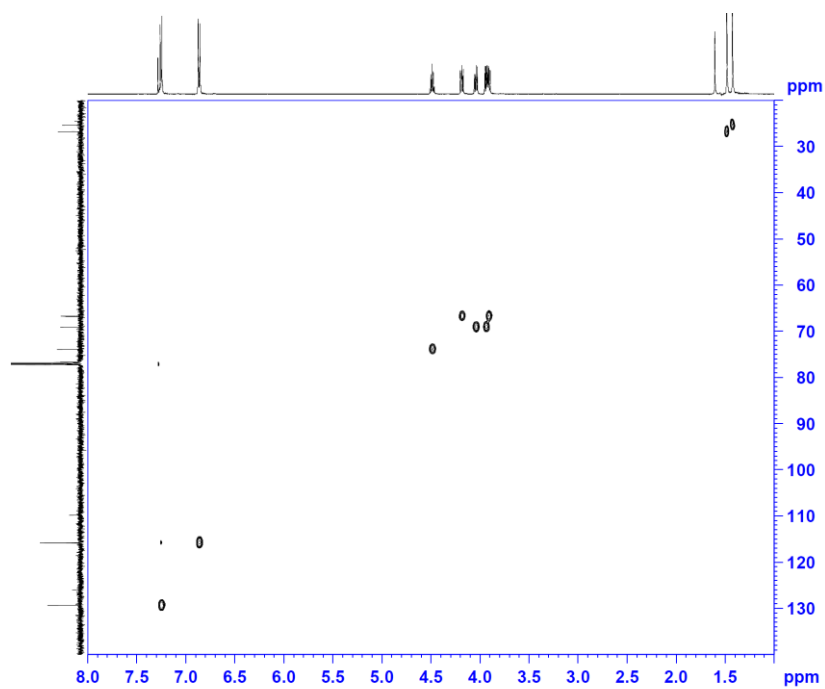
**Figure S12.** <sup>13</sup>C-NMR and DEPT spectra (CDCl<sub>3</sub>, 125 MHz) of **4**.



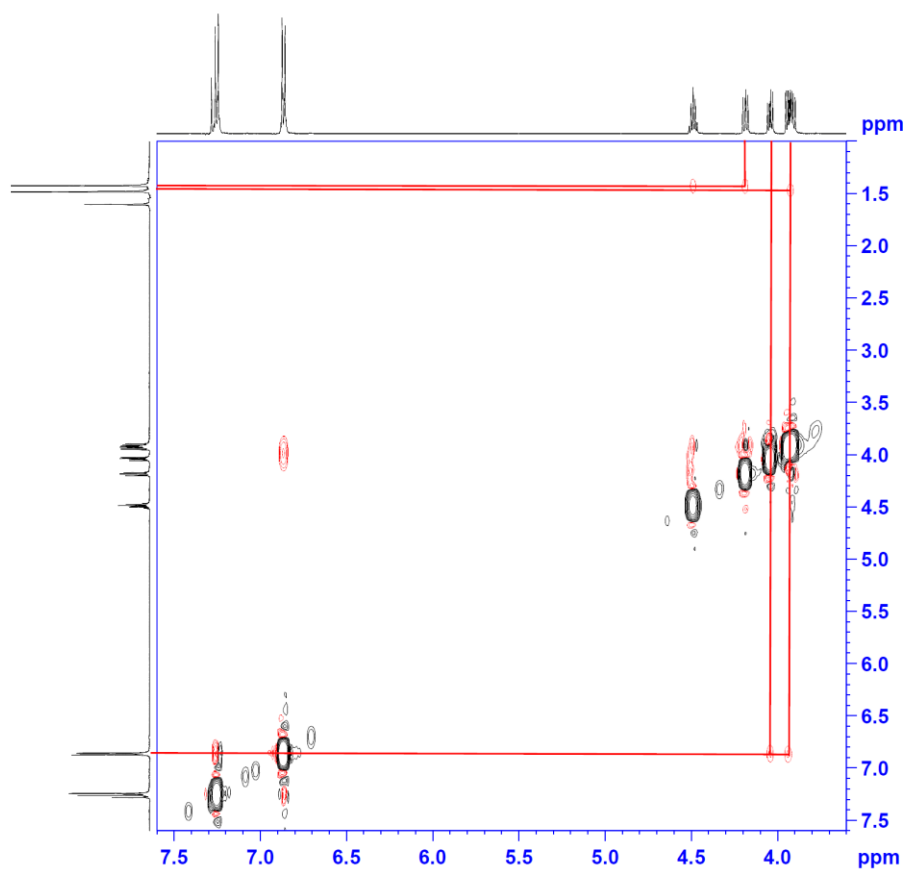
**Figure S13.**  $^1\text{H}$ - $^{13}\text{C}$  heteronuclear correlation spectrum (HMQC) of **3**.



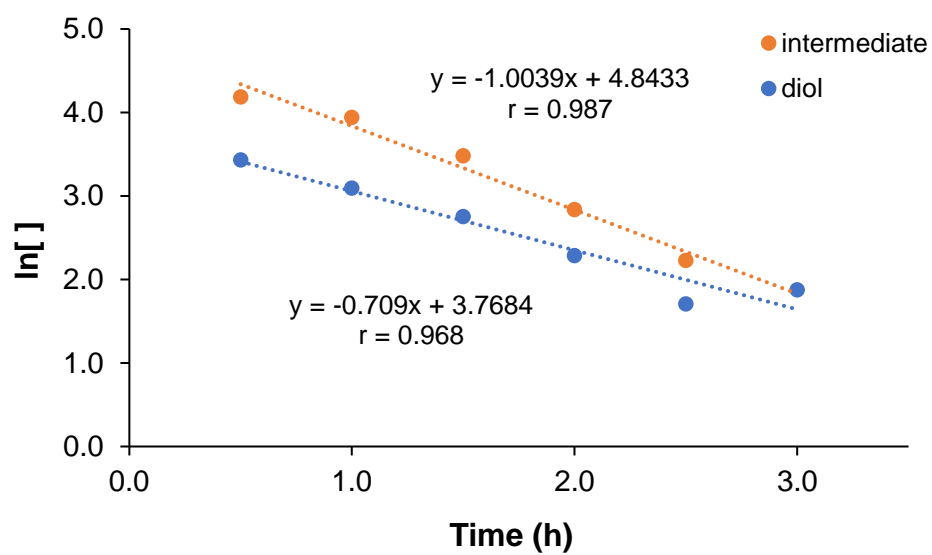
**Figure S14.**  $^1\text{H}$ - $^1\text{H}$  homonuclear correlation spectrum (NOESY) of **3**.



**Figure S15.**  $^1\text{H}$ - $^{13}\text{C}$  heteronuclear correlation spectrum (HMQC) of **4**.



**Figure S16.**  $^1\text{H}$ - $^1\text{H}$  homonuclear correlation spectrum (NOESY) of **4**.



**Figure S17.** Rate constants determination for evolution of diol **2** and intermediate **I<sub>2-4</sub>** corresponding to the acetalization catalyzed by **HT-S** at 40 °C.

**Table S5.** Recycling experiment with catalyst **N-HT-S**.

<b>Run</b>	<b>Time</b>	<b>Yield <sup>a</sup></b>
1	2 h	94%
2	2 h	98%
3	2 h	95%
4	24 h	91%
5	27 h	90%

<sup>a</sup> Isolated yield without purification by column chromatography.

**Table S6.** Recycling experiment with catalyst **HT-S**.

<b>Run</b>	<b>Time</b>	<b>Yield <sup>a</sup></b>
1	20 min	98%
2	30 min	98%
3	40 min	98%
4	40 min	97%
5	50 min	94%
6	60 min	95%
7	60 min	96%
8	60 min	96%
9	60 min	95%
10	70 min	96%

<sup>a</sup> Isolated yield without purification by column chromatography.